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**MACHINES.
TECHNOLOGIES.
MATERIALS 2025
PROCEEDINGS**

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**SCIENTIFIC-TECHNICAL UNION OF MECHANICAL ENGINEERING - INDUSTRY 4.0
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Designing and modeling of grapple buckets

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Abstract: The paper presents the process of automated designing and parametric modeling of grapple buckets using the CAD system. The presented grapple buckets are designed for bulk materials and they are part of the grapple construction. Grapples are installed on cranes, excavators and other specialized machines. A methodology has been developed that covers all stages of design – from the creation of 3D models of the parts, sub-assemblies and assemblies, to the generation of design documentation, using template files. The presented approach allows the creation of parametric models with multiple configurations, which cover different variants of the products. Using the presented methodology leads to a reduction in the volume of created documentation and optimizes the design process. Data management within a PDM system ensures centralized storage, actuality and accessibility of information.

KEYWORDS: GRAPPLE BUCKETS, GRAPPLES, CRANES, EXCAVATORS, METHODOLOGY, DEVELOPMENT, 3D MODELS, AUTOMATED DESIGNING, PARAMETRIC MODELING, CAD SYSTEM, TEMPLATE FILES, DESIGN DOCUMENTATION, PDM SYSTEM

1. Introduction.

The paper introduces the process of developing grapple buckets with a CAD system. The parts, sub-assemblies and assembly of the bucket are parametrically designed.

Grapple buckets are designed for bulk materials and they are part of the grapple construction. The grapples are used for loading and unloading of various materials. They are mounted on cranes, truck cranes, scrap gondolas, light trucks, excavators and other specialized machines [1, 2].

Grapples are the structure consisting of Grapple buckets (Left and Right), a Central body, a Rotator and a Carrier. Through the buckets, the grapple grabs materials. The Left and Right buckets are attached on the Central body. The Rotator rotates the grapple during operation. The Carrier attach the grapple to the corresponding machine.

The grapple buckets are designed and developed with the SolidWorks CAD system [3]. Using the CAD systems significantly facilitates the process of developing the product. The design is performed in the three-dimensional space. 3D models of the parts, sub-assemblies and assemblies are developed. The process of creating the design documentation is automated by using template files.

2. Literature review.

2.1. Parametric modeling of products with a CAD system.

Creating 3D models is an important step in the product development process. 3D models can be used to create new design documentation. In 3D models of parts and assemblies, the additional information is set by attributes (users and systems ones). The information from the attributes can be used for documentation management with PDM system, as well as by other departments in a company with ERP system [3, 4, 5, 6].

Based on the 3D models, the technology for parts manufacturing is also developed – adding or removing holes, determining allowances for mechanical processing, pre-processing of certain surfaces, and others [7]. This requires the creation of new design and technological documentation to be used in production. The 3D models are used to create the cutting files for the parts, which are made of sheet metal.

Very often the product is divided into sub-assemblies which are pre-manufactured and then a fixture is used to assemble them into the final product [8].

The next steps are developing the necessary fixtures for assembling, welding, machining and control [9, 10].

This means that product design is a long process that is implemented in multiple stages. Each stage is characterized by its own specific features [11, 12, 13, 14, 15].

Parametric modeling is used when the designed products have many versions and varieties. A single 3D model is created, where different versions of the product are presented with configurations. This leads to a small amount of design documentation for each

specific product. The accumulated big amount of documentation can cause many problems, for example – how to manage, organize, classify and archive the documentation; who has the right to change and update it; the documents should always be with the latest version; which other departments of the company have access to this documentation and the specialized information they are interested in [5, 6].

Parametric modeling reduces the time of the design process and increases its efficiency. Design automation is achieved, which means that designers do not have to redesign the product when there is a change in the dimensions of the model. Parametrically can be modeled dimensions of the model, geometric constructions in the model (*Features*), constraints between parts in an assembly (*Mates*), etc.

2.2. Design documentation management through PDM system. Application of ERP systems.

PDM (Product Data Management) systems are specialized software used to manage product data. The term "product data" means all information related to the product. PDM systems manage arrays of data and information necessary for the design, manufacture and assembling of the products, as well as for their maintenance. The functionalities of PDM systems have been improved and in addition to the Design and Engineering Departments, they are now used by other departments such as Supplies, Production and Maintenance. This means that PDM systems are developed to manage and control the huge volume of electronic data created by CAD/CAM/CAE systems.

In the PDM system, each configuration of the 3D model of the grapple bucket is entered as a separate product. The system automatically fills in the fields of the Product card, based on the information set in the bucket model with attributes for each configuration. If necessary, additional information can be entered manually in the item card.

PDM systems are used when there is a big amount of design, technological, production and other types of documentation accompanying the products that are developed, manufactured and supported. PDM systems solve the problems associated with the maintenance, storage and actuality of documentation related to the products [3].

ERP systems (Enterprise Resource Planning) are used in all processes necessary for the functioning of a company - people management, finances, production, availability of materials or products, supply system, services, orders, etc. One of the main functions of ERP systems is the shared database, i.e., the employees from different departments operate with the same information for their specific needs [4].

Both systems are part of the so-called Product Lifecycle Management (PLM). This means that a specific product is tracked from the creation of the product concept to the end of its life cycle.

3. Parametric modeling of the Grapple Buckets.

3.1. 3D models of the buckets.

The paper presents an approach to improve the efficiency of the product design process by using parametric modeling. The CAD system SolidWorks was used [3].

The SolidWorks CAD system enables parametric modeling through *parametric tables* or through *configurations*.

The paper discusses the parametric modeling approach using configurations. During modeling, configurations are created in 3D models of parts, sub-assemblies and assemblies.

Different models of grapple buckets differ in purpose, dimensions and construction. Fig. 1 shows the considered grapple bucket [1, 2].

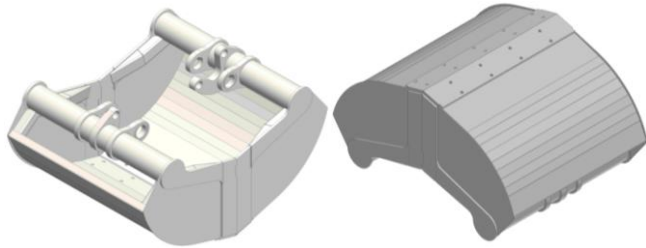


Fig. 1. Grapple bucket 1000 mm.

The grapple buckets of this model are three types, which differ in length – 640 mm (623016201), 800 mm (623016204) and 1000 mm (623016207).

A set of grapple buckets (Left and Right) is manufactured for each grapple. Each of them consists of pre-assembled sub-assemblies and parts.

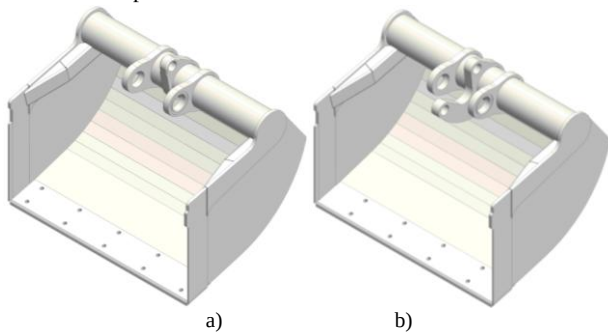


Fig. 2. Components of the Grapple Bucket 1000 mm.
a) Left Bucket, b) Right Bucket

Fig. 3 shows the 3D models of the sub-assemblies from which the grapple buckets are made.

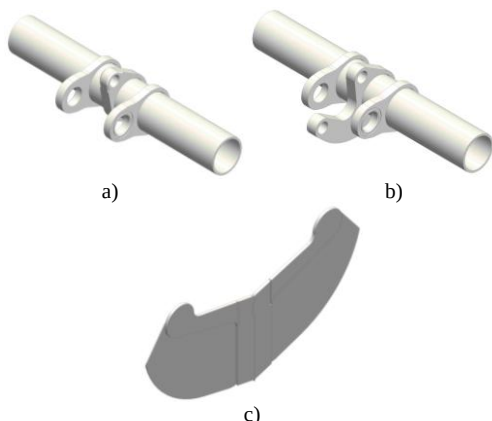


Fig. 3. Sub-assemblies of the Grapple Bucket 1000 mm.
a) Left Tube, b) Right Tube, c) Bucket Side

Each of the parts from the sub-assemblies is developed by using different 3D modeling commands – *Extruded Boss/Base*, *Revolved Boss/Base*, *Extruded Cut*, *Revolved Cut*, etc. Parts that are made from sheet metal are created using the module *Sheet Metal*, in this way their unfolding is obtained. The parts are assembled into individual sub-assemblies, which are assembled into the assembly of the bucket [8, 9].

The development of 3D models is consistent with the manufacturing technology of the buckets, as well as with the possibility of using the models from the PDM and ERP systems.

A design methodology has been used in the development and modeling of the products with the SolidWorks CAD system. This makes the modeling process easier and reduces the time required for designing. Fig. 4 shows a summarized algorithm of the methodology for designing grapple buckets.

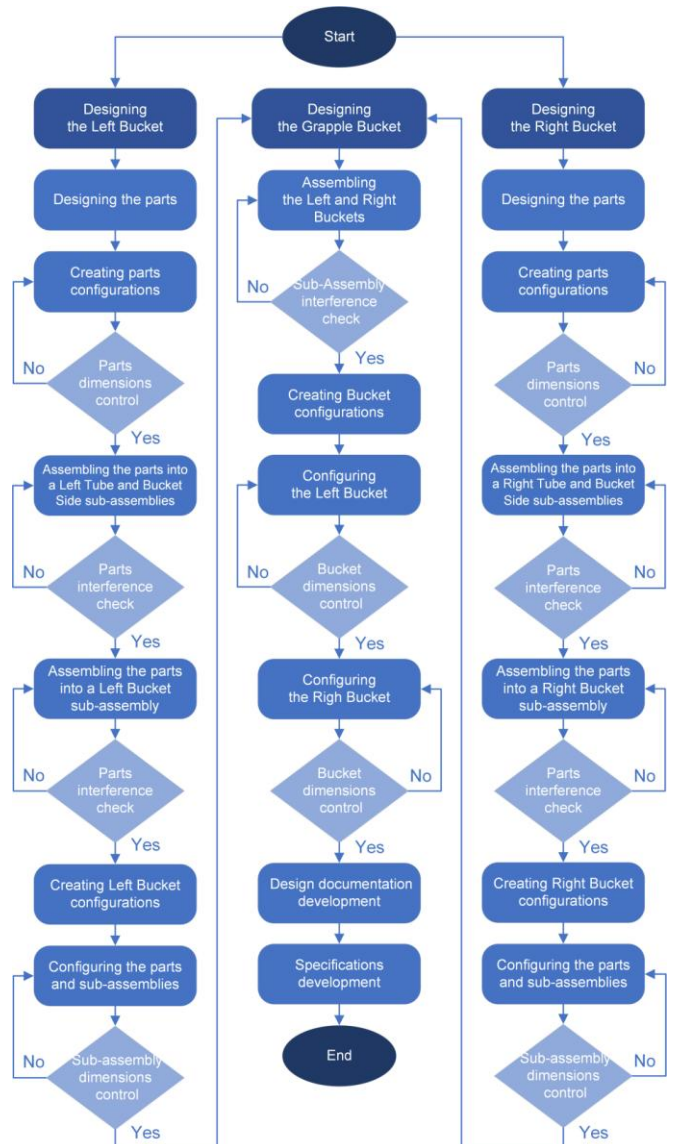


Fig. 4. Grapple buckets design algorithm.

A single 3D model of the 600 mm (623016201) length grapple bucket has been developed firstly. After that, the 800 mm (623016204) and 1000 mm (623016207) length grapple buckets has been modeled through configurations.

Each grapple bucket consists of two main sub-assemblies – Left Bucket and Right Bucket. The paper presents an example with Left Bucket, 1000 mm. The same approach is used to design the Right Bucket.

3.2. Parametric modeling of Left Tube and Right Tube.

An example with a Left Tube, 1000 mm sub-assemblies is considered. The 3D model of the Left Tube is parametrically modeled through configurations and Fig. 5 shows its components.

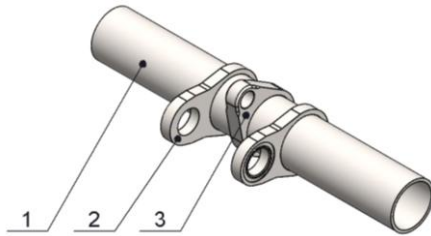


Fig. 5. Components of the Left Tube, 1000 mm.

1 – Tube, 2 – Lateral Ear, 3 – Central Left Ear

Part with position 1 is parametrically modeled and has configurations of different lengths.

To model parametrically the 3D model of the Left Tube, configurations are created in the *Configuration Manager* (Fig. 6). Each configuration represents a different length of the tube. For the corresponding bucket lengths, the Left Tube has the following configurations – 640 mm (623016201-LH-01), 800 mm (623016204-LH-01) and 1000 mm (623016207-LH-01).

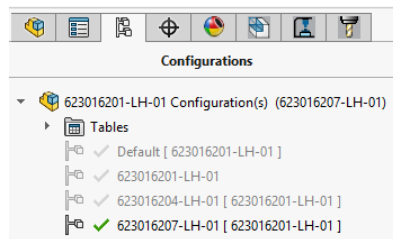


Fig. 6. Configurations in the 3D model of the Left Tube.

The next step is to configure the parts in the assembly of the tube. Part with positions 1 (Tube) contains configurations with different lengths. For each configuration of the Left Tube, the corresponding configuration of the part is set.

Fig. 7 shows the configurations of the Left Tube and the specified configurations of the Tube part. For the corresponding lengths of the Left Tube sub-assembly, the Tube part has the following configurations – 640 mm (599050297), 800 mm (599050296) and 1000 mm (599050241).

Modify Configurations			
Configuration Name	599050297-1@623016201-LH-01		
	Suppress	Configuration	Fixed
Default	☐	Default	☐
623016201-LH-01	☐	599050297	☐
623016204-LH-01	☐	599050296	☐
623016207-LH-01	☐	599050241	☐
< Creates a new configuration. >			

Fig. 7. Configuring the Tube part.

The configurations of the Tube part have different drawing numbers. The parts differ only in length, so one drawing of the part must be created with a parametric table including each of the variants.

Fig. 8 shows different lengths of the Left Tube, parametrically modeled with configurations.

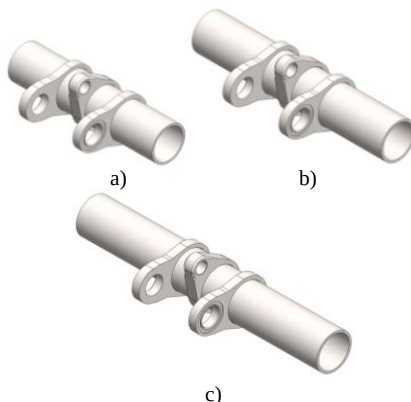


Fig. 8. Parametric models of the Left Tube.

a) 640 mm, b) 800 mm, c) 1000 mm

The same approach is used to design the Right Tube sub-assembly. The 3D model of Right Tube is also parametrically modeled with configurations.

3.3. Parametric modeling of Left Bucket and Right Bucket.

An example with a Left Bucket, 1000 mm is considered. The 3D model of the Left Bucket is parametrically modeled through configurations and Fig. 9 shows its components.

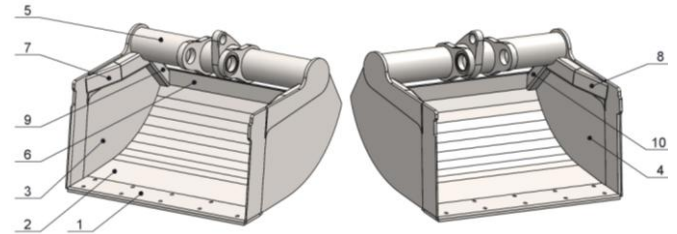


Fig. 9. Components of the Left Bucket, 1000 mm.

1 – Blade I, 2 – Bottom, 3 – Bucket Side I, 4 – Bucket Side II,
5 – Left Tube, 6 – Horizontal Rib,
7 – Rib I, 8 – Rib II, 9 – Rib III, 10 – Rib IV

Parts with positions 1, 2 and 6 are parametrically modeled and they have different length configurations. The Left Tube sub-assembly, position 5, is also parametrically modeled and has configurations with different lengths.

To model parametrically the 3D model of the Left Bucket, configurations are created in the *Configuration Manager* (Fig. 10). Each configuration represents a different length of the bucket. Buckets differ in length – 640 mm (623016201-LH), 800 mm (623016204-LH) and 1000 mm (623016207-LH).

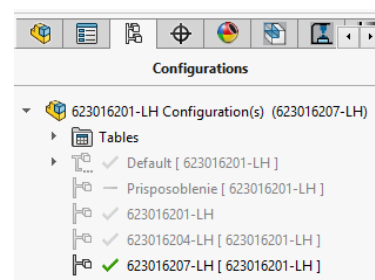


Fig. 10. Configurations in the 3D model of the Left Bucket.

The next step is to configure the parts in the assembly of the bucket. Parts with positions 1 (Blade I), 2 (Bottom) and 6 (Horizontal Rib) contain configurations with different lengths. For each configuration of the bucket, the corresponding configuration of the parts is set.

Fig. 11 shows the configurations of the Left Bucket and the specified configurations of the Blade I part. For the respective lengths of the bucket, the Blade I part has the following configurations – 640 mm (624016196), 800 mm (624014393) and 1000 mm (624014394).

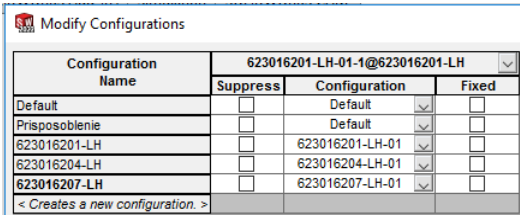
Modify Configurations			
Configuration Name	624016196-1@623016201-LH		
	Suppress	Configuration	Fixed
Default	☐	Default	☐
Prisposoblenie	☐	Default	☐
623016201-LH	☐	624016196	☐
623016204-LH	☐	624014393	☐
623016207-LH	☐	624014394	☐
< Creates a new configuration. >			

Fig. 11. Configuring the Blade I part.

The configurations of the Blade I part have a different drawing number. The parts differ in construction – they have different lengths and different number and position of the holes. This requires the creation of a separate drawing for each of the variants.

The sub-assemblies in the assembly of the bucket should be configured. This is the Left Tube sub-assembly, it also has configurations with different lengths.

Fig. 12 shows the configurations of the Left Bucket and the specified configurations of the Left Tube. For the respective lengths of the Left Bucket, the Left Tube sub-assembly has the following configurations – 640 mm (623016201-LH-01), 800 mm (623016204-LH-01) and 1000 mm (623016207-LH-01).



Configuration Name	Suppress	Configuration	Fixed
Default	☐	Default	☐
Prisposoblenie	☐	Default	☐
623016201-LH	☐	623016201-LH-01	☐
623016204-LH	☐	623016204-LH-01	☐
623016207-LH	☐	623016207-LH-01	☐
< Creates a new configuration. >			

Fig. 12. Configuring the Left Tube sub-assembly.

Fig. 13 shows different lengths of the Left Bucket, parametrically modeled with configurations.

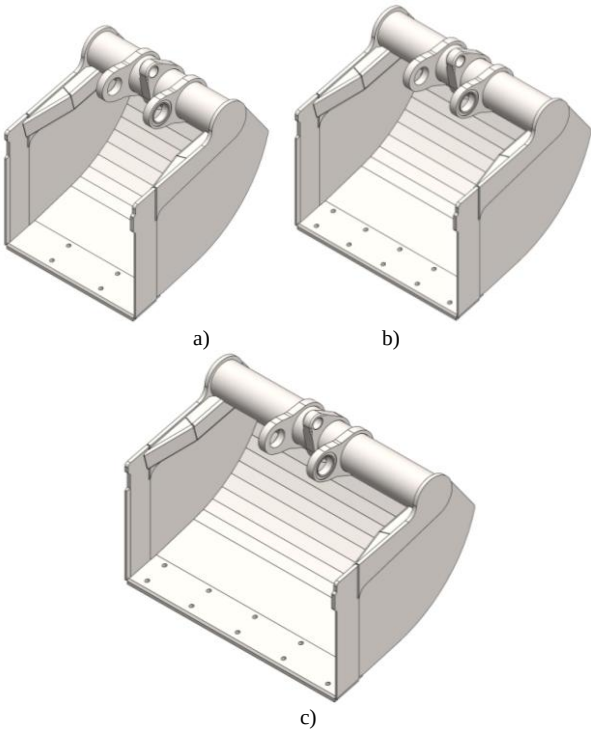


Fig. 13. Parametric models of the Left Bucket.
a) 640 mm, b) 800 mm, c) 1000 mm

The same approach is used to design the Right Bucket. The 3D model of Right Bucket is also parametrically modeled with configurations.

3.4. Parametric modeling of the Grapple Bucket.

The 3D model of the Grapple Bucket is parametrically modeled and has configurations with different lengths. The bucket model is an assembly consisting of a Left Bucket and a Right Bucket, assembled through the main planes *Front Plane*, *Top Plane* and *Right Plane* (Fig. 14).

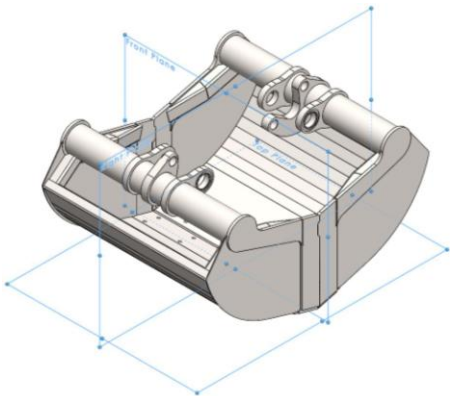


Fig. 14. Assembling the main components of the bucket.

To perform assembling, the 3D models of the sub-assemblies have in the same place coordinate systems and planes, that match with the coordinate system *Origin* and the main planes *Front Plane*, *Top Plane* and *Right Plane* in the 3D model of the bucket (Fig. 15).

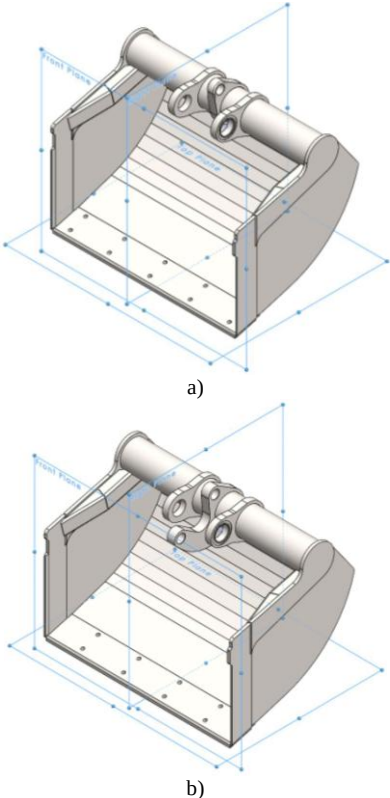
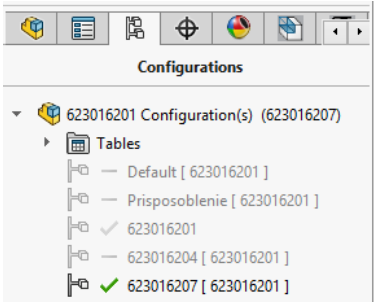


Fig. 15. Main planes in 3D models.
a) Left Bucket, b) Right Bucket

To model parametrically the 3D model of the Grapple Bucket, configurations are created in the *Configuration Manager* (Fig. 16). Each configuration represents a different length of the bucket. Buckets differ in length – 640 mm (623016201), 800 mm (623016204) and 1000 mm (623016207).

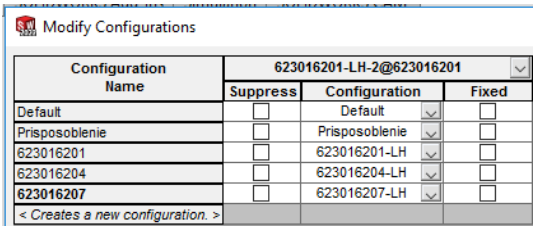


Configurations
623016201 Configuration(s) (623016207)
Tables
Default [623016201]
Prisposoblenie [623016201]
623016201
623016204 [623016201]
623016207 [623016201]

Fig. 16. Configurations in the 3D model of the Grapple Bucket.

The next step is to configure the sub-assemblies in the assembly of the bucket. The Left Bucket and the Right Bucket also contain configurations. For each bucket configuration, the corresponding configuration from the specific sub-assemblies is set.

The following figure shows the configurations of the Grapple Bucket and configurations which are set for the Left Bucket.



Configuration Name	Suppress	Configuration	Fixed
Default	☐	Default	☐
Prisposoblenie	☐	Prisposoblenie	☐
623016201	☐	623016201-LH	☐
623016204	☐	623016204-LH	☐
623016207	☐	623016207-LH	☐
< Creates a new configuration. >			

Fig. 17. Configuring the Left Bucket.

Configuring the Right Bucket is implemented using the same approach.

Fig. 18 shows different grapple bucket lengths, parametrically modeled with configurations.

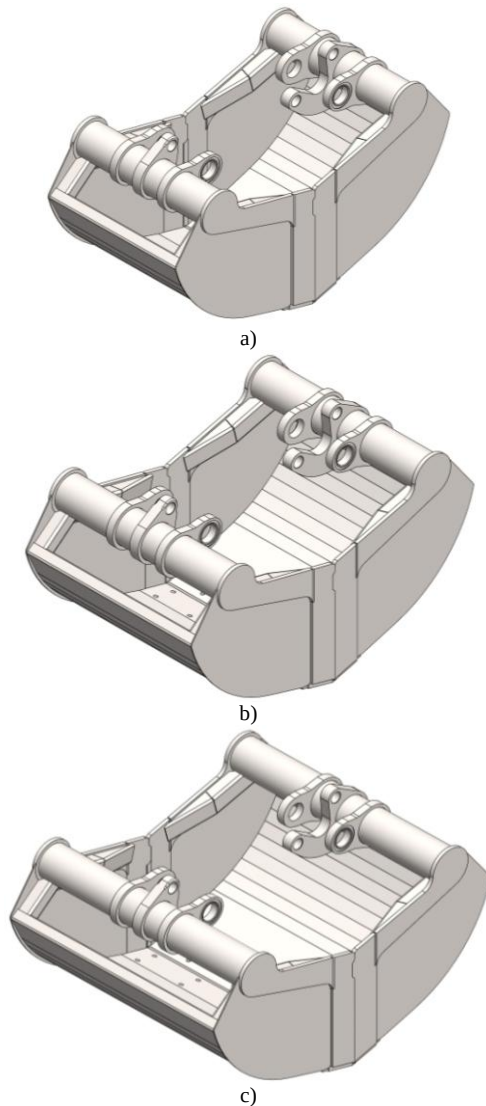


Fig. 18. Parametric models of the Grapple Bucket.
a) 640 mm, b) 800 mm, c) 1000 mm

3.5. Design documentation of the Grapple Buckets.

Complete design documentation has been made for the developed grapple buckets. The process of its creation is automated by using document *Template files* in the SolidWorks CAD system.

Template files for the *Part* and *Assembly* documents are used to create the 3D models of the parts and assemblies. In the template of each 3D model of the part or assembly, from the *File Properties* menu, in the *Summary Information* window, in the *Custom* section the required attributes (users and systems ones) are selected. The attributes for each configuration are filled in the *Configuration Specific* window [5, 6].

After the attributes of the 3D models having been filled in, the design documentation of the product (drawings and specifications) is developed. For the considered example of a grapple bucket, drawings of all parts, sub-assemblies (Left Tube, Right Tube, Left Bucket and Right Bucket) and the assembly of the Grapple Bucket are developed.

To develop the drawings, template files for *Drawing* documents are used, which represent the standard drawing formats – A4, A3, A2, A1 and A0. Information from the attributes is automatically visualized in the drawing tables, in the assembly drawing specifications and in individual product specifications [5, 6].

The following figures show examples of completed templates for the Tube part (599050241) and the Left Tube sub-assembly (623016207-LH-01).

The following attributes are used in the template of each *Part* – Description (Name of the Part), PartNo (Drawing number), AssemblyNo (Assembly from which the Part is), Weight (Weight of the Part – command "SW-Mass@Part1.SLDPR1"), Material (Material designation – command "SW-Material@Part1.SLDPR1"), Assortment (Type and dimensions of the workpiece), TYPE (Manufacturing operations), DrawnBy (Developed the document), CheckedBy (Checked the document), ApprovedBy (Approved the document), Revision (Document revision).

Property Name	Type	Value / Text Expression	Evaluated Value
1 Description	Text	Tube	Tube
2 PartNo	Text	599050241	599050241
3 AssemblyNo	Text	623016207-RH-01, 623016207-LH-01	623016207-RH-01, 623016207-LH-01
4 Weight	Text	"SW-Mass@599050241.SLDPR1"	25.152
5 Material	Text	"SW-Material@599050241.SLDPR1"	1.0545 (S355N)
6 Assortment	Text	Tube Ø140x8	Tube Ø140x8
7 TYPE	Text	Mechanical processing	Mechanical processing
8 DrawnBy	Text	N.Stankov	N.Stankov
9 CheckedBy	Text	-	-
10 ApprovedBy	Text	-	-
11 Revision	Text	A	A
12 <Type a new property>			

Fig. 19. Template of the Tube part.

The following figure shows the drawing of a Tube part, with an automatically filled table, based on the attributes specified in the 3D model of the part.

Scale		Weight, kg	Title	
1:5	25.152		Tube	
Drawn	N. Stankov	22.2.2025 r.	Material	Sheet
Checked	-	22.2.2025 r.	1.0545 (S355N)	1/1
Approved	-	22.2.2025 r.	Assortment	
Tolerances, unless noted: EN 22768-1 m			Tube Ø140x8	
			Drawing No	Rev.
			599050241	A

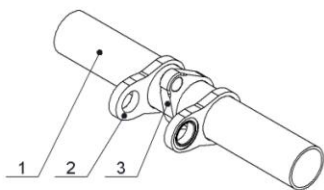
Fig. 20. Drawing of the Tube part.

The following attributes are used in the template of each *Assembly* – Description (Name of the Assembly), PartNo (Drawing number), AssemblyNo (Assembly of which the current Assembly is), Weight (Weight of the Assembly – command "SW-Mass@Assem1.SLDPR1"), TYPE (Manufacturing operations), DrawnBy (Developed the document), CheckedBy (Checked the document), ApprovedBy (Approved the document), Revision (Document revision).

Property Name	Type	Value / Text Expression	Evaluated Value
1 Description	Text	Left Tube	Left Tube
2 PartNo	Text	623016207-LH-01	623016207-LH-01
3 AssemblyNo	Text	623016207-LH	623016207-LH
4 Weight	Text	"SW-Mass@623016207-LH-01.SLDASM"	36.808
5 TYPE	Text	Welding	Welding
6 DrawnBy	Text	N.Stankov	N.Stankov
7 CheckedBy	Text	-	-
8 ApprovedBy	Text	-	-
9 Revision	Text	A	A
10 <Type a new property>			

Fig. 21. Template of the Left Tube sub-assembly.

The following figure shows the drawing of the Left Tube sub-assembly, with an automatically filled table and specification, based on the attributes set in the 3D model of the assembly.



No	Part №	Description		Qty.	Weight, kg	Assortment
1	599050241	Tube		1	25.152	Tube Ø140x8
2	624050307	Lateral Ear		2	3.997	Sheet metal 30x169x286
3	624050306	Central Left Ear		1	3.663	Sheet metal 40x169x242
		Scale 1:10	Weight, kg 36.808	 Title Left Tube		
Drawn	N.Stankov	22.2.2025 r.		Material	Assortment	Sheet 1/1
Checked	-	22.2.2025 r.				
Approved	-	22.2.2025 r.				
Tolerances, unless noted: EN 22768-1 m				Assembly No 623016207-LH		Drawing No 623016207-LH-01
						Rev. A

Fig. 22. Drawing of the Left Tube sub-assembly.

After the 3D models of the parts, sub-assemblies, assemblies and all drawings of the buckets have been developed, the next step is their management by the specialized systems – PDM and ERP. The design documentation is managed by the PDM system Solid-Works Enterprise PDM [3]. The various departments of the company have access to this information and operate with it through the ERP system Microsoft Dynamics NAV ERP [4].

Fig. 23 shows an example card of Grapple Bucket 1000 mm (623016207) in the ERP system.

[illegible]

Fig. 23. Card of the Grapple Bucket 1000 mm.

4. Methods for modeling Grapple Buckets.

A certain product can be designed by applying two main methods – *traditional modeling* or *parametric modeling*. Both approaches are applied in practice. Depending on the products being developed, the method to be used is also chosen.

The paper discusses the approach for parametric modeling through configurations. Grapple buckets are being modeled. The modeling of 3D models of parts and assemblies as well as the creation of drawings and product specifications, are presented.

The parametric modeling approach through configurations has been chosen because of the advantages it has. The reason for using this approach becomes clear after comparing the two modeling methods.

4.1. Traditional modeling of Grapple Buckets.

In the traditional modeling approach, a 3D model is created for each bucket size. The 3D model of the bucket is an assembly composed of parts and sub-assemblies. All parts are modeled in advance. Parts that are structurally the same but differ in length, for different bucket models must be modeled as separate parts. There are 5 such parts in the grapple buckets. These parts are Tube, Blade I, Blade II, Bottom and Horizontal Rib (Fig. 5 and Fig. 9). Some of the parts are the same for all buckets and they are modeled once. After modeling the parts, they are assembled into the 3D model of the buckets. Table 1 shows the grapple buckets and their components.

Table. 1. Grapple buckets, designed using the traditional modeling approach.

Bucket size, mm	640	800	1000
Number of unique parts, that do not require changes	11		
Number of parts that require changes	5	5	5
Total number of parts	34	34	34
3D model of the bucket (Assembly)	Yes	Yes	Yes

This approach has many disadvantages:

- a large number of parts that must be modeled in advance;
- a large number of assemblies to model;
- possible modeling errors due to the large variety of 3D models of parts and assemblies;
- creation of a big amount of design documentation – drawings, drawings of assemblies, specifications;
- redesigning the product when the size is different.

All these leads to a decreased efficiency of engineering work during design and an increased possibility of errors.

4.2. Parametric modeling of Grapple Buckets.

In parametric modeling through configurations, a single 3D model of the bucket is modeled and it includes all its variations (Table 2). The 3D model of the bucket is composed of parts and sub-assemblies. All parts are modeled in advance. Parts that differ only in length are modeled parametrically through configurations. The parts are assembled into sub-assemblies, which are modeled parametrically. The sub-assemblies are assembled into the assembly of the bucket, which is also modeled parametrically.

Table. 2. Grapple buckets, designed using the parametric modeling approach.

Bucket size, mm	640	800	1000
Number of unique parts, that do not require changes	11		
Number of parts that require changes	5		
Total number of parts	34		
3D model of the bucket (Assembly)	A single 3D model for all buckets		

Parametric modeling through configurations achieves high design efficiency, which is an advantage of the method. This is especially important when designing and modeling with CAD systems.

According to the data presented in the Table. 1 and Table. 2, the following conclusions can be made:

- in traditional modeling, the total number of parts and buckets that need to be modeled is bigger – **26 Parts** and **3 Buckets**, compared to **16 Parts** and **1 Bucket** in parametric modeling;
- parametric modeling significantly increases the efficiency of engineering work - the volume of design documentation created is reduced and the time required for modeling products is reduced;
- as a result of the smaller amount of design documentation, the number of errors in product modeling is significantly reduced;

- reducing design time is especially important in modern working conditions, where engineering departments consist of only a few people;

- parametric modeling is an approach that should be used for products with many variants and variations, as is the case with the considered grapple buckets.

5. Conclusion.

1. An approach for designing specialized products using a CAD system is presented. The implementation of parametric modeling through the SolidWorks CAD system allows significant automation in the design of grapple buckets. The use of configurations facilitates the creation of various product variants, reducing the time and volume of manually created documentation. Parametric modeling facilitates the process of developing products that have many variants and varieties.

2. The use of attributes (user and systems ones) in the 3D models of parts and assemblies enables the automated creation of design documentation (drawings of parts, drawings of assemblies and specifications).

3. The proposed approach of modeling through configurations improves the efficiency of engineering work, by reducing the design time, the volume of the designing documentation and errors during product design.

4. The presented integration between the 3D models and PDM system allows effective management of a large volume of design documentation. The files can also be used by other company departments through ERP system.

5. The presented approach is applicable to other similar products, which emphasizes its universal nature. In this way, similar problems related to the development and management of documents can be solved.

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Exergy analysis of 160 MW three cylinder steam turbine segments

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Abstract: Exergy analysis of three cylinder steam turbine segments is performed in this research. The highest mechanical power of 47389.66 kW is developed in the first segment (Seg. I, which actually represents the entire HPC – High Pressure Cylinder). Intermediate Pressure Cylinder (IPC) is the dominant mechanical power producer of all cylinders and it develops 48.95% of cumulative mechanical power produced in the whole turbine. The outlet Low Pressure Cylinder (LPC) segments (Seg. VII and IX) have the highest exergy destructions and the lowest exergy efficiency (equal to 61.27%) of all turbine segments. The best exergy performance shows IPC segments – Seg. V has the lowest exergy destruction (equal to 363.84 kW), while Seg. II has the highest exergy efficiency (equal to 94.04%) of all turbine segments. Outlet LPC segments (Seg. VII and IX) are the most sensitive to the ambient temperature change – their exergy efficiency decreases for 3.19% when the ambient temperature increases from 5 °C to 45 °C.

KEYWORDS: EXERGY ANALYSIS, COMPLEX STEAM TURBINE, TURBINE SEGMENTS, THE AMBIENT TEMPERATURE CHANGE

1. Introduction

Currently, the majority of electricity worldwide is produced by various kinds of steam turbines [1, 2]. However, steam turbines which can be found in power plants can also be used for many other purposes, the electricity production is a dominant, but not the only task which steam turbine can accomplish [3, 4].

In the conventional thermal or nuclear power plants are used high power steam turbines which are usually composed of several cylinders connected to the same shaft [5, 6]. Medium and low power steam turbines can be composed of one or two cylinders, what depends on the required mechanical power production [7, 8]. Turbine cylinders are usually composed of several turbine stages [9]. However, in exploitation can also be found auxiliary low power steam turbines which are composed of only one Curtis stage (such steam turbines are often used for the pump drive) [10].

Steam turbine cylinders can be single flow or dual flow cylinders, while dual flow cylinders can be symmetrical and dissymmetrical. In single flow turbine cylinder steam expands in just one direction, while in the dual flow cylinder steam expands simultaneously in two directions [11].

This paper presents exergy analysis of a three cylinder steam turbine segments. Such exergy analysis allows insight in the operation of different turbine cylinder parts, the cylinder is not considered as a whole (as in the standard exergy analysis). The observed steam turbine has nine segments overall – produced mechanical power, exergy efficiencies and exergy destructions are presented and compared for all segments. It is also analyzed and observed which turbine segment is the most sensitive to the ambient temperature change.

2. Description and operating regime of the analyzed steam turbine and turbine segments

Observed steam turbine consists of three cylinders which are connected to the same shaft: High Pressure Cylinder (HPC), Intermediate Pressure Cylinder (IPC) and Low Pressure Cylinder (LPC), Fig. 1. HPC and IPC are single flow cylinders, while LPC is a dual flow symmetrical cylinder, therefore both LPC parts (left part LPC-L and right part LPC-R) have the same operating parameters and performance. The shaft on which are connected all cylinders is used for the electric generator drive.

Exergy analysis is performed for all steam turbine segments (what requires division of each cylinder to the segments). Cylinder segment is a part of each cylinder defined between the cylinder inlet, outlet and steam extractions. Each turbine cylinder division to the segments allow insight into the operation performance of different cylinder parts, which cannot be found in the standard plant or turbine exergy analysis [12, 13].

HPC did not possess any steam extractions, therefore HPC has actually only one segment defined between HPC inlet and HPC outlet (HPC is its own segment, Seg. I).

IPC has three steam extractions, which allows IPC division on the four segments. The first IPC segment (Seg. II) is defined between IPC inlet and first IPC extraction (operating point 6, Fig. 1) which deliver a certain steam mass flow rate to the components of high pressure feedwater heating system. Second IPC segment (Seg. III) is defined between first and second IPC extractions (operating points 6 and 7, Fig. 1). Second IPC extraction delivers a certain steam mass flow rate to the deaerator. Third IPC segment (Seg. IV) is defined between second and third IPC extractions (operating points 7 and 8, Fig. 1), where third IPC extraction delivers a certain steam mass flow rate to the components of low pressure condensate heating system. The last, fourth IPC segment (Seg. V) is defined between third IPC extraction and IPC outlet (operating point 9, Fig. 1).

LPC is a dual flow cylinder – steam mass flow rate enters in the cylinder at its central part (operating point 9, Fig. 1) and simultaneously expands through both LPC parts (one half of the steam mass flow rate expands through LPC-L and other half expands through LPC-R). LPC-L and LPC-R have identical steam operating parameters at the inlet, in the extractions and at the outlet which confirms that LPC is symmetrical cylinder (steam temperature, pressure and mass flow rate are identical in operating points 10 and 11 and in operating points 13 and 14, Fig. 1). Each LPC part has two segments which are defined between LPC inlet and steam extraction and between steam extraction and LPC part outlet. For the LPC-L mentioned two segments are marked as Seg. VI and Seg. VII, while for the LPC-R segments are marked as Seg. VIII and Seg. IX. At the end of expansion in both LPC parts (operating points 13 and 14, Fig. 1), remaining steam mass flow rate is delivered to main condenser for condensation [14].

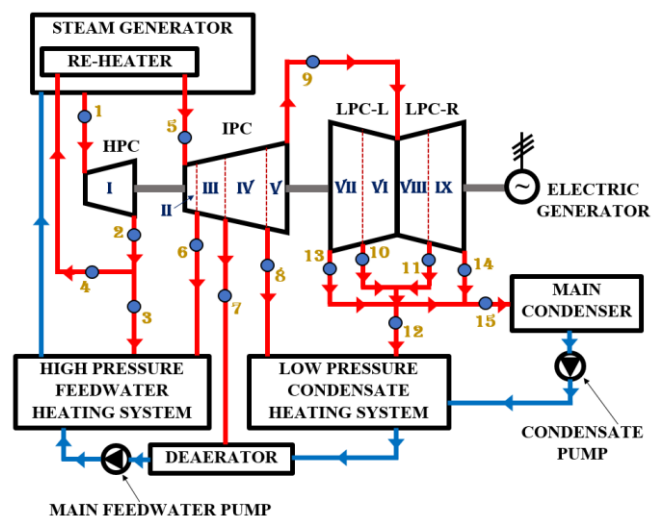


Fig. 1. General scheme of the three-cylinder 160 MW steam turbine along with operating points required for the exergy analysis and each cylinder marked segments (I – IX)

Steam expansion process in all cylinders and segments of the observed turbine in Specific enthalpy/Specific entropy (h/s) diagram is shown in Fig. 2. Exergy analysis of any steam turbine cylinder or segment requires data of the real (polytropic) steam expansion process [15]. Data of real expansion process can be properly obtained only from the measurements inside power plant during the exploitation, therefore these data involve all the losses which occur during operation. Losses in the real steam expansion process are proportional to the increase in steam specific entropy, as presented in Fig. 2, so steam specific entropy must continuously increase from the inlet until the outlet of each turbine cylinder or segment. Only in the ideal (isentropic) steam expansion process from the inlet until the outlet of each turbine cylinder or segment, steam specific entropy will remain constant, but the observation of such ideal expansion process is not required in the exergy analysis [16]. It should be highlighted that operating point's numeration and segments marking presented in Fig. 2 are in relation to operating point's numeration and segments marking from Fig. 1.

Steam expansion process is identical in both LPC parts (LPC-L and LPC-R) because LPC is a symmetrical dual flow cylinder, Fig. 2. At the LPC outlet (operating points 13, 14 and 15, Fig. 1 and Fig. 2) steam is slightly under the saturation curve (wet steam) which can be transferred to condensate in main condenser.

Fig. 2 is obtained from known steam operating parameters in each operating point (presented in Table 4) by using NIST-Refprop 9.0 software [17].

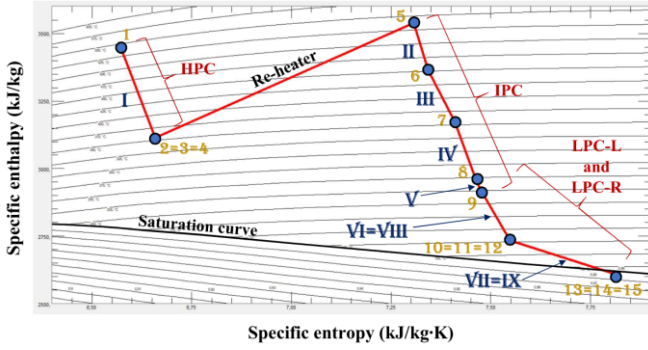


Fig. 2. Specific enthalpy/Specific entropy (h/s) diagram of steam expansion process throughout steam turbine and all its segments

3. Exergy analysis equations

In comparison to energy analysis which did not consider parameters of the ambient (ambient pressure and temperature), exergy analysis considers ambient parameters (parameters of the ambient in which observed system, component or component part operates). Second law of thermodynamics is a baseline for an exergy analysis [18, 19].

3.1. Overall equations, balances and principles

During the exergy analysis of any system, component or component part, a few overall equations, balances and principles must always be satisfied [7]. These equations, balances and principles will be presented in this subsection.

The overall exergy balance equation, valid for any system, component or component part at steady state is [9]:

$$\dot{X}_{\text{heat}} - P - \sum \dot{E}x_{\text{outlet}} + \sum \dot{E}x_{\text{inlet}} - \dot{E}x_{\text{Destruction}} = 0, \quad (1)$$

where P is mechanical power. $\dot{E}x$ is a fluid total exergy flow, which can be defined by an equation [8]:

$$\dot{E}x = \dot{m} \cdot \varepsilon, \quad (2)$$

where $\dot{m}$ is a fluid mass flow rate and ε is fluid specific exergy. Fluid specific exergy (ε) is defined by an equation [20]:

$$\varepsilon = (h - h_0) - T_0 \cdot (s - s_0), \quad (3)$$

where h is fluid specific enthalpy, s is fluid specific entropy, T is a temperature and index 0 is related to the ambient state. Exergy transfer by heat at the temperature T ($\dot{X}_{\text{heat}}$) is defined as [6]:

$$\dot{X}_{\text{heat}} = \sum (1 - \frac{T_0}{T}) \cdot \dot{Q}_{\text{heat}}, \quad (4)$$

where $\dot{Q}_{\text{heat}}$ is an energy transfer by heat. Mass flow rate balance for any system, component or component part is [4]:

$$\sum \dot{m}_{\text{inlet}} = \sum \dot{m}_{\text{outlet}}. \quad (5)$$

3.2. Equations for the exergy analysis of turbine segments

Equations for the exergy analysis of the observed turbine segments are defined according to the recommendations from the literature [21-23]. Indexes and segments marking applied in all equations from this subsection are related to the operating points and markings shown in Fig. 1 and Fig. 2. In Table 1 are presented equations for each turbine segment developed mechanical power calculation, while in Table 2 and Table 3 are presented equations for each turbine segment exergy inlet and exergy outlet calculation.

Table 1. Equations for each segment developed mechanical power calculation

Segment	Developed mechanical power	Eq.
I	$P_{\text{Seg.I}} = \dot{m}_1 \cdot (h_1 - h_2)$	(6)
II	$P_{\text{Seg.II}} = \dot{m}_5 \cdot (h_5 - h_6)$	(7)
III	$P_{\text{Seg.III}} = (\dot{m}_5 - \dot{m}_6) \cdot (h_6 - h_7)$	(8)
IV	$P_{\text{Seg.IV}} = (\dot{m}_5 - \dot{m}_6 - \dot{m}_7) \cdot (h_7 - h_8)$	(9)
V	$P_{\text{Seg.V}} = (\dot{m}_5 - \dot{m}_6 - \dot{m}_7 - \dot{m}_8) \cdot (h_8 - h_9)$	(10)
VI	$P_{\text{Seg.VI}} = \frac{\dot{m}_9}{2} \cdot (h_9 - h_{10})$	(11)
VII	$P_{\text{Seg.VII}} = (\frac{\dot{m}_9}{2} - \dot{m}_{10}) \cdot (h_{10} - h_{13})$	(12)
VIII	$P_{\text{Seg.VIII}} = \frac{\dot{m}_9}{2} \cdot (h_9 - h_{11})$	(13)
IX	$P_{\text{Seg.IX}} = (\frac{\dot{m}_9}{2} - \dot{m}_{11}) \cdot (h_{11} - h_{14})$	(14)

Table 2. Equations for each segment exergy inlet calculation

Segment	Exergy inlet	Eq.
I	$\dot{E}x_{\text{Seg.I,inlet}} = \dot{m}_1 \cdot \varepsilon_1$	(15)
II	$\dot{E}x_{\text{Seg.II,inlet}} = \dot{m}_5 \cdot \varepsilon_5$	(16)
III	$\dot{E}x_{\text{Seg.III,inlet}} = (\dot{m}_5 - \dot{m}_6) \cdot \varepsilon_6$	(17)
IV	$\dot{E}x_{\text{Seg.IV,inlet}} = (\dot{m}_5 - \dot{m}_6 - \dot{m}_7) \cdot \varepsilon_7$	(18)
V	$\dot{E}x_{\text{Seg.V,inlet}} = (\dot{m}_5 - \dot{m}_6 - \dot{m}_7 - \dot{m}_8) \cdot \varepsilon_8$	(19)
VI	$\dot{E}x_{\text{Seg.VI,inlet}} = \frac{\dot{m}_9}{2} \cdot \varepsilon_9$	(20)
VII	$\dot{E}x_{\text{Seg.VII,inlet}} = (\frac{\dot{m}_9}{2} - \dot{m}_{10}) \cdot \varepsilon_{10}$	(21)
VIII	$\dot{E}x_{\text{Seg.VIII,inlet}} = \frac{\dot{m}_9}{2} \cdot \varepsilon_9$	(22)
IX	$\dot{E}x_{\text{Seg.IX,inlet}} = (\frac{\dot{m}_9}{2} - \dot{m}_{11}) \cdot \varepsilon_{11}$	(23)

Table 3. Equations for each segment exergy outlet calculation

Segment	Exergy outlet	Eq.
I	$\dot{E}x_{\text{Seg.I,outlet}} = \dot{m}_2 \cdot \varepsilon_2 + P_{\text{Seg.I}}$	(24)
II	$\dot{E}x_{\text{Seg.II,outlet}} = \dot{m}_6 \cdot \varepsilon_6 + (\dot{m}_5 - \dot{m}_6) \cdot \varepsilon_6 + P_{\text{Seg.II}}$	(25)
III	$\dot{E}x_{\text{Seg.III,outlet}} = \dot{m}_7 \cdot \varepsilon_7 + (\dot{m}_5 - \dot{m}_6 - \dot{m}_7) \cdot \varepsilon_7 + P_{\text{Seg.III}}$	(26)
IV	$\dot{E}x_{\text{Seg.IV,outlet}} = \dot{m}_8 \cdot \varepsilon_8 + (\dot{m}_5 - \dot{m}_6 - \dot{m}_7 - \dot{m}_8) \cdot \varepsilon_8 + P_{\text{Seg.IV}}$	(27)
V	$\dot{E}x_{\text{Seg.V,outlet}} = \dot{m}_9 \cdot \varepsilon_9 + P_{\text{Seg.V}}$	(28)
VI	$\dot{E}x_{\text{Seg.VI,outlet}} = \dot{m}_{10} \cdot \varepsilon_{10} + (\frac{\dot{m}_9}{2} - \dot{m}_{10}) \cdot \varepsilon_{10} + P_{\text{Seg.VI}}$	(29)
VII	$\dot{E}x_{\text{Seg.VII,outlet}} = \dot{m}_{13} \cdot \varepsilon_{13} + P_{\text{Seg.VII}}$	(30)
VIII	$\dot{E}x_{\text{Seg.VIII,outlet}} = \dot{m}_{11} \cdot \varepsilon_{11} + (\frac{\dot{m}_9}{2} - \dot{m}_{11}) \cdot \varepsilon_{11} + P_{\text{Seg.VIII}}$	(31)
IX	$\dot{E}x_{\text{Seg.IX,outlet}} = \dot{m}_{14} \cdot \varepsilon_{14} + P_{\text{Seg.IX}}$	(32)

Exergy destruction of each turbine segment is calculated as:

$$\dot{E}x_{\text{Seg.}(i),\text{Destruction}} = \dot{E}x_{\text{Seg.}(i),\text{inlet}} - \dot{E}x_{\text{Seg.}(i),\text{outlet}} \quad (33)$$

Each turbine segment exergy efficiency is calculated as:

$$\eta_{\text{Seg.}(i),\text{EX}} = \frac{P_{\text{Seg.}(i)}}{\dot{E}x_{\text{Seg.}(i),\text{inlet}} - \dot{E}x_{\text{Seg.}(i),\text{outlet}} + P_{\text{Seg.}(i)}} \quad (34)$$

In Eq. 33 and Eq. 34 index i represents each turbine segment ($i = \text{I, II, III, IV, V, VI, VII, VIII, IX}$).

4. Steam operating parameters required for the turbine segments exergy analysis

Exergy analysis of each observed turbine segment is performed by using steam operating parameters measured in each operating point (Fig. 1 and Fig. 2) during the plant exploitation. These operating parameters are found in [24] and presented in Table 4. During the plant exploitation, in each operating point from Fig. 1 and Fig. 2 are measured steam temperature, pressure and mass flow rate - other steam operating parameters presented in Table 4 are calculated using NIST-Refprop 9.0 software [17].

As the exergy analysis considers parameters of the ambient (ambient temperature and pressure), in exergy analysis of any system, component or segment is required the definition of the base ambient state [25]. In this research, the base ambient state is defined by the ambient temperature of 25 °C and the ambient pressure of 1 bar.

Table 4. Steam operating parameters for the turbine segments exergy analysis

O. P. *	Temp. (°C)	Pressure (bar)	Mass flow rate (kg/s)	Specific enthalpy (kJ/kg)	Specific entropy (kJ/kg·K)	Specific exergy (kJ/kg) **
1	538	129.19	145.10	3440.6	6.5738	1485.20
2	355	36.33	145.10	3114.0	6.6586	1133.30
3	355	36.33	10.10	3114.0	6.6586	1133.30
4	355	36.33	135.00	3114.0	6.6586	1133.30
5	538	32.39	135.00	3540.3	7.3062	1366.60
6	452	18.04	7.60	3365.2	7.3429	1180.40
7	356	8.37	7.44	3174.1	7.4091	969.63
8	244	3.20	8.01	2955.0	7.4638	734.21
9	226	2.69	111.95	2920.3	7.4747	696.26
10	128	0.92	2.35	2733.5	7.5482	487.54
11	128	0.92	2.35	2733.5	7.5482	487.54
12	128	0.92	4.70	2733.5	7.5482	487.54
13	65	0.25	53.63	2610.4	7.8094	286.61
14	65	0.25	53.63	2610.4	7.8094	286.61
15	65	0.25	107.25	2610.4	7.8094	286.61

* O. P. = Operating Point (according to Fig. 1 and Fig. 2)

** At the base ambient state

5. Results and discussion

Developed mechanical power in each segment of the analyzed steam turbine, which is one of the essential parameters in the exergy analysis of any turbine segment, is presented in Fig. 3.

Observing all segments, it can easily be concluded that the highest mechanical power of 47389.66 kW is developed in the first segment (Seg. I, which actually represents the entire HPC). Each of the first three IPC segments (Seg. II, III and IV) develop notable mechanical power, while the last IPC segment (Seg. V) develop the lowest mechanical power of all turbine segments, Fig. 3. Low pressure steam turbine cylinder is symmetrical one, therefore both LPC parts (LPC-L and LPC-R) develop the same mechanical power. Symmetrical LPC segments (Seg. VI and VIII, as well as Seg. VII and IX) also develop identical mechanical power.

In three cylinder steam turbines, LPC is usually the dominant mechanical power producer [13]. Steam turbine analyzed in this paper strongly deviates from this general guideline. In the observed steam turbine, IPC is the dominant mechanical power producer of all cylinders and it develops 48.95% of cumulative mechanical power produced in the whole turbine. HPC develops 29.68%, while

LPC (both LPC parts) develops only 21.37% of cumulative mechanical power produced in the whole turbine.

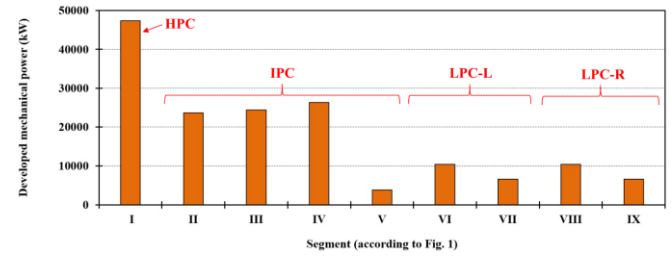


Fig. 3. Developed mechanical power in each segment of the analyzed steam turbine

Exergy destruction and exergy efficiency of each steam turbine segment at the base ambient state are presented in Fig. 4.

The outlet LPC segments (Seg. VII and IX) have the highest exergy destructions and the lowest exergy efficiency (equal to 61.27%) of all turbine segments. At least some turbine stages in the LPC outlet segments operate with wet steam (water droplets in wet steam increase losses) and in supersonic regime, so high exergy destruction and low exergy efficiency of the outlet LPC segments can be expected. The best exergy performance shows IPC segments – Seg. V has the lowest exergy destruction (equal to 363.84 kW), while Seg. II has the highest exergy efficiency (equal to 94.04%) of all turbine segments. HPC (Seg. I) has high exergy destruction, just slightly lower than outlet LPC segments, however such high exergy destruction did not notably ruin its exergy efficiency (Seg. I exergy efficiency is equal to 92.81%).

Considering turbine cylinders, in three cylinder steam turbines IPC usually shows the best performance (because IPC did not operate with the steam of the highest pressure in the plant as HPC and neither one IPC stage did not operate with wet steam as occur in some LPC outlet stages) [4, 13]. For the observed steam turbine, it can be concluded that HPC and IPC shows very similar exergy performance, so clear dominance of the IPC in relation to other cylinders did not exist for steam turbine analyzed in this paper.

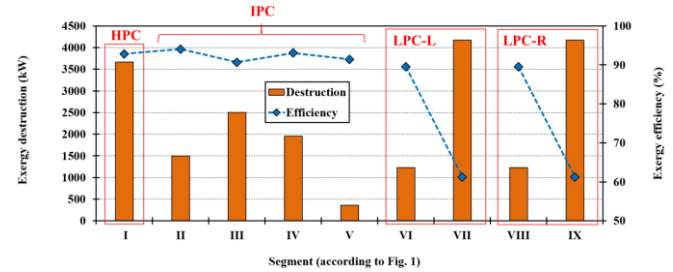


Fig. 4. Exergy destruction and exergy efficiency of each analyzed steam turbine segment at the base ambient state

Final part of the observed steam turbine segments research was related to each segment sensitivity on the ambient temperature change. During the ambient temperature variation, the ambient pressure remains always the same and identical as at the base ambient state. For each segment is valid a conclusion that increase in the ambient temperature simultaneously increases segment exergy destruction and decrease segment exergy efficiency. However, the intensity of exergy destruction and exergy efficiency change during the ambient temperature variation differs from one turbine segment to another.

Decrease in exergy efficiency of each steam turbine segment during the ambient temperature increase from 5 °C to 45 °C is presented in Fig. 5. From Fig. 5 is clear that LPC is much more sensitive to the ambient temperature change in comparison to other two cylinders. Outlet LPC segments (Seg. VII and IX) are the most sensitive to the ambient temperature change – their exergy efficiency decreases for 3.19% when the ambient temperature increases from 5 °C to 45 °C. Seg. II of the IPC is the least sensitive to the ambient temperature change – increase in the ambient temperature from 5 °C to 45 °C decreases Seg. II exergy efficiency for 0.71% only.

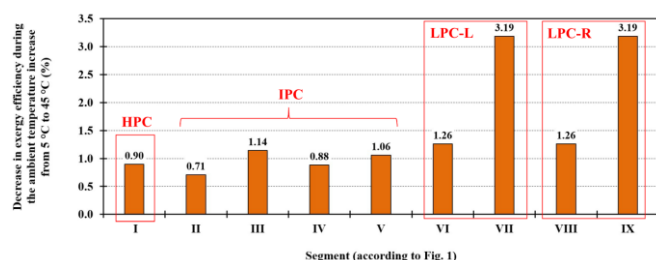


Fig. 5. Decrease in exergy efficiency of each steam turbine segment during the ambient temperature increase from 5 °C to 45 °C

6. Conclusions

Exergy analysis of complex three cylinder steam turbine segments is performed in this paper. The most important conclusions are:

- The highest mechanical power of 47389.66 kW is developed in the first segment (Seg. I, which actually represents the entire HPC).
- In the observed steam turbine, IPC is the dominant mechanical power producer of all cylinders and it develops 48.95% of cumulative mechanical power produced in the whole turbine. HPC develops 29.68%, while LPC (both LPC parts) develops only 21.37% of cumulative mechanical power produced in the whole turbine.
- The outlet LPC segments (Seg. VII and IX) have the highest exergy destructions and the lowest exergy efficiency (equal to 61.27%) of all turbine segments.
- The best exergy performance shows IPC segments – Seg. V has the lowest exergy destruction (equal to 363.84 kW), while Seg. II has the highest exergy efficiency (equal to 94.04%) of all turbine segments.
- HPC (Seg. I) has high exergy destruction, just slightly lower than outlet LPC segments, but sufficiently high exergy efficiency (Seg. I exergy efficiency is equal to 92.81%).
- LPC is much more sensitive to the ambient temperature change in comparison to other two cylinders (HPC and IPC).
- Outlet LPC segments (Seg. VII and IX) are the most sensitive to the ambient temperature change – their exergy efficiency decreases for 3.19% when the ambient temperature increases from 5 °C to 45 °C.

7. Acknowledgment

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Changes in the mass moment of inertia of the planetary gear mechanism reduced to the axis of the sun gear depending on the ratio between the radii of the sun and planet gears

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Abstract: Direct Planetary gear mechanism normally consists of a single central gear at the center, an internal (ring) gear around the outside, some number of planet gears that go in between and a planet carrier. They are used to reduce or multiply the number of revolutions. For correct selection of the torque of the driving machine, it is necessary to know the moment of inertia of the planetary gear mechanism, which directly depends on the mass moment of inertia of the planetary gear mechanism and the angular acceleration. In this paper is analyzed the impact of the ratio of the radii of the central and planet gears on the mass moment of inertia of the planetary gear mechanism reduced to the axis of the central gear at a constant diameter of the internal gear. The changes in the reduced mass moment of inertia depending on the number of planet gears was also analyzed.

Keywords: PLANETARY MECHANISM, REDUCED MASS MOMENT OF INERTIA, PLANET CARRIER, SUN GEAR, PLANET GEARS

1. Introduction

Planetary gears can provide high transmission accuracy and support high torque transmission. They belong to the group of epicyclic mechanisms and are most often used for torque transmission and speed transformation. They can be used as reducers, when the angular velocity of the output shaft is lower than the angular velocity of the input shaft through which energy is supplied to the system, or they can be used as multipliers, when the angular velocity of the input shaft is lower than the output shaft angular velocity.

Planetary gears normally consist of; a single sun gear at the center (a), an internal (ring) gear around the outside (b), some number of planets (c) that go in between and a planet carrier H. Planets are the same size, at a common center distance from the center of the planetary gear, and held by a planetary carrier. During the operation of the planetary train, the input power drives the sun gear to rotate, and further drives the surrounding planetary gears which are housed in a rotating planetary carrier. Then the output shaft connected with the planetary carrier connects rotates to output the torque and rotating speed.

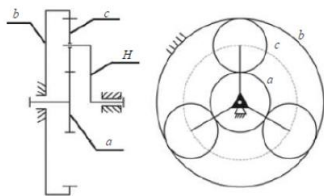


Fig.1. Planetary gear: (a) sun gear, (b) internal (ring) gear, (c) planets, H (planet carrier)

Number of planet gears can be different and they are typically spaced at regular intervals around the sun. More planet gears allow better force balance and lower forces by more load sharing. However, number of the additional planet gears do not change the kinematic but dynamic characteristics of the mechanism.

During the operation of planetary mechanisms, there are three phases in their movement: the start-up phase, the stationary mode of operation and the stopping phase. During the first phase, the speed of the driving member increases from zero to maximum and the work of the driving forces is greater than the work of the resistance forces. In the stationary mode, the work of the driving forces for a full cycle is equal in absolute value to the work of the resistance forces. During the stopping phase, the speed of the driving member decreases to zero, and the work of the resistance forces is greater than the work of the driving forces. (The resistance forces are understood as all the forces that oppose the movement of the mechanism).

It is necessary the drive unit to enable uninterrupted operation of the mechanism in all three phases, i.e. to provide a sufficiently large

input torque that will be transmitted to the drive shaft of the planetary gear and to provide appropriate angular velocity of the input shaft at which the material moment of inertia is reduced.

The general form of the Lagrange equation of motion

$$\frac{d}{dt} \left(\frac{\partial E_k}{\partial \dot{q}_i} \right) - \frac{\partial E_k}{\partial \dot{q}_i} + \frac{\partial E_p}{\partial \dot{q}_i} = Q_i$$

applied to a planetary gear train is ;

$$\frac{d}{dt} \left(\frac{\partial E_k}{\partial \dot{\varphi}} \right) - \frac{\partial E_k}{\partial \dot{\varphi}} + \frac{\partial E_p}{\partial \dot{\varphi}} = M_i \quad (1)$$

where E_k is kinetic energy of the mechanism, φ is angle of rotation (generalized coordinate), $\dot{\varphi}$ is angular velocity (generalized velocity), M_i is generalized moment. The member $\frac{\partial E_p}{\partial \dot{\varphi}}$ takes over the action in the mechanism of the forces from the weight and the elasticity of the members. These forces can be neglected since their influence is very small and the elasticity of the members of the planetary mechanism can be avoided by increasing their stiffness [1]. For efficient use of equation (1), we reduce the moments M_i of all members of the planetary mechanism to the input shaft and replace the total action of the thus reduced moments with a single member M^* for which we further use the term reduced moment of inertia. The work of the reduced moments with respect to the possible displacements is equal to the total work of all forces acting on the mechanism with respect to the possible displacements, so equation (1) takes the form [2-3]

$$\frac{d}{dt} \left(\frac{\partial E_k}{\partial \dot{\varphi}} \right) - \frac{\partial E_k}{\partial \dot{\varphi}} = M^* \quad (2)$$

We replace the mass material moments of inertia of all the components of the planetary mechanism with a single material moment of inertia I^* reduced to the input shaft. I^* is called the reduced mass material moment of inertia.

$$I^* = \sum_{i=1}^n \left[J_{Si} \left(\frac{\omega_i}{\omega} \right)^2 + m_i \left(\frac{v_{Si}}{\omega} \right)^2 \right] \quad (3)$$

The following equations apply to planetary mechanisms:

$$E_k = \frac{I^* \cdot \dot{\varphi}^2}{2} \quad (4)$$

$$\frac{\partial E_k}{\partial \dot{\varphi}} = I^* \cdot \dot{\varphi} \quad (5)$$

$$\frac{\partial E_k}{\partial \dot{\varphi}} = \frac{1}{2} \frac{dJ^*}{d\varphi} * \dot{\varphi}^2 \quad (6)$$

$$\frac{d}{dt} \left(\frac{\partial E_k}{\partial \dot{\varphi}_i} \right) = J^* * \ddot{\varphi} + \frac{dJ^*}{d\varphi} * \dot{\varphi}^2 \quad (7)$$

By substituting (4), (5), (6) and (7) into (2) we obtain the following nonlinear differential equation (8) :

$$J^* * \ddot{\varphi} + \frac{1}{2} \frac{dJ^*}{d\varphi} * \dot{\varphi}^2 = M^* \quad (8)$$

By solving (8), we determine the value of the actual angular velocity of the input (drive) shaft on which the sun gear is mounted and on which the reduction is performed.

$$\omega_i = \sqrt{\frac{2}{J_i^*} \int_{\varphi_0}^{\varphi_i} M^* * d\varphi + \frac{J_0^*}{J_i^*} * \omega_0^2} \quad (9)$$

$M^* = M^*(\varphi, \omega, t) = M_p - M_0$, where M_p is moment of motion, M_0 is moment of resistance forces. J_0^* is the reduced mass moment of inertia at the initial moment.

Considering that in gear mechanisms the transmission ratio does not change during operation, the reduced mass moment of inertia remains unchanged, i.e. $J_0^* = J_i^*$ equitation (9) will be [4] :

$$\omega_i = \sqrt{\frac{2}{J_i^*} \int_{\varphi_0}^{\varphi_i} M^* * d\varphi + \omega_0^2} \quad (10)$$

From equitation (10) it is obvious that the angular velocity depends on the reduced mass moment of inertia J^* .

2. Mass moment of inertia reduced to the axis of the sun gear

In this paper we will further examine changes in the mass moment of inertia of the planetary gear mechanism reduced to the axis of the sun gear depending on the ratio between the radii of the sun gear and planet gears. For this purpose, we first determine the expression for calculating the mass moment of inertia reduced on the sun gear using the law of kinetic energy [5-6].

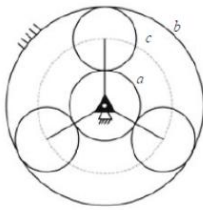


Fig.2. Scheme of planetary mechanism

$$E_k^* = E_{Ka} + N E_{Kc} + E_{KH} \quad (11)$$

E_k^* – kinetic energy of the system

E_{Ka} -sun gear kinetic energy

E_{Kc} - kinetic energy of one planetary gear

E_{KH} - planet carrier kinetic energy

N - number of planetary gears

$$E_k^* = J^* * \frac{\omega_a^2}{2} \quad (12)$$

ω_a [1/s] - angular velocity of the sun gear

$$E_{Ka} = \frac{J_a * \omega_a^2}{2} = \rho_a \pi h R_a^4 \frac{\omega_a^4}{4} \quad (13)$$

$$J_a = \frac{m_a * R_a^2}{2}; m_a = \rho_a \pi h R_a^2, \text{ where}$$

J_a - mass moment of inertia of sun gear [$\text{kg} \cdot \text{m}^2$],

m_a [kg]–sun gear mass, R_a [m]– sun gear radius, h -gear thickness

ρ_a [kg/m^3] - specific density of the material from which the sun gear is made

$$E_{Kc} = \frac{J_c * \omega_c^2}{2} + \frac{m_c * v_{sr}^2}{2} \quad (14)$$

$$J_c = \frac{m_c * R_c^2}{2}; m_c = \rho_c \pi h R_c^2, \text{ where}$$

J_c -mass moment of inertia of one planet gear [$\text{kg} \cdot \text{m}^2$]

R_c -planetary gear radius, v_{sr} - planetary gear center speed

$$v_{sr} = (R_a + R_c) * \omega_H$$

ω_H [1/s] - angular velocity of the planet carrier

ρ_c [kg/m^3] - specific density of the material from which the planetary gears are made

At the mutual contact points of the sun gear with the planetary gears, the peripheral speed is

$$R_a * \omega_a = R_c * \omega_c \rightarrow \omega_c = (R_a / R_c) * \omega_a$$

By substituting the previously mentioned equations into equation (13) we obtain

$$E_{Kc} = \rho_c * \pi * h * \frac{R_a^2}{2} * \frac{R_c^2}{2} * \omega_a^2 + \rho_c * \pi * h * \frac{R_c^2}{2} * (R_a + R_c)^2 * \omega_H^2 \quad (15)$$

$$E_{KH} = \frac{J_H * \omega_H^2}{2} \quad (16)$$

where

$$J_H = m_H * \frac{(R_a + R_c)^2}{2}, m_H = \rho_H * \pi * (R_a + R_c)^2 * h$$

m_H [kg] - mass of of the carrier H,

J_H - mass moment of inertia of the carrier H [$\text{kg} \cdot \text{m}^2$]

ρ_H [kg/m^3] - specific density of the material from which the carrier H is made,

Using above equations for J_H and m_H equitation (15) will be transformed in

$$E_{KH} = \rho_H * \pi * h * \frac{(R_a + R_c)^4}{2} * \frac{\omega_H^2}{2} \quad (17)$$

According to the Willis Method for epicyclic mechanisms, for the ratio of angular velocities in the planetary mechanism we obtain[7-9]

$$i_{1H} = \frac{\omega_a}{\omega_H} = 1 - \frac{d_b}{d_a} = 1 - \frac{(R_a + 2R_c)}{R_a} \quad (18)$$

$$\frac{\omega_a}{\omega_H} = \frac{-R_a}{2 * R_c}$$

where $R_b = R_a + 2R_c$

By substituting equations (12), (13), (15), (17) and (18) into equation (11), the following expression is obtained:

$$J^* = (\rho_a * \pi * h) * \frac{R_a^4}{2} + N(\rho_c * \pi * h) * \frac{2R_a^2 R_c^2 + R_a^2 (R_a + R_c)^2}{4} + \rho_H * \pi * h_H * (R_a + R_c)^4 * \frac{R_a^2}{4 * R_c^2} \quad (19)$$

Further, using equation (19), the values for the reduced mass moment of inertia were calculated. It was assumed that all parts of the planetary mechanism are made of same material with a density ρ [kg/m³]. The radius of the internal (ring) gear (b) remains constant during the calculations. Thickness of the carrier h_m , which has shape of a circular plate, is $h_H = h/k$, where h is thickness of gears.

$$J^* = (\rho * \pi * h) * \left(\frac{R_a^4}{2} + \frac{N * (2R_a^2 R_c^2 + R_a^2 (R_a + R_c)^2)}{4} + \frac{(R_a + R_c)^4 * R_a^2}{4 * k * R_c^2} \right) \quad (20)$$

From equation (20) it is clear that for a given density of the material from which the mechanism is made and a given thickness of the gears, the value of the reduced mass moment depends on the ratio between the radii of the central and planetary gears, on the thickness of the carrier h which is taken to have the shape of a circular plate and on the number of planet gears.

3.Results and discussion

The calculation performed below is for $h_H = h/4$, i.e. the thickness of the planet carrier is 4 times less than the thickness of the gears. In table 1 are given values for reduced mass moment of inertia depending on the ratio between the radii of the sun gear and planet gears.

Table 1: Reduced mass moment of inertia of the planetary gear mechanism reduced to the axis of the sun gear depending on the ratio between the radii of the sun and planet gears for 3,4,5,7 and 9 planet gears

Reduced mass moment of inertia for 3,4,5,7 and 9 planet gears depending on the ratio between the radii of the sun gear and planet gears									
i_{aH}	Ra	Rc	Ra/Rc	$J^*_{(N=3)}$	$J^*_{(N=4)}$	$J^*_{(N=5)}$	$J^*_{(N=7)}$	$J^*_{(N=9)}$	
0,176	0,85	0,075	11,3	6,608	6,764	6,921	7,234	7,547	
0,250	0,8	0,1	8,00	3,228	3,360	3,493	3,759	4,024	
0,333	0,75	0,125	6,00	1,813	1,925	2,037	2,262	2,486	
0,429	0,7	0,15	4,67	1,113	1,207	1,301	1,489	1,677	
0,538	0,65	0,175	3,71	0,724	0,802	0,880	1,037	1,194	
0,667	0,6	0,2	3,00	0,490	0,554	0,619	0,749	0,878	
0,818	0,55	0,225	2,44	0,340	0,393	0,446	0,552	0,658	
1,000	0,5	0,25	2,00	0,239	0,282	0,325	0,411	0,497	
1,222	0,45	0,275	1,64	0,170	0,204	0,238	0,307	0,375	
1,500	0,4	0,3	1,3	0,120	0,147	0,173	0,227	0,281	
1,857	0,35	0,325	1,08	0,084	0,104	0,125	0,166	0,206	
2,333	0,3	0,35	0,86	0,057	0,072	0,087	0,117	0,147	
3,000	0,25	0,375	0,67	0,038	0,048	0,059	0,080	0,101	
4,000	0,2	0,4	0,50	0,023	0,030	0,037	0,050	0,064	
5,667	0,15	0,425	0,36	0,013	0,017	0,021	0,028	0,036	
9,000	0,1	0,45	0,22	0,006	0,007	0,009	0,013	0,016	
19,00	0,05	0,475	0,10	0,001	0,002	0,002	0,003	0,004	

In the upper half of Table 1, when the gear ratio is greater than 1, the planetary gear mechanism functions as a multiplier. In this case, the angular velocity of the sun gear is less than the angular velocity of the carrier. For a gear ratio of 1, the radius of the sun gear is twice the radius of the planet gears and in this case the angular velocities of the sun gear and the planet gears are equal. When the gear ratio is greater than 1, the planetary gear mechanism functions as a reducer and in this case the angular velocity of the sun gear is greater than the angular velocity of the carrier.

The dependence of the reduced mass moment of inertia on the ratio between the radii of the sun gear and planetary gears is shown at figure 3.

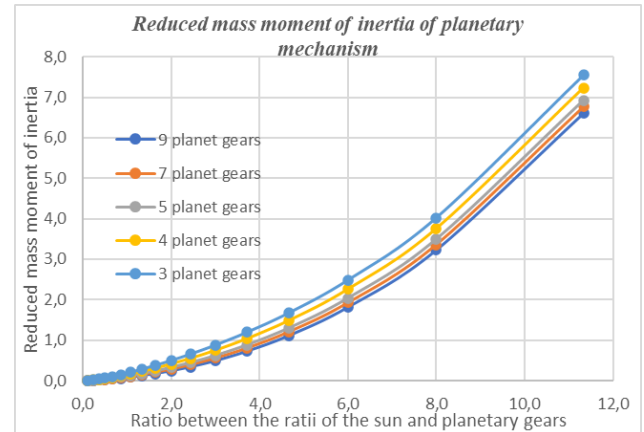


Figure 3: Reduced mass moment of inertia of planetary mechanism

When the number of planet gears is 9, the reduced moment of inertia as a function of the ratio of the radii of the sun gear and planet gears is given by the approximate equation

$$y = 0.0517x^2 - 0.0073x + 0.0243 \quad (21)$$

Based on the data in the Table 1 and Figure 3 it can be concluded that with an increase in the ratio between the radii of the sun gear and the planet gears, the value of the mass moment of inertia of the planetary mechanism reduced to the sun gear axis also increases. This dependence is described by second order curve - parabola. By increasing the number of planet gears, the coefficient in front of the quadratic element (21) also increases.

In table 2 are represented calculated the percentage increases of the reduced mass moment of inertia when the planetary mechanism has 4, 5, 7 or 9 planet gears compared to the reduced moment when there are only 3 planet gears.

Table 2. Percentage increases of the reduced mass moment of inertia when the planetary mechanism has 4, 5, 7 or 9 planet gears

Increase in the reduced mass moment of inertia with 4, 5, 7 and 9 planetary gears compared to the reduced mass moment of inertia with 3 planetary gears [in percentage]					
i_{aH} ratio	Ra/Rc	$J^*_{N=4}/J^*_{N=3}$	$J^*_{N=5}/J^*_{N=3}$	$J^*_{N=7}/J^*_{N=3}$	$J^*_{N=9}/J^*_{N=3}$
-0,176	11,33	2,37	4,74	9,48	14,22
-0,250	8,000	4,11	8,23	16,46	24,69
-0,333	6,000	6,18	12,36	24,72	37,08
-0,429	4,667	8,45	16,90	33,80	50,70
-0,538	3,714	10,83	21,65	43,31	64,96
-0,667	3,000	13,24	26,47	52,94	79,41
-0,818	2,444	15,62	31,25	62,50	93,75
-1,000	2,000	17,96	35,92	71,84	107,76
-1,222	1,636	20,21	40,42	80,85	121,27
-1,500	1,333	22,36	44,71	89,42	134,14
-1,857	1,077	24,37	48,73	97,46	146,19
-2,333	0,857	26,21	52,42	104,84	157,26
-3,000	0,667	27,86	55,71	111,43	167,14
-4,000	0,500	29,28	58,56	117,12	175,67
-5,667	0,353	30,45	60,91	121,81	182,72
-9,000	0,222	31,37	62,74	125,47	188,21
-19,00	0,105	32,02	64,05	128,09	192,14

The results of the table 2 are graphically shown on the figure 4.

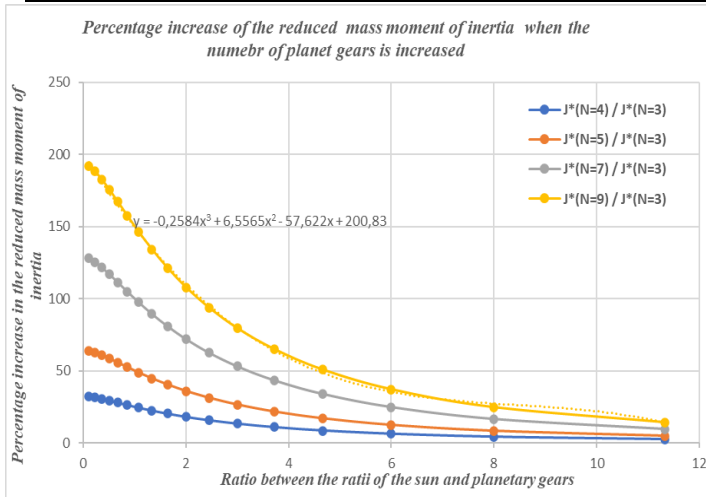


Figure 4: Percentage increases of the reduced mass moment of inertia when the planetary mechanism has 4, 5, 7 or 9 planet gears compared with 3 planet gears mechanism

From the data in Table 2 represented at Figure 4 it can be concluded that at the same ratio of the radii of the sun gear and planetary gears, with an increase in the number of planetary gears, the value of the reduced mass moment of inertia also increases. The percentage increase in the reduced mass moment of inertia when the number of planet gears increases is greatest at the largest planetary mechanism ratios, that is, at the smallest ratios of the radii of the sun gear and planet gear. With an increase in the ratio between the radii of the sun gear and planetary gear, this percentage increase decreases. It follows that the percentage increase is smaller in cases when the planetary mechanism works as a multiplier than when it works as a reducer.

4. Conclusion

By changing the ratio of the radii of the sun gear and planet gears in planetary mechanisms, the mass moment of inertia reduced to the sun gear axis is also changed, and with it the reduced torque M^* is also changed, which directly affects the characteristics of the planetary mechanism. On the other hand, an increase in the reduced mass moment of inertia can be achieved by increasing the number of planetary gears. The number of planetary gears does not affect the kinematic characteristics, but it does affect the moment of inertia.

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Dynamic behavior of RC structures with post-tensioned slabs

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Abstract: The seismic behaviour of RC structures is one of the main factors in the design of buildings. For the best performance, a series of factors such as: the geometry in plan and height, the physical characteristics of the materials used, time of construction or the choice of the type of horizontal structure are listed. The scope of this presentation is the impact of post-tensioned slabs and dynamic response of RC structure using the response spectrum method according to EC2 & EC8, by 3D Model FEM solution (ETABS software).

Keywords: SEISMIC BEHAVIOUR, POST-TENSIONED, RESPONSE SPECTRUM

1. Introduction

Construction technology has achieved great development in the last years. Today's demand from construction companies, citizens for good quality, safety and short period of construction constitute the criteria for the architectural and structural solution.

Post-tensioning technology has its beginnings a long time ago. But their use has advanced not only in the construction of bridges, walls with anchors, but also in the foundations and floors of civil buildings.

A great research-scientific work was done in Switzerland, Holland, Denmark and Germany for the use of post-tensioning technology, which is still unknown in some countries [8],[12]. Joint efforts by researchers, design engineers and pre-construction firms resulted in relevant standards and recommendations helping to promote the widespread use of this form of construction, their inclusion in European norms (see prEN1992-2004) [3].

The calculation of post-tensioned slabs bonded or non-bonded, the loads combination are now included in FEM solution, modeling all the structure in ETABS software [4], according to EC0, EC2 and EC8 criteria.



Fig.1 Anchoring devices [9]



Fig. 2 Hydraulic pump



Fig. 3 Storage of tendons[11]

2. Analysis

2.1 Design principles of post-tensioned structures

The Ultimate Limit State (ULS) for members pre-stressed in bending and axial loading is based on the assumptions given in EC2 6.1(2) [1].

- The distribution of deformations in pressed concrete is accepted according to the hypothesis that plane sections remain plane [2].

- Calculated stresses in pressed concrete are taken as ηf_{cd} ($\eta=1$, for $f_{ck} \leq 50\text{MPa}$). The maximum deformations in the extreme fibers of pressed concrete will be considered equal to $\epsilon_{cu3} = 0.0035$, $f_{ck} \leq 50\text{MPa}$ (prEN1992-1-1 Table 3.1)[3]

- The tensile strength of concrete is not taken into consideration.

- The deformations in the connected post-tensioned tendons and in the usual additional reinforcement (compression or tension) are the same as those of the surrounding concrete [9].

The Serviceability Limit State (SLS) for pre-stressed elements is based on the following assumptions given in prEN1992 paragraph 7.2 [3]

- The elastic behavior is limited by the stress values in concrete given in EC2 7.2(3) [3].

- In general, it is necessary to calculate the stresses due to the loads that act immediately after pulling the tendon as well as after the development of all losses [9]; in both cases the effects of permanent load and utilization on deformations and forces in the tendon can be neglected [1],[10].

Post-tensioned elements in concrete are checked in three stages:

- In assembly
- In charge of service
- In holding capacity

Pre-stressed flexural elements are classified on the basis of tensile strength f_t , ultimate fiber tensile stress from pre-stressing at service load (BS 4.1.3) [5] as follows:

Class 1 (no cracking) – no tensile bending stresses

Class 2 (with invisible cracks) – with tensile stresses from bending

Class 3 (cracked) – with tensile bending stresses and with surface crack widths as follows:

- Crack width ≤ 0.1 mm for exposure class elements XS2, XS3, XD2, XD3, XF3 and XF4 (prEN1992-1-1, Table 4.1)
- Crack width ≤ 0.2 mm for all elements [

For the design of post-tensioned concrete, the engineer must also specify the load (PT) coming from the tendon (balancing load)[11]. Below are the possible combinations of PT load in different phases.

- The combination according to the condition of the initial service without considering the losses for a long time

1.0D + 1.0PT- TRANSFER

- Combination according to the limit state of the service considering the losses for a long time

1.0D + 1.0PT
 1.0D + 1.0L + 1.0PT- FINAL
 - Combination according to the limit state of destruction for PT-FINAL+HP

2.2. Loads in the models taken in the study

Dead Load DL

- volume weight of concrete = 24 kN/m³

(Super Dead Gk)

- floor layers = 1.8 kN/m²

- ceiling = 0.5 kN/m²

- furniture, temporary partitions = 1.0 kN/m²

- walls = 2.5

Gk = 5.8 kN/m²

Qk (Live Load LL) P = 2 kN/m² (for residence)

For design, the combination of extremes (Envelope) was taken:
 The results for each case of loading and combination are compared giving the min and max values [4].

2.3 Dynamic analysis in ETABS

Modal analysis determines the type and number of shapes that are analyzed in the model. Although an unlimited number of shapes can be defined, in most cases the first shapes are sufficient to represent the dynamic response of the structure. For a certain number of oscillation forms, the results of each modal analysis give their own values (period, cyclic frequency, circular frequency). The dynamic analysis of the structure can be performed with the method of eigenvalues or with the method of Ritz vectors.

- Analysis of eigenvalues (Eigenvector Analysis): determines the forms of free oscillations and the frequencies of the system which provide a complete information about the behavior of the object.

- Analysis with Ritz vectors (Ritz-vector Analysis): Shapes are determined by taking into consideration the spatial distribution of the dynamic load, which gives more accurate results than using the same number of natural shapes. The forms of Ritz vectors do not represent the internal characteristics of the structure as the eigenvalue method does. [3],[4]

Response Spectra, EC-8 Pren 1998-1:2003(E)

$a_{gR}=0.27g$

$\gamma_i=1$

$S=1.35$, $T_B(s)=0.20$, $T_C(s)=0.8$, $T_D(s)=2$;

$q=q_0 \cdot k_w \geq 1.5$; DCM, Frame system; $q=3.9$

EC-8; Tabela 5.1, $q_0=3.0 \cdot \alpha_u/\alpha_1$; $\alpha_u/\alpha_1=1.3$; $k_w=1$

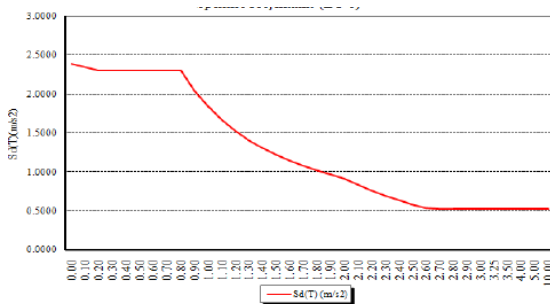


Fig. 4 Design spectrum according to EC-8

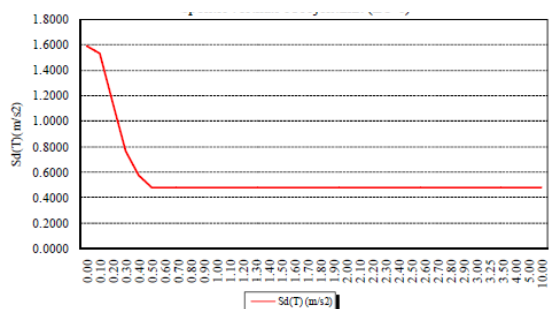


Fig. 5 Vertical design spectrum according to EC-8

Table. 1 Mechanical characteristics of materials [2], [5]

Name	Material	E (MPa)	Coefficient Poisson ν	Pesha Volumore (kN/m ³)	Rezistenca Ilogaritese
Steel	Rebar BS 4449-2005 (Klasa C)	200000		76.98	$F_{yk}=500$ MPa, $F_{yk}=625$ MPa
Steel	Rebar DIN 488-2-2009 (Klasa C)	200000		76.98	$F_{yk}=500$ MPa, $F_{yk}=540$ MPa
Steel	Rebar D.M. 14/01/2008 (Klasa C)	200000		76.98	$F_{yk}=450$ MPa, $F_{yk}=540$ MPa
Concrete	C25/30	31000	0.2	24	$f_{ak}=25$ MPa
Concrete	C30/37	32000	0.2	24	$f_{ak}=30$ MPa
Concrete	C35/45	34000	0.2	24	$f_{ak}=35$ MPa

Table 2. Mechanical characteristics of tendons [6], [7], [11]

Diametri	Standarti	Tipi i kavos	Diametri nominal	Masa per ajezi te gjatesise	Rezistenca ne Tërheqje (Kufiri i Keputjes) f _{tk}	Sforcimi ne Kufirin e Elasticitetit $\epsilon=0.1\%$ (fy)	Ngarkesa Tendonit ne Kufirin e Keputjes (P _{tk})	Ngarkesa e Tendonit ne Kufirin e Elasticitetit $\epsilon=0.1\%$ (P _{0.1k})	Moduli i Elasticitetit
			mm	g/m	MPa	MPa	kN	kN	MPa
(T15)	Pr EN 10138	standart	15.2	1095	1860	1600	260	224	196
		super	15.7	1170	1860	1600	279	240	196
(T15)	ASTM A779/90 pr EN 10138	kompakt	15.2	1290	1860	1600	300	258	196
0.5" (T13)	BS 5896/80	standart	12.5	750	1860	1600	164	139	196
		super	12.9	785	1860	1600	186	158	196
	ASTM A416/98	Grade 270	12.7	775	1860	1600	183.7	156	196
0.6" (T15)	Standard francez pr EN 10138	standart	15.2	1095	1860	1600	260	230	196
		super	15.7	1170	1860	1600	279	248	196
0.6" (T15)	BS 5896/80	standart	15.2	1090	1860	1600	232	167	196
		super	15.7	1180	1860	1600	265	225	196
0.6" (T15)	ASTM A416/98	Grade 270	15.24	1095	1860	1600	248	220	196
0.6" (T15)	ONORM B 4258	standart	15.2	1095	1860	1600	248	220	196
		super	15.7	1170	1860	1600	265	236	196
0.6" (T15)	ASTM A779/90	kompakt	15.2	1290	1860	1600	300	264	196

3. Objective and research methodologies

To make comparisons between R/C slabs and post-tensioned slabs, a typical plan (30x30) m has been studied and 4 possible solutions for the structure have been given. The procedure for designing post-tensioned slabs in the ETABS program is a novelty of this version [4], which, together with the frame structure, models post-tensioned slabs, giving a clearer overview of the characteristics of R/C structures and the effects of post-tensioning during dynamic analysis. The program performs hyper-static analysis, which redistributes the moments that come as a result of the post-tensioning of the slab in undetermined structures. The design of the slab begins with the definition of the calculation belts in two directions perpendicular to each other. Usually these belts correspond to the area of the slab supports. The axial forces, moments and shear forces for a belt are determined by analysis based on the Wood-Armer technique [4], [5] and the bending calculation is based on the Ultimate Limit State. The columns and beams are ordinary R/C and their dimensioning is made referring to the recommendations of EC-2 [3] and the fulfillment of the criteria according to the SLS serviceability and ULS ultimate limit state.

The structures were considered fixed to the base, so the soil-structure interaction was not taken into account (it is not the object of this study).

Modeling of a 5-story reinforced concrete building with (10x10)m spaces; Dual system (Frame + R/C Wall).

The calculations include 4 types of reinforced concrete slabs with beams in two directions (10x10)m:

- Model IIIa, $h_s=35$ cm, R/C;
- Model IIIb, $h_s=35$ cm, PT
- Model IIIc, $h_s=30$ cm, PT;
- Model IIId, $h_s=27.5$ cm, PT

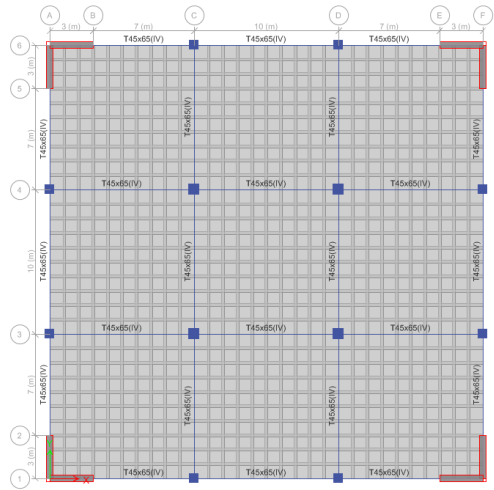


Fig. 6 Plan of R/C building

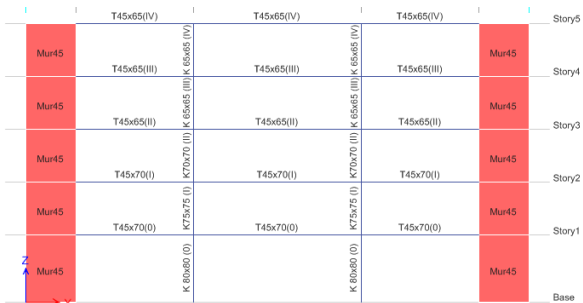


Fig. 7 Model with 5- Level (dimension)

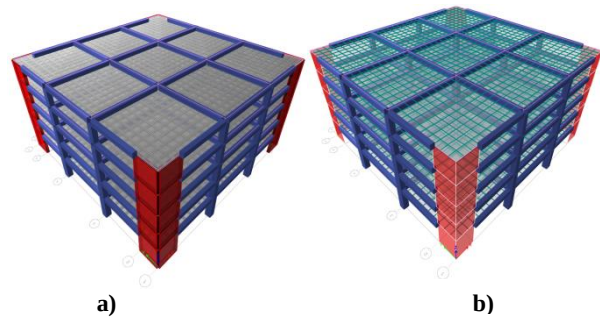


Fig. 8 Model 3D a) R/C slabs b) PT- slab

3. Results

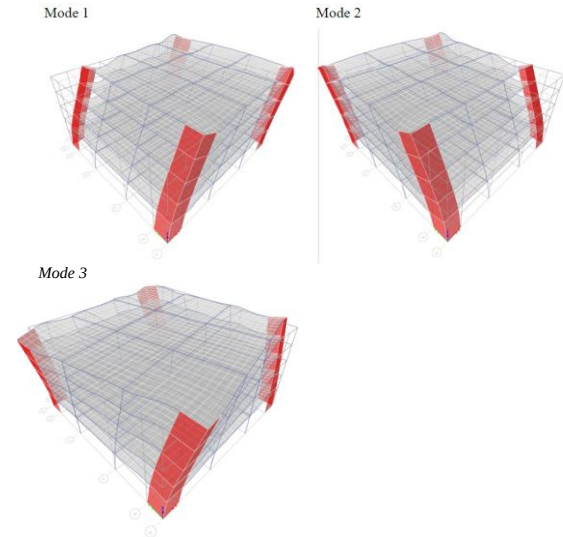


Fig. 9 Mode Shape of the model

Table 3. Periods and frequency

	Modes	Period (sek)	Frequency (cik/sek)	Cyclic frequency, rad/sek
Model IIIa	Mode 1	0.479	2.088	13.1185
	Mode 2	0.479	2.088	13.1203
	Mode 3	0.272	3.673	23.0756
Model IIIb	Mode 1	0.444	2.25	14.1368
	Mode 2	0.444	2.253	14.1548
	Mode 3	0.260	3.848	24.1747
Model IIIc	Mode 1	0.440	2.27	14.2642
	Mode 2	0.440	2.273	14.2841
	Mode 3	0.257	3.891	24.447
Model IIId	Mode 1	0.438	2.281	14.3325
	Mode 2	0.438	2.284	14.3534
	Mode 3	0.256	3.913	24.589

Table 4. Based Shear Force

Level	Model IIIa	Model IIIb	Model IIIc	Model IIId
V	4136.163	4137.7891	3999.3447	3929.9465
IV	7114.9173	7090.7821	6854.7061	6736.667
III	9180.5111	9168.1858	8862.4794	8709.8069
II	10561.9907	10561.9907	10179.5705	10003.5617
I	11277.2665	11212.6678	10836.6806	10648.962

Table 5. Seismic Force

Level	Model IIIa	Model IIIb	Model IIIc	Model IIId
V	4582.5471	4498.2924	4358.8871	4219.0361
IV	2978.7543	2952.993	2855.3614	2806.7205
III	2065.5938	2077.4037	2007.7733	1973.1399
II	1381.4796	1393.8049	1317.0911	1293.7548
I	715.2758	650.6771	657.1101	645.4003

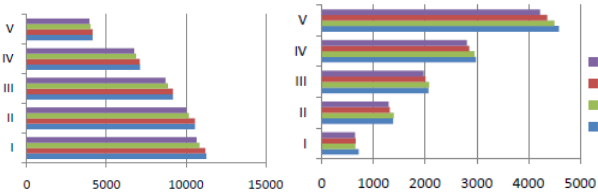


Fig. 10 a) Based Shear Force b) Seismic Force

Table 6. Relative floor displacements (Inter-storey Drift)

Level	Model IIIa	Model IIIb	Model IIIc	Model IIId
V	0.002683	0.002365	0.002572	0.002704
IV	0.002814	0.002433	0.002398	0.00238
III	0.002649	0.002291	0.002253	0.002235
II	0.002159	0.001955	0.001924	0.001908
I	0.001094	0.001098	0.001086	0.001081

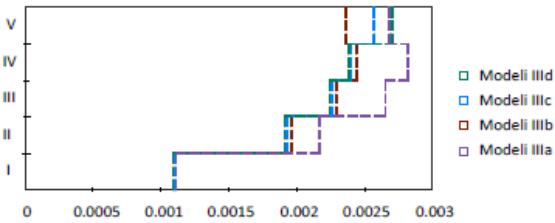


Fig. 11 Inter-story drift for model III

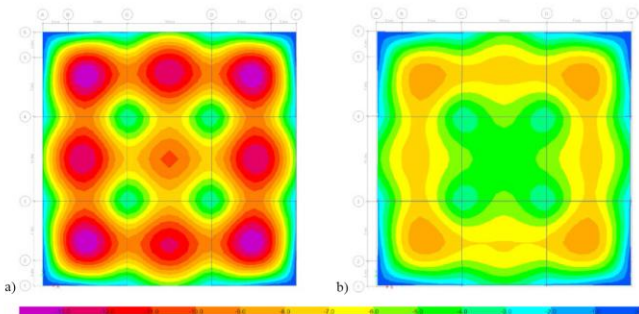


Fig. 12 Displacement Uz, a) RC slabs b) PT-slabs

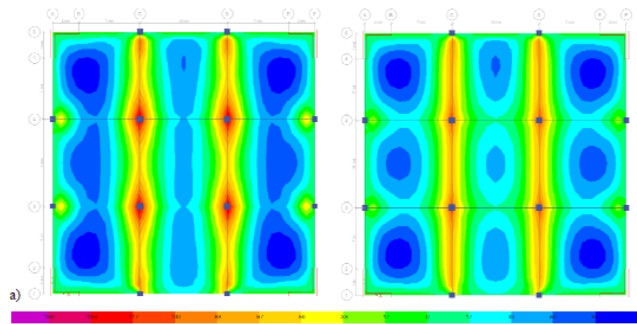


Fig. 13 Diagram of Moment M_{1-1} , a) RC slabs b) PT-slabs

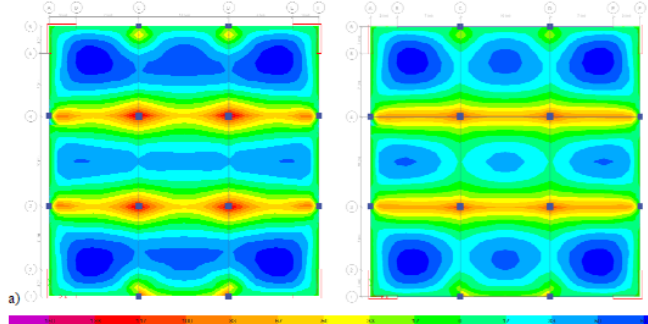


Fig. 14 Diagram of Moment M_{2-2} , a) RC slabs b) PT-slabs

4. Time-history analysis

Time-history analysis (THA) deals with the study of the response of structures to loads that vary with time, reflecting their response analytically for each value of the time variable. Time-domain analysis can be linear or nonlinear. The general equation of dynamic equilibrium that is solved:

$$M \ddot{u}(t) + C \dot{u}(t) + K u(t) = r(t)$$

where: K - stiffness matrix; C - damping matrix, M - diagonal mass matrix; u , $\dot{u}$, $\ddot{u}$ are the displacement, velocity and acceleration of the structure and r - is the acting force.

For structures under the action of seismic loads, the values of displacements - u , velocities - $\dot{u}$, accelerations - $\ddot{u}$ are relative to the ground motion. While on the right side of the equation the effective load $r(t)$ represents the seismic action caused by the ground movement with acceleration - $\ddot{u}_g$.

Other types of analysis (RSA) deal with the study of the response of structures to determine the amplitude of the maximum response.

Nonlinear analysis in the time domain (TH) performed by the ETABS software, otherwise known as FNA (fast nonlinear analysis), has a very efficient use in the case when many structural elements have linear behavior and some of them are predetermined with nonlinear behavior.

In the models taken in the study, nonlinearity is predefined through the localization of plastic hinges at the beam supports in the columns, for the cases of slabs with ordinary and post-tensioned reinforcement.

The response of the structure is again presented in the time domain by choosing as factors:

- the based shear force according to the two directions Ox and Oy ;
- the displacements U_x and U_y , the accelerations A_x and A_y for a point on the upper floor of the structures

In this study, the dynamic analysis was performed using the time domain (TH) data for two loads in the program:

the data (LACC_NOR-1.TH) and (LACC_NOR-2.TH), respectively.

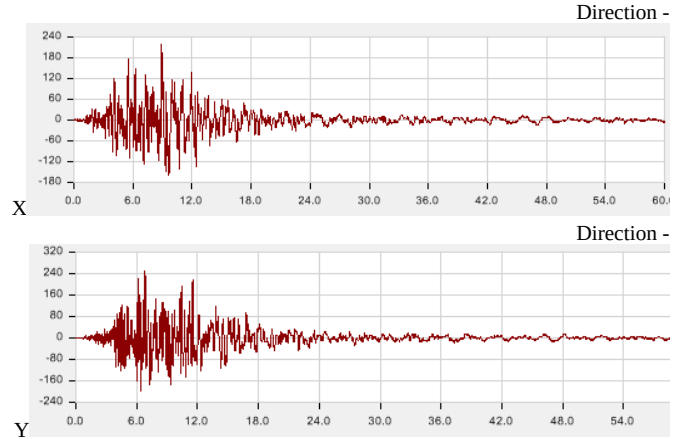


Fig. 15 Time history LACC-NOR

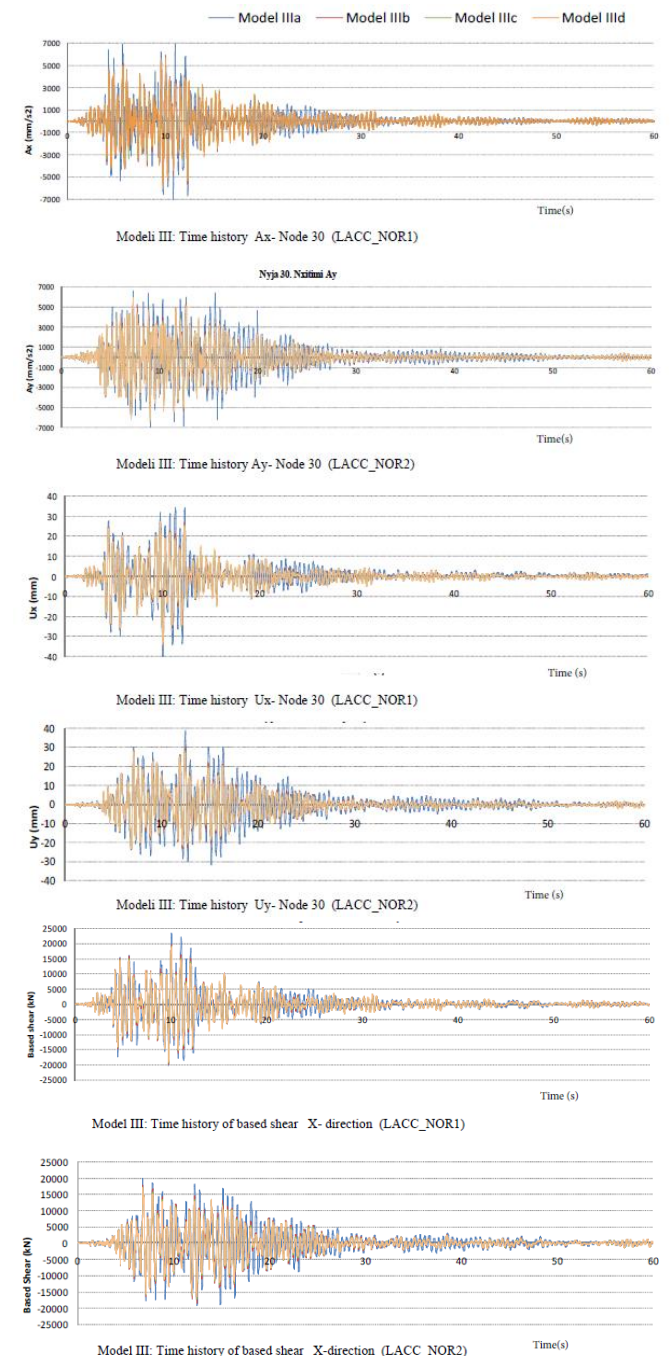


Fig. 16 Time history of Node 30

5. Conclusions

5.1 Conclusions for the Response Spectra Analysis

From the summary of the results of the static and dynamic analysis, from all the combinations made according to EC0, EC8 using the response spectrum method, it is noted that:

- For the same thickness of the slab, the solution with ordinary reinforcement results in larger deformations than the solution with post-tensioning.
- The significant increase in the resistance capacity of the slab in the case of the placement of post-tensioning tendons, better fulfilling the requirements of the code for the Ultimate Limit State (ULS). For the same external load, the internal forces in the structural elements are reduced.
- Increasing the rigidity of the structure due to the contribution of the tendons as an integral part of the structure's modeling. This is a direct result of the use of material with very superior mechanical characteristics in relation to concrete and ordinary reinforcement.
- Visible reduction of the relative displacements of the floor (inter-storey drift) better fulfilling the requirements of the code for the Serviceability Limit State (SLS).

5.2 Conclusions for the time-domain analysis

From the time-history analysis with the selected function: Direction -X (LACC_NOR1) and Direction - Y (LACC_NOR2) from the ETABS 2016 program, it is noted:

- The time response of the acceleration factors A_x and A_y ; displacements U_x and U_y for a point on the upper floor has smaller values in the case of horizontal structures realized with post-tensioned (PT) slabs. The value of the above response for the case with post-tensioning is reduced in slabs with smaller thickness.
- The time response of the base shear force B_x and B_y is reduced in models with PT slabs and with smaller thickness
- This change is not constant throughout the response time: in the interval of the largest amplitudes of the seismic action, the change is more noticeable (time interval 5-25 sec). In the rest of the interval when the amplitudes of the seismic action decrease, the response change is reduced and in some cases the same response amplitudes are observed.
- For the dynamic calculation methodology in the frequency domain according to the response spectrum (RSA) where the dimensioning is based on the maximum response values, it is clear that the level of displacements and force factors in the structure is greater.

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Thermal Effects of High-Speed Machining: Analysis of Cutting Zone Temperatures, Tool Behavior, and Cutting Forces in C45 Steel Turning at Elevated Speeds

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Abstract: High-speed machining (HSM) significantly influences the thermal dynamics of the cutting process, particularly in the tool-workpiece interaction zone. At elevated cutting speeds, the reduced tool-workpiece contact time minimizes heat transfer to the workpiece, concentrating thermal energy in the cutting zone. This results in localized material softening, reduced cutting forces, and enhanced process efficiency. This study investigates the thermal effects of HSM by experimentally turning C45 steel using a cubic boron nitride (CBN) tool at cutting speeds ranging from 800 m/min to 1800 m/min. Cutting forces were measured and analyzed in conjunction with temperature distributions within the cutting zone and tool. The experimental results were compared against predictions from the analytical Oxley machining model and numerical simulations using DEFORM software. The analysis revealed the dependence of cutting speed on cutting zone thermal properties, providing insights into material behavior under high-speed conditions. Furthermore, the study identified optimal cutting speeds that balance thermal effects and cutting process stability. These findings contribute to the definition of appropriate high-speed machining parameters, ensuring effective heat dissipation and stable tool performance.

Keywords: HIGH-SPEED CUTTING, MODELING, CUTTING FORCESS

1. Introduction

High-speed cutting (HSC) significantly differs from conventional machining due to its elevated cutting speeds, which influence various aspects of the cutting process, including cutting force development, heat generation, chip formation, and surface integrity (1). Unlike conventional machining, HSC often results in lower cutting forces due to material softening at high temperatures, improved surface quality, and a reduction in built-up edge formation. Additionally, heat dissipation mechanisms vary between the two processes, with HSC directing the majority of heat into the chip rather than the cutting tool or workpiece.

Given these complexities, numerical simulations, particularly Finite Element Method (FEM) simulations, have become an essential tool for analyzing and optimizing machining processes. Simulations allow for the detailed investigation of cutting forces, temperature distribution, stress development, and tool wear without the constraints and costs of physical experiments. The fundamental FEM simulation was based on an orthogonal 2D cutting model. Several frameworks have been developed within 2D FEM models, incorporating specialized machining methods. Hashemi et al. (2) developed an explicit Lagrangian elastic-plastic finite element model to simulate chip separation, segmentation, and interaction in high-speed orthogonal cutting. Their model incorporates a fracture algorithm to dynamically simulate chip formation and breakage, enabling a detailed analysis of chip geometry, stress, and strain distributions in critical regions. In the study by Hussain et al. (3), the effects of PCBN tool edge chamfering parameters on cutting forces, cutting temperature, and chip morphology during the hard turning of GCr15 were investigated. Using both experimental tests and Deform Finite Element Simulation, the research analyzed how variations in cutting speed, feed rate, and depth of cut influence surface quality and tool life. Karpat et al. (4) studied machining with negative rake angle chamfered tools and analyzed the influence of the dead metal zone on friction conditions and temperature distribution. They used slip-line field analysis and an analytical thermal model to predict temperature fields at the tool-chip and tool-workpiece interfaces.

However, with advancements in hardware capabilities and improvements in computational solvers, complex 3D FEM simulations are becoming increasingly popular. Kyratsis et al. (5) introduces the fundamentals of 3D Finite Element Method (FEM) applied in standard machining processes such as turning, drilling, and milling.

Moreover, simulations provide insights into machining behavior that are difficult to obtain through experimental means alone.

This study aims to leverage FEM simulations alongside analytical modeling to analyze the cutting forces and temperature distribution during high-speed cutting. By comparing simulation results with experimental measurements, this research seeks to enhance the understanding of force reduction mechanisms at high cutting speeds and contribute to the optimization of cutting speed for improved efficiency and reducing the magnitude of cutting forces.

2. Material and methods

The analytical cutting model and FEM simulation build upon the Vopát et al. (6) experimental work. A total of 11 turning experiments were conducted, with cutting speeds ranging from 800 to 1800 m/min, during which the cutting forces were measured. The cutting experiments were conducted using CNGA 120408 turning inserts. A DMG CTX alpha 500 turning center was utilized for the machining tests. The workpiece material was C45 steel, shaped as a cylindrical rod with an external diameter of 130 mm. To measure the cutting force components, a Kistler 9257B static dynamometer was employed. The cutting parameters were set with a depth of cut of 0.4 mm and a feed rate of 0.13 mm/rev.

Both the analytical Extended-Oxley model and the 3D FEM cutting simulation were performed under identical cutting conditions and utilized the same material model. The results of the cutting force measurements from the experiment, analytical model, and simulation are summarized in the graph presented in Figure 4.

2.1. Extended-Oxley Analytical model

Pantalé et al. (7) present an enhanced implementation of the Extended-Oxley analytical model for orthogonal metal cutting, originally proposed by Lalwani (8). This implementation addresses previous challenges related to solution non-uniqueness, accuracy, and computational efficiency. Extended-Oxley Analytical model predicts cutting forces and temperatures during orthogonal cutting by solving a set of nonlinear equations. The algorithm minimizes the imbalance in cutting forces, ensuring equilibrium between predicted and actual forces. The Python programming language and the LMFIT library to solve the nonlinear equations efficiently. It incorporates the Johnson–Cook thermo-elastoplastic flow law to describe material behavior under varying strains, strain rates, and temperatures.

2.2. DEFORM 3D cutting model

The FEM simulation was conducted in accordance with the experimental tests, utilizing DEFORM 3D software. To optimize computational efficiency, the tool geometry was simplified to include only the surfaces in contact with the workpiece, thereby reducing the number of elements in the tool model. A finer mesh was applied in the region closest to the workpiece to enhance the accuracy of the simulation results. The cutting tool was modeled as a rigid body, while the workpiece was defined as a plastic deformable object.

The meshing parameters were configured with a minimum node size of 0.025 mm in the cutting zone to ensure precise modeling of material deformation. Chip separation was governed by an adaptive remeshing criterion, allowing the mesh to dynamically refine in the cutting zone to maintain high accuracy. The material behavior of C45 steel was modeled using the Johnson–Cook constitutive equation, consistent with the analytical Oxley model. Thermal properties of both the tool and workpiece were set according to the DEFORM 3D material database. The initial setup of the 3D cutting model in DEFORM is presented in Figure 1.

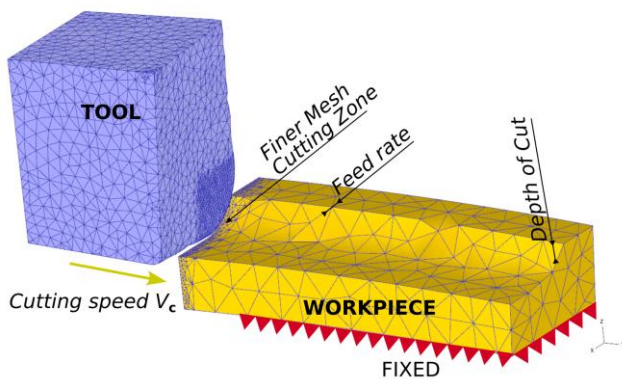


Fig.1 DEFORM 3D FEM cutting model setting

Friction at the tool-chip interface was modeled using a constant coefficient of friction, set to 0.1. The simulation employed an incremental Lagrangian solver to accurately capture large plastic deformations during the cutting process. The iterative computation continued until the predefined cutting time was reached, ensuring convergence and accuracy in force and temperature predictions. Results of the temperature mapping are shown in Figure 2.

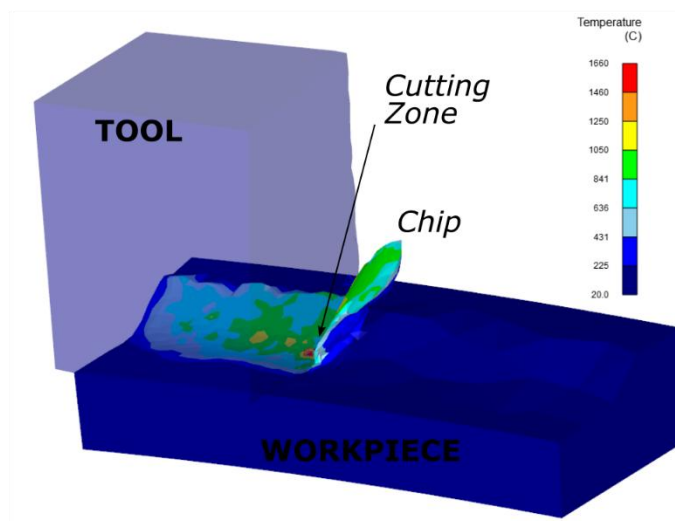


Fig.2 3D FEM simulation result

3. Result and discussion

3.1. Cutting force

The primary objective of this study was to compare the cutting forces measured in previous experimental work Vopát et al. (6) with those predicted by the Oxley analytical model and the 3D FEM simulation using DEFORM. A representation of the progression of cutting forces obtained from the FEM simulation is presented in Figure 3. The simulation results indicate that the cutting forces initially increased to a certain value before stabilizing.

It is important to highlight that the force-time history begins at the onset of the cutting process, approximately 0.2 μ s after initiation. This extremely short duration may limit the ability to obtain a fully comprehensive prediction of the cutting forces throughout the entire machining process.

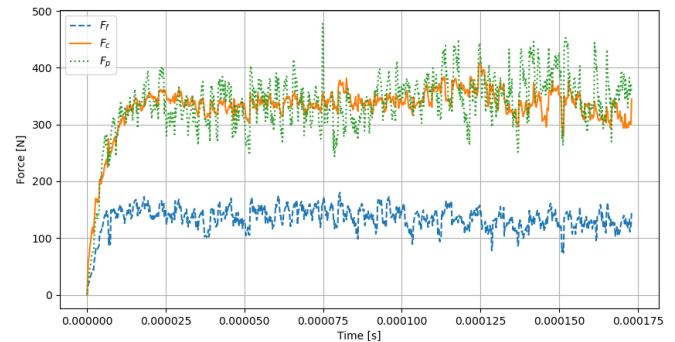


Fig. 3 Cutting forces obtained from 3D FEM DEFORM simulation

The solid lines represent the components of the cutting forces measured in the experiment Vopát et al. (6). The largest force, F_c , corresponds to the main cutting speed vector. The component F_f is of approximately the same magnitude but slightly lower. The values of the force components F_p and F_f are primarily influenced by the cutting conditions, specifically the depth of cut and feed rate. Focusing on the main cutting force F_c , an initial increase can be observed, followed by a gradual decline beyond a cutting speed of approximately 1100 m/min. This speed can be considered a critical point at which the mechanical properties of the material change due to temperature effects, leading to material softening.

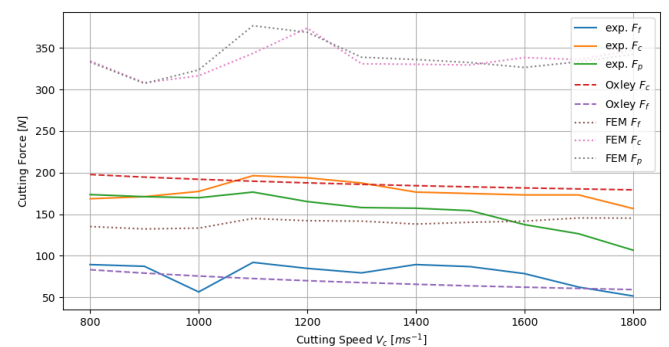


Fig. 4 Overview of cutting forces in relation to cutting speed

The analytical Oxley model provides predictions only for the F_c and F_f components of the cutting forces, as it is based on an orthogonal cutting assumption and solves for two-dimensional (2D) cutting conditions. The predicted cutting force components are represented by dashed lines, which exhibit a decreasing trend. This observed trend supports the theory that the machined material undergoes thermal softening as the temperature increases. However, since the Oxley model is an analytical approach, it does not allow for the precise identification of the critical point where the thermal balance shifts material softening, as can be determined through experimental measurements.

The dotted lines show the forces obtained by 3D FEM simulation. Comparing the values with the experimental results, the simulations show that the force component F_f is the smallest, while F_c and F_p are slightly higher. A more rigorous calibration of the material model and boundary conditions could lead to more comparable experimental and simulation results. However, the simulation shows a similar behaviour of the cutting model as the experiment. The forces initially increase, with a gradual decrease in cutting forces at a speed of approximately 1100 to 1200 ms as in the experiment. In the case of the complex 3D FEM simulation and the experiment, we find agreement in the dependence of the cutting forces on the cutting speed.

3.2. Tool Temperature Distribution

The thermal balance in the cutting process plays a crucial role, significantly influencing not only the machining operation itself but also surface quality, tool life, and overall process efficiency. As increased temperatures lead to material softening, this phenomenon can be strategically utilized to reduce cutting forces. Although temperatures were not measured during the experimental tests, both the analytical Oxley model and the FEM simulation allow for temperature monitoring and analysis. These models provide insights into temperature distribution on the cutting tool as well as within the cutting zone of the workpiece. Figure 5 illustrates the temperature distribution along the cutting edge of the tool, highlighting the thermal effects in the machining process.

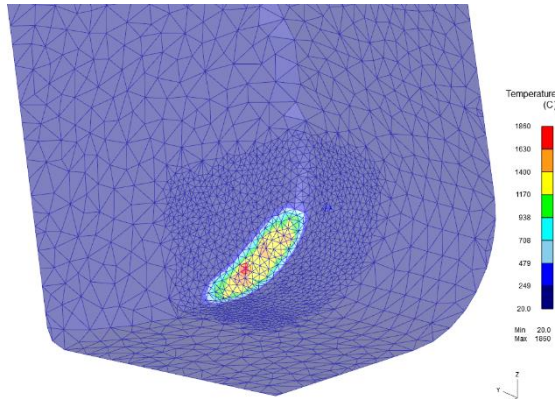


Fig. 5 Temperature distribution on the cutting edge of the tool

When analyzing temperature in the FEM cutting simulation, it is important to consider that the simulation duration is limited to only a few tenths to several microseconds. As a result, the thermal process does not reach a steady-state condition within this timeframe. It can be inferred that, for instance, the temperature of the cutting tool would continue to rise until a thermal equilibrium is achieved between heat generation and dissipation. Figure 6 presents the temperature distribution at tool point near the cutting edge of the tool over time from the initiation of the cutting process. The results indicate a continuous increase in temperature, suggesting that further heating would occur beyond the simulated timeframe.

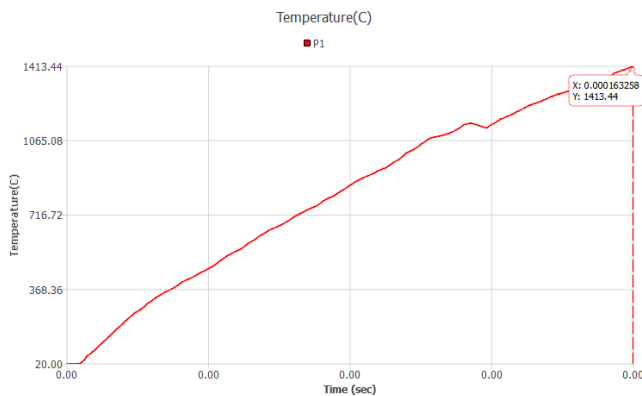


Fig. 6 Temperature distribution at tool point near the cutting edge

3.3. Heat analysis of the cutting process

Figure 6 provides an overview of the cutting zone, including the chip shape and the overall temperature distribution within the workpiece. Additionally, a histogram representing the workpiece temperature is presented, where the highest frequency corresponds to 20°C, which is expected given that the machining process was conducted at room temperature. The histogram also provides insight into the characteristic temperature distribution within the workpiece.

The workpiece temperatures, along with the maximum tool temperature, are plotted against the cutting speed in Figure 7. From this analysis, it can be observed that the maximum temperature of both the tool and the workpiece occurs at a cutting speed of approximately 1000 m/min. Notably, this corresponds to the speed at which the lowest cutting forces were recorded.

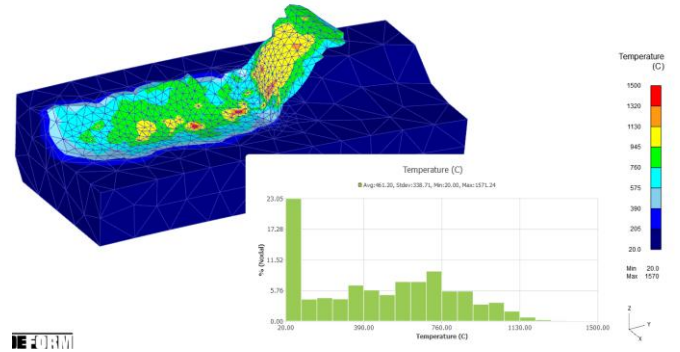


Fig. 7 Temperature distribution in cutting zone (DEFORM simulation)

It is also evident from the relationship between tool and workpiece temperatures and cutting speed that beyond a peak at 1000 m/min, the tool temperature increases while the workpiece temperature decreases. Figure 8 shows an illustration of this relationship. This phenomenon is attributed to the rate of chip evacuation from the cutting zone. According to heat balance theory, approximately 90% to 95% of the heat generated is dissipated through the chip. As a result, a higher chip evacuation rate leads to a reduction in the overall workpiece temperature under the given cutting conditions.

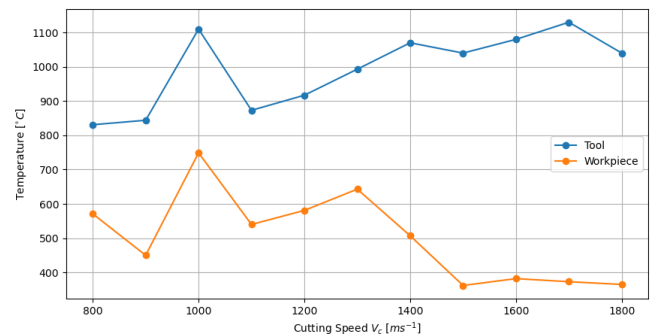


Fig. 8 Tool and workpiece temperature in relation to cutting speed

4. Conclusion

This study demonstrates the limitations of analytical modeling in predicting cutting forces and temperature distribution in high-speed machining. While the Oxley analytical model provides useful insights into cutting forces, its inherent assumptions and two-dimensional orthogonal cutting make it insufficient for accurately capturing the complex thermal and mechanical interactions in the cutting zone.

Finite Element Method (FEM) simulations offer a more detailed and flexible approach, enabling the analysis of cutting forces, chip formation, and temperature distribution under realistic cutting conditions. However, despite its advantages, the FEM simulations in

this study were constrained by the short simulation time, preventing the thermal process from reaching equilibrium. As a result, while FEM provides more accurate cutting force predictions than the analytical model, its ability to precisely analyze temperature evolution remains limited.

Moreover, the accuracy of FEM simulations strongly depends on the precise calibration of the material model and the correct definition of boundary conditions. In this study, the material behavior and heat transfer properties were consistently applied in both the analytical and FEM simulations to ensure a valid comparison. However, achieving reliable temperature predictions in FEM requires a well-calibrated material model that accurately represents strain, strain rate, and temperature-dependent behavior.

Future research should focus on extending FEM simulation durations to allow for thermal stabilization and validating temperature predictions through experimental measurements. Additionally, further refinement of material models and boundary conditions will enhance the accuracy and reliability of FEM simulations, ultimately improving the understanding of high-speed machining processes.

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Large-scale distortion analysis of the welding and thermal straightening process chain

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Abstract: An coupled analytic-numerical model for calculation of distortions arising by welding fabrication is introduced. Target of the analytical model is the calculation of the inherent strains after the local thermal-mechanical influence of the welding or thermal straightening process. Following the fabrication processing chart the strains are loaded on an elastic FE-model of the structure and the residual stresses and distortions of the whole structure are calculated. The consideration of welding and thermal straightening scenarios, inclusively the assembling stages, is done by taking into consideration the intermediate variation of the strain state in the FE-model of the structure at every processing step. The important physical relations are demonstrated. The model is intended to be used for solving industrial tasks, i.e. intending acceptable precision and calculation time as well as low simulation costs.

Keywords: COMPUTATIONAL WELDING MECHANICS, WELDING DISTORTIONS, THERMAL STRAIGHTENING, INHERENT STRAIN, FE-SIMULATION,

1. Introduction

The welding distortions are still one of the main problems in welding technology. It decreases the quality of the welded structure, complicate the smoothly accomplishment of the manufacturing process and increase the cost of post treatments. Since several decades, thermal straightening is an established technique for improving welding structures, figure 1. By the thermal straightening additional deformations are set into the structure, which lead to decreasing the out-of-plane distortion and increasing the in-plane shrinkage. Prediction and control of the welding distortions and the effect of the thermal straightening process, during the early stages of a designing cycle, allows avoiding expensive engineering correction that could occur later.



Fig. 1 Railway carriage after thermal straightening, ©ALSTOM Transport Deutschland GmbH, Salzgitter[1].

In the praxis, the straightening process guidance and parameters are chosen on the background of practical experience, complemented with additional time- and cost-intensive experiments. Unfortunately, it is impossible to capture the influence of the different structural stiffness, materials and residual stresses state after welding with this approach.

Another approach seems to be a welding - thermal straightening distortion simulation, based on the computational welding mechanics. Particularly in welding, analytical [1-8] or numerical [9-15] models achieve a solution to the problem. The application of analytical models is strongly limited due to the necessary simplifications of the physical phenomena. However, the numerical models are free from such limitations. They cover a large number of topics; one of which is the prediction of the residual distortions caused by the mutual influence of highly localized heat input, the material behavior, the initial inhomogeneous temperature and stress-strain states and the response of the whole structure as well.

Despite the benefits the numerical models, one great difficulty of their industrial utilization is the complexity of the problem formulation and necessity of number of input data that, in many cases, are not available. In addition, the conventional numerical thermal-mechanical welding simulations, combined with straightening simulations are not feasible, as they require significant computation costs and highly qualified personnel. These difficulties rise when the subject of the simulation is a large, complex welded structure, which often makes the employment of numerical methods in the praxis inefficient. Hence, there is a great demand for simple welding simulation models and approaches, which allow for quick distortions prediction. The technical literature gives among the works [5-9] a set of validated applications that are limited to simple welded joints [14-17].

To answer this need, an innovative solution approach for simulation of welding distortions has been developed in earlier works [16,18]. The approach is an integration of analytical and numerical calculation's procedures in a hybrid model. The analytical procedure calculates the shrinkage volume and strains distribution in it, representing the weld seams, based on the theoretical works of Okerblom [2], Gatovskij [4] and Vinokurov [5] implemented in a novel enhanced model [18]. The calculated strains are then applied to the global FE model in order to predict the distortions of the structure during the welding stages, as well as their residual state [18].

The aim of this article is to demonstrate the analytical models for calculation of inherent strain by welding and thermal straightening processes as well as their implementation it into the welding simulation procedure. Thus, to suggest an engineering tool that reduces the development time, costs and increases the efficiency of manufacturing.

2. Concept of the coupled analytical-numerical model

By welding, the analytical approach calculates the shrinkage volume, representing the weld seams, based on the theoretical works [3-7]. The introduction of complex semi-empirical relations allows to take into consideration the relevant factors and to reach the need adequacy. The calculated plastic strains are then loaded (as inherent strain) on the global FE model in order to calculate the distortions of the structure during the welding (after every one weld), as well as the global stress field. For this purpose, an elastic FE-analysis, which is fast and needs a simple material data definition, is carried out. The stress state in the structure, obtained from the numerical calculation, returns to the analytical one, using a loop statement for every next weld. This procedure allows taking into consideration the influence of the welding sequence as well as the changes of the clamping conditions and variation of the structure restrain.

The solution of the deformations problem, in the thermal straightening, bases on the hypothesis for prolonged accumulation of plastic strains, caused by the irregular thermal-deformational

field around the heat source, the restrain intensity and the variation of the stress state as well. This governs the basic approach for solving such problem (figure 2), in which a thermal-deformation calculation is carried out, taking into account the nonlinear material behaviour, as well as the field of initial elastic strains. The last one could be due to residual stresses, caused either after welding or already completed straightening works as well. That means the simulation of straightening follows the simulation of welding and consists of sequential calculations of plastic, i.e. inherent, strain of every one heat spot.

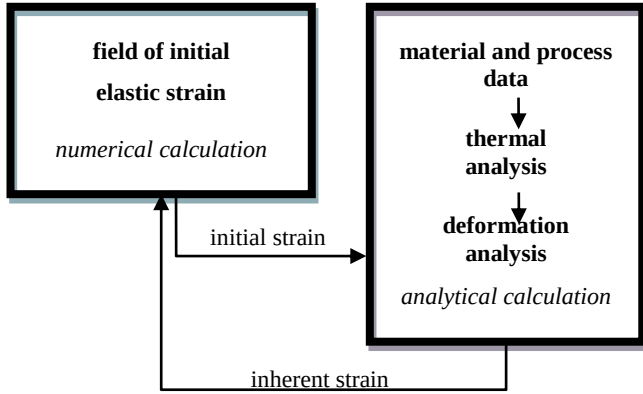


Fig. 2 Coupled analytical-numerical analysis

On this place is not worth to underline that the both, welding and thermal straightening simulations may be executed in an arbitrary sequence, following various manufacturing charts of the fabrication plan.

3. Analytical model

3.1 Welding

Purpose of the analytical model (also shrinkage model) is determination of the area where plastic strain appears. As mentioned above, scientific background of the model for welding build the plastic strains evolution under temperature dependent ideal elastic-plastic material behavior [3,5-7]. In case of long welds, the mechanical problem can be considered as uniform stress-strain in the weld cross section with known distribution of the maximal temperatures. The thermal strain (expansion and shrinkage) in the longitudinal direction is constraint by the surrounding cold metal, which realizes infinity restrain. After this model the zone with the plastic strains w_{pz} is defined by certain maximal temperatures, i.e. by the maximal thermal strain $\alpha(\Delta T_{max})$ equal to yield strain ε_Y plus initial strain ε_0 and $2\varepsilon_Y$, figure 3.

For the longitudinal component of the inherent strain, it is governed by the equation:

$$\varepsilon_x^* w_{pz} s = \int_0^s \int_0^\infty \varepsilon_x^{pl} dy dz = s \int_{\varepsilon_Y + \varepsilon_0}^{2\varepsilon_Y} y(\varepsilon_{max}^{th}) d\varepsilon_{max}^{th},$$

and for the transverse component, respectively:

$$\varepsilon_y^* = \frac{1}{w_{pz} s} \int_0^s \int_0^\infty \varepsilon_y^{pl} dy dz.$$

Herby the ε^* is the inherent strain to the corresponding component direction (x or y), s is the thickness of the sheets, w_{pz} is the width of the plastic deformed zone, ε_Y is the strain at the yield stress, ε_0 is the initial strain (that is elastic in nature), ε^{pl} are the plastic strain components (x or y), ε_{max}^{th} is the thermal strain at the maximal temperature.

Different influences on plastic strains, for example relation of the heat input to the thickness, external and internal stiffness, strain hardening, existing plastic strains in the weld, solid state transformations, clamping conditions or multi-run welding can be considered by semi-empirical correction functions [6,17]. Capturing all these effects on the arising distortions simultaneously is not possible as they also affect each other. In the technical literature is among the works [5-9] a set of applications published and compared with experimental results for simple welded joints [14-17] or large [16,18].

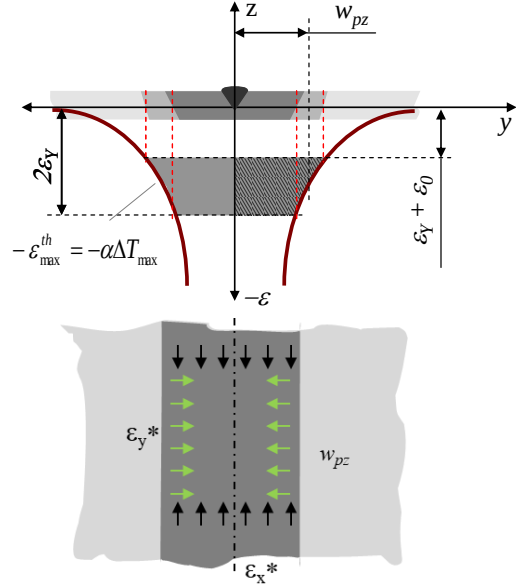


Fig. 3 Formation of plastic strain by welding

The distribution of maximal temperatures can be obtained by derivation of the quasi-stationary temperature field. In that order, diverse analytical solution can be used. The current model for welding, for example, uses a combination of moving point heat source in semi-infinity body and moving line heat source in thin plate [18,19]. The combination factor depends on the welding process, heat input and the thickness of the sheets.

3.2 Heat spot

If the heat source is fixed, as it by thermal straightening with heat spot is, the kinetics of plastic strain evolution vary. Assuming a large circular disk, subject of heating at its center and followed by cooling, an axial symmetric mechanical problem should be considered, figure 4.

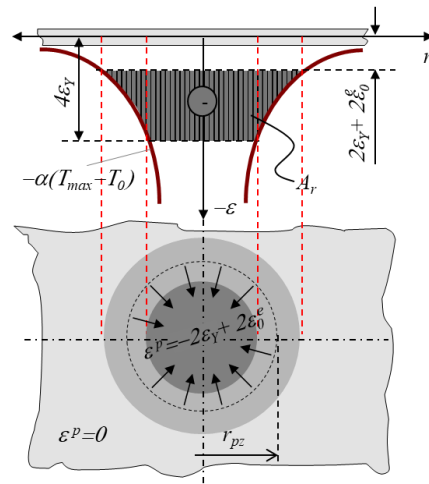


Fig. 4 Radial distribution of the plastic strains after heat spot.

In this case, the surrounding cold metal behaves elastically during both heating and cooling, and the strain occurring in it is equal to the elastic strain in the heated core. Hence, in a stress free disc the plastic strains arise in domain between maximal thermal strain $\alpha(\Delta T_{max})$ equal to yield strain $2\varepsilon_Y + 2\varepsilon_0$ and $4\varepsilon_Y$ respectively, see plastic deformed zone r_{pz} on figure 4. Presence of any initial strain will shift the thermal strain curves with its value and sign, like to heat line problem [21,22]. Thus, the shrinkage volume (v_r in $\text{mm}^2 \text{ mm/mm}$) in case of heat spot is given by the equation:

$$v_r = \int_{(2\varepsilon_Y + 2\varepsilon_0)}^{4\varepsilon_Y} A_r d\varepsilon^{th}$$

where A_r is the area bordered by the maximal thermal strain and integral limits $2\varepsilon_Y + 2\varepsilon_0$ and $4\varepsilon_Y$ respectively. Hence, the radius of plastic deformed zone is:

$$r_{pz} = \sqrt{\frac{v_r}{\pi(2\varepsilon_Y + 2\varepsilon_0)}}$$

To apply the model in the overall calculation procedure an appropriate solution of the thermal problem is need. In the common case, the solutions with instantaneous line heat source [19-23], enhanced to non-uniform initial temperature T_0 , suggested by [24] give satisfactory results.

3. Application of the analytical-numerical model

To demonstrate the capability of the coupled analytical-numerical model a welding simulation, followed by simulation of a thermal straightening is carried out. The subject is a qualitative demonstration, i.e. plausibility proof of the model. To do it, a complex structure with 8 welds was chosen [20], figure 4. It is made from two T-profiles being joined with the plate by weld 1 and 2, two L-profiles joined with the plate by weld 3 and 4 and with the T-profiles by the welds 5, 6, 7 and 8. Each wall thickness is 3 mm. The distance between T-profiles is 800 mm. The material is a mild structural steel. For the purpose of simulation, reference welding process and parameters are chosen.

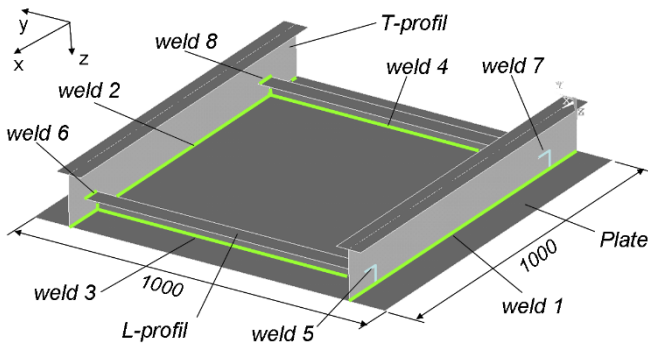


Fig. 5 Model of the complex structure with dimensions and welds (upside down)

Object of the analysis are the distortions in the sheet, closed by the frame. The blades are not considered by the analysis. According to [20] great angular distortions should be expected if at the beginning the L-profile and the T-profile are welded together (welds 5 – 8), afterwards they are joined to the sheet by welds 1 and 2 simultaneously and finally by 3 and 4.

For the distortion calculation using the hybrid model a finite element model with shell elements was generated. For the purpose of simulation, the mesh is very coarse and just refined in the supposed areas of plastic deformation, where the structure is loaded. Thus, in those domains the element size is about 20 mm (according to the analytical solution). For the reason of free forming, only the node in the middle of the plate ($x=500, y=500$) has fixed DOF (translation and rotation). The loading is realized by longitudinal

and transverse strains, linear distributed along the thickness, in three steps according to the welding scenario.

The strains in the longitudinal and transversal directions, as well as their centroid are calculated by means of the analytical model, stated above, under stress free condition. At this stage, a reference temperature distribution is applied. For the third step, the residual stresses after the second step (tension in the plate and compression in L-profile along welds 3 and 4) are taken into consideration. The obtained results are presented exemplary by the field of displacement in Z-direction (out-of-plane), figure 6.

The distortions, obtained by the coupled analytical-numerical model satisfy qualitatively the expected [20]. The average values of calculated bending and transverse shrinkage in the plate, between the T-profiles, in the middle of the structure, after welding are as follow: $\Delta Z = 3 \text{ mm}$ and $\Delta Y = 0,2 \text{ mm}$.

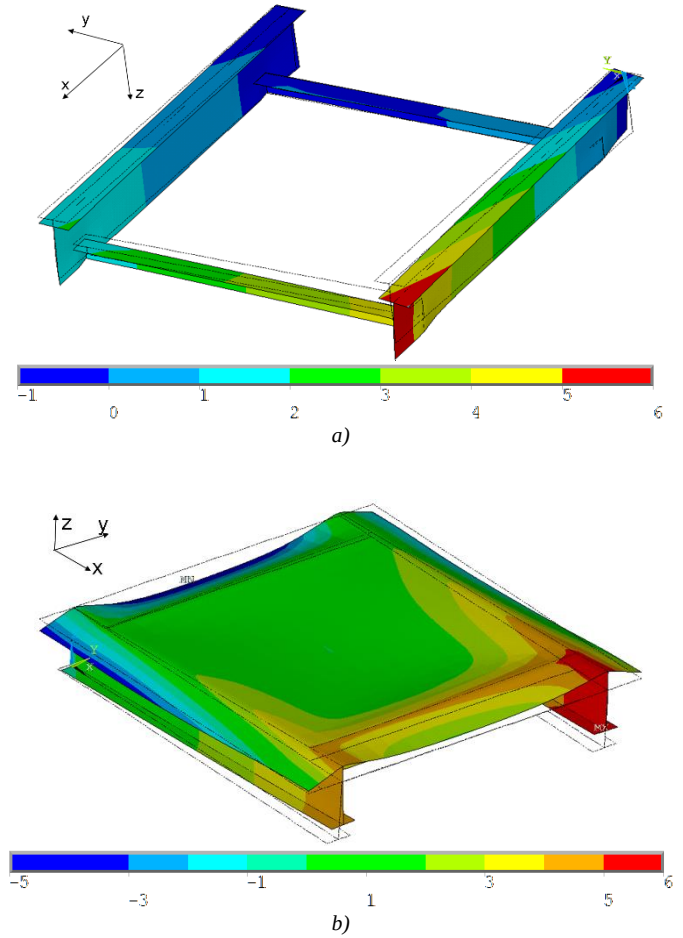


Fig. 6 Bending of the structure (displacement in z-direction, mm), scaling x75: (a) after welding the profiles, (b) after welding entire structure

Based on the calculated welding distortions a thermal straightening simulation is carried out. It is done by means of 46 heat spots, with distance about 100 mm each other. The process parameters are also reference, so that the nominal width of plastic zone corresponds to about 20 mm. The heating sequence follows a spiral trajectory, from outside to the middle, in 46 load steps, figure 7.

The inherent strains for every next spot and direction are recalculated, taking into consideration the actual strain (or stress) vector from the current element. Hence, it varies from spot to spot in the range between several times and zero. At this stage the recalculation was done according to the relation from [18,24]. The calculation time for the entire simulation, welding and straightening, took 5 – 10 min. The obtained results, after the last

spot, confirm the expected result – straightening the sheet (figure 8) and accumulating additional shrinkage in the plane. The calculation shows a reduction of the bending line to less than 1 mm, figure 9 (a). The in-plane shrinkage of the structure (exemplar distance between the T-profiles), rise from 0,2 mm to 0,25 mm (figure 9 (b), blue curve).

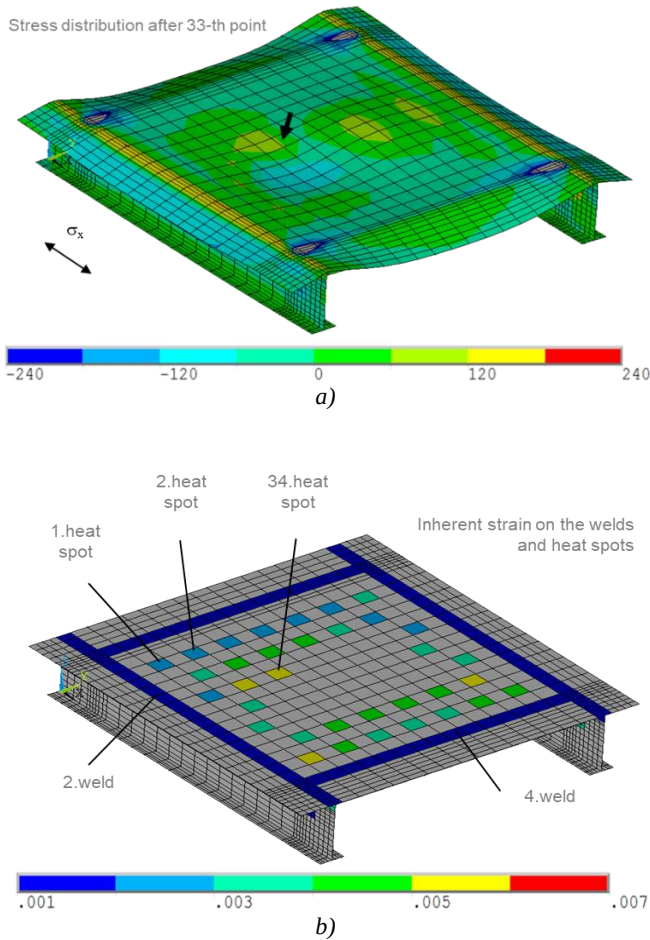


Fig. 7 Loading of the 34 heat spot: a) stress field (in MPa) after 33. heat spot, b) inherent strain, loaded to the elements at 34. heat spot

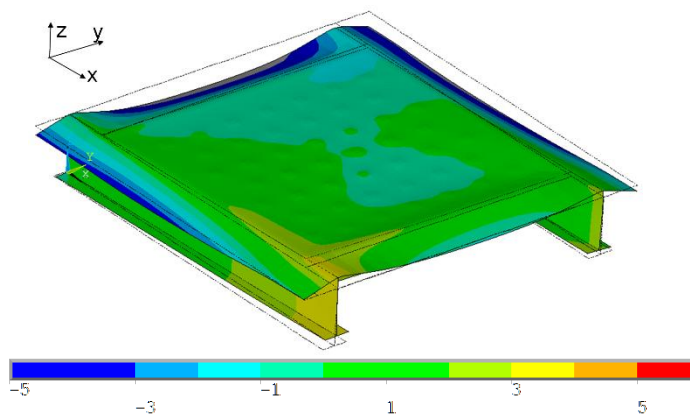


Fig. 8 Bending of the structure after straightening (displacement in z-direction, mm), scaling $\times 75$

The examination of intermediate steps shows that not all heat spots are reasonable, i.e. some of them don't affect the distortions or cause a negative effect. For example, the angular distortions after heat spot 41 are equal to the final and after 44 spot they reach the minimum. Never less, it is an evidence that the model provides

possibilities to determine the structural shrinkage as well as to analyze or optimize the straightening procedure.

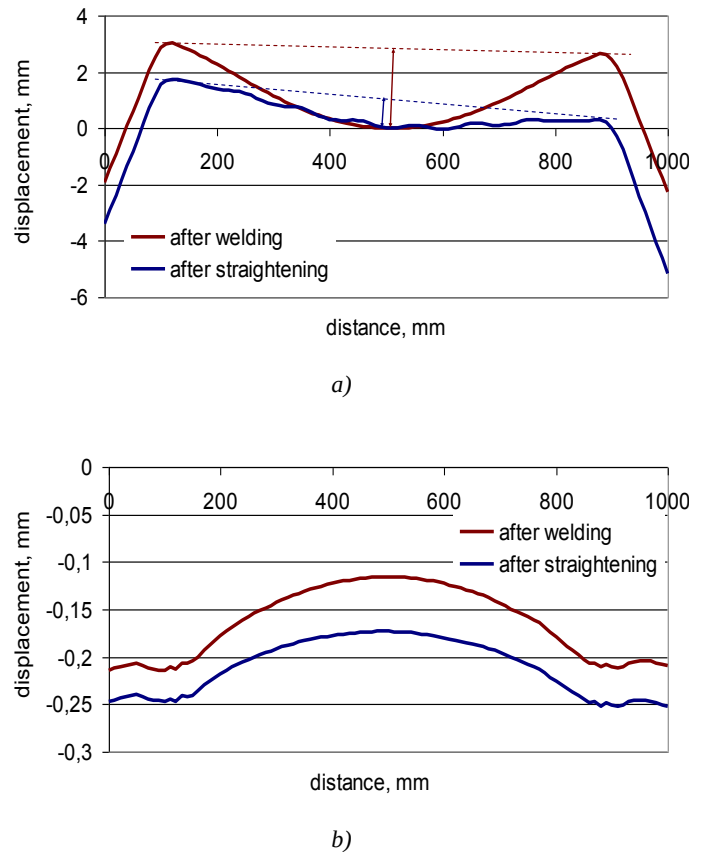


Fig. 9 Comparison between “after welding” and “after straightening”: (a) out-of-plane displacement, (b) transverse shrinkage

4. Conclusions

To find the optimal process guiding and parameters for a thermal straightening of welded structures as well as the resulting shrinkage, the procedure of time- and cost-intensive experiments is still commonly used. For this purpose, the employment of the contemporary thermal-mechanical finite-element-simulation is increasing. However, in range of large structures with multiple weld seams, the complete thermal-mechanical FE-simulation is not rational.

The limitations of the common thermal-mechanical FE-simulation, regarding the distortions calculation of large welded structures are summarized. An abstract about the existing simplified approaches as well as their inadequateness is given. Based on the need for fast and adequate precise approaches for calculating welding distortions in large structures a novel and practically relevant model is suggested and demonstrated.

To demonstrate model capability an analysis of welding distortions, after welding and subsequent thermal straightening, in a large structure has been carried out. Furthermore, the qualitative interaction of the thermal straightening and the welding processes is analyzed for one reference scenario. For this purpose, the variation of the stress state in the structure, during welding and straightening is taken into consideration. The results of the analytic-numerical hybrid model match the expertise and the expectations.

The example is performed using ANSYS® FE-code. The calculation time was about several minutes, on a common PC. The coupled analytic-numerical model shows enormous potential regarding to the reduction of the computation time, as well as significant reduction of the pre-processing time. However, there is a

demand for future research and development activities over finding the proper equation for the maximal temperature distribution, considering the most used flame and induction heat sources, as well as cooling conditions due to fixing tools. In addition, the process parameters, derivation of the material parameters for different structural materials as well as different numerical model parameters should be characterised.

Abbreviations

DOF	degree of freedom
IP	integration points
A_r	is the area bordered by the maximal thermal strain and integral limits $2\varepsilon_Y + 2\varepsilon_0$ and $4\varepsilon_Y$ respectively
b_{pz}	width of the plastic deformed zone
r_{pz}	radius of the plastic deformed zone
v	shrinkage volume
ΔY	overall in-plane shrinkage in Y-direction
ΔZ	maximal out-of plane displacement
ΔT_{max}	maximal temperature increment
T_0	initial temperature
α	thermal expansion coefficient
ε_Y	strain by yield limit
ε_0	initial elastic strain (residual strain from preliminary load steps)
ε^*	inherent strain
ε^{el}	elastic strain
ε^{pl}	plastic strain
ε^{th}	thermal strain

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Plasma jet surface hardening of tool steel - computational investigation using different modelling approaches

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Abstract: The article presents a comparison of different computational approaches used to study the surface treatment of a NiCrMoV steel with 0.55% Carbon content by a plasma jet. Metallographic observations show formation of martensite layer due to high cooling rate by heat transfer into the bulk steel. Heat affected zone (HAZ) was 1 mm deep and 10-12 mm wide. The treated surface maximum hardness was measured at 763 HV0.1 whereas untreated steel was at 210 HV 0.1. The computation indicates a hardening sink near the surface that went undetected in the experimental observations. The ability of the different computational approaches to explore the process related phenomena and to give reliable results is put into discussion.

Keywords: SURFACE MODIFICATION, SURFACE HARDENING, TOOL STEEL, HARDNESS SIMULATION, HAZ-SIMULATION, FE-SIMULATION

1. Introduction

A NiCrMoV high-strength low alloy steels (HSLAS) incorporates high-strength into high ductility [1]. Among others, this steel is increasingly used as tool steel for applications such as molds for plastic, die casting, extrusion and forging. However, the use of these steels is limited by their poor toughness at the highest strength levels. Commonly, the quenching and tempering technique is employed to produce a useful combination of strength and toughness for heavy section NiCrMoV steel components [2]. A hardening in furnace with slow ramping and prolonged holding time is generally recommended [3]. Often the whole part does not need to be treated.

In many industrial applications only specific area of a part made out of a tool steel needs to be hardened. This provides hard and wear resistant surface while preserves ductile core of the part to increase fatigue and crack resistance [4]. Among others, surface hardening techniques laser and plasma jet treatments have hugest power density, however the plasma systems in general are much less expensive and safe to operate. In comparison with inert gases, the higher energy that is required for dissociation and ionization of the molecular gases translates into a higher plasma enthalpy. High plasma enthalpy is desirable as it allows higher materials processing rates [5-8].

However, controlling the plasma surface hardening process to achieve the desired properties remains a challenge. The experimental methods are often limited and do not allow a full understanding of the processes. Supplementing those with computational methods allows the problem to become more clearly understood.

The target of the simulation is computing of hardness distribution after the surface treatment and thus to explain the experimental observations and to extend them. To simulate the process of hardening two appropriate approaches can be recognized. The first approach - continuous cooling time approach (CCT) is based on thermo-metallurgical analysis, which describes the diffusion and non-diffusion driven solid-state transformations by means of phase proportions as function of temperature and time. It can also involve an analysis of carbon diffusion due to carburisation. However, the current study omits this. The second approach - maximal temperature, austenitisation and cooling time, from German "Spitzentemperatur, Austenitisierung- und Abkühlzeit" (STAAZ) is a phenomenological representation of the influence of the thermal cycle. It uses certain parameters from the local temperature cycle (heating rate, maximal temperature, austenitization time and cooling rate) and compares them with known material data, e.g. hardness.

2. Materials and experimental set-up

In this study, a tool steel surface treatment by plasma jet is taken into consideration. The material of the test specimens is low-

alloyed NiCrMoV steel with 0.55% Carbon (see Table 1). The initial steel condition is soft annealed with hardness of HV 210.

The plasma torch is operated at 36 kW input power. The torch traverse speed and standoff distance is adjusted to ensure the substrate heating above austenitizing temperature (850°C) for this steel but without melting (see Figure 1 and Table 2).

Table 1: Chemical composition, wt. % [9]

C	Si	Mn	Cr	Ni	Mo	V
0.55	0.3	0.7	1.1	1.7	0.5	0.1

Table 2: Process parameter

Parameters	Units	Value
Arc current	A	300
Voltage	V	110
Plasma gas total flow	l/min	45
Plasma gas power	kW	19.8
Distance to substrate	mm	25-30
Travel speed	m/min	0.838

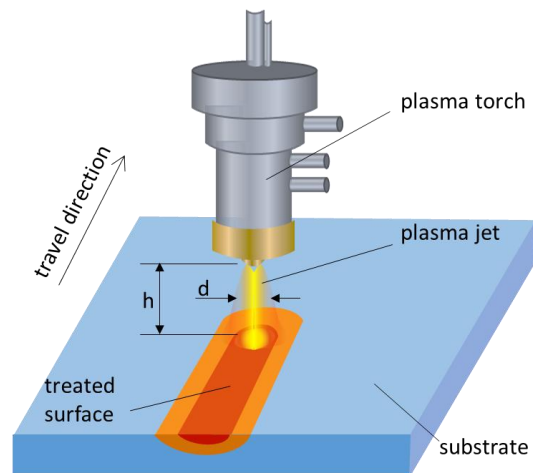


Fig. 1 Plasma jet process to surface hardening

During the experiments thermocouples type K is used to record the temperature-time history. The hardness is measured using UCI-Method (Ultrasonic Contact Impedance) on hardness scanner UT100-V (Producer BAQ GmbH, Germany). The measurement was carried out with load 0.1 kg (HV 0.1) and grid size 0.2x0.2mm. Due to limitations of the measurement method, the scanning area has an offset of 0.2 mm from the top surface, see Figure 2-b). Metallographic observations show formation of martensite layer due to high cooling rate by heat transfer into the bulk steel. Heat affected zone (HAZ) was ca. 1 mm deep and 10-12 mm wide. The

treated surface maximum hardness is measured at 763 HV0.1 whereas untreated steel was at 210 HV0.1.

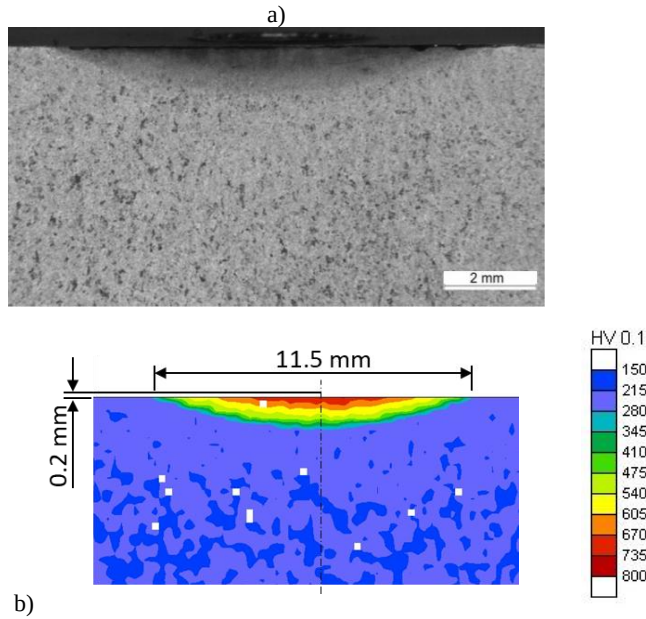


Fig. 2 Metallographical analysis of the sample cross-section: a) macrostructure and b) hardness distribution

3. Modelling approach

Temperature field

Background for the computations is transient numerical (using finite element method) calculation of temperature field. Concerning the geometry of the body and the parameters of the process a 2D heat conduction model was build. The assumption for 2D problem in the cross-section, i.e. high-speed heat source, is verified by comparison between analytical solution for moving and fast moving heat source and the numerical calculation with simplified heat source as well. It showed that the 2D model satisfies the process conditions and also that the specimen geometry corresponds to semi-infinity body, i.e. the requirement to the boundary conditions at the free surfaces is not severe. The mesh properties are adjusted and proved by means of h-method and the time steps - p-method. The material properties (conduction coefficient and enthalpy increment) of the model are get from [10, 11] and defined as temperature dependent in the range between room temperature and 1400 °C. The initial temperature is set to 22 °C, assumed as room temperature. At the free surfaces a convective boundary condition with film coefficient of $25 \times 10^{-6} \text{ W/(mm}^2\text{K)}$ was optionally set.

To simulate the heat input a heat source model normally distributed on the upper surface is used:

$$q_2(x, t) = \frac{q_{eff}}{\pi r_0^2} e^{-\frac{x^2 + (v(t-t_0))^2}{r_0^2}}, \text{ W/mm}^2$$

where q_{eff} is the effective heat power (12 kW), r_0 is the distribution radius 7.6 mm, v is the travel velocity (6.38 mm/s) and t_0 is the time offset (2 s). The efficiency as well the distribution radius were adjusted regarding the metallurgical investigations (ref. Figure 2). The simulation is performed until cooling down to room temperature.

The computed distribution of the pick temperatures indicates a maximal value on the surface in the middle of the heated zone about 1250 °C and dimensions of the HAZ (assumed 800 °C isotherm): 11 mm width and 1 mm deep, see Figure 3. Figure 4-a illustrates temperature-time curves from different points in the HAZ, marked

with their distance in transverse direction and from the surface. The results show extremely high heating and cooling rates (Figure 4-b) and short austenitization time Figure 4-a. The temperature rates on the surface are little higher.

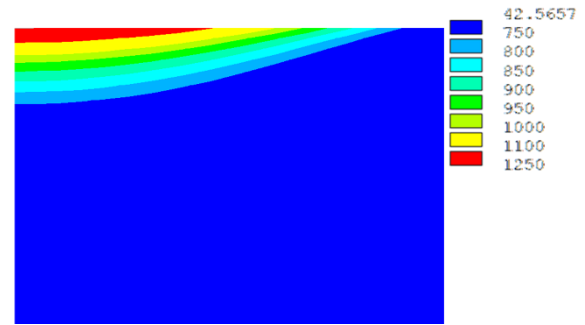


Fig. 3 Distribution of the pick temperatures in °C (specimen cross section, symmetrically split)

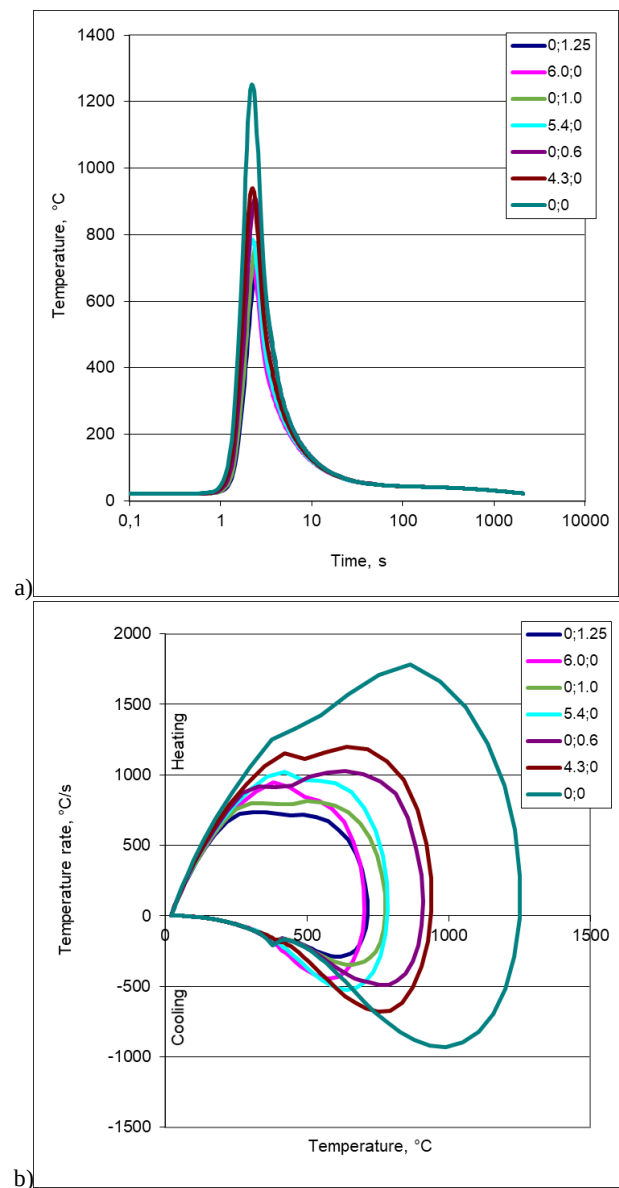


Fig. 4 Results from the temperature field analysis in several points of HAZ: a) temperature cycles and b) curves of the temperature rate

CCT approach

Concerning the first approach two reactions are taken into account – transformation of the initial phase (labelled p1) into austenite “austenite + carbide” (labelled p4) during heating and decomposition of the austenite into martensite (labelled p3) during cooling. To solve the austenitization problem the Leblond’s model [12] is used. It allows simulation the diffusible phase transformation process, considering the continuous time-temperature-austenitization diagram. The model gives the austenite phase proportion evolution dp/dt and is written as:

$$\frac{dp_4}{dt} = \frac{p_{eq,4}(T) - p_4}{\tau_4(T)},$$

where the austenite proportion at equilibrium $P_{eq,4}(T)$ changes from 0 to 1 in the temperature range between A_{c1} and A_{c3} and the time delay function $\tau_4(T)$ takes into account the variation of the transformation temperatures depending on the heating rate. Because of absence of exact data, a steel grade 50CrMo4 is chosen as reference material for austenite calculation from [10].

The martensite decomposition (martensite phase proportion, p3) is simulated using Koistinen-Marburger model [13]. The model has two parameters: martensite start temperature M_s and coefficient k , which depends on the martensite end temperature. The parameters are usually constants but they can be also variables (e.g. functions of C%), but not a function of maximum temperature. In our case, the equation becomes:

$$p_m = p_3 = p_4[1 - \exp(-k(M_s - T))],$$

where $M_s = 225$ °C according to [10] and $k = 0.008$ – guess value (based on experience).

Generally, the results after this approach are only precise for a domain with average pick temperatures according to the cooling time diagram. In this case, it means that the representative range is about 1000 °C.

The calculation of the hardness variation can be done by means of mixing rule in respect to the phase proportions. It means that the hardness of every one phase should be known. Assuming Leslie’s model (for steels with more than 0.5 %C) [14] the maximal hardness of martensite (HV_m) can be calculated as follow:

$$HV_m = HV_4 = 1667C - 926C^2 + 150 = 786.74$$

According to the Maynier’s model [15] (for low-alloyed steels) it is:

$$HV_m = 127 + 949C + 27Si + 11Mn + 8Ni + 16Cr + 21\log(CR)$$

Near HAZ boundary, where the cooling rate (CR) is equal to 1.1736×10^6 °C/h, it gives $HV_m = 823.4$ and at the point with maximal cooling rate ($CR = 3.348 \times 10^6$ °C/h) the resulting hardness is $HV_m = 833$. The hardness of the retained austenite usually is assumed as 100 HV.

The development of the phases in certain points in relation to temperature or time can be tracked on Figure 5. In the middle of the HAZ, on the upper side (red curve) the austenitisation reach quickly with increasing the temperature a value of 100% where 1.2 mm under the surface (blue curve) the austenite formation reaches only about 8% (Fig.5-b). That is due to solution of initial phase (Figure 5-a). During the stay above martensite start temperature, i.e. in the time interval between ca. 2 s and 7 s, there is no visible change in the phase proportions. After the 7-th second the entire HAZ simultaneously cools down the martensite start temperature (ref. Figure 4-a) and the martensite transformation begins. At about the 100-th second the velocity of martensite transformation process reduces drastically. Figure 5-c indicates that after the cooling to the

room temperature the final phase proportion at all four points does not match exactly the austenite proportion. This means that part of the austenite remains, i.e. retained austenite.

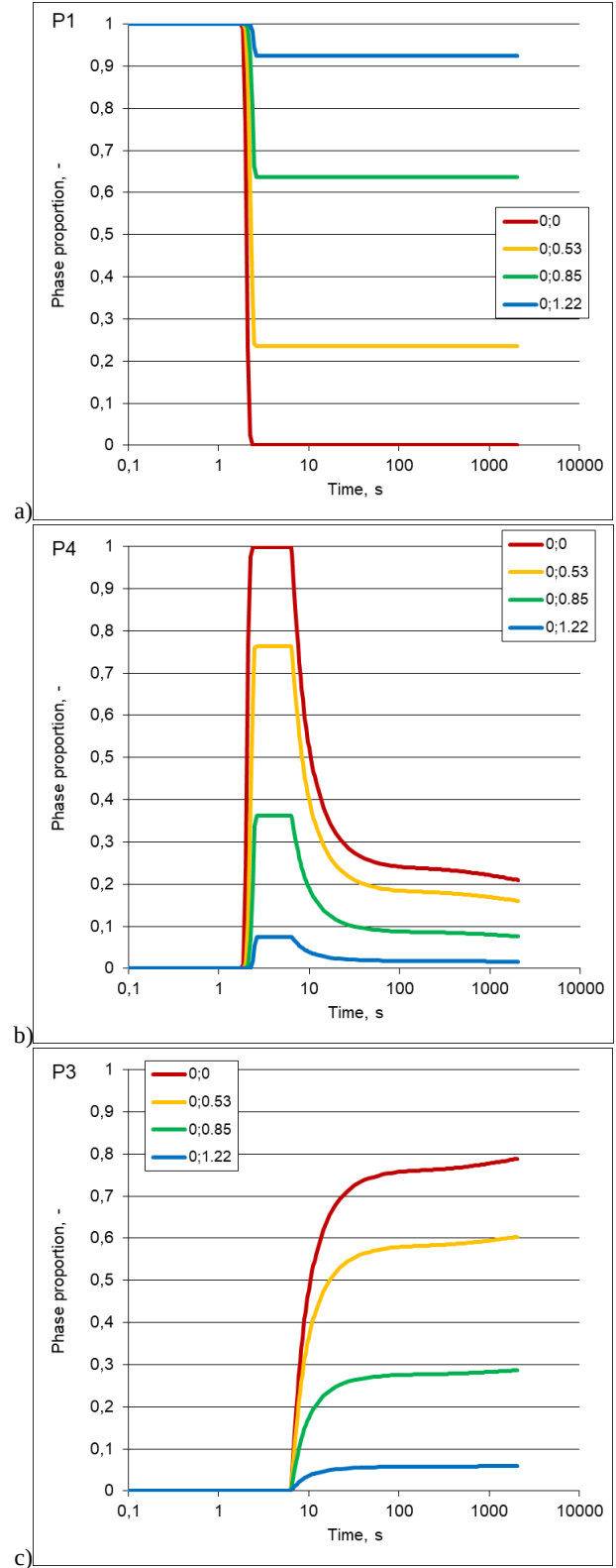


Fig. 5 Variation of the temperatures and phase proportions with the time in four locations with different depth in the middle of the HAZ: a) initial phase “p1”, b) austenite “p4” and c) martensite “p3”

The results from the performed calculation indicate a presence of about 20 % retained austenite (p4) in the highly heated region of HAZ (over 1200 °C), the rest 80% is martensite (p3), see Figure 6. In the transition zone of the HAZ, which is about 5 mm wide (i.e. maximum temperature up to 900 °C), the proportion is about 10 % p4 and 20 % p3. In the core of the HAZ, i.e. in the range of

maximum temperatures from 1000 °C to 1200 °C, the proportion is 75-79 % martensite (p3) and 15-20 % retained austenite (p4).

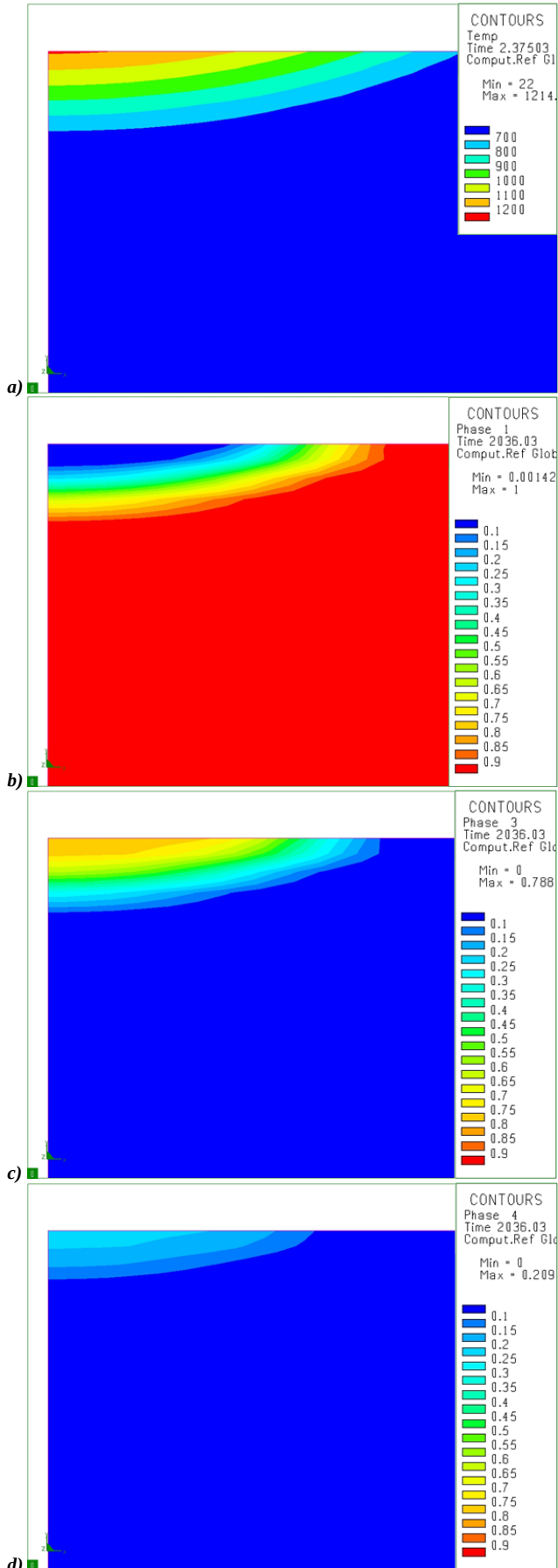


Fig. 6 Phase proportions after cooling down to room temperature: a) temperature field at 2.375 s (0.375 s after the source passing through the section), b) initial phase, c) martensite and d) austenite (retain austenite)

The calculated distribution of the hardness using both Mayner's and Leslie's models is shown on the Figure 7. The oscillations of the HAZ border are due to reduced integration points by the elements formulation, i.e. element average and should be ignored by the interpretations. The profile indicates a continuous increasing of the hardness in the HAZ from the initial one, i.e. from the base metal, (210 HV) to a maximal value of 738 HV according to Mayner's model (Figure 7-a) and 670 HV according to Leslie's model (Figure 7-b). Furthermore, the Leslie's model predicts a narrow zone in comparison to the Leslie's model.

Disadvantage of this approach is that the influence of the maximum temperature is not captured.

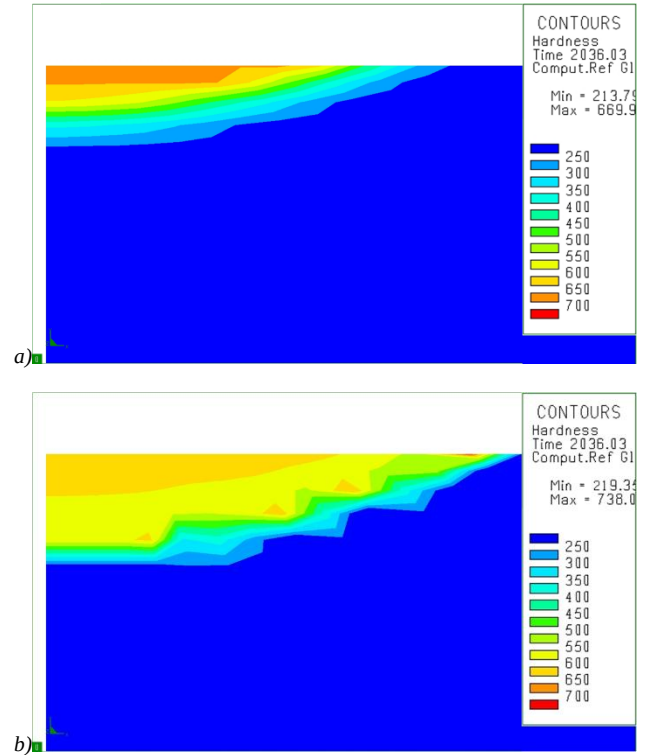


Fig. 7 Variation of the temperatures and phase proportions with the time in four locations with different depth in the middle of the HAZ: a) initial phase "p1", b) austenite "p4" and c) martensite "p3"

STAAZ approach

The second approach allows taking into consideration the influence of the maximal temperature on the martensite formation or hardness (STAAZ model) [16]. Originally, the model requires experimentally obtained hardness data from physical simulation under variation of the austenitization temperature and time as well as the cooling time, collected in a data cloud (STAAZ-cloud). The created data cloud is then directly coupled with the thermal analysis. The interpolation between the sets in the data cloud gives the property value for particular temperature cycle, i.e. to every element node of the FE-model:

$$[p] = [N] \cdot [P]^T$$

where $[p]$ is the interpolated property vector (for example phase proportion, hardness, yield stress etc.) and $[P]$ is the vector with the reference values to the 2, 3 or 4D combination matrix $[N]$. In current study, the used interpolation routine is numerical – Lagrange interpolation [17].

For the simulation of the particular problem a STAAZ-cloud from literature data for 50CrMo4 [10], as arbitrary material, is generated (i.e. based on the same data as for the previous modelling approach). In the current case, the STAAZ-cloud involves HV values depending on maximal temperature and temperature rate.

The austenization time is indirectly considered by its relation to the maximal temperature. The initial hardness is set to HV 225.

The creation of the STAAZ-cloud model consists of conversion of (quench hardness-austenitisation temperature curves) using the data for HV 0.1. For the data assignment, the temperature cycle parameters are transformed to their sets of maximal temperature (T_{max}) and cooling time between 800 °C and 500 °C ($t_{8/5}$). For this purpose, the individual quench curves must be analysed and prepared for the application. During the evaluation and preparation of the data sets, a plausibility check is carried out, supported by literature data of similar materials. The STAAZ-cloud is then defined in a 9x6 matrix, graphically presented in Figure 8.

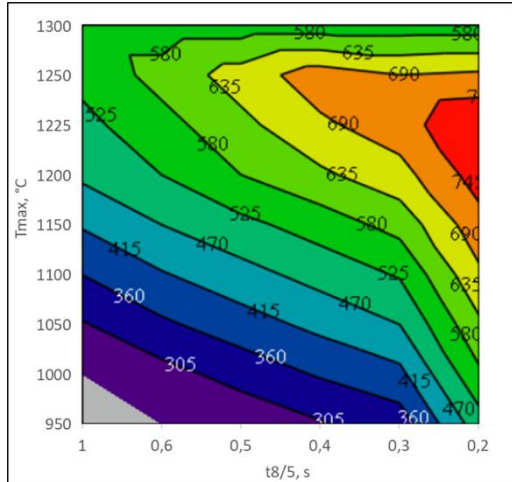


Fig. 8 STAAZ-cloud (HV values) as function of maximal temperature T_{max} and cooling time $t_{8/5}$

The results of the calculated hardness demonstrate that the depth and width of the hardened zone is comparable to that obtained using the previous calculation approach. The maximal calculated hardness reaches a value of about 820 HV. However, a softening effect on the surface layer in the middle of the HAZ is clearly observable, Figure 9. This hardness sink can be explained with the diminution of the martensite start temperature with increasing of the maximal temperature and thus increasing of the retained austenite fraction (compare to Figure 6-d). The maximal hardness correspond the temperature range 1000 – 1100 °C.

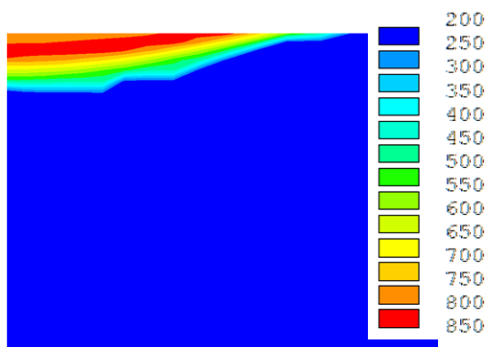


Fig. 9 Calculated distribution of hardness (HV)

4. Comparison between CCT and STAAZ approaches

The computation models created, on the base of the respective approaches, are used to calculate the hardness in the HAZ of the single heated track as a function of the calculated temperature history. In order to calibrate the various models, the same original metallurgical data are used. Figures 10 and 11 demonstrate the comparisons between the measured and calculated hardness

distributions, using the CCT and STAAZ approaches, respectively. The calculated hardness distribution, employing both hardness models created on the CCT approach, is, in general, underestimated, primarily due to the disregard of the influence of maximum temperature. The calculated hardness values in the HAZ using the STAAZ approach (red curve of Figure 11) demonstrate a strong correlation with the measurements. The reduction in hardness in the areas of overheating, i.e. the creation of retained austenite phase (compare to Figure 6-d), is well represented by this model. Despite the data being collected for similar alloy and the austenitising time being disregarded to minimise the preparation effort, a negligible discrepancy is observed between the calculated and measured hardness results. However, the calculations allow clear and consistent statements to be made.

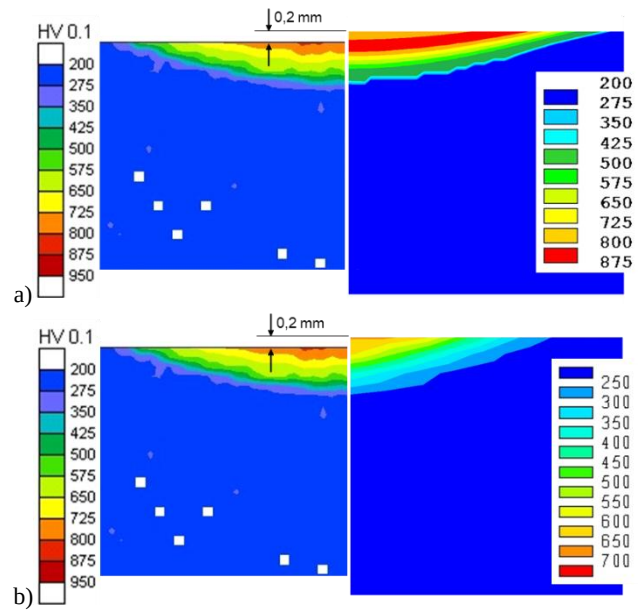


Fig. 10 Comparison of measured and calculated hardness profile using: a) STAAZ and b) Maynier

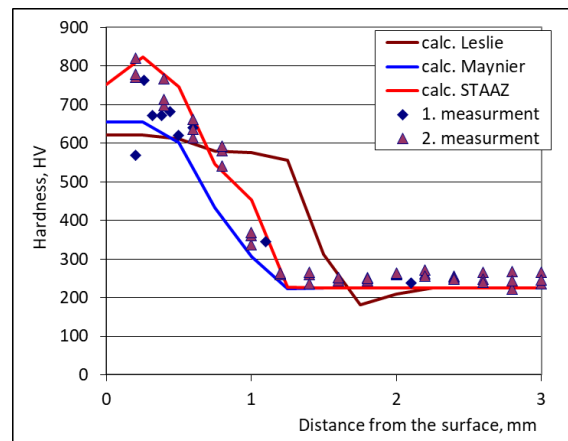


Fig. 9 Comparison between measured (two repetitions) and calculated hardness distribution (Leslie, Maynier and STAAZ) on the axis of the specimen cross-section

The CCT approach was realised with the FE-code Sysweld (ESI Group, France) and the STAAZ approach with ANSYS APDL (ANSYS Inc., USA).

By the CCT approach, the effect of the maximal temperature cannot be captured because the M_s is not function of T_{max} . Because of the missing of exact data some of the results (exact phase proportion and HV) have only qualitative character. So, object for future investigation should be a generation of austenitization diagram and data sets with hardness values for calibration of the

STAAZ model as well as the influence of the carbonisation on the hardness variation. Furthermore, a determination of the carbon flow and calibration of the diffusion model is also necessary. It is noteworthy that an increase in the C-content due to carburisation will result in a decrease in M_s , consequently leading to a greater amount of retained austenite on the surface, i.e. a reduction in hardness.

5. Conclusions

The article demonstrates the ability of computation to predict the hardness distribution after the surface treatment, to explain and extend the experimental observations as well as to show the complex interactions.

- The CCT approach may give reliable data for phase distribution, but its coupling with the hardening models does not provide sufficient results for calculation of hardness distribution.
- The STAAZ approach provides a reliable calculation of hardness. Due to the essential influence of maximal temperatures is only able to recognise the hardening sink near the surface.
- Nevertheless, the application of both approaches serves a good base for understanding on resulting properties after plasma jet surface hardening, and therefore useful for the process optimization.
- The model creates an appropriate base for further development for taking into consideration the carburisation effect of the plasma.

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Modeling of martensite distribution in stainless steel wire during thermomechanical processing

Моделирование распределения мартенсита в проволоке из нержавеющей стали в процессе термомеханической обработки

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Аннотация: В данной статье проведен ряд теоретических исследований нового комбинированного процесса многоциклового термомеханической обработки, включающей волочение проволоки и последующее охлаждение в жидком азоте. Анализ температурных полей и мартенситной составляющей во всех рассмотренных моделях показал, что для обеих толщин самым эффективным вариантом является пониженная скорость волочения и отсутствие промежуточного подогрева. При этом повышение скорости деформирования резко снижает уровень образования мартенсита в осевой зоне. Наименее эффективным вариантом является использование повышенной толщины заготовки при повышенной скорости волочения и отсутствии подогрева до комнатной температуры, который для при повышении толщины заготовки выполняет роль локального охлаждения осевой зоны заготовки.

КЛЮЧЕВЫЕ СЛОВА: ПРОВОЛОКА, МОДЕЛИРОВАНИЕ, ВОЛОЧЕНИЕ, СТАЛЬ, КРИОГЕННАЯ ДЕФОРМАЦИЯ.

1. Введение

Производство длинномерных деформированных полуфабрикатов, таких как прутки, профили, проволока, является ключевой задачей современной металлургии. Эти материалы широко применяются в различных отраслях, от машиностроения и строительства до электроники и авиации.

Современное производство проволоки сталкивается с серьезными вызовами, обусловленными устаревшими технологиями. Используемое оборудование отличается высоким энергопотреблением и низкой производительностью, что негативно сказывается на рентабельности производства и конкурентоспособности компаний на мировом рынке.

Особую актуальность приобретает производство проволоки из аустенитных коррозионностойких сталей, спрос на которые неуклонно растет. Эти стали, составляющие около 80% мирового рынка коррозионностойких сталей, характеризуются уникальными свойствами, делающими их незаменимыми в широком спектре отраслей.

В данной работе предлагается новая технология деформирования проволоки, основанная на криогенном охлаждении. Суть ее заключается в следующем: проволока, проходя через фильеру, подвергается холодной обработке, основанной на использовании низких температур. Разработанная технология позволит измельчить микроструктуру и получить мартенситную составляющую в проволоке.

Моделирование занимает центральное место в современной науке и технике. Оно позволяет нам исследовать сложные физические явления, прогнозировать их поведение и, что немаловажно, разрабатывать новые технологии или совершенствовать уже существующие. Для разработки и оптимизации технологии деформирования проволоки с криогенным охлаждением используются компьютерные модели. Модели позволяют изучить влияние различных параметров процесса на структуру и свойства проволоки (температура, скорость деформации, диаметра проволоки), а также провести оптимизацию технологического процесса.

Особую роль моделирование играет в материаловедении. Изучение микроструктуры материалов, их свойств, а также процессов их деформации и обработки, — все это является предметом многочисленных исследований, которые зачастую невозможно провести без применения компьютерного моделирования.

Новизной данной статьи является моделирование распределения мартенсита в проволоке из нержавеющей

стали в ходе нового совмещенного процесса волочения с криогенным охлаждением.

2. Моделирование в программном комплексе Deform

При рассматриваемой термомеханической обработке нержавеющей аустенитной проволоки будет происходить фазовое превращение аустенита в мартенсит. При этом интенсивность данной трансформации будет зависеть как от геометрических параметров (начальный диаметр проволоки и значения обжатий в каждом проходе), так и от технологических параметров (распределение температуры по сечению, которая зависит от скорости деформирования в волоке и времени охлаждения в жидком азоте). Для оценки данных фазовых трансформаций были использованы 8 моделей, построенные при изучении усилий:

- 1) с 6 мм до 5 мм при 500 мм/с с подогревом заготовки до 20°C после каждого охлаждения в азоте;
- 2) с 6 мм до 5 мм при 1000 мм/с с подогревом заготовки до 20°C после каждого охлаждения в азоте;
- 3) с 6 мм до 5 мм при 500 мм/с без подогрева заготовки;
- 4) с 6 мм до 5 мм при 1000 мм/с без подогрева заготовки;
- 5) с 9 мм до 7 мм при 500 мм/с с подогревом заготовки до 20°C после каждого охлаждения в азоте;
- 6) с 9 мм до 7 мм при 1000 мм/с с подогревом заготовки до 20°C после каждого охлаждения в азоте;
- 7) с 9 мм до 7 мм при 500 мм/с без подогрева заготовки;
- 8) с 9 мм до 7 мм при 1000 мм/с без подогрева заготовки.

Для корректного моделирования фазовых превращений необходимо в базу материала ввести термокинетическую диаграмму, характеризующую фазовые переходы в зависимости от температуры и времени (Рис. 1).

Полученные ранее картины распределения температурных полей позволяют оценить распределение мартенсита по сечению заготовки после каждой стадии термомеханической обработки. Для начального состояния было задано условие 100% аустенита, которые может превращаться в мартенсит. Поэтому для количественной оценки достаточно рассматривать только содержание мартенсита (доля аустенита составляет остаточную часть от 1).

На рис. 2 приведены картины распределения мартенсита в сечении заготовки для модели волочения с 6 мм до 5 мм при 500 мм/с с подогревом заготовки до 20°C. Отчетливо видно, что в отличие от температурных полей, здесь нет характерной однородности по длине заготовки. Поэтому ранее использованный точечный метод оценки для данного

параметра не подходит. Поскольку основной интерес представляет содержание мартенсита после всех стадий термомеханической обработки, было решено рассматривать только последнюю стадию охлаждения в жидком азоте. Оценку мартенситной составляющей в этом случае удобно проводить по шкале, которая показывает минимум и максимум содержания мартенсита в сечении заготовки на данной стадии.

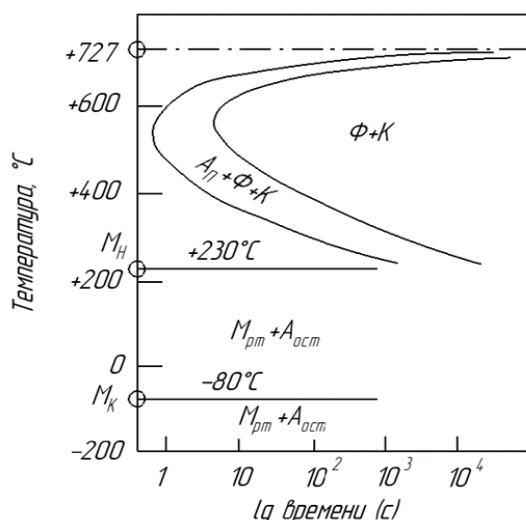


Рис. 1 Термокинетическая диаграмма для стали AISI-316

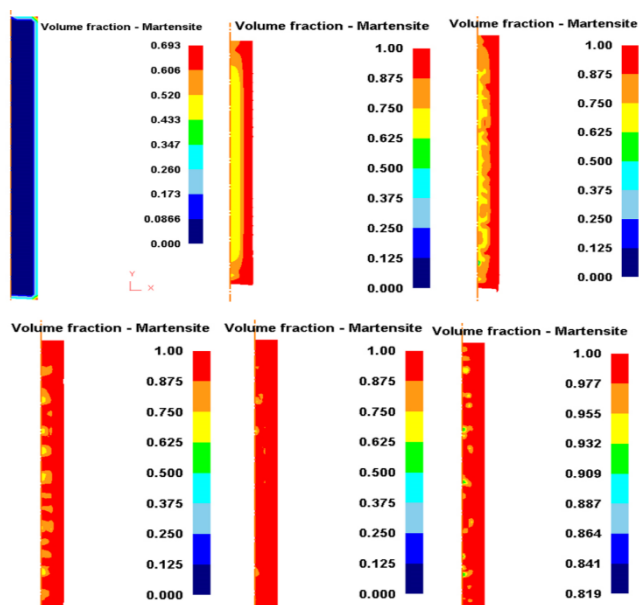


Fig. 2 Распределение мартенситной составляющей в модели волочения с 6 мм до 5 мм при 500 мм/с с подогревом заготовки до 20°C

Для сравнительного анализа на рис. 3 были представлены результаты моделирования фазовых превращений всех 8 моделей в порядке их упоминания и проведенного ранее рассмотрения температурных полей. Сравнивая полученные картины распределения мартенсита и ранее полученные графики температурных полей, можно сделать вывод о том, что уровень образования мартенсита находится в прямой зависимости от интенсивности охлаждения металла. Так при обработке тонкой заготовки с 6 мм до 5 мм наиболее эффективными условиями, при которых мартенсит образуется почти во всем сечении, является пониженная скорость волочения и отсутствие промежуточного подогрева (Рис. 3, верхний ряд). В этом случае уровень мартенсита в сечении составляет 88%. Включение в данные условия промежуточного нагрева снижает образование мартенсита до 82%. Здесь необходимо отметить тот факт, что данные значения уровня

мартенсита зафиксированы в осевой зоне, тогда как на поверхности уровень мартенсита составляет 100%.

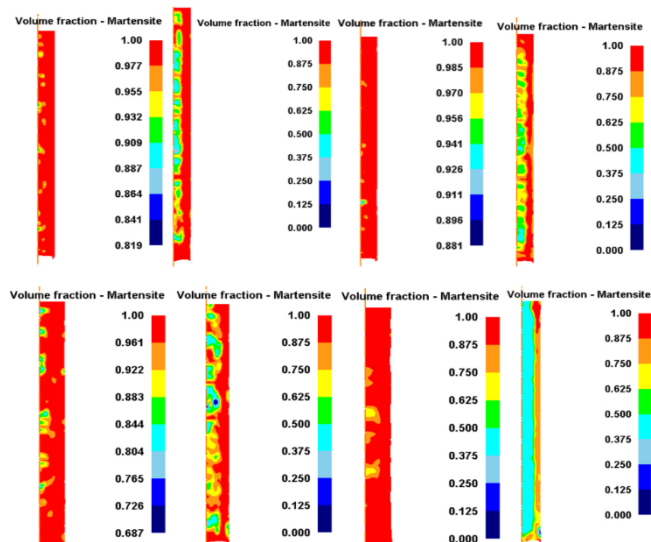


Fig. 3 Сводное распределение мартенситной составляющей после всех стадий термомеханической обработки

Повышение скорости волочения, как уже было отмечено ранее, приводит к снижению охлаждения заготовки (особенно в осевой зоне). Это приводит к резкому снижению образования мартенсита в данной области до 40-45% в случае с подогревом заготовки и до 50-55% в случае без подогрева заготовки.

Использование заготовки увеличенной толщины дает аналогичные результаты (Рис. 3, нижний ряд), хотя и с более низким уровнем образования мартенсита. Самым эффективным вариантом является пониженная скорость волочения и отсутствие промежуточного подогрева, в этом случае минимальный уровень мартенсита в осевой зоне составляет 70-75%. Наличие промежуточного нагрева снижает образование мартенсита до 68%. Здесь можно отметить тот факт, на поверхности уровень мартенсита по-прежнему составляет 100%, снижаясь до 95% при наличии промежуточного нагрева. Повышение скорости волочения резко снижает уровень образования мартенсита в заготовке повышенной толщины. При наличии подогрева заготовки до комнатной температуры уровень мартенсита составляет примерно 48% в осевой зоне и 90% в поверхностной зоне. При отсутствии промежуточного подогрева до комнатной температуры уровень мартенсита составляет примерно 38% в осевой зоне и 80% в поверхностной зоне. Такой результат является следствием получаемого температурного поля, когда осевая зона после охлаждения в жидком азоте имеет температуру выше комнатной.

4. Заключение

Анализ температурных полей и мартенситной составляющей во всех рассмотренных моделях показал, что для обеих толщин самым эффективным вариантом является пониженная скорость волочения и отсутствие промежуточного подогрева. При этом повышение скорости деформирования резко снижает уровень образования мартенсита в осевой зоне. Наименее эффективным вариантом является использование повышенной толщины заготовки при повышенной скорости волочения и отсутствии подогрева до комнатной температуры, который для при повышении толщины заготовки выполняет роль локального охлаждения осевой зоны заготовки.

5. Финансирование

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Modeling of microstructure evolution during the combined process “radial-shift broaching with drawing” by cellular automata method

Моделирование эволюции микроструктуры в ходе совмещенного процесса «радиально-сдвиговая протяжка с волочением» методом cellular automata

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Аннотация: В данной статье проведено исследование влияния совмещенной технологии радиально-сдвиговой протяжки и последующего волочения на эволюцию микроструктуры прутков двумя методами моделирования – ЖМАК и клеточными автоматами. Анализ результатов показал, что результаты моделирования имеют весьма высокую степень сходимости между собой, поэтому можно рекомендовать использовать любой из этих методов для получения информации о размере зерна. Если же необходимо получение данных об изменении формы зерен, то в этом случае пригодным методом будем Cellular Automata. В этом случае можно будет отследить изменение формы зерен в разных направлениях.

КЛЮЧЕВЫЕ СЛОВА: ПРУТОК, МОДЕЛИРОВАНИЕ, СТАЛЬ, РАДИАЛЬНО-СДВИГОВАЯ ПРОКАТКА.

1. Введение

Современные методы обработки металлов давлением широко распространены и позволяют решать самые разнообразные задачи, в том числе производство деталей (и полуфабрикатов) заданной формы с улучшенным комплексом механических свойств. Традиционные методы, такие как прессование, ковка, штамповка и прокатка, направлены на получение изделий заданной геометрической формы (профиля) и размеров. Кроме того, некоторые методы, например метод интенсивной пластической деформации (ИПД), позволяют создавать сверхтонкие структуры материалов с повышенным уровнем механических свойств.

Получение материалов с мелкозернистой структурой после пластической деформации основано на использовании методов ИПД. Целью ИПД является накопление больших степеней деформации (истинная степень деформации от 6-8 и более). Но у методов ИПД имеется большое количество недостатков, одним из основных это ограничение по размерам заготовки.

Для получения длинных цилиндрических полуфабрикатов с улучшенными механическими свойствами был изучен и применен метод прокатки с радиальным сдвигом (РСП). Ряд исследований показал, что этот метод может быть использован для обработки практически любых металлов и сплавов, в том числе с высокой прочностью и низким сопротивлением деформации. По сравнению с обычной спиральной прокаткой, процесс FSR позволяет получить более мелкозернистую структуру и сжать металл по всему объему прутка. Такой процесс также улучшает свойства проката за счет получения уникальной микроструктуры. Результаты исследования [1] показывают, что при малых углах подачи в центральной части заготовки появляется рыхление металла. При увеличении угла подачи с 18 до 240 металл сжимается и интенсивно обрабатывается для получения более мелкозернистой структуры.

Исследования показывают, что метод РСП позволяет получить градиентную мелкозернистую структуру. Материал в поверхностной области деформируется более интенсивно, формируя как мелкозернистую микроструктуру за счет динамической рекристаллизации, так и крупнозернистую микроструктуру, образующуюся в результате развития процесса рекристаллизации. В центральном слое сохраняется деформированная нерекристаллизованная структура. В результате после РСП удастся сформировать так называемую градиентную структуру, которая одновременно придает обрабатываемому материалу высокую прочность и вязкость. При этом конечный уровень механических свойств заготовки сопоставим с показателями, полученными методом ИПД.

В 2001 году на факультете МГТУ им. Г.И. Носова были начаты прикладные исследования по радиально-сдвиговой протяжке (РСПр) [2]. Ее прототипом является радиально-

сдвиговая прокатка. Возможность совмещения процессов ПВП и волочения рассматривалась в работе [8]. Использование традиционного волочения в качестве заключительного процесса после различных процессов ИПД является хорошим вариантом, так как при этом устраняются все недостатки предварительной деформации (например, неоднородность геометрии, пористость и т.д.).

2. Моделирование в программном комплексе Deform

Для моделирования эволюции микроструктуры методом Cellular Automata необходимо использовать уже заранее рассчитанные модели с полями всех параметров (напряжения, деформации, скорости течения и т.д.). Для возможности сравнения с результатами модели ЖМАК, при расчете клеточными автоматами в каждой модели рассматривались две точки – в центре заготовки и на расстоянии 0,5 мм на поверхности в области контакта металла с валками, что позволяет оценить максимальный уровень проработки металла. Помимо этого, было решено рассчитывать микроструктуру в обозначенных точках в двух направлениях (поперечном и продольном), поскольку данный метод предоставляет визуальную картину зерен и имеется возможность оценки не только изменения их размера, но и формы. Для расчета всех моделей использовалось построение окна с размерами 100 x 100 мкм. Исходная структура со средним размером зерна 25 мкм представлена на Рис. 1.

На рисунках 2 - 4 представлены результаты расчета микроструктуры для моделей 30-25-20 при трех температурах.



Рис. 1 Исходная микроструктура

Анализируя полученные данные, можно отметить результаты расчета клеточными автоматами имеют высокую сходимость с результатами в модели ЖМАК, особенно в поперечном направлении. В продольном направлении на поверхности сохраняется достаточная однородность формы зерен, тогда как в центральной области наблюдается интенсивное удлинение зерен за счет действия растягивающих сил при волочении и ламинарного течения по схеме РСПр. При температуре 900°C интенсивность удлинения зерен снижается из-за активации механизма статической рекристаллизации, который приводит к формированию новых зерен. В таблице 1 приведены сравнительные данные среднего размера зерен по обоим методам расчета микроструктуры.

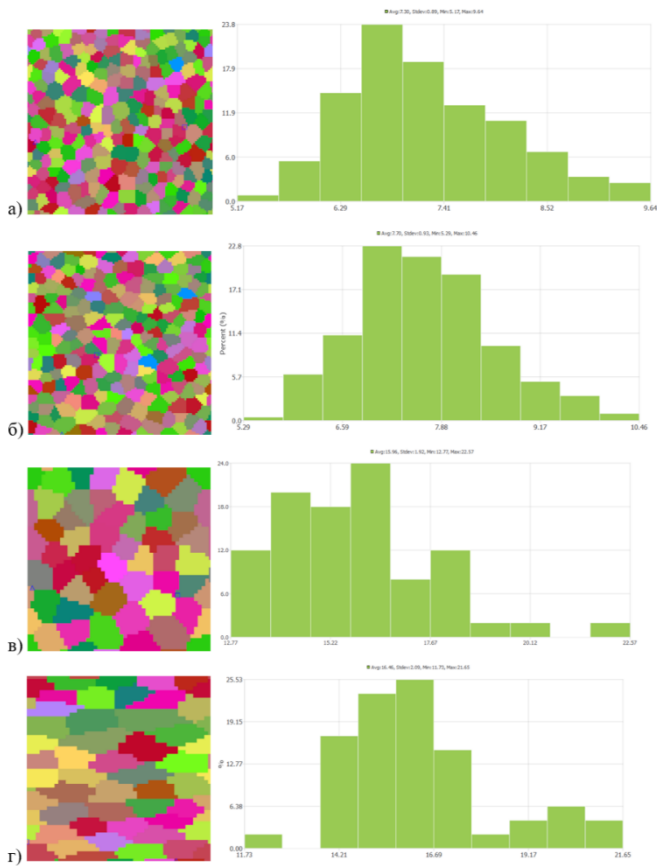


Fig. 2 Микроструктура модели 30-25-20 при 20°C: а – поверхность, поперечное направление; б – поверхность, продольное направление; в – центр, поперечное направление; г – центр, продольное направление

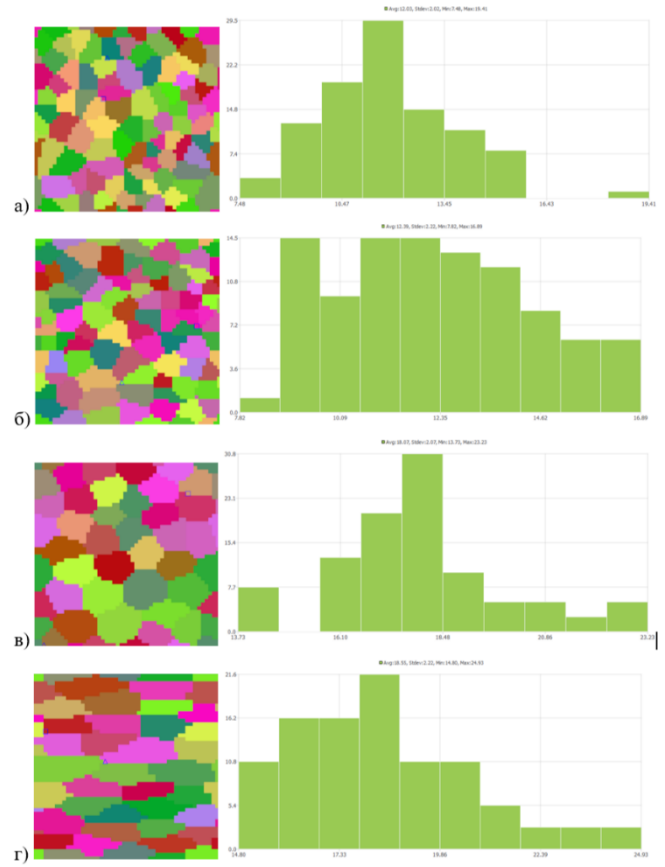


Fig. 3 Микроструктура модели 30-25-20 при 500°C: а – поверхность, поперечное направление; б – поверхность, продольное направление; в – центр, поперечное направление; г – центр, продольное направление

Таблица 1 - Средний размер зерен в моделях 30-25-20, мкм

Температура	Направление	Поверхность		Центр	
		СА	ЖМАК	СА	ЖМАК
20°C	поперечное	7,3	8	15,96	16
	продольное	7,7		16,46	
500°C	поперечное	12,03	12	18,07	18
	продольное	12,39		18,55	
900°C	поперечное	14	14	20,27	20
	продольное	14,33		20,95	

Как в случае с моделями 30-25-20, при анализе результатов моделей 30-27-23 была выявлена высокая сходимость результатов расчета микроструктуры с результатами метода ЖМАК как на поверхности, так и в центре (см. Таблица 2). Здесь также наблюдается удлинение зерен в продольном направлении в центральной области заготовки, на поверхности зерна имеют более равноосную форму из-за турбулентной схемы течения металла за счет конструктивного расположения валков стана РСП.

При анализе результатов моделей 20-18-16 была выявлена высокая сходимость результатов расчета микроструктуры с результатами метода ЖМАК как на поверхности, так и в центре (см. Таблица 5.3). Здесь практически не наблюдается эффекта удлинения зерен в продольном направлении, поскольку центральные слои в этих моделях получают крайне незначительное измельчение зерен. Поверхностные слои также прорабатываются весьма слабо. Основной причиной такого эффекта является резкое снижение единичных обжатий, а также использование заготовки уменьшенного диаметра. В результате очаг деформации в валках формируется в прямолинейной области валков, что резко снижает угол закручивания поверхностных слоев – в этом случае целесообразно говорить не о радиально-сдвиговой, а об обычной поперечно винтовой прокатке.



Fig. 4 Микроструктура модели 30-25-20 при 900°C: а – поверхность, поперечное направление; б – поверхность, продольное направление; в – центр, поперечное направление; г – центр, продольное направление

На рисунках 5 - 7 представлены результаты расчета микроструктуры для моделей 30-27-23 при трех температурах.

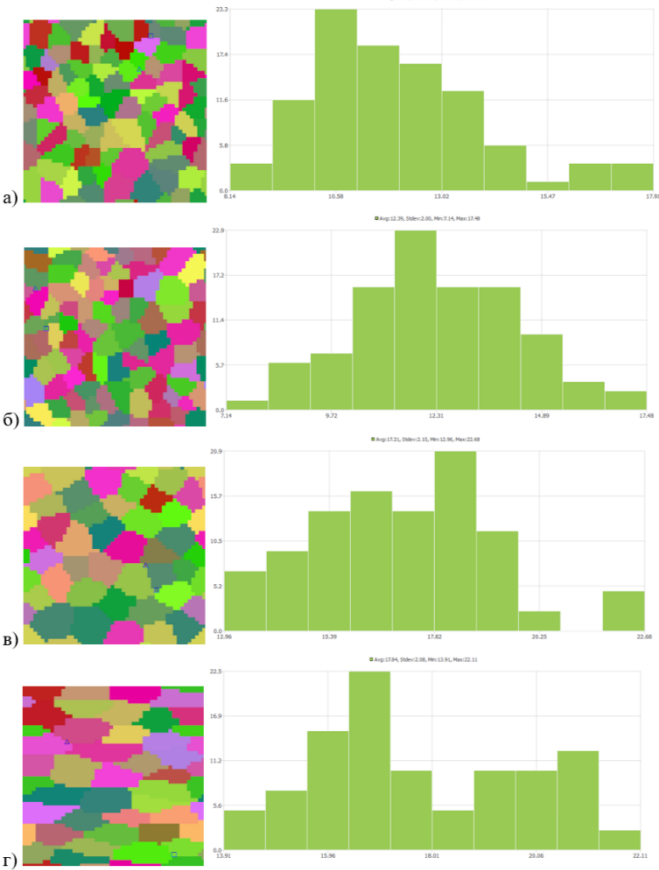


Fig. 5 Микроструктура модели 30-27-23 при 20°C: а – поверхность, поперечное направление; б - поверхность, продольное направление; в – центр, поперечное направление; г - центр, продольное направление

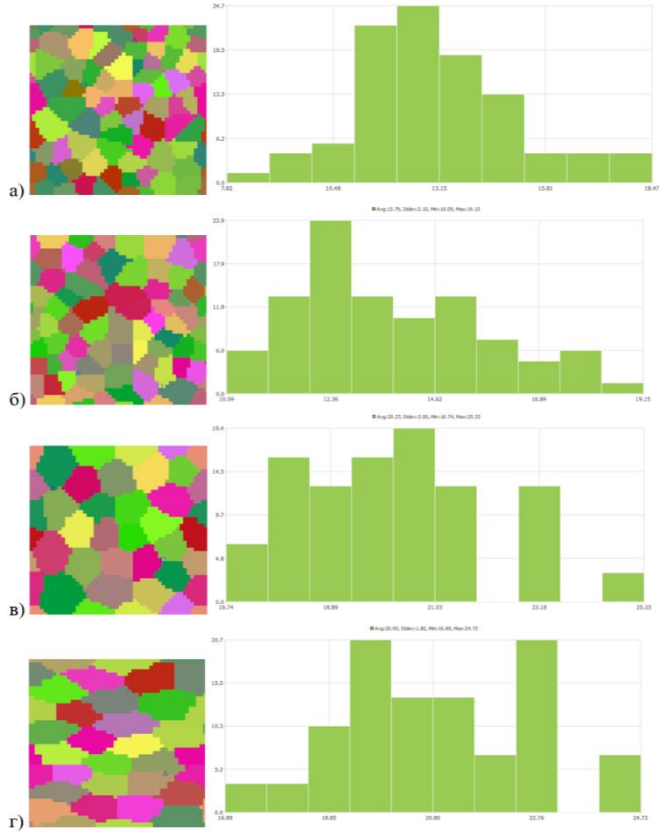


Fig. 6 Микроструктура модели 30-27-23 при 500°C: а – поверхность, поперечное направление; б - поверхность, продольное направление; в – центр, поперечное направление; г - центр, продольное направление

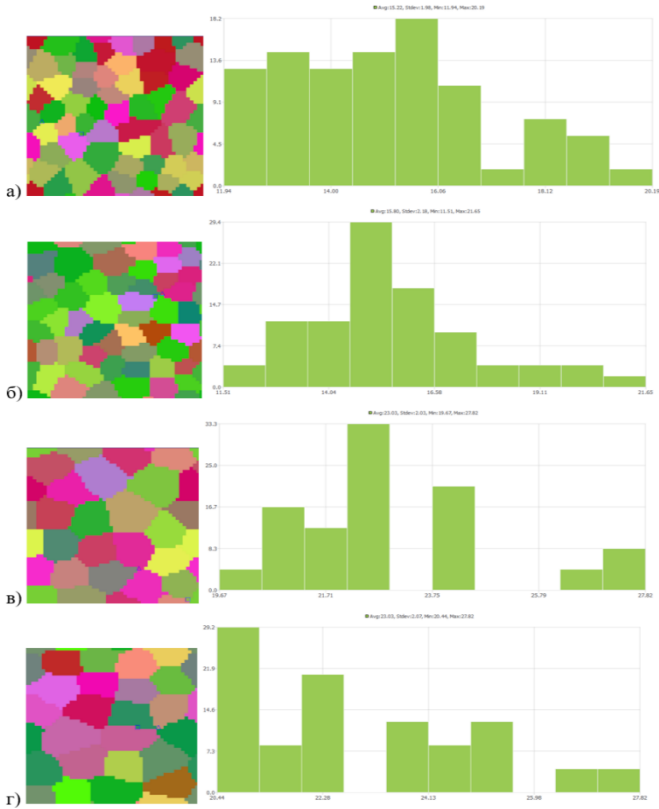


Fig. 7 Микроструктура модели 30-27-23 при 900°C: а – поверхность, поперечное направление; б - поверхность, продольное направление; в – центр, поперечное направление; г - центр, продольное направление

Таблица 2 - Средний размер зерен в моделях 30-27-23, мкм

Температура	Направление	Поверхность		Центр	
		CA	JMAK	CA	JMAK
20°C	поперечное	12,17	12	17,21	17
	продольное	12,39		17,84	
500°C	поперечное	13,21	13	20,27	20
	продольное	13,79		20,95	
900°C	поперечное	15,22	15	23,03	22
	продольное	15,8		23,03	

На рисунках 8 - 10 представлены результаты расчета микроструктуры для моделей 20-18-16 при трех температурах.

Таблица 3 - Средний размер зерен в моделях 20-18-16, мкм

Температура	Направление	Поверхность		Центр	
		CA	JMAK	CA	JMAK
20°C	поперечное	19,95	20	23,53	25
	продольное	20,6		24,06	
500°C	поперечное	21,72	22	24,06	25
	продольное	22,13		24,62	
900°C	поперечное	23,03	23	25,89	25
	продольное	23,53		26,6	

5. Финансирование

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Fig. 8 Микроструктура модели 20-18-16 при 20°C: а – поверхность, поперечное направление; б - поверхность, продольное направление; в – центр, поперечное направление; г - центр, продольное направление

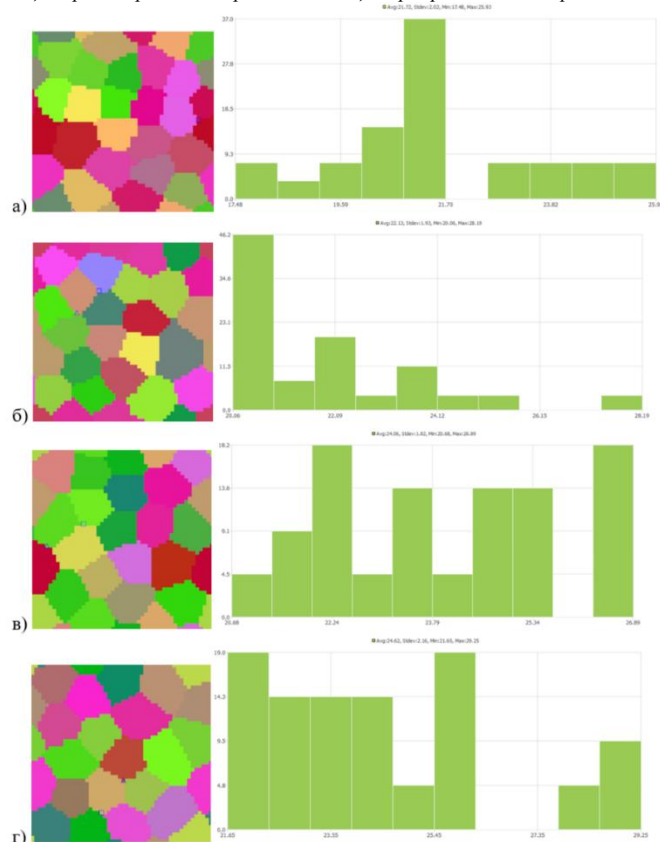


Fig. 9 Микроструктура модели 20-18-16 при 500°C: а – поверхность, поперечное направление; б - поверхность, продольное направление; в – центр, поперечное направление; г - центр, продольное направление

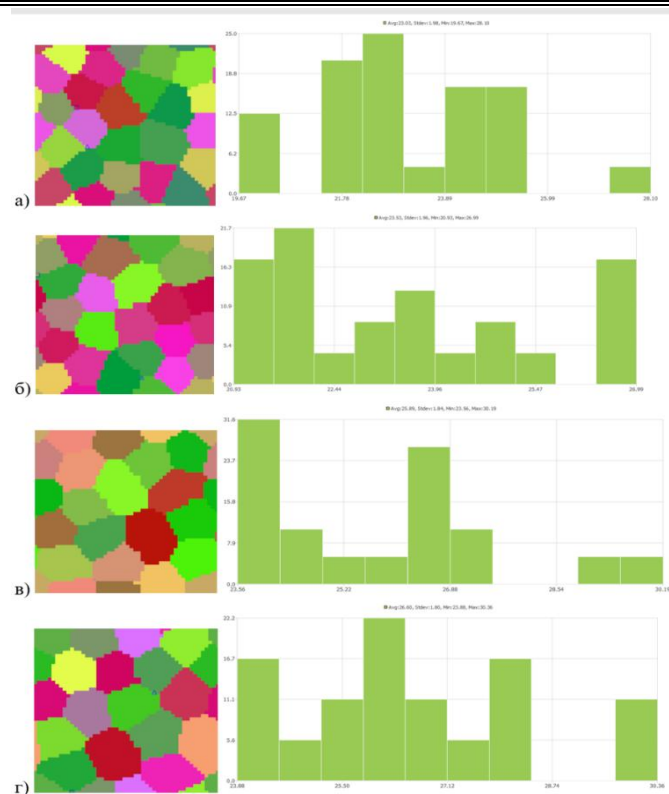


Fig. 10 Микроструктура модели 20-18-16 при 900°C: а – поверхность, поперечное направление; б - поверхность, продольное направление; в – центр, поперечное направление; г - центр, продольное направление

4. Заключение

Из полученных данных моделирования микроструктуры можно сказать, что деформирование заготовки по схеме 30-25-20 при комнатной температуре является самым эффективным способом, поскольку позволяет измельчить исходное зерно более чем в 3 раза на поверхности заготовки. При этом использование схемы 30-27-23 при комнатной температуре дает двукратное измельчение исходного зерна. Проработка центральной области заготовки во всех рассмотренных моделях носит слабо выраженный характер, достигая 35%-ого уменьшения в самых оптимальных условиях.

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Composite modifications obtained through various technological approaches based on inorganic binders and foam glass materials

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Abstract: Composite structural modifications based on hydraulic inorganic binders and foam glass aggregate fractions were prepared. Formwork moulds were developed for the moulding of composite test bodies using different technological approaches. A series of experimental specimens meeting the requirements for standard laboratory tests were obtained. The role of the technological regimes used in the formation of the structural characteristics and performance of the composites is examined. The existing possibilities for further modification and potential application of the obtained composite materials are analysed.

Keywords: COMPOSITE MODIFICATIONS, FORMWORK MOULDS, INORGANIC BINDERS

1. Introduction

The intensive expansion of large urbanized territories is accompanied by the active construction and reconstruction of a diverse building stock, the establishment of industrial zones and the development of transport infrastructure. In this aspect, the quality of the construction and renovation activities carried out is dependent on the technological and operational characteristics of the building materials used [1-3]. At the same time, the modern development of construction is today associated with the active application of innovative materials that meet the requirements for energy efficiency and adequate noise insulation of buildings [3-5].

On the other hand, the growing quantities of waste products (of industrial, construction and household nature) create conditions for the emergence of environmental and other accompanying problems in their collection and disposal. A main approach to reducing the negative impact of these factors on the state of the environment is the implementation of complex strategies for long-term waste management and recovery [6, 7]. Greater deployment of appropriate green technologies, providing for the efficient recycling of a variety of waste materials with a view to their use as raw materials, is essential for a sustainable improvement of environmental security [8-10]. Cost-effective production technologies have been developed for the production of separate categories of foam silicate materials by utilizing significant amounts of waste glass (of various origins) [11-13]. One basic technological approach is the thermal foaming of powder fractions of waste glass (obtained after grinding) in the presence of suitable foaming additives [11, 12]. The development allows for the recycling of waste materials that are practically non-degradable in the natural environment. For the realization of the production process, systems of specialised equipment, applicable in conjunction with standard equipment, have been developed [11]. Foam glass materials are used in the construction for thermal insulation of buildings (of various types), installations and equipment [14].

Composite materials involving inorganic binders and granular foam glass have been developed [15-19]. The resulting composite materials combine the specific characteristics of the components used and are suitable for the preparation of non-load-bearing, non-combustible, water-resistant, long-lasting thermal and acoustic insulation panels, boards, profiles etc [15-18]. A promising opportunity for further functional development of the developed composites is their structural modification by using different technological approaches. In relation to this, a preliminary study across a wide thematic range has been carried out [20-34].

The experimental research work carried out led to the conclusion that the existing traditional laboratory formwork moulds (of different materials) are inappropriate for forming test bodies when using some of currently employed technological approaches. Therefore, there arose a methodological need to apply formwork moulds meeting the requirements of the developed composite materials.

The aim of the present work is to present the main design characteristics and application possibilities of innovative formwork moulds, tailored to the specificity of the technological methods used for the preparation of composite experimental specimens based on

inorganic binders. Composite modifications obtained in laboratory conditions by using different technological modes are considered. The main structural characteristics of the obtained materials and the prospects for their potential application are analyzed.

2. Materials and methods

The development uses various raw materials, components, technological additives and standard equipment: laboratory balances Kern type ABJ220-4NM, platform balances with a weighing capacity of up to 10 kg, a ball mill with a working volume of 50 L (at a working speed of approx. 260 rpm), agate mortar, a set of sieves, laboratory baths, a perforated spatula, a grid stand, a granulator, programmable muffle furnace LM-312.11, waste glass (origin - municipal waste), foaming additive (glycerine or calcium carbonate), sodium silicate (module 3.0), Portland cement (CEM I 52.5 R, white CEM I 52.5 N or CEM I 42.5 N), gypsum, technical silicone, etc. Perlite (mainly in a fraction of up to about 3 mm), reinforcing fibres, etc. were used as additional components for some of the formulations. The quantities of waste glass provided were subjected to a two-stage grinding process (from initial coarse crushing to obtain powder fractions).

In accordance with the basic requirements for performing various standard laboratory tests of construction materials, sets of formwork moulds were developed for the preparation of composite test bodies:

- cubes with dimensions 100x100x100 mm for compressive strength testing;
- prisms with dimensions 100x100x400 mm for flexural tensile strength testing;
- plates with dimensions 250x250x50 mm for studying the thermal conductivity coefficient.

The technological process for the preparation of the designed formwork moulds involves preparing a model and a gypsum mould with silicone inserts.

The obtained formwork moulds consist of two gypsum side parts with silicone inserts and a bottom part (fig. 1 and 2) with an additional silicone element (overlay or insert). This allows for the application of different technological schemes for moulding composite structure specimens. The silicone inserts of the side parts end on both sides with tensioning elements that fix them to the gypsum parts so as to prevent their deformation when in use. In the molding process, the silicone inserts form the sides of the test bodies, while the bottom forms their lower part.

The design of the bottom of the main formwork moulds includes a drainage system as a structural element which allows the separation of part of the liquid phase (from the introduced mixture consisting of cement mortar and foamed silicate aggregates). Separate sets of formwork moulds have been developed: with a bottom of solid gypsum with perforated holes (and an additional silicone overlay) or with special bottom made of a modified silicone insert with an integrated sieve (or grid element) and silicone overlay. The incorporated sieve is made of corrosion-resistant material with a mesh size smaller than the minimum grain size of the fractions of lightweight additive materials used. The insertion of the silicone overlay upon the bottom blocks the outlet holes and

prevents the liquid phase leaking out. At the same time, additional formwork moulds with a monolithic gypsum bottom (without perforations) and a silicone insert were developed as well.



Fig. 1. Formwork mould for the preparation of composite specimens.



Fig. 2. Formwork mould for the preparation of composite specimens.

A series of composite specimens involving lightweight silicate aggregates and hydraulic binders were prepared in laboratory conditions.

Fractions of foamed glass granules were obtained by applying controlled thermal foaming (at temperatures of up to 900°C) to a primary granulate prepared from a powder fraction of waste glass, a foaming additive and a plasticizer.

Cement mortars (with different values of w/c ratio) were prepared, to which foam-silicate aggregates and additional components were introduced. The complete wetting of the foam-glass aggregates in the cementitious solutions is considered as a recommended technological condition. This allows individual surface pores to close and ensures the presence of a surface layer of binder in the contact areas of adjacent aggregates. After intensive homogenisation, the resulting mixtures were distributed by pouring them into the prepared formwork moulds.

The design features of the formwork moulds allow, by using different technological approaches, to obtain composite specimens with different structures.

Regardless of the compositions and technological methodology used, all specimens were subjected to a residence time (in the formwork moulds) of up to 48 h, after which they were carefully removed from the formwork moulds.

The main differences between the individual composite modifications obtained by the different technological approaches consist in the varying specificity of the structure thus formed and the amount of residual cement phase in the final specimens.

3. Results and discussion

From the prepared mixtures (cement mortar and granules), experimental specimens were moulded by controlled drainage of a portion of the liquid phase through the perforated holes or the embedded sieve at the bottom of the formwork moulds. The implementation of this technological process involves mounting the formwork moulds on metal stands, under which are placed laboratory vats provided for the collection of the draining liquid phase. The application of a water cement w/c ratio in the range of 0.6 to about 0.9 enables the full use of the existing technological

capabilities of the methodology and obtaining materials with relatively low mass.

Placing the silicone overlay stops liquid phase drainage at a predetermined technological stage and allows influencing effectively the formation of the structural specificity of the obtained final samples. The method provides adequate control of the available cement mortar and of the bulk density and mass values of the resulting specimens.

Due to the reduced presence of binder, the final specimens thus prepared are characterized by relatively lower mechanical properties. This effect can be partially limited through the use of higher strength cements (CEM I 52.5 R or others), the introduction of suitable plasticising additives, modifiers, etc. For some product categories for which there are no regulatory requirements for increased mechanical properties, this composite structure allows to obtain various modifications tailored to a specific functional role.

The use of different fractions of foam-silicate aggregates with an appropriate ratio of the radii of the individual granules allows obtaining materials with a variety of structures (fig. 3).



Fig. 3. Structure of an experimental specimen (fragment).

Due to the gradual drainage of a significant part of the cement solution, the prepared specimens have as a characteristic structural feature the availability of direct contact between individual aggregates and the formation of interconnected gaps between adjacent granules. Composite modifications with relatively uniformly sized interconnected gaps allow for the preparation of drainage elements potentially applicable in different operating conditions.

In the experimental specimens prepared with the use of only larger fractions of foamed glass aggregates, a structure with distinct and relatively large interconnected free gaps was observed.

A promising option for additional modification of the obtained composite materials is their further technological processing by impregnation with mixtures of inorganic binders solutions of and non-combustible thermal insulation fillers (perlite or others).

On the basis of two aggregates fractions with a suitable radii ratio, it is possible to form a relatively compact composite structure in which the introduced smaller aggregates are positioned in the free spaces between the granules of the larger fraction. At the same time, the foam glass granules obtained in real technological conditions are characterized by a variety of individual shapes, in a number of cases different from an ideal sphere. Therefore, the software-generated three-dimensional models of composite systems built on the basis of aggregates with ideal spherical shape provide only an approximate idealized view of the state of the structure and the possibilities for optimising the materials.

Developed in addition to the technological approach considered here is an alternative version which provides for an initial distribution in the form of a layer of the prepared mixture (cement mortar-foam granules) placed on a separate grid stand. This allows for a more efficient drainage of a significant part of the liquid phase. After a process time of 5 to 10 min, the resulting residual mixture is introduced into the formwork moulds.

A comparative series of composite test specimens was prepared without draining the liquid phase (following the classical method). The specimens were made using formwork moulds with a silicone overlay pre-placed on the bottom (prior to the introduction of the prepared cement mortar-silicate aggregate mixture), which prevent

free drainage of the liquid phase. Another technological option is the use of formwork moulds with a monolithic bottom (without perforation) and a silicone insert.

The molded compact specimens are characterized by a typical composite structure formed by the cement phase (acting as a matrix) and fractions of glassy aggregates distributed in the volume of the material. The resulting composites represent a potential fully functional alternative to different types of standard thermal insulating lightweight concretes obtained with the participation of conventional lightweight aggregates.

This technological method enables the preparation of relatively more massive composite samples with increased mechanical characteristics, but at the same time with higher values of bulk density and relatively higher mass of the material. Observation of the prepared cross-sections of the moulded experimental specimens revealed mainly extensive zones with a predominant concentration of foam silicate aggregates (fig. 4).

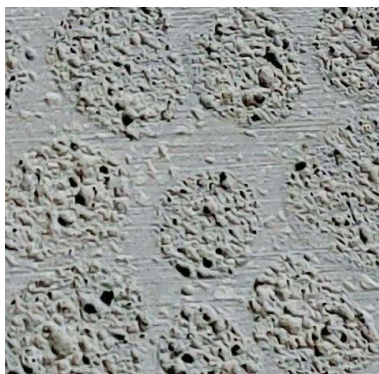


Fig. 4. Section (fragment) of experimental composite specimens.

At the same time, the cross-sections of some of the obtained composites showed the presence of limited local areas composed predominantly of massive cement phase. The considered technological method is suitable for moulding composite specimens mainly at w/c ratio of about 0.5 or lower values.

An appropriate technological option for the preparation of other composite modifications is the initial co-introduction (in the prepared cement mortar) of the necessary fractions of granulated foam glass and of other additional components (expanded perlite and/or reinforcing fibres, etc.).

The performance characteristics of the base components used in the development (Portland cement and foam-silicate aggregates) ensure the production of durable, waterproof and non-combustible materials.

The main technological guidelines for the optimization of the structure and functional capabilities of composites is the use of appropriate fractions of lightweight additive materials, the application of appropriate proportions between the amounts of cement mass and foam silicate aggregates, the use of more prolonged and intensive homogenization of the prepared mixture (cement mortar - granules), etc.

4. Conclusions

Formwork moulds kits were developed to mould experimental specimens with composite structure based on inorganic hydraulic binders and foam glass aggregates. The design characteristics of the formwork moulds allow for the application of different technological approaches in the moulding process: by controlled drainage of part of the cement mortar or without removal of part of the liquid phase. The specimens thus prepared can be used for laboratory tests to determine compressive strength, flexural tensile strength and to study the thermal conductivity coefficient.

The specificity of the used compositions and the capabilities of the individual technological schemes offer a possibility to obtain a variety of composite modifications. A comparative analysis of the specimens prepared under different technological regimes has

revealed characteristic structural differences and variations of the amount of cement phase, all of which determine the performance properties of the composites and the potential opportunities for their functional application.

A possible alternative use of the prepared formwork moulds lies in the moulding of experimental specimens obtained from other materials, subject to the necessary technological and methodological compatibility.

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Building eco-friendly houses with construction coatings, bricks and panels based on sedimentary rocks

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Abstract: *In the development of new composite structural elements based on sedimentary rocks (clays) as inorganic binders and foam composite materials, their physical characteristics are presented for the construction of recipe compositions and the determination of their complex new properties. The role of the components of the compositions, together with organic reinforcing fiber materials such as straw, rice husks and others, basic parameters of the technological regime and other factors for ensuring adequate workability of the prepared composite compositions for obtaining building coatings and elements - bricks and panels for building ecological houses - is considered. The determination of the necessary technological characteristics of the composite mixtures ensures their effectiveness.*

KEYWORDS: SEDIMENTARY ROCKS (CLAYS), PLASTICITY, DENSITY, VISCOSITY

1. Introduction

In recent years, luxury homes made from sedimentary rock-based materials have begun to be built in Europe, the US and Australia, with more than 10,000 in Australia alone. Obviously, this is not about some fashion extravagance, because a similar trend can be observed in the construction of public buildings such as schools, hospitals, shops, tourist sites, museums, churches, etc. More and more universities, academic institutions and construction companies have become involved in the development of new compositions and technologies for the production of such materials.

There is a growing trend to consider natural materials used in construction as the materials of the future, mainly because of their energy and performance efficiency and the proven betterment of quality of life that they bring. It should be noted that buildings constructed using bricks, panels and mortars based on unfired sedimentary rocks absorb and release airborne moisture faster and to a greater extent than those using other materials (e.g. 30 times more than baked bricks). This is a characteristic that brings about a balance in the indoor climate. Unbaked products based on this kind of building material are able to conserve heat, isolate noise and save energy [1, 2]

The topical significance of the proposed material lies in the discovery of the fundamental interactions between the elements in the sedimentary rock-based recipe compositions which can contribute to the creation of a non-combustible and non-flammable thermal and acoustic insulating eco-composite building material. It can be obtained in two varieties:

(a) as coarse and fine eco-friendly coatings, by adding organic and inorganic modifiers (hemp stalk fibres, pieces of different lengths of wheat, rye, oats, or barley, rice husk, marl, glass and other fibres and waddings) to the basic raw materials;

(b) as heat-treated building elements for construction and renovation, where the material is enriched by adding suitable modifiers (heat-treated rice husk, expanded clay, expanded perlite, vermiculite, foam glass granules, etc.) to the basic raw material.

In order to ensure adequate energy efficiency, modern construction makes wide use of external thermal insulation systems for various types of buildings [3].

The research objectives of the present paper are of exceptional importance for the development and the future use of an innovative multifunctional eco-friendly composite building material based on plastic sedimentary rocks and possessing thermal and acoustic insulating properties.

A further possibility of applying different fractions of foam glass granules is their introduction, together with organic fibres, as lightweight additive materials.

Bulgaria offers the necessary conditions for the implementation in the construction industry of products and materials based on sedimentary rocks.

The increasing production capacities in various industries and the intensive utilization of the resulting products in various

economic sectors and in everyday life are accompanied by the generation of significant quantities of waste materials of different origins [4-6]. The existing quantities of waste products such as waste glass, straw and rice husks represent a significant environmental problem with an alarming prognosis for its growth in the long term. Effective and safe waste management requires the existence of established landfills and an organized system of collection and transportation using specialized transport equipment [12-14]. A partial but very good solution to the problem is the development and implementation of production technologies through which a number of waste products can be recycled waste-free and then be used as primary raw materials in the production of a wide range of products.

Due to their high durability, significant chemical resistance and specific thermophysical and mechanical properties, glassy waste materials are considered as a fully usable raw material [7]. There are already industrial production methods implemented for the utilization of vitreous waste raw materials and the production of thermal insulating foam glass materials, mainly from differently sized granules [8-10].

A team from IMSETHC-BAS has been working on improving the foam glass production technology and developing innovative equipment for obtaining foam glass granules based on patents and utility models. At the same time, the team organizes the use of organic and inorganic fibres to strengthen the resulting mixtures.

The individual technological stages of the production cycle have been optimised. The laying of the coatings has been related to determining the time required to give them the necessary strength. Adequate regimes for the processing of the building elements - bricks and panels - have been developed, tailored to the specificity of the waste raw materials used and with a focus mainly on the use of a specific drying mode that can make the elements as strong as necessary.

A further possibility for creating the composite material recipes lies in employing different compositions of the base material - sedimentary clays and different fractions of foam glass granules introduced as lightweight additive materials, as well as in the use of differently sized fibres.

Innovative composite building elements have been developed using granulated foam glass and inorganic binders, with clays as the main component. Owing to their characteristics, the prepared composites represent a potential alternative to a number of traditional and standard thermal insulation building products. An experimental industrial line has been developed for the production of granules, panels and other elements with potential application in construction [11-14].

What deserves particular attention is the type and size of the fibres used. When the reinforcing material for the obtained clay products is straw, it must be cut to a certain size, the longitudinal pieces being mainly up to 5 mm long for coatings and up to 10 mm long for products such as bricks and panels. Another fibre material

is rice husks, treated in the same way as straw but without the need of cutting.

To further modify the material, a variety of research data with a broad thematic scope were analyzed. At the same time, the experimental results obtained in additional laboratory studies are a prerequisite for further expanding the technological possibilities for the preparation of new composite products based on sedimentary rocks.

The aim of the present work is to determine the technological characteristics of composite mixtures applicable for the preparation of different modifications of experimental specimens for coatings and separately for reinforced and unreinforced bricks and panels. The research carried out takes into account the existing experience in the field of composite materials [15-19] and the laboratory analytical methods presented in the specialized literature [20,21].

2. Description of experimental thermal insulation composites based on sedimentary rocks

1.1. Creation of thermal insulation compounds for coatings

Table 1 presents the experimental recipe compositions (series A.1 - A.4) developed on the basis of a dry mix consisting of clay, sand, foam glass granules up to 2 - 3 mm in diameter (Fig. 1.), fibre - rice husks (Fig. 2.), modifying additives (perlite and zeolite) and water at a water to dry mix ratio (B/CS) of 30% to 100% for coatings and 25% to 100% for articles, respectively.

The composition of the mixtures is in units of weight.



Fig. 1. Foam glass granules



Fig. 2. Rice husks

The availability of an optimal amount of water needed to obtain a mixture of normal density (viscosity) is one of the most important indicators of the obtained working mixture. This density should be neither low nor high, but just right to allow good adhesion to the work surface for application by hand or with a shotcrete machine. The drying temperature is very important. Drying at freezing temperatures is not permitted.

Compositions A.1	Compositions A.2
Composition A.1.1 Dry mixture (DM) = clay (C) : sand (S) : granules (G) : fibre (F) + water (W) C/S/G/F = 25+25+25+25	Composition A.2.1 Dry mixture = clay (C) : sand (S) : granules (G) : zeolite (Z) : fibres (F) + water (W) C/S/G/Z/F = 35+25+25+10+5
Composition A.1.2 Dry mixture = clay (C) : sand (S) : granules (G) : fibre (F) + water (W) C/S/G/F = 45+25+30+5	Composition A.2.2 Dry mixture = clay (C) : sand (S) : granules (G) : zeolite (Z) : fibres (F) + water (W) C/S/G/Z/F = 30+25+20+20+5
Composition A.1.3 Dry mixture = clay (C) : sand (S) : granules (G) : fibre (F) + water (W) C/S/G/F = 35+35+30+0	Composition A.2.3 Dry mixture = clay (C) : sand (S) : granules (G) : zeolite (Z) : fibres (F) + water (W) C/S/G/Z/F = 35+20+20+25+0
Composition A.1.4 Dry mixture = clay (C) : sand (S) : granules (G) : fibre (F) + water (W) C/S/G/F = 35+30+35+0	Composition A.2.4 Dry mixture = clay (C) : sand (S) : granules (G) : zeolite (Z) : fibres (F) + water (W) C/S/G/Z/F = 35+20+20+25+0
Compositions A.3	Compositions A.4
Composition A.3.1 Dry mixture = clay (C) : sand (S) : granules (G) : perlite : fibre (F) + water (W) C/S/G/P/F = 35+25+20+10+10	Composition A.4.1 Dry mix = clay : sand : granules : perlite : zeolite, fibre + water C/S/G/P/Z/F = 40+20+20+10+5+5
Composition A.3.2 Dry mixture = clay (C) : sand (S) : granules (G) : perlite : fibre (F) + water (W) C/S/G/P/F = 30+15+35+5+5	Composition A.4.2 Dry mix = clay : sand : granules : perlite : zeolite, fibre + water C/S/G/P/Z/F = 35+25+25+05+5+0
Composition A.3.3 Dry mixture = clay (C) : sand (S) : granules (G) : perlite : fibre (F) + water (W) C/S/G/P/F = 35+35+20+10+0	Composition A.4.3 Dry mix = clay : sand : granules : perlite : zeolite, fibre + water C/S/G/P/Z/F = 35+15+25,10+10+5
Composition A.3.4 Dry mixture = clay (C) : sand (S) : granules (G) : perlite : fibre (F) + water (W) C/S/G/P/F = 30+20+25+20+05	Composition A.4.4 Dry mix = clay : sand : granules : perlite : zeolite, fibre + water C/S/G/P/Z/F = 40+20+10+10+20+0

1.2. Data for determining the parameters of the composite mixture

1.2.1. For coatings

The choice of clay for coatings depends on its composition, homogeneity, particle size, moisture content and some other physical parameters.

The clay deposit is selected according to its composition in order to achieve the desired strength of the coating. Clays with more iron oxide content (red clays) and others with more aluminium oxide content (grey clays) can be used. After drying, the clay is ground to a particle size of 0.5 – 1.0 mm and the ingredients are dosed and homogenised. The studies were carried out with plastic sedimentary rock (clay) from the region of the town of Troyan. Solid mineral and biological inclusions such as quartz, belemnites, etc. were removed from the clay. Sands of different particle sizes – from 1.0 to 4.0 mm – were used.

Typically, coatings are not baked but dried at the natural ambient temperature. They are usually made up to 5.0 mm thick, therefore the size of the foam glass granules is small – up to 2.0 or maximum 3.0 mm and so are the sand ones.

The greater the excess water in the resulting mixture, the greater the drying distortion will be. Moreover, there is a danger of delamination of the mixture, which severely degrades its properties. Thus, any water layers that may have formed can freeze in winter working conditions and will cause the coatings to crack and crumble. It is therefore not recommended to work in winter conditions.

1.2.2. For bricks and panels

Table 1. Experimental compositions in weight %

The materials used to make products such as bricks and panels do not differ from those for coatings except in the size of the components involved.

Depending on the type and size of the product, it can be reinforced using different materials, mainly steel grids for panels with larger dimensions, as well as modifications of the recipes with water glass modulus 1.4 ($\text{Si}_2\text{O}_2/\text{Na}_2\text{O}$) and sodium silicochloride (Na_2SiF_6).

The times for drying and removal of the formwork of the obtained elements are determined experimentally according to the thickness of the products.

The quantity of fibres and their size is also determined experimentally.

The quality of the articles - mainly their strength - depends on the time and pressure during the filling the formwork.

The determination of the normal density, i.e. the viscosity, is carried out using a vicat apparatus.

1.2.3. For coatings

Once the dough (mixture) is ready, it must retain its plasticity for some time before it can be used and processed. The drying time is determined on the same sample as the one previously used to establish the normal density.

1.2.4. Preliminary drying and final drying

These are parameters of the coating compositions and articles which depend on the condition of their gel masses. The evaporation of water from the gel masses causes surface and deep drying of the articles.

To prevent cracking of the articles, it is particularly important to observe the amount of water prescribed in the recipes, as well as the drying time and speed, all of which must be adjusted according to the composition.

A study of the physical and mechanical properties of compositions used for the preparation of thermal insulation coatings and articles

The water in the compositions is intended to be up to 30 or 25 % over the total weight. The viscosity of the coatings varies from 100 to 120 mN.s/m² depending on the formulation of the dry mix. The viscosity of the articles was not measured as the material for them is not flowable but plastic.

The preliminary drying time of the articles is within 24 h before placing them in a drying oven for final drying.

Table 2. Physical and mechanical properties

Composition type	Characteristic		
	Viscosity, cP, [mn.s/m ²]	Compressive strength, [kg/cm ²]	Thermal conductivity coefficient, λ , [W.m-1.K-1]
For coatings			
120	140	0.1-0.15	6
120	140	0.1-0.15	6
100	120	0.1-0.15	5.5
100	120	0.1-0.15	5.5
For articles			
-	150	0.15-0.17	12
-	150	0.15-0.17	12
-	135	0.15-0.17	12
-	135	0.15-0.17	12

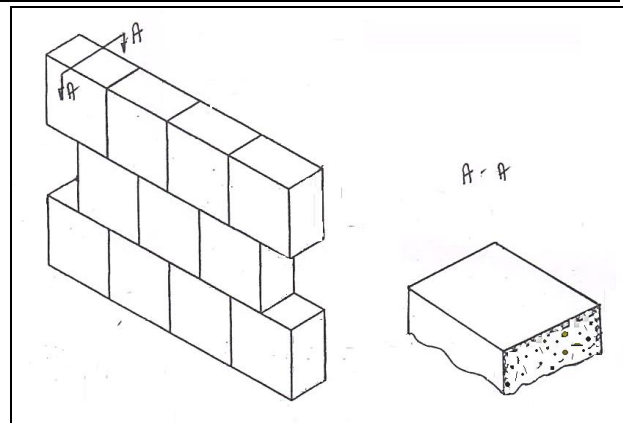


Fig. 3. Diagram of brickwork for walls

Figure 3 presents a diagram of brick masonry, according to composition A.1. – clay, sand, foam glass granules, rice husks.

2. Conclusions

With the increase of heat and sound insulation additives, the viscosity of the coatings decreases from 120 to 100 cP.

With the increase of heat and sound insulation additives (foam glass granules, perlite, zeolite, etc.), the drying time of the coatings decreases from 6 to 5.5 h, and for the obtained products it is 12 h.

With the increase of additives in the brick and panel composite, the compressive strength decreases from 150 to 135 kg/cm².

As a result of the present and forthcoming studies, it will be possible to correlate the obtained dynamic characteristics concerning the thermal and acoustic insulation of the modified sedimentary rocks, as well as the relationship with the static characteristics – the strength of the specimens of the different recipe compositions, the drying time of the mixtures, the viscosity of the coatings, etc., i.e. a decrease in some characteristics at the expense of an increase in others and vice versa. It will be possible to create mathematical models with which to optimise the compositions for different specific purposes.

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Electrical and thermal monitoring for remote diagnostics of X-ray generators

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Abstract: Many industries, like the aviation industry and border control, rely on X-ray inspection systems for their security operations. These systems are state of the art complex electromechanical machines that need to be maintained by highly qualified service specialists. The most critical part of each x-ray inspection system is the x-ray generator. In this article, we propose a technique for remote diagnostics based on electrical and thermal telemetry monitoring. We have studied the most important electrical and thermal parameters of various conventional X-ray generators equipped with stationary anode X-ray tubes, up to 300kV acceleration voltage, and their correlation to the operational condition of the generator. The experimental results, obtained in a real operational environment, show that a limited number of monitoring parameters are enough to properly estimate the condition and diagnose some failures in X-ray generators. A simplified physical model, based on electrical and thermal parameters, has been proposed and compared with the results.

Keywords: X-RAY GENERATOR, FAILURE, SENSORS, REMOTE DIAGNOSTICS, PREDICTIVE MAINTENANCE

1. Introduction

Modern X-ray inspection systems are mighty electromechanical machines that incorporate not only conventional X-ray generators but also a combination of much more accurate CT (Computer Tomography) modules, delivering high-resolution and 3D X-ray image reconstruction. Aside from their main function to generate and detect x-ray radiation, those systems are equipped with advanced computer algorithms for automatic image recognition of threats like weapons, explosives and other prohibited objects and substances. X-ray systems are widely used in various security applications, like luggage and cargo screening in the aviation industry as well as for borders and customs control.

In general, the X-ray inspection systems are subjected to very intense usage, and in most cases can be used in environmental conditions that vary in a wide range. For example, a regular airport hand-luggage scanner operates continuously generating x-ray emissions for long periods that may exceed 5 hours each. On other hand, the EDX (Explosive Detection Systems) used for inspection of checked luggage usually operate and generate continuously, without interruption, x-ray emission for even longer periods that may exceed 10-12 hours, executing thousands of luggage scans per day.

Considering the intense usage of X-ray inspection systems, their complexity, and their importance for security, the high availability became the most important KPI (Key Performance Indicator). This way problems with estimating their health status, making failure predictions, and taking actions for failure prevention became essential for every owner and user that needs high availability of such devices. Even in modern procurement regulations, this question occurs explicitly in the service contracts.

The most powerful tool for X-ray systems health status estimation and failure prediction is remote monitoring of technical parameters and environmental conditions. Using telemetry sensor data in real time, health status can be obtained using modern mathematical algorithms [1]. All this makes the remote monitoring and remote diagnostics of X-ray systems a "must-have" instrument for every service provider who wants to provide the highest possible availability but keeping the optimal maintenance cost [2].

The most critical and simultaneously the most loaded part of each X-ray system is the X-ray generator. For this reason, we're focusing our attention on a technique for remote diagnostics based on electrical and thermal telemetry monitoring of conventional X-ray generators.

2. Materials and methods

In this article, we propose a technique for remote diagnostics based on electrical and thermal telemetry monitoring. A limited number of important electrical, thermal, and environmental parameters, of conventional X-ray generators, are monitored

remotely in real-time. The studied generators are equipped with conventional stationary anode X-ray tubes (Fig. 1) which are mounted inside the generator body and immersed in insulator oil. The typical Hi-tension range for X-ray inspection purposes starts from 140 up to 300kV acceleration voltage.

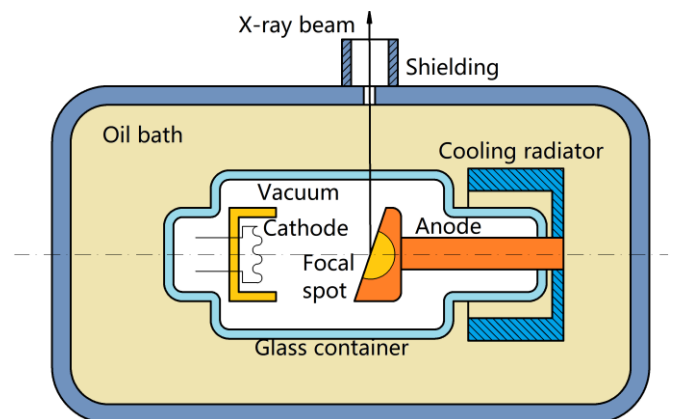


Fig. 1 General representation of an x-ray tube [3].

To achieve remote diagnostics of X-ray generators it isn't necessary to monitor all the X-ray system parameters. The most important parameters, subject to remote monitoring in real-time are the following 8:

Electrical parameters:

1. Positive Hi-tension (from +70 to +150 kV)
2. Negative Hi-tension (from -70 to -150 kV)
3. Cathode current (filament current)
4. Anode current (electron beam)

Thermal parameters:

1. Ambient temperature inside the housing of the X-ray inspection system
2. Surface temperature of the X-ray generator

Environmental parameters:

1. Environmental temperature
2. Relative humidity

To successfully monitor these parameters in real-time, we're using sensor networks comprising voltmeters, thermometers, and humidity sensors. Those located inside the housing of the X-ray system consider "Internal sensors". Environmental parameters are measured by "External sensors". Data collection and data transfer are established using the commercial infrastructure of the PM4X LPR™ solution represented in Figure 3. All sensors for this

particular study are installed on more than 10 X-ray inspection systems of different types operating in a real environment. The data have been collected over 2 years.

The monitored electrical parameters are absolute indicators because they are directly related to the current operational condition of the generator, and they can be easily used for remote diagnostics. In contrast, the thermal and environmental parameters cannot be used directly for condition diagnostics if their values still fit the threshold range predetermined by the manufacturer. Of course, if their values exceed the threshold, usually it is too late for a reaction and the failure is imminent.

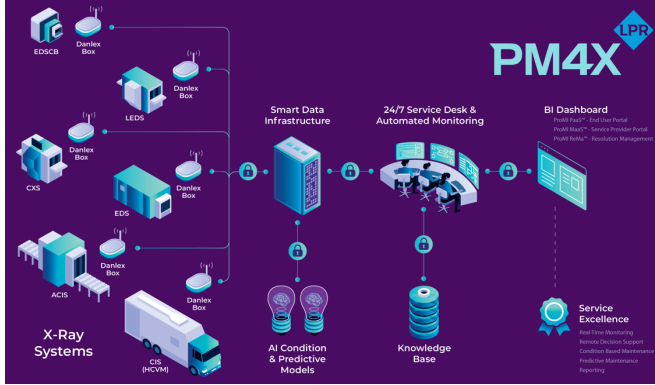


Fig. 2 PM4X LPR infrastructure for remote monitoring [2].

For this reason, we have developed and tested in practice a simplified physical model dedicated to estimating the internal temperature of the X-ray tube components by only measuring the surface temperature of the generator body [3]. The results of this model determine four operational regimes and only the first one assures the normal operation. All other regimes lead to long-term or short-term failures that can be diagnosed or even prevented. Table 1 represents an example for x-ray generator with 140 kV accelerating voltage operational regimes as a function on the surface temperature.

Table 1: Relation between the X-ray tube operational regime, measured surface temperature and estimated temperature X-ray tube cooling element [3].

Operational regime	Defined internal temperature range at X-ray tube cooling element	Measured generator external surface temperature
Normal operation	$10^{\circ}\text{C} \leq T_i < 80^{\circ}\text{C}$	$10^{\circ}\text{C} \leq T_{out} < 51^{\circ}\text{C}$
Slow degradation	$80^{\circ}\text{C} \leq T_i < 250^{\circ}\text{C}$	$51^{\circ}\text{C} \leq T_{out} < 80^{\circ}\text{C}$
Faster degradation	$250^{\circ}\text{C} \leq T_i < 300^{\circ}\text{C}$	$80^{\circ}\text{C} \leq T_{out} < 87^{\circ}\text{C}$
Imminent Failure	$T_i > 300^{\circ}\text{C}$	$T_{out} > 87^{\circ}\text{C}$

All the thresholds are set based on physical models in combination with the engineering data provided by the manufacturers for each X-ray generator component.

3. Algorithm for remote diagnostics of X-ray generator

The general algorithm consists of three steps.

The first step is to collect the sensor data. PM4X LPR™ solution collects data from internal and external sensors. If the thresholds are exceeded even on this step the diagnostic can be obtained and failure determined.

The second step is to plug this data into ML (Machine Learning) models. Utilizing predetermined thresholds and historical data these models can derive dynamically adapted threshold values

of the measured parameters corresponding to the environment and the feedback from the next step.

The third step uses predictive modeling to forecast the condition based on the current state estimated by the dynamically adapted thresholds. Here is also the feedback to step two for next iteration of dynamic adaptation of thresholds.

If the thresholds do not adapt, it means that the X-ray system works in its nominal safe conditions.

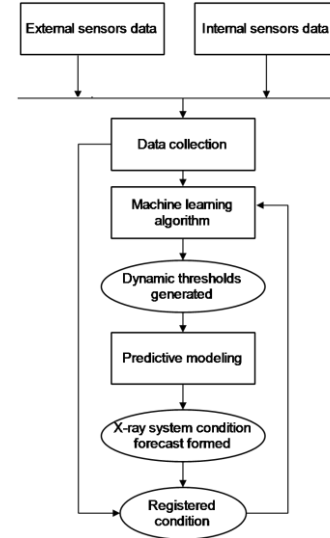


Fig. 3 General algorithm for health status and condition forecast estimation

These three steps are excellent for remote diagnostics of the current state but for credible failure probability estimation, these steps are insufficient. In such cases, the algorithm changes into an advanced form. Figure 4 shows an advanced algorithm for estimation of failure probability based on sensor telemetry data combined with additional data sources like, X-ray OS (Operating System) log messages and history maintenance reports. Detailed description of this algorithm is published under the patent application EP3955707A1 [4].

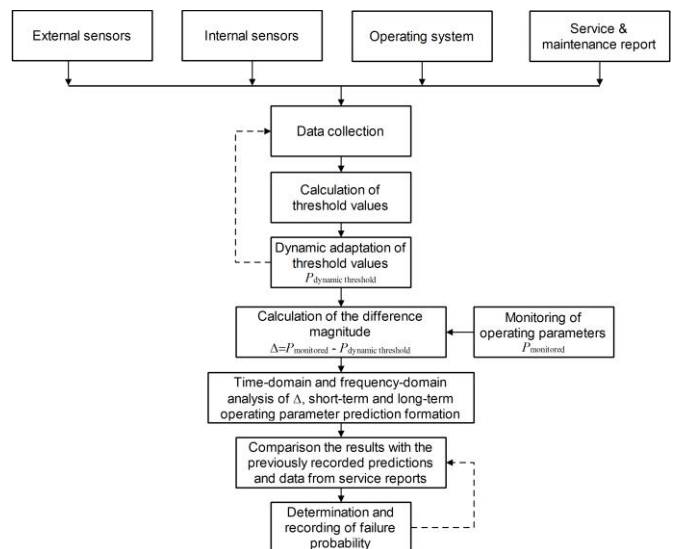


Fig. 4 Advanced algorithm for health status and failure probability estimation [4]

4. Results and discussion

Using remote monitoring and improved algorithms we succeeded in remotely diagnosing and forecasting the status of X-ray generator in real operational conditions.

In the case of successfully powered-on and stand-by state of X-ray system, the absence of cathode (filament) current is an absolute indicator for failure of the generator and its power supply.

During the execution of the X-ray scanning procedure, the absence of positive or negative Hi-tension is an absolute indicator of failure of the generator and its power supply.

Measuring the surface temperature of the generators helps to estimate the wearing rate of the x-ray tube. An interesting experimental result is the prolonged operation at the lower range of the “Slow degradation” regime, this will not activate thresholds warnings but could lead to up to 50% shortened lifespan of the generator.

5. Conclusions

Tested in practice, real-time remote monitoring allows the collecting of real-time data from electrical and thermal parameters and makes remote diagnostics not only possible but reliable. Using simple algorithms, the remote diagnostic and condition forecasting of X-ray generators is achievable with the monitoring of only 8 parameters per generator. Forecasting the condition could initiate on-time corrective actions to prevent sudden failures.

6. Acknowledgements

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Optimization of a casting technology with the tools of Magmasoft software package

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Abstract: The latest version of MAGMASOFT software package – MAGMA6.1 is used to simulate and optimize a casting formation. The alloy AlSi7Mg was used for this purpose. The casting is intended for the production of a wire connector for high voltage power transmission networks. Die casting method is used. The results obtained from the stimulation of pouring and crystallization of the casting are presented. Based on the located defects and the analysis performed, changes of the technology were made, which ultimately ensure the formation of a defect-free and soundness casting.

Keywords: COMPUTER SIMULATION, CASTING FORMATION, OPTIMIZATION

1. Introduction

Mathematical modeling of the processes of casting formation from various types of alloys has long become an integral part of the process of designing and optimizing foundry technologies. It finds its widest application in its software implementation. The incontrovertible favorite in this area is the MAGMASOFT software package^[1].

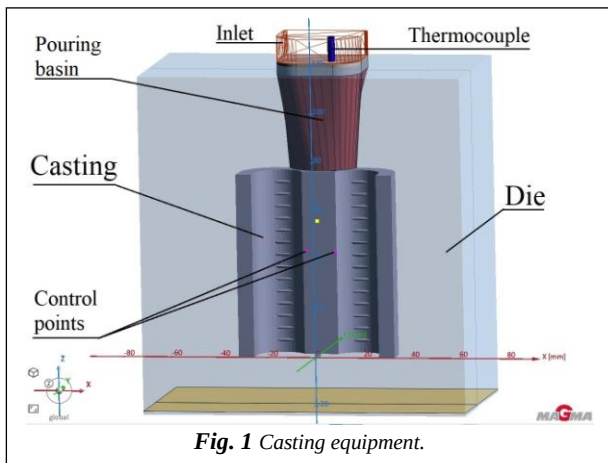
Created by the company MAGMA Giessereitechnologie based in Aachen, Germany^[2], the package is continuously developed and enriched by the company's employees, through the development of numerous scientific projects with various units of leading German institutes such as the Foundry Institute in Aachen, Max Planck Institute, etc. The mathematical models included in the program are constantly updated, reflecting most of the most important scientific developments in the field of materials science, whether they are the subject of doctoral dissertations or scientific publications. Both the mathematical models are being extended and improved, covering an ever-wider range of phenomena and processes, as well as the database, which allows the treatment of an ever-wider range of materials and alloys, as well as casting methods. It is a well-known fact that MAGMASOFT has no competition in terms of the built-in database in the package.

In addition to successfully optimizing the casting technologies, MAGMASOFT is also successfully used in the research and formation of new materials and alloys^[3,4].

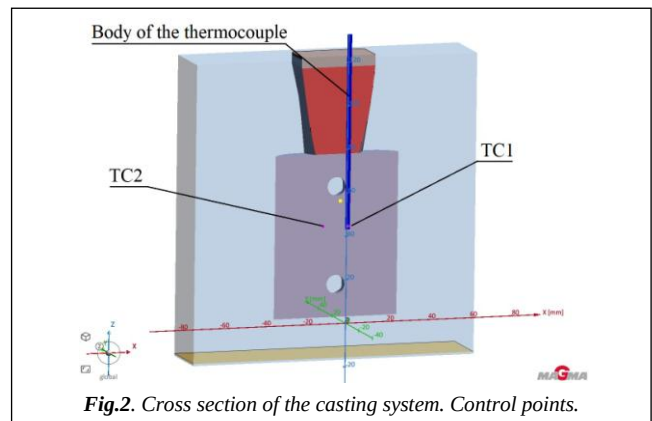
In this work, the capabilities of the latest version of MAGMASOFT – MAGMA6.1 are illustrated on the example of a casting for the production of a contact detail for the needs of high-voltage power transmission networks. The geometry of the casting equipment and the casting conditions are given in the next section.

2. Casting equipment and conditions

The geometry of heuristically build casting equipment after rebuilding with the MAGMASOFT's modeler is presented on Fig.1.



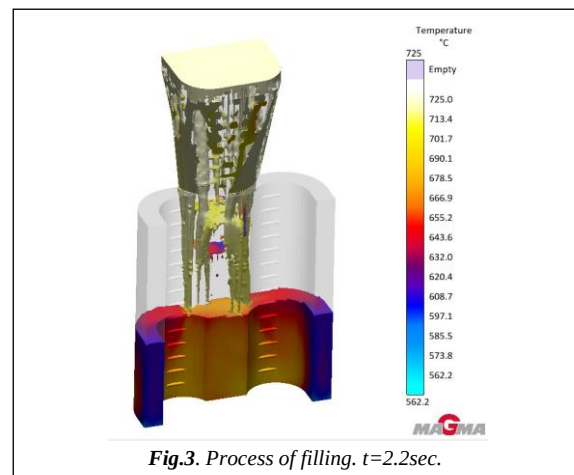
The working surface of the mold is coated with „Wolfraco“ coating. It is a graphite-based consistency. It is diluted with water, has good application and gives smoothness to the surface of the casting. A thermocouple is mounted inside the casting which sensitive end is visualized with the control point TC1(Fig.2).

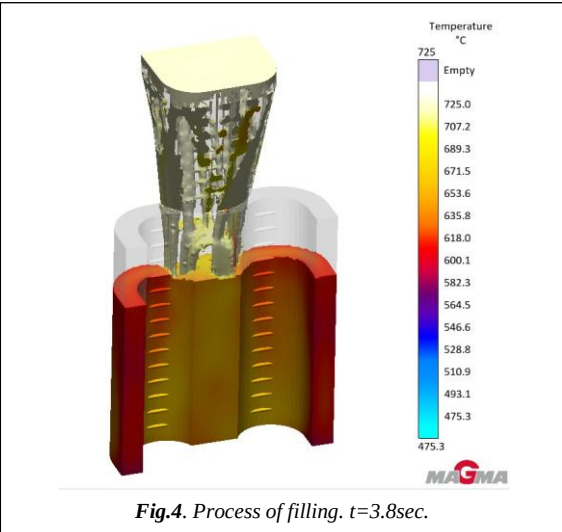


The symmetrical point of TC1 with respect to the vertical plane of symmetry is labeled TC2. "INLET" is the software inlet of the liquid metal when it filling the mold cavity. Die casting method is used to simulate the processes of pouring and crystallization. The process of filling of the casting with molten metal is presented in next section.

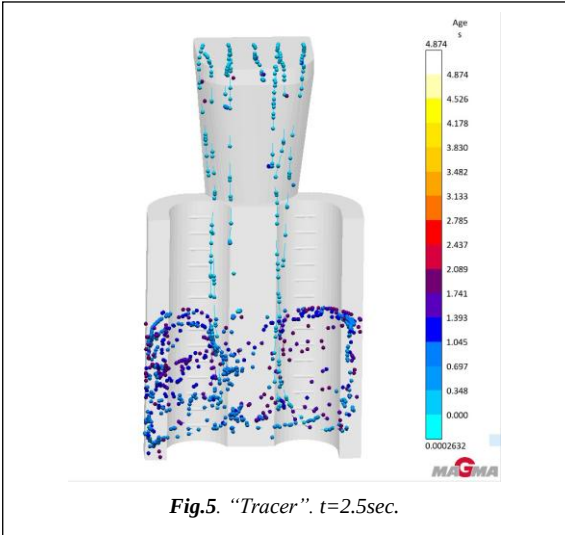
3. Results of the filling process

The following two figures show two consecutive moments of the process of filling the mold cavity with liquid metal. The temperature field is color-code represented. The process occurs by maintaining a full cup.



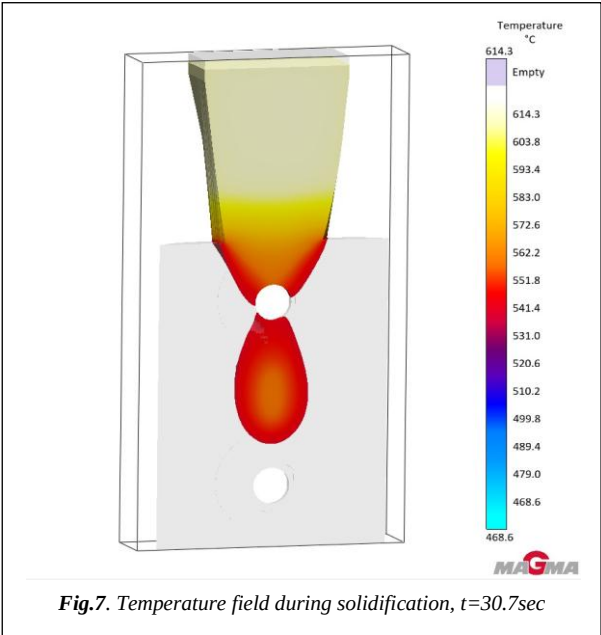
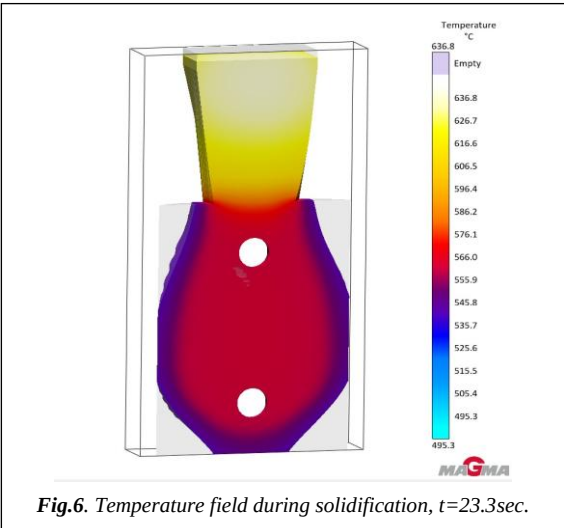


The software offers a "Tracer" tool that allows the trajectories of individual fluid particles to be traced during the filling. Its visualization is shown on Fig.5 in a concrete moment of time.

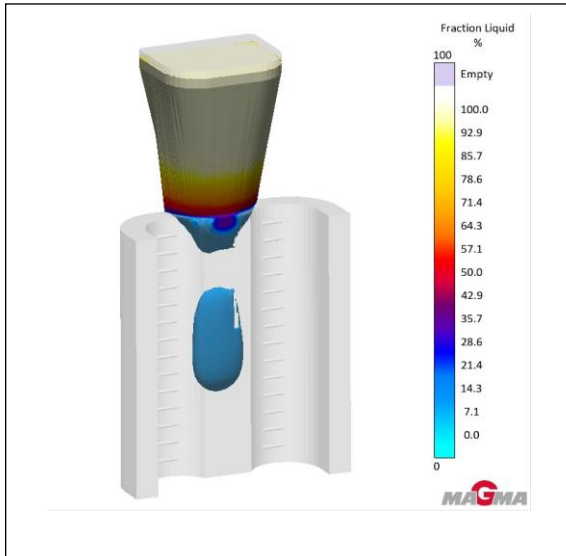
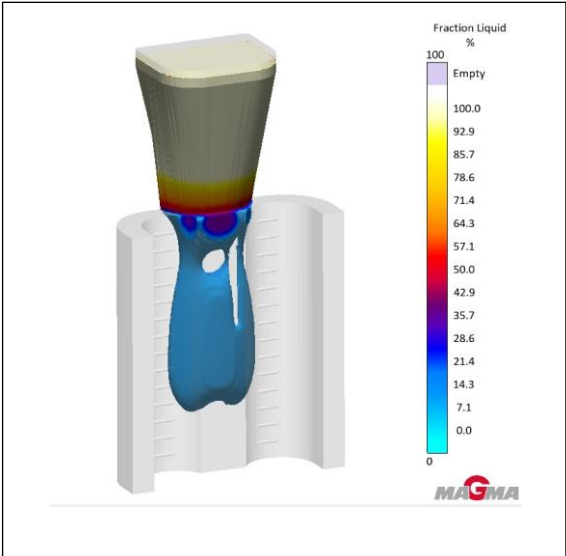


4. Results of the solidification

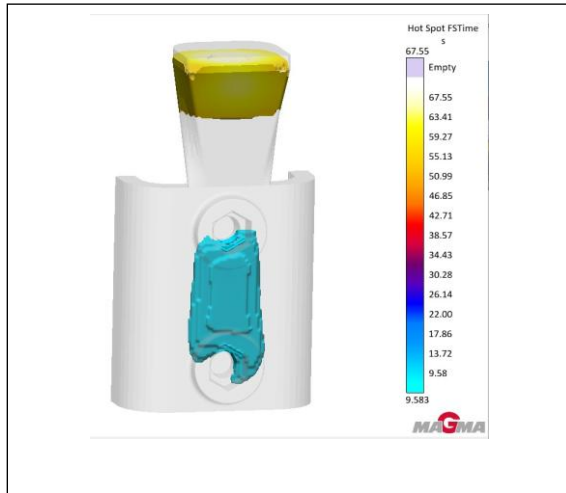
As a result of the simulation of the crystallization process of the casting, its temperature field was obtained. It is presented at two characteristic moments in time in Fig. 6 and Fig. 7. The solidifying phase in the casting is visualized as transparent to highlight the liquid phase zone.



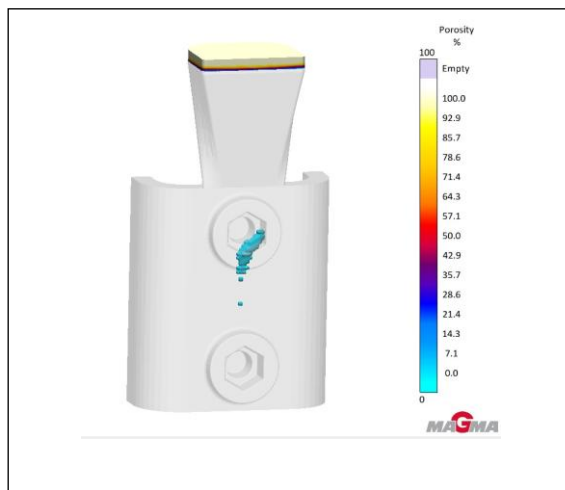
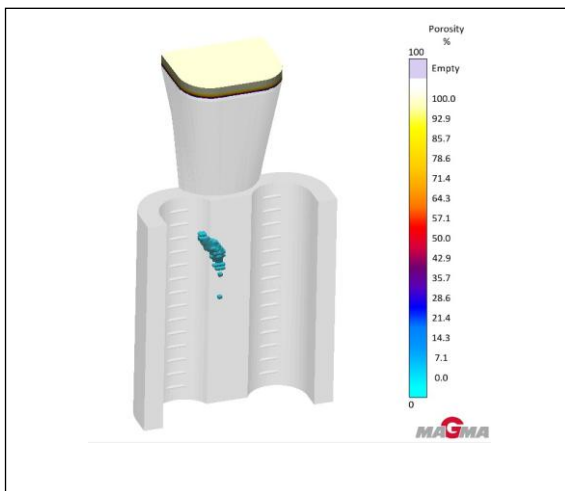
In Fig. 7, a thermal hot spot between the two holes of the part is clearly visible. This finding is also confirmed by the evolution of the "Fraction Liquid" criterion, presented in Fig. 8 and Fig. 9.



This thermal hot spot is also confirmed by the "Hot Spot FS Time" criterion in Fig.10.



All these criteria indicate that a mass deficit and a high risk of porosity should be expected in the outlined area. This is also confirmed by the "Porosity" criterion shown in Fig. 11 and Fig. 12.



Localized porosity is represented by a view from both sides of the casting. For greater clarity, the free of defect part of the casting is represented as transparent.

5. Optimization

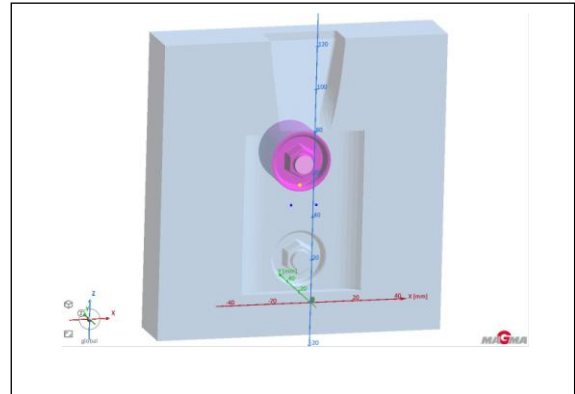
The analysis of the evolution of the temperature field during the crystallization indicates that, on the one hand, the filling cup cools down too early and fails to sufficiently supply the mass deficit in the casting, and on the other hand, the insert forming the upper

opening crosses the feeding path and contributes to the formation of the defect.

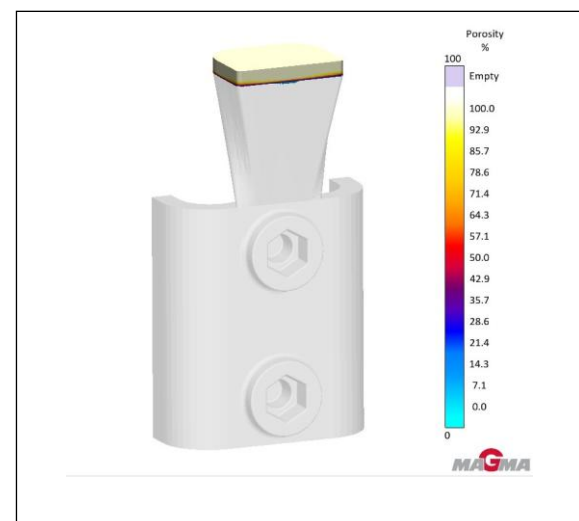
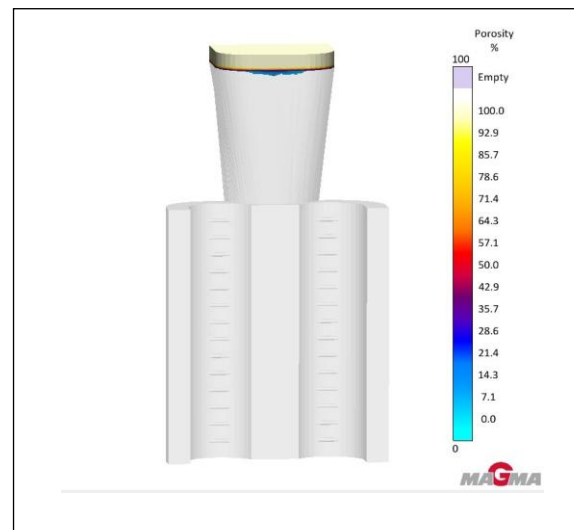
To eliminate these undesirable effects, the following changes were made:

- The working surface of the filling cup was treated as being covered with an insulating coating with high thermal resistance;
- To form the upper opening of the part, a new steel insert was constructed, the contact surface of which with the casting is assumed to be covered with the same coating.

The geometry of the new insert is shown in Fig.13.



After these changes, the feeding of the casting has already been significantly improved, flowing consistently from its lower end to the filling cup without closing thermal nodes. Under these conditions, crystallization forms a defect-free casting. This is evidenced by the "Porosity" criterion presented in Fig.14 and Fig.15.



6. Thermocouple inertia estimation

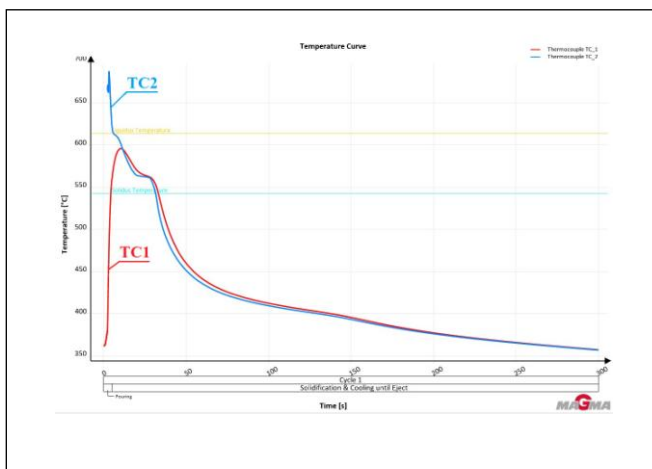
Thermocouples used to measure temperatures in metal melts are built into a protective body /usually a tube made of heat-resistant material/ for the following reasons:

- they work at high temperatures;
- they work in a chemically highly aggressive environment;
- need for electrical insulation.

Thus constructed, the thermocouple is a body with its own thermophysical parameters, which exchanges heat with the environment and forms its own temperature field. The sensitive tip of the thermocouple measures the temperature at the point of contact with this temperature field.

Therefore, the measured temperature does not coincide with the temperature of the point in the medium that we want to measure.

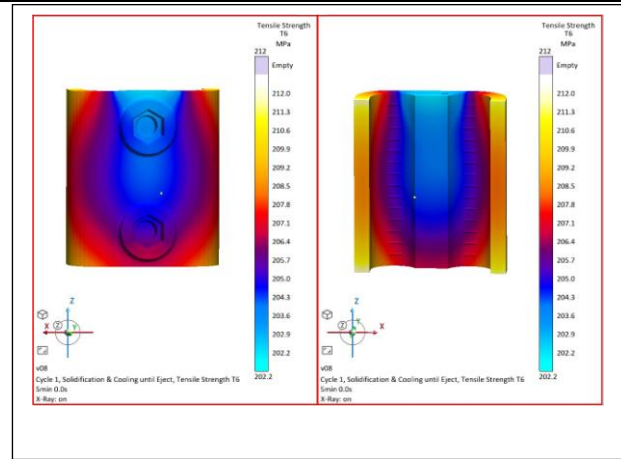
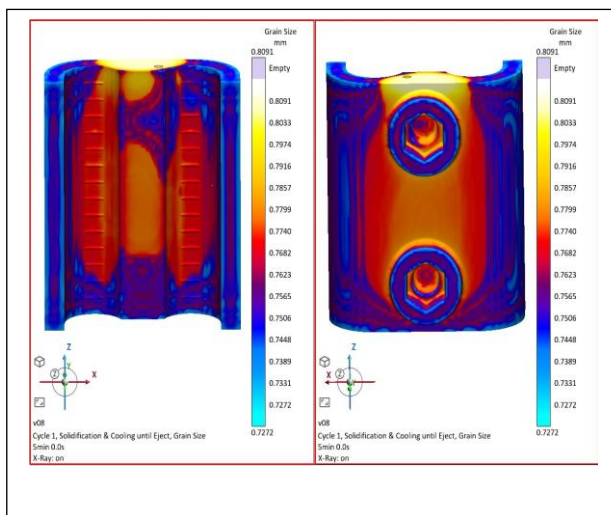
To get an idea of the inertia of the thermocouple used in our case, we have chosen a second control point in the casting /TC2, Fig.2 /, which has the same temperature as the first /TC1, Fig.2 / because it is symmetrical to the vertical plane of symmetry of the casting equipment. Fig. 16 shows the temperature curves at the control points.



It is clearly visible that there is a serious difference in the behavior of the two curves, especially at the beginning of the process, which means that this effect must be taken into account when interpreting the data from such measurements.

7. Prediction of structural and mechanical properties

One of the great advantages of computer simulation is that we can obtain realistic predictions of the structures and mechanical properties of the formed details without making laborious and expensive experimental measurements. Two such prediction results are presented in Fig. 17 and Fig. 18.



8. Conclusion

The authors hope that the above is a convincing illustration of the efficiency and expediency of using modern computer simulation in the optimization and improvement of foundry technologies. They are widely used both in the formation of new materials and alloys, and in the identification of parameters that are difficult to measure [5].

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Measurement comparison between 3D model of the promotional school windmill shaft and two different prototypes made on a 3D printer Ender 3 S1 Plus

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Abstract: Parents bought me a 3D printer three years ago. Many products have already been made on it, for friends and even a teacher who came up with the idea of making a promotional school windmill. A few of our classmates got together to cooperate on this project. Our task was to follow the demanding construction process, from planning to concepts, detailing, planning and production of technical documentation. In this article, I would like to present my work and the knowledge I have acquired through a school project: from conceptualization in the SolidWorks, to the production of a prototype on an Ender 3 S1 Plus machine. The 3D printer will produce the component with two different additive thermoplastic materials, PLA and PETG. If there is any significant difference in the accuracy of manufacturing the component with different materials will be shown by measuring on the Atos Q scanner.

Keywords: RAPID PROTOTYPING, ENDER 3 S1 PLUS, THERMPLASTICS, ATOS Q MEASUREMENT

1. Introduction

The first step for faster product development was computer-aided design (CAD) software system that makes engineers more productive. The product CAD data was so effective, that the system was expanded throughout the enterprise connecting everyone involved in designing and building products. Design review meetings and communication are critical to the success of any product development project.

For design engineering, rapid prototyping is a most powerful tool for conceptualization, form and fit review, functional analysis, and pattern generation. These applications are also relevant to the manufacturing process. Prototypes are often used to answer two types of questions: "Will it work?" and "How well does it meet the customer needs?" When used to answer such questions, prototypes serve as learning tools.

Result of two different rapid prototyping techniques is shown in Fig. 1.



Fig. 1 Result of Rapid Prototyping for learning and communication purpose. Exhaust and foot holder BMW motorcycle component made with DMLS (left side) and with CNC rapid prototyping technology (right side of Fig.) [1]

2. From idea to modeling of windmill components

One way to think about the development process is as the initial creation of a wide set of alternative product concepts and then the subsequent narrowing of alternatives and increasing specification of the product until the product can be reliably and repeatably produced by the production system.

The conceptual design phase includes the definition of the product architecture and the decomposition of the product into subsystems and components. The final assembly scheme for the production system is usually defined during this phase as well. The output of this phase usually includes a geometric layout of the product, a functional specification of each of the product's subsystems, and a preliminary process flow diagram for the final assembly process.

We all have ideas. And so, in our class, with the support of our teacher-mentor, we set ourselves a development challenge: to make a prototype of a windmill that would serve to promote the school. First, a concept was created, based on the sketch presented in Figure 2.

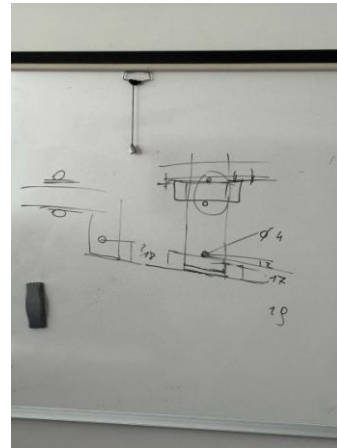


Fig. 2 Sketch of our final concept, made on a school blackboard.

Each of us students in the group took charge of one component of the assembled windmill. Each of us had to make a 3D model of the component in the SolidWorks program, draw a workshop drawing of the component and make a prototype. My mentor assigned me to make the component – the windmill shaft.

The Solidworks software we learned to use in our Technical school. SolidWorks is a software for solid modeling computer-aided design (CAD), computer-aided engineering (CAE). The complete software includes also tools for analyses and simulations, such as Finite Element Analysis, which supports modeling, design, and collaborative work. Figure 3 shows a 3D model of the shaft.

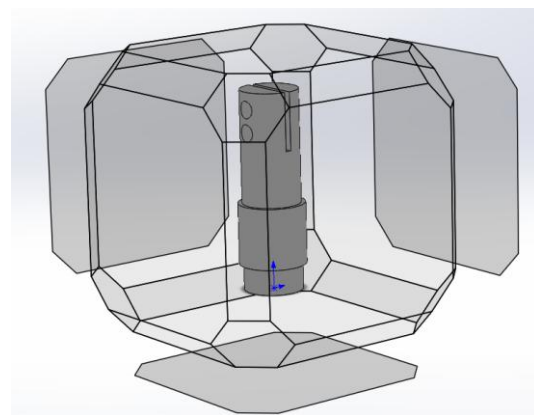


Fig. 3 3D model of windmill shaft in SolidWorks program..

The SolidWorks user base ranges from individuals to large corporations and covers a wide cross-section of manufacturing market segments. Its core industries include industrial equipment,

high-tech, life sciences, home and lifestyle, architecture, engineering, and construction. Figure 4 shows a drawing of the shaft.

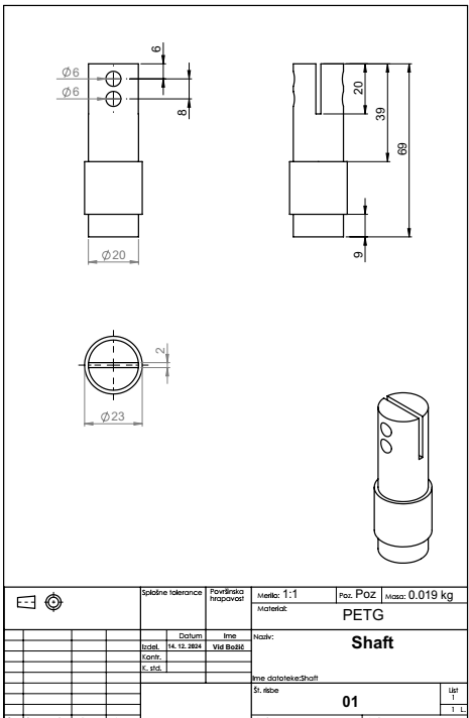


Fig. 4: Technical drawing of windmill shaft.

Rapid prototyping and 3D printer: Ender-3 S1 Plus

The next stage was the production of the shaft using rapid prototyping technology. If you own a 3D printer, software matters. That is why Cura is an open source slicing application for 3D printers. It was created by David Braam who was later employed by Ultimaker, a 3D printer manufacturing company, to maintain the software.

Ultimaker Cura is used by over one million users worldwide and handles 1.4 million print jobs per week. It is the preferred 3D printing software for Ultimaker 3D printers, but it can be used with other printers as well. It works by slicing the user’s model file into layers and generating a printer-specific g-code. Once finished, the g-code can be sent to the printer for the manufacture of the physical object. The open source software, compatible with most desktop 3D printers, can work with files in the most common 3D formats such as STL, OBJ, X3D, 3MF.

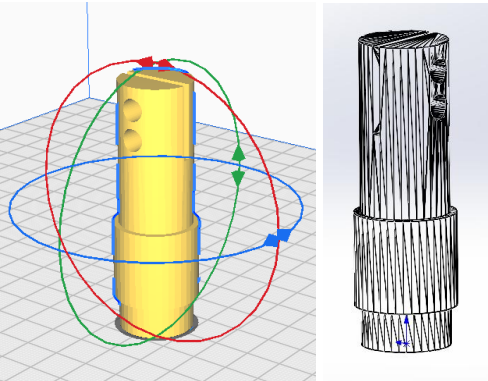


Fig. 5: 3D model in Ultimaker Cura program.

The Ender-3 S1 Plus resembles any number of open-frame 3D printers, including close to a dozen in Creality’s Ender-3 stable alone. The printer is made up of a handful of essential parts. The

base, measuring about 33 by 38 by 5 centimeters, houses the printer’s electronics, power supply, and ports (a USB-C port and an SD card slot).

The mentioned printer is suitable for beginners who are still learning about the 3D printing process. This is my first 3D printer, and while it’s very easy to set up and start printing, it does take some time and research for the user to get the hang of it. Most prints took anywhere from 1 to 5 hours or even more depending on the size and level of quality detail.

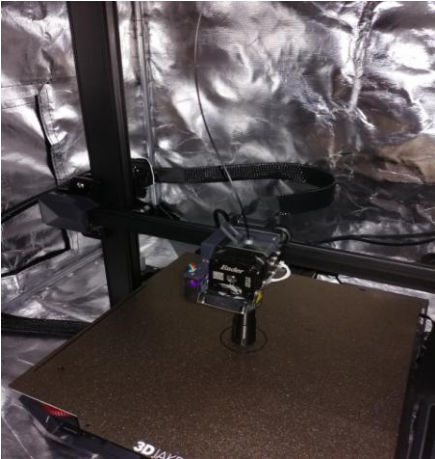


Fig. 6: My first 3D printer.

- Basic printer Ender-3 S1 Plus specifications:
- Print size: 300 x 300 x 300 mm
 - Print speed: 100 mm/s
 - Filament: 1.75 mm PLA, PETG
 - Working mode: online or SD offline
 - File format: STL, OBJ, G-code
 - Net Weight: 6.7 kg
 - Layer Thickness: 0.1-0.4 mm
 - Nozzle Diameter: 0.4 mm
 - Printing accuracy: ± 0.1 mm
 - Nozzle temperature: 255 °C
 - Heater temperature: 110 °C

The printer, as I mentioned, works with a process called fused deposition modeling (FDM). It works as follows:

- Filament: The printing material is available in spools of thin plastic filament, similar to a thick string.
- Heating and Extruding: The printer heats the filament until it melts. The molten plastic is then extruded through a nozzle.
- Layer by layer: The nozzle moves precisely, depositing layer by layer onto the build platform, slowly building a 3D object.

Filament PLA and PETG

Many materials are used as filament on 3D printer machines. I decided to compare two different materials: PLA and PETG.

Thermoplastic polymers such as PLA (polylactide), PETG (glycol-modified polyethylene terephthalate) and other types of plastics are usually obtained from natural sources such as corn, sugar cane, potatoes or petroleum, depending on the base material component. PLA is the most widely used plastic filament material in FDM 3D printing due to its low melting point, high strength, low thermal expansion and good layer adhesion, although it has poor heat resistance unless annealed.

PETG delivers significant chemical resistance, durability, and formability for manufacturing. PETG has good strength and durability, as well as being more impact resistant and better suited to higher temperatures. It is also widely used in 3D printing since the glycol prevents the problems such as overheating and becoming cloudy and fragile. With good adhesion between layers, minimal

deformation during printing, good resistance in low temperature environments, chemical resistance against bases and acids, PETG is becoming the favored material for 3D printing.

I got the best visual results from both PLA and PETG prototypes after a week of testing, varying the printing parameters. The best PLA prototype was made with the following parameters:

Print nozzle temperature: 200 °C

I used this temperature for final PLA prototype. This temperature enables optimal melting of the material, as it is the highest limit that still ensures good adhesion and prevents problems such as clogging of the nozzle and uneven extrusion of the material.

Printing substrate temperature: 70 °C

This temperature is recommended for materials such as PLA due to the good adhesion of the first layer. The stability of the first layer is critical to a successful print finish, as irregularities in the initial layers can cause problems throughout the process.

Print speed: 50 mm/s

Reducing speed did increase the print time, but I expected the quality of the final product to be much better, as printing slower reduces defects such as uneven surfaces, excessive layer overlap or edge irregularities.

Speed of filling (Infill): 50 mm/s

This speed was chosen as a compromise between the printing speed of the inner layers and the quality of the structure.

External wall printing speed: 25 mm/s

To print the outer walls, I reduced the speed too, as I assumed that this would improve the quality of the outer surfaces and increase the smoothness of the edges.

Extruder movement speed: 100 mm/s

I significantly reduced the speed of the extruder movement to 100 mm/s as I assumed this would improve the quality of the model.

Response time: 20 mm/s

For the response time, I reduced the value to 20 mm/s to increase the accuracy when applying the material, especially during fast movements of the print head.

The best PETG prototype was made with the following parameters:

Print nozzle temperature: 235 °C

Printing substrate temperature: 85 °C

Print speed: 50 mm/s

Speed of filling (Infill): 50 mm/s

External wall printing speed: 25 mm/s

Extruder movement speed: 100 mm/s

Response time: 20 mm/s

Measurement of two prototypes and comparison with 3D model

The company Merilna tehnika s.p. has its laboratory in Unec, close to Postojna. The director of the company is Mr. Samo Strle. Samo has many years of experience in the field of measurement and quality. He just had some time to show me the measuring instruments and machines that he has in his premises. I learned a lot about contourograph, roughness meter, altimeter, optical meter and two 3D measuring machines: Zeiss and Dea.

We decided to measure both prototypes and compare them with the 3D model on the ATOS Q 3D scanner. ATOS Q is an industrial 3D scanner that can be used in a variety of applications due to its wide range of measuring areas. In addition to incorporating many of the features of our high-end automated systems, ATOS Q's lightweight design makes it easy to transport from one location to another, providing users with added convenience and flexibility. One of the major advantages is that with ATOS, the entire part's surface is captured. ATOS blue light 3D scanners help alleviate this challenge by picking up every visible area. This results in incredibly detailed, accurate data full of millions of XYZ points that create a geometric part in minutes.

These scanners register the surface information of an object with the help of reflected light from its surface. 3D scanning spray

is an evaporating solution that works to matt out reflective and transparent surfaces. With a dedicated spray, we prepared both prototypes for measurement, to ensure much more efficient 3D scanning and complete scan results. Nejc, an employee of company, introduced me to the machine and measurement with ATOS Q. Figure 7 shows the measurement process - scanning the PLA prototype and blue light for high-quality measurement capture.

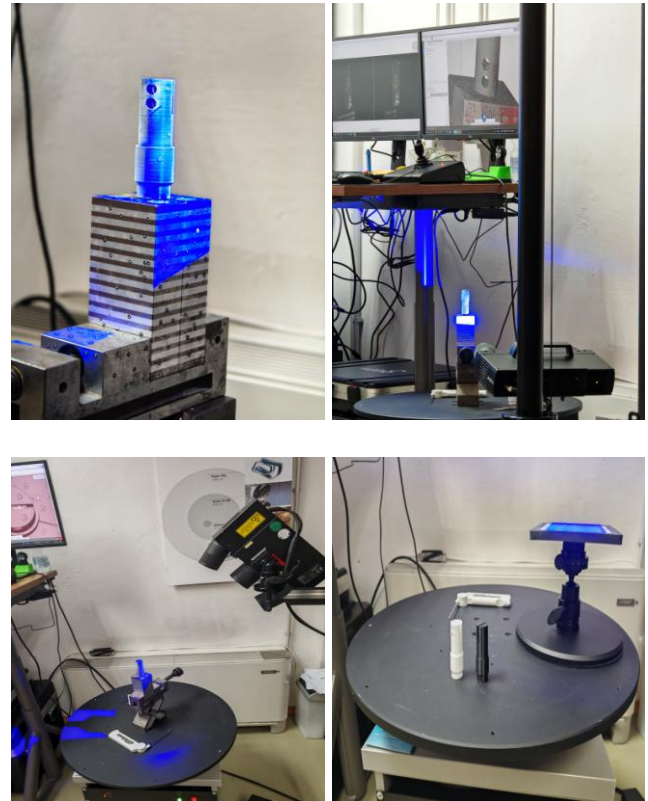
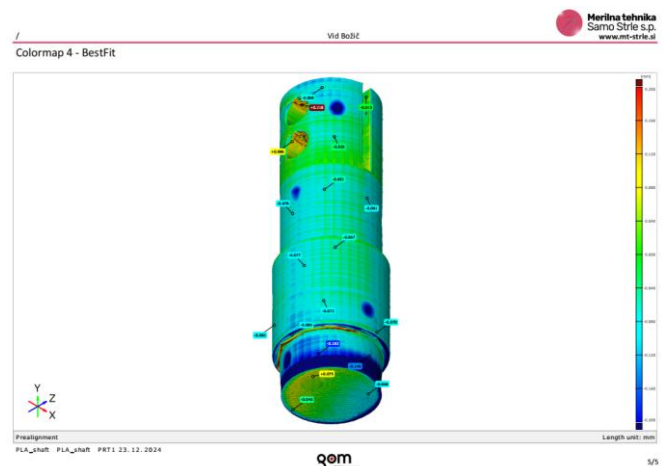


Fig. 7: Measurement of PLA prototype with ATOS Q [2].

GOM Inspect is the comprehensive, user-centric 3D metrology software that tackles every challenge. It has one user interface that handles the entire workflow, which covers complex inspection tasks in different applications. With GOM [3] inspect software we can easily visualize surface inspections and generate reports of measuring results.

Figure 8 presents the results of measurements of the PLA prototype relative to the 3D model. A color gradient towards dark blue means a deviation of the prototype measurement relative to the 3D model in a negative direction, while a color gradient towards dark red means a deviation of the prototype measurement relative to the 3D model in a positive direction.



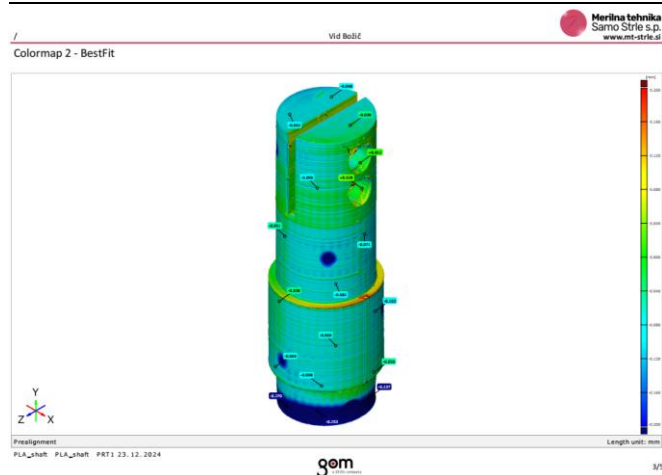


Fig. 8: Results of measurement of PLA prototype with GOM software.

The measurements surprised me greatly, as the results of the PLA prototype are extremely good: the largest deviation from the 3D model was at the shaft end: - 0.270 mm and in the upper hole: + 0.218 mm.

Figure 9 presents the results of measurements of the PETG prototype relative to the 3D model.

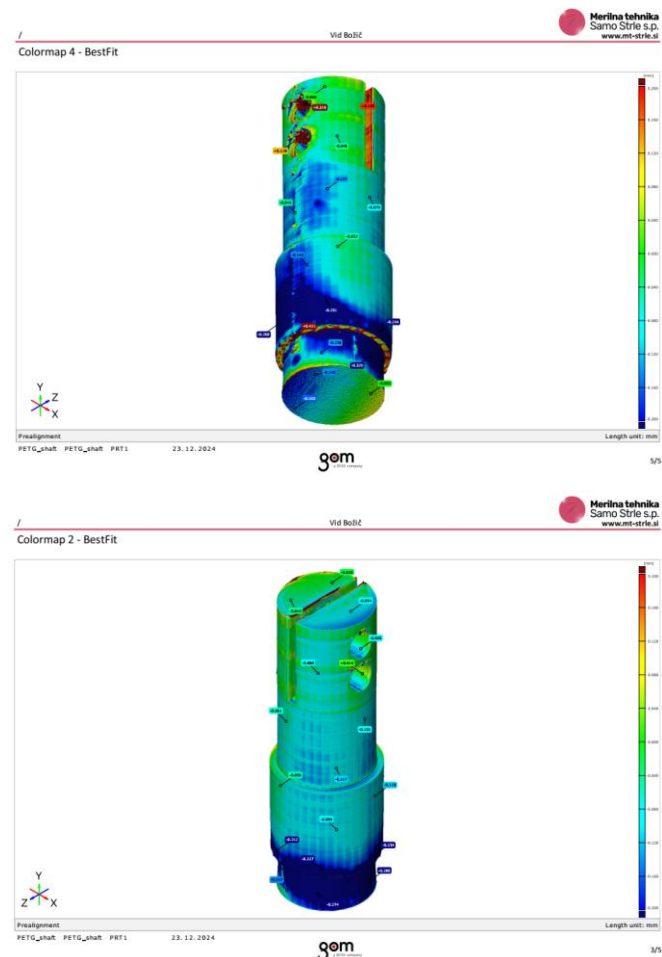


Fig. 9: Results of measurement of PETG prototype with GOM software.

A slightly larger deviation from the 3D model was measured in the PETG prototype. The largest deviation from the 3D model was at the shaft end: - 0.325 mm and in the upper hole: + 0.310 mm.

A comparison between the two prototypes is shown in the figure 10. The difference between them is greatest on the underside of the shaft and ranges from -0.405 mm to + 0.223 mm.

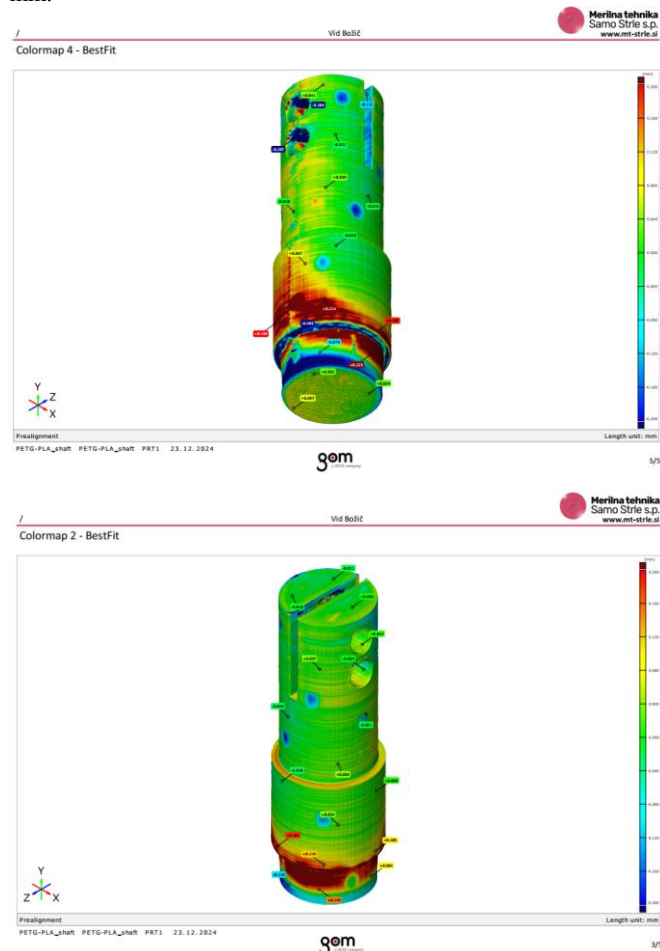


Fig. 10: Comparison results of measurement of PLA and PETG prototype.

Conclusions

Our project team is almost at the end of its concept. We have to combine, assemble all the prototype components into the final assembly and test our first windmill prototype on the planned object. I am satisfied with my work, acquired knowledge and experience. I used my previous knowledge in the field of 3D modeling and rapid prototyping technology. The results after measuring the two first prototypes surprised me. I expected greater deviations from the 3D model. We can achieve good results with a relatively cheap printer. The results of the prototypes could possibly be further improved by optimizing the settings of the machine's 3D printer. However, this task should wait for me, if the team agree on making a real functional prototype.

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Approach of Artificial Intelligence to accelerate FEM simulations

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Abstract: FEM Simulation is widely used in engineering practice. The use in small and medium-sized companies is partially limited due to the high workload and the time required for the simulation calculations. In recent years, the use of Artificial Intelligence (AI) has been increasingly adopted, emerging as an exciting and promising area of research. This article presents a methodology for the implementation of artificial intelligence in the simulation process of a part and an assembly. This methodology includes phases to integrate AI into the CAD model preparation process, as well as the definition of contact conditions, fixtured reactions, and external forces. Artificial intelligence can process a large volume of previous calculations, allowing it to analyse and automate these preparation steps AND thus increase the accuracy of simulations.

KEYWORDS: ARTIFICIAL INTELLIGENCE, FEA, MACHINE LEARNING, DEEP LEARNING

1. Introduction

Artificial intelligence (AI) has penetrated many areas of industry and society. It has recently become increasingly clear that without artificial intelligence, companies will find it relatively difficult to manufacture competitively in the future.

Machine learning is a branch of artificial intelligence (AI) that enables computers to learn from experience and recognize patterns in data without being explicitly programmed. Algorithms and statistical models are used to analyze data, identify patterns and make predictions or decisions. There are different types of machine learning, with two of the most prominent approaches being supervised learning and unsupervised learning.

The Finite Element Analysis (FEA) is used to predict forces, deformations, and stresses in engineering systems. To achieve this, designs are first created in CAD. This model is then meshed, material properties are assigned, and the acting forces are applied. The decision regarding the model's level of detail and meshing (idealization) must be purpose-driven, as such calculations typically take several days to complete. Computation time increases exponentially with the level of detail and mesh density. If the level of detail is too coarse, no conclusions or incorrect conclusions can be drawn from the analysis results. Conversely, if the level of detail is too fine, the efficiency of the analysis decreases.

Small and medium-sized enterprises (SMEs) make up 99% of all companies in the EU [1]. With the help of various action programs, the competitiveness of SMEs is to be promoted through research and development and access to funding is to be simplified for the relevant companies. The strategies for improving conditions for SMEs also take digitalization into account. In addition, new considerations for the use of AI for process optimization have been initiated.

Artificial intelligence in design offers enormous potential in mechanical engineering to drive productivity, quality and innovation. By integrating AI technologies, companies can create more efficient production processes that meet today's dynamic market conditions. Nevertheless, there are also challenges to overcome, particularly with regard to data security, system integration and the shortage of skilled workers. Close collaboration between researchers, engineers, manufacturers and regulators is crucial to fully realize the benefits of artificial intelligence in mechanical engineering. [1].

AI has a special application in small and medium-sized companies, where there are many customers with individual needs. These customer needs can be optimized with AI-based programs. While conventional CAD systems are often geared towards general standards, AI-supported systems can be customized to automatically take specific requirements into account.

By solving the task, the employees can be strongly supported, as the modelling and idealization decisions require a lot of know-how and are made by approximation. This also results in the benefit of an accelerated development process.

2. Phases of the implementation of artificial intelligence

To perfect the specific application of AI in mechanical engineering, certain steps must be taken. First, it is important to identify areas in the production process where the use of artificial intelligence makes sense. However, it is important to evaluate the potential benefits and associated challenges of each application. The possible advantages, such as improved product quality, reduced downtime or increased efficiency, should be considered.

The customer is the one who can best define the current situation and the desired improvements in the work process. On this basis, AI specialists can make suggestions for achieving the customer's goals by making the best use of the possibilities offered by artificial intelligence. This collaboration ensures that the implemented solution is not only functional but also meets the specific needs and requirements of the company.

The introduction of artificial intelligence in a company takes place in certain phases. These include the initial analysis of processes to determine where AI can best be implemented. They also include the collection of the data required to train the system and finally the actual implementation of artificial intelligence. Diagram 1 defines the sequence and key phases of AI implementation.

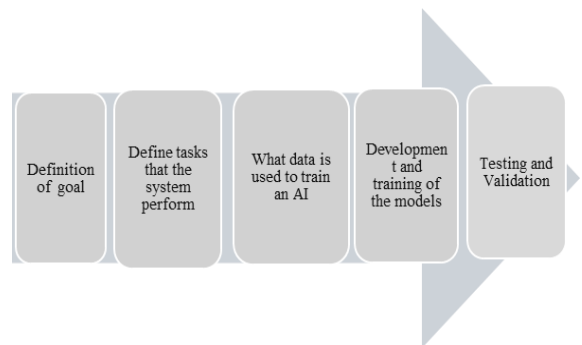


Diagram 1 Phases of the implementation of artificial intelligence

The process begins with an analysis of the situation in the company and the definition of focal points in the work process. These form the goals that we want to achieve with the implementation of artificial intelligence. It is important to evaluate the potential benefits and associated challenges of each application. Particular attention is paid to the following benefits: improved product quality, reduced downtime or increased efficiency.

When implementing artificial intelligence in a process, it is important to clearly define the specific tasks that the system will perform. This ensures that the development is specifically geared towards achieving the set goal.

Before formulating specific tasks, it is necessary to analyze the current workflows. This includes mapping the workflows and identifying key stages; determining manual and repetitive actions that

can be automated; analyzing weak points and frequent errors that can be eliminated with AI; examining complex processes that require analysis of large data volumes. Once the processes have been analyzed, specific tasks that the system needs to perform can be formulated.

The appropriate system is selected on the basis of these tasks. The decision is whether to use a system that merely automates the processes through a defined algorithm or whether to apply artificial intelligence where the system is able to make decisions on its own. Once specific requirements of the application are defined, suitable AI models or solutions should be developed or acquired. The selection of the right AI models is crucial to achieve optimal results.

Machine learning is a branch of artificial intelligence that enables computers to learn from experience and recognize patterns in data without being explicitly programmed. Algorithms and statistical models are used to analyze data, identify patterns and make predictions or decisions.

The implementation of artificial intelligence is possible if data and information are available that provide the necessary basis for evaluating the process that is to be optimized. This data forms the basis for analysis and processing in order to obtain meaningful information. If artificial intelligence is to be used in mechanical engineering, it is important to prepare the data in such a way that it is suitable for analysis.

Obtaining the data required for artificial intelligence and processing it are the next phases in the implementation process of artificial intelligence. When collecting data, it is crucial to cover all possible cases of the situation under investigation. The quality of the data used to train a model determines the accuracy of its performance.

The use of artificial intelligence requires access to large amounts of data in order to train meaningful models and make accurate predictions. Companies must ensure that their data is adequately protected during collection, storage and processing.

The final phase is related to the development and training of artificial intelligence models. This is a process in which algorithms are created and trained to perform tasks related to data processing, decision-making, or pattern recognition. The main goal is to build intelligent systems that can "learn" from data and perform specific tasks in a manner similar to human intelligence.

3. Methodology in FEM Analyses and defining the goal we want to solve with AI

In modern digital design, the process begins with the creation of a CAD model in a CAD system, which represents a precise and detailed three-dimensional digital model of the structure. Subsequently, an FEM (Finite Element Method) analysis is performed using a CAE (Computer-Aided Engineering) system, which is an important step in verifying the structure under various loads.

During the design and development of machines and components, a strength analysis is carried out to determine stresses and deformations. This makes it possible to check whether the developed design can withstand the loads to which it will be subjected and how it will behave under different load conditions. These calculations and analyses are of crucial importance for the safety, efficiency and durability of the respective mechanisms and components.

FEM simulation can also be used to analyze structures that are not clearly defined. This simulation is considerably faster compared to conventional methods for calculating loads. Therefore, companies that design various components and systems are recommended to integrate such software into their work. Depending on the scope of the analysis, the simulation can be divided into two main categories:

- Simulation for strength analysis of a component / part and,
- Simulation for strength analysis of an assembly.

This classification enables more precise planning and application of the appropriate analysis methods, depending on the specific engineering problem.

FEM simulation uses a part or assembly created in a CAD system. In order to proceed with the simulation, preparation of the model and the assembled unit is required. This preparation can be roughly divided into two parts:

- Geometric refinement of the model – including simplification and optimization of the geometry, removal of insignificant elements, and adding additional/auxiliary elements.
- The definition of contact conditions fixtured reactions, and external forces necessary for calculating stress and deformations.

This preparatory work requires considerable experience and knowledge as it determines the accuracy and efficiency of the subsequent calculations. The experience is based on information from previous calculations as well as knowledge from disciplines such as material strength. In addition, the experience of a long-time employee with practical knowledge of the behavior of components and assemblies can be extremely valuable in defining the boundary conditions correctly.

The use of artificial intelligence in this process makes sense for two main reasons:

- AI can process a large volume of previous calculations and determine the most efficient method for model preparation.
- Based on the collected information, AI can automate the refining of the CAD model, as well as the definition of fixtured reactions, contact conditions, and external loads.

The goal being pursued is the introduction of AI to support the process of preparing the CAD model and defining the support reactions, reactive connections and forces. The implementation of artificial intelligence in this process would lead to a significant reduction in preparation time and an increase in the accuracy of the simulations.

4. Definition of tasks that AI must solve in the CAD model refining process and the process of defining contact conditions and fixtured reactions

This section examines the process of preparing CAD models and assembly, as well as the preparatory actions the author has encountered throughout their long-standing practice. Based on this, the tasks that can be solved with the help of artificial intelligence are defined.

It is shown how the integration of AI in simulation of parts and assembly facilitates and optimizes the corresponding operations. The introduction of AI allows the automation of repetitive tasks, reducing preparation time and increasing the accuracy of results, which significantly improves the efficiency of the entire FEM simulation process.

The analysis of operations makes it possible to identify the specific actions carried out at each stage. Based on this, the tasks that artificial intelligence must perform are formulated. Additionally, the ways in which AI implementation can facilitate the respective operations will be presented.

4.1. Definition of tasks that AI must solve in the Part Simulation

Refining the CAD model before initiating the FEM simulation is a crucial step in ensuring both the accuracy and efficiency of the calculations. This process involves simplifying geometry by removing elements that do not significantly impact the simulation results, such as small geometric features or structural details that are not subjected to considerable loads.

The fastening elements in an assembly can be defined through contact conditions or replaced with simplified models. When using the second approach, we reduce the computational time of the simulation. First, we need to remove the fastening elements from the assembled unit in the CAD system, and then, in the simulation software, introduce simplified models. These are routine tasks that usually take a lot of technological time, and defining the simplified models requires a competent thought process from the operator. Therefore, the introduction of artificial intelligence is appropriate to assist in this process.

AI can automate the removal of fastening elements in CAD models and generate simplified models for simulations, thus saving time and reducing the complexity of the task. AI could also assist in determining the best approximation for simplified models based on the specific characteristics of the assembly, further optimizing the process. Fig.5

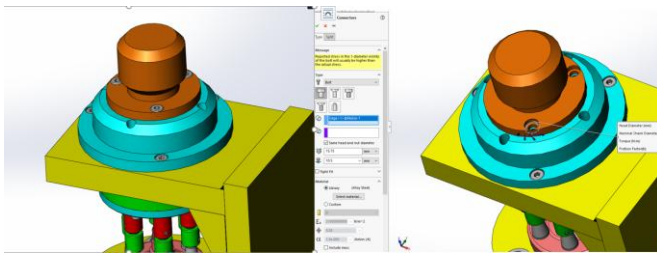


Fig. 5 Definition of Fastening Elements by Connectors (Bolts) in FEM Simulation

Table 2 presents the tasks involved in implementing AI for preparing the CAD assembly for simulation. For each task, the advantages of using artificial intelligence are outlined.

№	Tasks	Description
1	Definition of fastening elements	In FEM analysis, bolts can be replaced with simplified models, such as Bolt (bolt connection) or Check Interaction (interaction check).
2	Defining component contact	Determination of contact areas between components, including type of contact ("frictional", "bonded" or "free").
3	Defining of mesh	An incorrect mesh can lead to inaccurate results. optimization of mesh quality and size to achieve a balance between accuracy and efficiency in meshing
4	Interference Detection	Ensures that simulations or further analysis are accurate since interference could affect the results.

Table 2: Tasks for Refining the assembly for FEM Simulation

5. Data and information needed for training AI by FEM Simulation

The implementation of artificial intelligence in an organization requires careful data management, as this is the basis for its efficient functioning. With AI, the focus is on creating algorithms and models that enable computers to learn from data. Instead of being explicitly programmed for each task, the algorithms are trained through examples and experience, allowing them to improve over time.

The first step in data collection and integration is to identify relevant data sources and merge them. For FEM simulations, the classic approach involves an expert performing the analysis based on their experience, using manuals, technical literature and standards

such as DIN. This process requires considerable effort and time, which limits the possibilities for faster and more scalable implementation of artificial intelligence solutions.

In order for artificial intelligence to utilize the same knowledge available in specialized literature, a second system – the autoencoder – is introduced during training. An autoencoder is a type of artificial neural network used for unsupervised learning. It compresses the input data into a latent-space representation and then reconstructs the output, allowing the system to learn patterns and features from the data.

Training artificial intelligence to simulate deformations requires numerous results from simulations of components and assemblies that have already been carried out and are specific to the components used in a company. However, companies often do not have enough digitized data. The digitization of data and information from previous simulations is therefore of great importance for the introduction of AI. For AI models to be accurate and reliable, the data must be consistent, well-organized and of high quality. The standardization of data formats and structures is crucial to avoid errors during processing.

6. Conclusiones

The problem presented in the report aims to significantly expand the scope and efficiency of artificial intelligence. The proposed analysis and approach can be used to tackle a wide range of tasks and develop a fully functional model. The results can be systematized as follows:

1. A systematic methodology for the implementation of artificial intelligence in the simulation process has been proposed.
2. An analysis of the preliminary refinement of the model and the assembled unit before FEM simulation has been conducted, highlighting the challenges where the author identifies advantages in the implementation of AI.
3. Definition of tasks that AI must solve in the CAD Part refining process and the process of defining contact conditions and fixtured reactions.
4. Definition of tasks that AI must solve in the CAD Assembly refining process and the process of defining contact conditions and fixtured reactions.
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The influence of vibrations on the friction of removable electrical connectors during operation, and the causes of malfunctions in electrical contacts

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Abstract: This paper discusses contact interaction under mechanical stress and how vibrations change the physical properties of conductor materials, which affects their ability to provide reliable contact. It also analyzes the causes of contact failures, which allows us to better understand the mechanisms that lead to problems in the operation of electrical connectors. These factors can lead to serious consequences, including breakage and reduced reliability of connectors, and also considers the possibilities of improving the characteristics of electrical contacts at the stage of their creation, using various technologies for applying and creating an approximately ideal molecular structure of the contact surface of detachable electrical connectors, with an increase in the surface layer of the contact and a decrease in the coefficient of friction.

KEY WORDS: VIBRATION EFFECTS, TRIBOLOGY, TRIBOTECHNICS, ELECTRICAL CONTACTS, ELECTRICAL CONNECTORS.

Introduction

Modern electrical connectors play a key role in ensuring the reliability and efficiency of electrical and electronic systems, finding application in the automotive industry, aviation, telecommunications and consumer electronics. Detachable electrical connectors, in particular, allow for quick connection and disconnection of electrical circuits, which makes them indispensable in conditions of frequent replacement or maintenance of equipment. However, there are many factors that negatively affect their performance and durability, including vibrations that occur during operation. The relevance of this study is due to the fact that vibration effects can cause serious problems, such as deterioration of contact interaction, increased resistance, overheating and failure of connectors. Under conditions of constant mechanical impacts, it is important to understand how vibrations affect friction in detachable electrical connectors and what measures can be taken to minimize the associated risks. The article analyzes the effect of vibrations on the friction of connected contacts, identifies various types of vibrations and their sources that affect the quality of an electrical connection. Contact interaction under mechanical stress and how vibrations change the physical properties of conductor materials, which affects their ability to provide reliable contact, are considered. An analysis of the causes of contact failures was also carried out, which allows for a better understanding of the mechanisms that lead to problems in the operation of electrical connectors.

Results and discussion

Analysis of the Effect of Vibration on Friction of Connected Contacts. Vibrations that occur during the operation of electrical and electronic systems significantly affect the condition and reliability of removable electrical connectors. These mechanical vibrations can be caused by the operation of engines or external conditions, for example, in vehicles and industry. Understanding the effect of vibrations on friction in connected contacts is critical to ensuring their reliability and durability. Contact interaction in connectors is based on microscopic irregularities in the surfaces of the materials from which the electrodes are made. When connected, the contacts deform, increasing the contact area and reducing electrical resistance. However, vibrations can change the mechanical characteristics of connectors, causing microcracks on the contact surfaces, which leads to increased friction and changes in pressure distribution. At high vibration levels, a change in the contact structure occurs [1]. Vibrations can loosen connected elements, increasing wear and aging of materials, which makes the interaction between the contacts less predictable and can lead to a loss of sealing of the connection. Contaminants from mechanical impacts also reduce conductivity, and their accumulation under the influence of vibration aggravates the situation, creating difficulties in establishing stable contact. The durability of connectors can be affected by resonance phenomena that occur at certain vibration frequencies. If the vibration frequency coincides with the natural frequencies of the system, this can lead to significant vibrations in the connectors, causing additional energy that contributes to the formation of microscopic cracks and other damage.

Types of vibrations and their sources. Vibrations caused by mechanical influences can be classified into several types, each with its own sources and level of impact on electrical connectors. Understanding these types of vibrations, their amplitude, frequency and nature of impact is important for assessing the behavior and durability of connections in electrical systems. The first type is periodic vibrations caused by the operation of rotating machines such as generators, motors and compressors[2]. These vibrations have a fixed frequency corresponding to the rotation speed of mechanical units and can be transmitted through the structures on which the devices are installed. Periodic vibrations can cause resonance effects that amplify the vibrations and increase the loads on electrical connectors. The second type is random vibrations caused by various external factors such as impacts, jolts or atmospheric phenomena such as strong wind. These vibrations are characterized by amplitude and frequency that vary over time, making them unstable and difficult to predict. This increases the likelihood of damage to electrical contacts due to random loads. The third type is impact vibrations, caused by sudden dynamic impacts that are usually short in duration but high in intensity. Examples include equipment falling or sudden stops and starts of machines. Impact vibrations can cause instantaneous deformations in conductor and connector materials, which can lead to serious damage and reduced reliability of connections.

Contact interaction under mechanical stress. When using pluggable electrical connectors, it is important to consider the mechanical effects of vibrations on contacts, which may be caused by motors, adjacent machinery, vehicles or natural phenomena. These vibrations can degrade the interaction between contacts and reduce the reliability of the connections. Vibrations affect the mechanical properties of materials, causing temporary changes in the shape and parameters of contacts. Depending on the structure of the materials, this can lead to compaction or wear, which reduces the pressure on the contact surfaces and, therefore, the effectiveness of the connection [3]. Cyclic loads can also cause material fatigue and the formation of microcracks, which degrade the quality of contact and create risks for the reliability of the connection. These changes can affect both the surface and the internal layers of the material. In addition, vibrations can displace contacts, changing their position and reducing the effectiveness of the connection. Insufficient contact can lead to intermittent current, overheating and deterioration of the electrical properties. It is important to consider all these factors to ensure the reliability of electrical connectors.

Physical properties of conductor materials. Conductor materials have unique physical properties that affect their behavior under mechanical influences such as vibration. Key characteristics are electrical conductivity, mechanical strength, and corrosion resistance, which are important for understanding the effects of vibration loads on electrical connectors. The electrical conductivity of copper and aluminum depends on their atomic structure and free electron density. Vibrations can cause oscillations that lead to shifts in the crystal lattice and deterioration of contact characteristics. At high frequencies, fatigue cracks are possible, which reduces the reliability of connections [4]. Mechanical strength is also critical,

since vibration loads can cause deformations and accumulation of fatigue damage, which leads to a decrease in the strength of connectors and their failure. Low strength can contribute to micropressures and open pathways for corrosion. Corrosion resistance is important for the durability of connections, especially in aggressive environments where vibrations can aggravate corrosion processes. Taking all these factors into account is necessary to ensure the reliability of electrical connectors under mechanical influences.

Contact Failure Root Analysis . Vibration-induced electrical contact failures are a complex issue affecting the design, operation, and monitoring of removable electrical connectors. Understanding the causes of such failures requires consideration of many factors, including the mechanical properties of materials, the nature of the contact interaction, and the types of vibration. One of the main causes of failures is insufficient mechanical strength of connector materials. Vibration loads can cause uneven stress distribution, leading to fatigue failure. Different vibration frequencies can cause different vibration amplitudes, which changes the properties of materials and can lead to cracks or wear if permissible stress limits are exceeded [5]. The nature of the contact interaction between conductors is also important. Parameters such as the amount of pressure, the geometry of the contact surfaces, and the presence of contaminants affect the possibility of failure. Under constant vibration conditions, the contact may oscillate, which changes its quality. Incorrect angle of inclination, insufficient rigidity of the structure or inadequate clamping can lead to divergence of connections, increased resistance and overheating of contacts. The condition of the contact surfaces is also critical. Oxide and other coatings can form on electrical contacts, affecting their characteristics. Mechanical loads during vibration can cause a flickering contact, which hinders the transmission of electric current and leads to additional losses.

Methods for Minimizing Operational Risks . Effective risk management for removable electrical connectors requires an integrated approach to minimizing the impact of vibration on friction and contact reliability. It is important to consider many factors during the design, installation, and operation stages. The first step in reducing risks is careful selection of materials. Using conductors with high mechanical and electrical properties, such as alloys with high compressive strength and wear resistance, can reduce the likelihood of failure. The friction coefficients of different materials should also be taken into account to optimize contacting [6]. The manufacturing processes of connectors are also important. Controlling tolerances and surface roughness of contacts during production can significantly affect the amount of friction. Laser processing and special coatings can improve material compatibility and increase the service life of connectors. Also, the use of galvanic methods of applying and processing electrical contacts is usually the most effective and durable, with the least likelihood of failure of contact connectors along with other methods of processing and creating electrical contacts. Installation and operation must take into account possible mechanical effects. The location of connectors, vibration of surrounding equipment, and the level of attachment are critical. Engineers must design wiring diagrams that ensure secure attachment and reduce connector movement. Dynamic testing to standards can help identify vulnerabilities at the design stage. Regular maintenance also plays an important role in preventing failures. Connector condition checks can help identify and resolve problems before they occur.

Prospects for Research in Connector Reliability . Prospects for research in connector reliability open up new opportunities for improving technologies and increasing the efficiency of electrical systems. The relevance of the topic is due to the growing requirements for the reliability and durability of connectors under harsh operating conditions, where vibrations and mechanical loads can significantly affect their performance. One of the areas of research is an in-depth understanding of the processes occurring in contacts under the influence of vibrations. Current research shows that vibrations cause dynamic changes in contact zones, which affects normal and friction contact forces. To create

more accurate models predicting the behavior of connectors under various conditions, it is necessary to combine theoretical and experimental approaches [7]. Studying the interaction of materials from which electrical connectors are made is also important for improving reliability. The development of new alloys with improved physical and chemical properties can reduce the likelihood of oxidation and destruction of contact surfaces. The use of modern technologies, galvanic treatment of special coatings, allows us to create connectors that are more resistant to mechanical wear and vibration. Implementing active monitoring of the condition of connections during operation can significantly improve their behavior. Using sensors to monitor vibration and temperature allows early detection of potential faults and the implementation of the concept of predictive maintenance, which increases reliability and reduces replacement costs. Attention should also be paid to the study of model figures on which the mechanical characteristics of connectors are based. Modern numerical modeling methods can help in this process, providing a deeper understanding of the behavior of connectors under various operating conditions.

Conclusion

The study found that vibration effects from the operation of various mechanisms and equipment significantly affect the contact interaction in electrical connectors. These effects can change the friction coefficient, which worsens the quality of electrical contact and can lead to malfunctions. Vibrations can be both periodic and random, with varying intensity depending on the operating conditions. Contact interaction under mechanical stress was also analyzed in detail. The initial contact pressure and the mass of the moving parts play a key role in the reliability of connectors. Insufficient pressure can cause microcracks and increase contact resistance, which leads to overheating and other problems. Uneven achievement of the working pressure can negatively affect the durability of connectors, causing uneven load distribution and deterioration in contact quality. The physical properties of conductor materials are also significant. Different materials have different mechanical and electrical properties, which affects their behavior under vibration loads. In the course of the work, the main causes of malfunctions in electrical contacts were identified, including overheating, insufficient pressure and abrupt achievement of the working pressure. These factors can lead to serious consequences, including breakdowns and reduced reliability of connectors, and the possibilities of improving the characteristics of electrical contacts at the stage of their creation are considered. In this direction, the most promising is the study of applying galvanic coatings to electrical connectors, using various technologies for applying and creating an approximately ideal molecular structure of the contact surface of removable electrical connectors, with an increase in the surface layer of the contact and a decrease in the friction coefficient.

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Mooring system for fuel oil unloading

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Abstract: The aim of this paper is the calculation of the PLEM (Pipeline End Manifold) System of the 18" dia. subsea pipeline, according to the International Codes and Standards. A Geotechnical survey was carried out to define soil – structure interaction, effect of scour, soil stratigraphy and susceptibility to liquefaction. Then were defined the acting forces on PLEM, pipeline and submarine hoses taking in consideration the total buoyancy as well. The calculation was carried out for filled pipeline (in service), as well as for empty pipeline. The ground conditions along the sealines and at the location of the PLEM have been investigated by a dedicated additional site investigation, consisting of 12 no. vibrocore samplings and the related laboratory testing. The stability of PLEM was checked for several load combination. This calculation it was very important for environmental reason and shows that the structure meet the requirements of the project.

KEY-WORDS: PLEM, SUBMARINE PIPELINE, TOTAL BUOYANCY, LIMIT EQUILIBRIUM

1. Introduction

A fuel oil (FO) line 3.5 km long and ND 18" was designed and installed offshore in Vlora Bay to transfer fuel from vessels to onshore FO tanks located near the Combined Cycle Power Plant of Vlora. The plant should operate with fuel oil D2, because the natural gas is not available in Albania. A Pipeline End Manifold (PLEM) was installed, and the Mooring system capacity was to accommodate 15 T oil tanker, the quantity needed for one month operation of the plant in base load.

Due to environment reasons the offshore structures and pipelines should be reliable, despite of the high price of the erection.

Albania has a short history in the field of offshore pipeline constructions. Consequently, this paper is a new approach towards finding satisfactory solutions.

Investigations consisted of site activities and laboratory tests and the results are reported here below. The data collected provide a strong tool to compare with geotechnical analysis of structures and to calibrate the used models.

The behavior of the structure was checked under lateral loads to verify its reliability [1]. Moreover, the analysis of stiffness and soil–structure interaction were carried out. The analysis that provides information on soil composition is necessary to assess the susceptibility to liquefaction of natural on-site shallow soils.

2. Design data

Pipeline [2]	
Manufacture process	SAW /ERW
Nominal Diameter	18" = 457.20 mm
Inner diameter	438.16
mm Inner section	1507 cm ²
Assumed Wall Thickness	0.375" = 9.52 mm
Material	API 5L — X52
SMYS	52000 psi = 358.6 Mpa = 3655 kg/cm ²
Max Working pressure	15 barg (including surge)
Flow rate (max)	1500 m ³ /h
Max water depth	– 12.00 m
Max operative pressure	15 barg

Fluid [3]	
Type	Fuel Oil
Density (40°C)	856 kg/m ³
Viscosity (40°C)	2.7 cSt at 38°C

Submarine hoses [4]	
No	2
Nominal Diameter	8" = 203.2 mm
Material	reinforced rubber / synthetic rubber
Total length	35.0 m

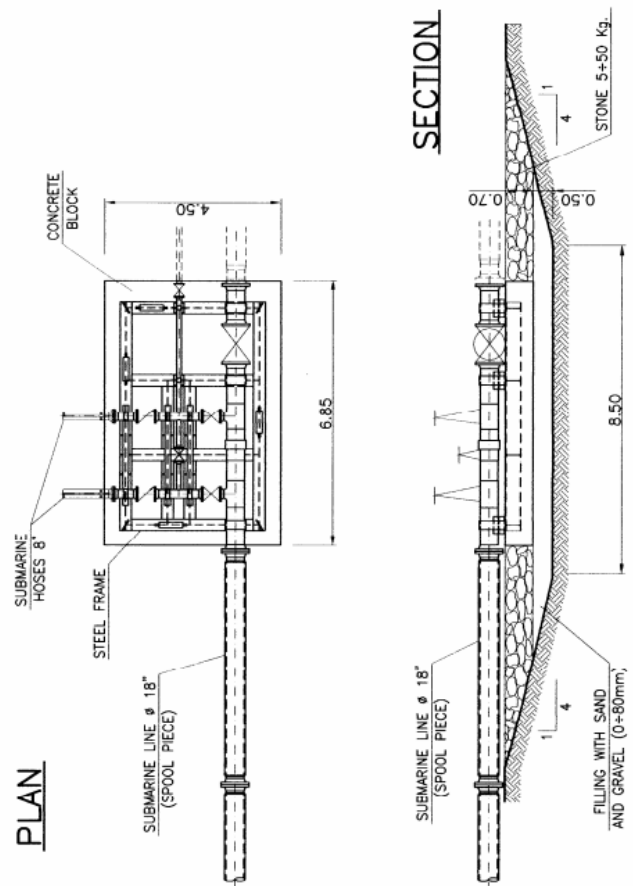


Figure 1. PLEM

3. Weights

With reference to the Figure 1, the following Weights and Buoyancy forces must be considered:

3.1 Weight in open air, empty pipe

Concrete block	6.85 m * 4.50 m * .70 m * 2.4 t/m ³	≈ 51.80 t
Steel frame		≈ 5.30 t
Piping dia. 18"		≈ 0.60 t
Piping dia. 8" - 4"		≈ 0.25 t
Valve – Flanges dia. 18"		≈ 2.25 t
Valves – Flanges dia. 8" - 4"		≈ 1.60 t
Total		≈ 62.00 t

3.2 Total buoyancy

Concrete block	$6.85 \text{ m} * 4.50 \text{ m} * 0.70 \text{ m} * 1.03 \text{ t/m}^3$	$\cong 22.20 \text{ t}$
Piping dia 18"	$0.452 \pi/4 * 7.00 \text{ m} * 1.03 \text{ t/m}^3$	$\cong 1.15 \text{ t}$
Piping dia 8" - 4"	$0.222 \pi/4 * 9.00 \text{ m} * 1.03 \text{ t/m}^3$	$\cong 0.28 \text{ t}$
Valves and flanges	Negligible	
Total		$\approx 23.60 \text{ t}$

3.3 Net Weight with empty pipes

Weight	$= 62.00 \text{ t}$
Buoyancy	$= -23.60 \text{ t}$
Total	$\approx 38.40 \text{ t}$

3.4 Net Weight with filled pipes (In-service)

Weight	$= 62.00 \text{ t}$
Buoyancy	$= -23.60 \text{ t}$
Fuel	$0.95 + 0.19 + 0.05 = 1.20 \text{ t}$
Total	$\approx 39.60 \text{ t}$

As a result of the above calculation:

$$\text{Weight} / \text{Buoyancy} = 62.00 / 23.60 = 2.627$$

4. Limit Equilibrium

The following forces could be applied on the PLEM structure:

- Line pressure (15 barg)

$$R_1 = 1507 \text{ cm}^2 * 15 \text{ barg} = 22600 \text{ kg} = 22.6 \text{ t}$$

(horizontal applied) [3]

- Pulling forces by submarine hoses (breaking load)

$$R_2 = 12000 \text{ kg} * \text{no. } 2 = 24000 \text{ kg} = 24.0 \text{ t}$$

(Applied over 25 m / 15 m i.e. - $\alpha = 25^\circ$) [3]

Drag forces are negligible in reason of partially embedded condition. The following stabilizing forces can be considered.

Horizontal stabilizing forces:

- Horizontal Base sliding (with sliding factor = 0.7 for sandy / gravely material) [4]

$$S_b = \text{Net weight} * 0.7 = 39.60 \text{ t} * 0.7 * 1.1 = 30.5 \text{ t}$$

- Soil Passive pressure, with .70 m of embedment ($\phi = 35^\circ$, $K_p = 3$, $\gamma' = 0.9 \text{ t/m}^3$) [4]

$$S_f = 0.5 * \gamma' * .702 \text{ m} * K_p * 4.5 \text{ m} = 0.5 * 0.9 * .702 * 3 * 4.5$$

$$S_f = 3.0 \text{ t}$$

$$S_{\text{tot}} = S_b + S_f = 33.5 \text{ t}$$

Based on the aforementioned forces the following conditions are verified:

- Horizontal equilibrium:

$$F_s = S_{\text{tot}} / (R_2 * \cos \alpha) = 33.5 / (24.0 * 0.9) = 1.55$$

- Vertical equilibrium

$$F_s = W / (R_2 * \sin \alpha) = 39.6 / (24.0 * 0.42) = 3.9$$

- Rotation equilibrium

$$M_{fl} = R_2 * \sin \alpha * 4.5 = 24.0 * 0.42 * 4.5 = 45.4 \text{ tm}$$

$$M_{s1} = W * 4.5 / 2 = 39.6 * 4.5 / 2 = 89.1 \text{ tm}$$

$$F_s = M_{s1} / M_{fl} = 89.1 / 45.4 = 1.96$$

5. Average Soil Pressure

The average soil pressure is:

$$p = W / A = 39.6 / (6.85 * 4.50) = 1.285 \text{ t/m}^2 = 0.13 \text{ kg/cm}^2$$

Which is admissible for sandy soil layer.

6. Results and discussion

Global PLEM behaviour under lateral loads has been modelled using the APILE2 software [5], which considers the non-linear behaviour of the soil by means of nonlinear horizontal load-displacement (p-y) curves.

The horizontal, vertical and rotation equilibrium was checked taking in consideration all the forces acting in the structure and considering the two cases; when the pipeline is empty and when the pipeline is filled with fuel oil (in service). The different buoyancy was considered for the two cases.

The safety factors are as below:

- For the horizontal equilibrium: $F_s = 1.55$
- For the vertical equilibrium: $F_s = 3.9$
- For the rotation equilibrium: $F_s = 1.96$

7. Conclusion

After a geotechnical inspection to define foundation type, in this paper it is analysed the stability of the PLEM. The PLEM is a steel frame connected with to the pipeline by means of a spool piece with flanged connection and with two submarine hoses.

There are considered two load combinations, and the result of this analysis is that PLEM structure is steady and reliable.

The offshore installation of the pipeline and PLEM was carried out successfully and the system for fuel oil unloading is still working properly.

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Assessment of the risk of electromagnetic emissions through monitoring for an object on the territory of the Metropolitan municipality

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Abstract: In many cases, the importance of electromagnetic fields or the so-called "electromagnetic smog" on human health, living organisms, and the environment is ignored or at least minimized. The electromagnetic field (EMF) is a combination of invisible electric and magnetic fields with charge that occur in nature. Today, the impact of electromagnetic fields on the environment is increasing from human activities with the development and application of telecommunication technologies. Measurement, continuous monitoring, database creation, and evaluation of electromagnetic field parameters in urban environments are important aspects of optimizing EMF levels to achieve a healthy living environment. The paper presents an analysis and evaluation of electromagnetic radiation measurements from base stations of mobile operators, at one site, in a lightly urbanized urban environment over a certain time interval. Initial measurements showed exceeding the levels of electromagnetic emissions according to national legislation. During the repeated measurements, it was found that mobile operators, after correcting the radiation from base station antennas, have brought the radiation levels within the permissible limits. This underscores the necessity of ongoing monitoring.

Keywords: ELECTROMAGNETIC RADIATION, ELECTRIC FIELD, ELECTROMAGNETIC POLLUTION, ENVIRONMENTAL LEGISLATION, EMF MONITORING

1. Introduction

The rapid development of the telecommunications industry has led to an oversaturation of the environment, especially the urbanized one, with technical sources of electromagnetic radiation. Electromagnetic radiation (EMR) is a wave propagating through space with an electric and magnetic component. Communication devices such as: mobile phones, cordless phones, local wireless networks and radio transmission towers, provide an electromagnetic field (emission) at the time of receiving and transmitting information. Existing and new radio communication technologies are leading to an increase in the number of base stations. When these facilities are placed in close proximity to or on public and residential buildings, in order to ensure greater traffic capacity and full radio coverage of the mobile phone service area, their penetrating radiation increases the voltage in the aforementioned buildings many times over. 2G, 3G, 4G, 5G antennas, with low and high range, continuously broadcasting the signals of mobile operators penetrate our homes and workplaces and can pose a serious threat to our health. Electromagnetic radiation is invisible to us, we cannot see, taste, or feel it, and this makes it particularly dangerous, as we have no way to protect ourselves from it. Radiation (EMF) passes through material or a person's skin, causing ionization that can damage chemical structures, and if the damage occurs to critical chemicals in cells (such as the DNA molecules that make up chromosomes in the cell nucleus), the cell can become damaged and lead to the formation of cancer [1-5].

In many cases, the importance of electromagnetic fields or the so-called "electromagnetic smog" on human health, living organisms, and the environment is ignored or at least minimized. European environmental policy currently includes more than 300 legal acts, including directives, regulations, decisions and recommendations [6,7]. They cover a wide range of measures to protect the environment from pollution through standards for air quality, waste management, water, nature conservation, control of industrial pollution, chemicals, background noise and climate change. Unfortunately, despite the growing public interest and concern, not enough attention is still paid to electromagnetic fields of artificial origin as a pollutant and the effects they cause on the environment, human health and living organisms. The fact is that the level of radiation from artificially created EMI in a number of cases significantly exceeds the level of electromagnetic fields from natural sources. Therefore, monitoring is necessary. To verify, assess, control and protect the population and the environment from exposure to electromagnetic radiation in accordance with the national standards and regulatory requirements adopted for each country [8-10].

Monitoring can be done for: source monitoring, environmental monitoring and individual monitoring, as appropriate. In this work we show individual monitoring. The rapid urbanization of the city

of Bankya, Sofia Municipality, Bulgaria, and the modern world's need for communication devices have led to the number of transmitting antennas and their power exceeding the maximum permissible standards in the Site under consideration. The purpose of this type of monitoring is to verify and assess the dose of electromagnetic fields received from the base station RADOST - SID SOF0318 with three sector antennas (A1: KATHREIN 80010292, A2: KATHREIN 742266, A3: KATHREIN 742266) near the studied site. Does the facility fall within the hygienically protected zone within the meaning of regulation [8] of the Bulgarian legislation on the maximum permissible levels of electromagnetic fields in populated areas and the determination of hygienically protected zones around radiating facilities?

2. Characterization methods

Two types of specialized mobile equipment were used. One of the devices is SPECTRAN, manufactured by the German company AARONIA AG, including a SPECTRAN HF 6060 device for measurements in the range of 1 MHz to 7 GHz, with a directional antenna type HyperLOG 7060. The measurement results are visualized in real time and recorded on a laptop computer on which the manufacturer's software is installed, allowing for settings, measurements, visualization and storage of the results in graphical and digital form.

The device was set to a measurement frequency range - from 700 MHz to 2500 MHz, to cover the emission range from the RADOST Base Station - SID SOF0318. The device is equipped with a directional HyperLOG 7060 antenna. It was set to a measurement frequency range of 0.68 – 8 GHz. Measurement data is recorded every 1000 ms. The data are stored in digital files and are combined with additional accompanying information, such as coordinates, a map of the area, location of the antennas, records of the spectrograms in graphical form for each measurement interval, and photographs of the measurement locations with the installed antennas.

The second device is the NARDA AMB 8057-03, for electromagnetic monitoring of the environment in the frequency range from 110 kHz to 7 GHz, providing capabilities for continuous autonomous control of electromagnetic radiation, even in points difficult to access for observation. It uses isotropic antennas to measure the characteristics of electromagnetic fields propagating in the environment and reaching the antenna from all directions in space (360°). The transmission of data to the server with the database is automatic, and there is also the possibility of remote setting of the device. The measurement results are stored in a specialized database containing information about the characteristics, frequency distribution and location (coordinates of the measurement points in a geographic information system GIS) of electromagnetic radiation. Mobile equipment NARDA AMB –

8057-3 provides capabilities for visualizing and reviewing measurement information for a selected time interval, date, week or day, for the entire frequency range (110 kHz – 7 GHz), a selected subrange of the frequency range (110 kHz – 7 GHz), peak values or average value for a specific frequency, a selected time interval of the day, to observe subtle effects in the distribution of radiated power for a given frequency.

3. Results and Discussion

Object - town Bankya, Sofia Municipality, Bulgaria. Measurements of electromagnetic fields in the frequency range 700 MHz to 2500 MHz were carried out with mobile equipment SPECTRAN HF 6060 in the time interval from 11:17 to 11:35 on 21.10.2015 (Fig.1). The measurement lasts for 18 minutes. The point from which the measurements were taken at the site is located ~ 25-30 meters in a straight line from the transmitting antennas of the base station. It should be noted that the measurements were conducted in a residential area, on a weekday, Wednesday. The area is without public and administrative buildings and it is assumed that the use of mobile communications is characterized by low traffic.



Fig. 1 Location of measuring equipment and arrangement of base stations around the research site.

Figure 2 shows the average values of the measurement results in three frequency ranges 943 MHz, 1690 MHz and 2152 MHz. Maxima are observed in the levels of measured EMI around $267 \mu\text{W}/\text{cm}^2$, at a frequency of 943 MHz. These measured values are twenty times above the permissible level of $10 \mu\text{W}/\text{cm}^2$, according to [8]. Excessive electromagnetic pollution has been measured. At frequencies 1690 MHz and 2152 MHz, the measured values are below the permissible level of $10 \mu\text{W}/\text{cm}^2$, $0.8990 \mu\text{W}/\text{cm}^2$ and $0.5070 \mu\text{W}/\text{cm}^2$, respectively.

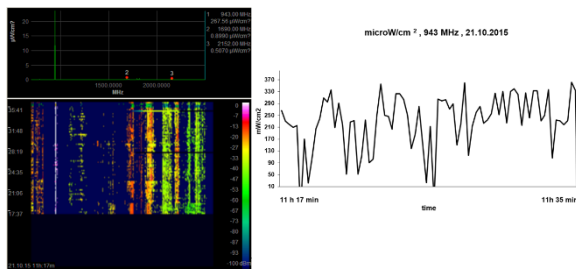


Fig. 2 Average values of the results of measuring the energy flux density of EMF.

In the same object, city Bankya, Metropolitan Municipality, Bulgaria, repeated continuous 24-hour measurements of electromagnetic fields with another mobile device NARDA AMB-8057-03 were carried out from May 27 to June 9, 2021 in the frequency range 100 kHz to 7 GHz. This frequency range covers the frequencies of all mobile operators operating on the territory of Bulgaria and allows to track the dynamics of electromagnetic radiation from the antennas, on weekdays and weekends. The point from which the measurements were made in the object is located ~ 25-30 meters in a straight line from the emitting antennas of a base station.

Figure 3 shows the average values of the measurement results in three frequency ranges 942 MHz, 1842 MHz and 2140

MHz. These frequency ranges were selected based on a recommendation from the EU Council [7].

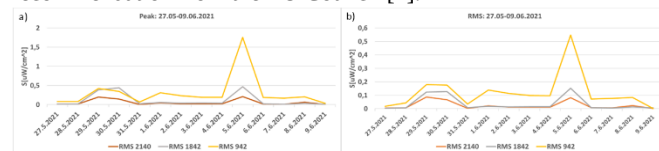


Fig. 3 Maximum peak values (a) and maximum average values (b) from measurements of electromagnetic fields in an object.

The maximum peak values of the measured power density of electromagnetic radiation EMF, for two of the frequency ranges 1842 MHz and 2140 MHz, are almost an order of magnitude lower than the maximum permissible standard of $10 \mu\text{W}/\text{cm}^2$. For a frequency of 1842 MHz, the measured values range from 0.011 to $0.47 \mu\text{W}/\text{cm}^2$, and for a frequency of 2140 MHz, they range from 0.007 to $0.21 \mu\text{W}/\text{cm}^2$. For the 942 MHz frequency range, the measured values vary in a wide range from 0.03 to $1.75 \mu\text{W}/\text{cm}^2$. For this frequency range, we should pay attention to the values obtained on June 5. This date is a holiday (Saturday).

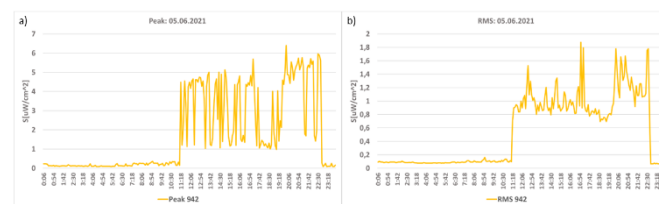


Fig. 4 Maximum peak values (a) and maximum average values (b) from measurements of electromagnetic fields in the facility for 05.06.2021.

Figure 4 shows the maximum peak and average values measured on 05.06.2021. The maximum peak values range from 0.083 to $6.39 \mu\text{W}/\text{cm}^2$. It is noteworthy that the highest values were measured in the evening hours, and after 10:30 p.m. the power of electromagnetic radiation decreases sharply.

As we have already specified, this is a residential area and the use of mobile sources on weekends is higher both for the date 05.06.2021 and the previous Saturday 29.05.2021. Accordingly, the level of the measured power density of electromagnetic radiation EMF has increased, but the obtained values are below the maximum permissible standard value of $10 \mu\text{W}/\text{cm}^2$.

The maximum average values of the measured power density of electromagnetic radiation EMF, for a frequency of 2140 MHz - vary from 0.002 to $0.08 \mu\text{W}/\text{cm}^2$. For frequencies of 1842 MHz and 942 MHz they vary in a wide range. For frequency 1842 MHz they vary from 0.004 to $0.15 \mu\text{W}/\text{cm}^2$, and for frequency range 942 MHz, from 0.002 to $0.17 \mu\text{W}/\text{cm}^2$. The measured values for the three frequencies are less than the standard limit values of $10 \mu\text{W}/\text{cm}^2$. Again, if we pay attention to the date 05.06.2021, Saturday, the measured values for the 942 MHz frequency range are higher. They are in the range of 0.07 to $1.87 \mu\text{W}/\text{cm}^2$, which is less than the maximum permissible values according to the standard of $10 \mu\text{W}/\text{cm}^2$.

From the measured power density of electromagnetic radiation EMF for all fourteen days of measurements, a recurring daily trend of a decline in the levels of radiated power from mobile operators is observed, after 10:30 p.m. to 6:00 a.m. - 6:30 a.m. This is explained by the end of the day and the intensive use of mobile communication devices. This trend of difference is repeated when comparing workdays and weekends.

Conclusions

The results obtained from measuring electromagnetic radiation in a site located in a lightly urbanized environment prove to us the need for monitoring. To carry out regular measurements and thus to check and control the parameters of all sources of EMF. To seek solutions to the problem of environmental pollution with this new

pollutant, including methods for analyzing the risk to human health and related solutions for the development of urban infrastructure.

During the initial measurements of electromagnetic fields in the frequency range of 943 MHz, maxima in the levels of measured EMF were observed $\sim 267 \mu\text{W}/\text{cm}^2$. These measured values are more than twenty times the permissible level of $10 \mu\text{W}/\text{cm}^2$. Excessive electromagnetic pollution has been detected. For the frequency ranges 1690 MHz and 2152 MHz, the measured values are below the permissible level of $10 \mu\text{W}/\text{cm}^2$. Cases of exceeding the maximum permissible standards under the Bulgarian National Standards in individual sites on the territory of Sofia Municipality are rather an exception. The presence of isolated cases of exceeding the permissible norms may be due to the degree of urbanization, inconsistency between the construction control authorities and those controlling the installation of new broadcasting antennas, their power and location. All this has a significant impact on the intensity of electromagnetic emissions. In this case, one of the frequencies at the date of measurement was not set to operate within the permissible radiated power level of $10 \mu\text{W}/\text{cm}^2$ [8].

During repeated continuous, all-day and continuous monitoring of fourteen days in the same site at the request of citizens and in the three frequency ranges 942 MHz, 1842 MHz and 2140 MHz, the levels of the measured radiated power of electromagnetic radiation are below the maximum permissible norm of $10 \mu\text{W}/\text{cm}^2$. This proves that after the initial measurements with high values, SRHI took action to eliminate the discrepancies, in accordance with the procedure specified in the regulatory framework and they were eliminated. Therefore, it is crucial to conduct monitoring and control of EMF to ensure a high level of protection of the population and the proper functioning of the Base Stations within the permissible norms.

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Investigation of extruded polyethylene blend films tensile creep elongation with different open holes configurations

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Abstract: In this article, tensile creep elongation of PE plastic films with two configurations of open holes stressed at the incremental temperatures of 23,30,37,43,51,58 degrees and a constant dead weight of 50 kg will be analyzed. The aim of the article is to analyze influence of the two configurations of open holes on the relative elongation. The test was conducted with the same blend, under the same test conditions. The test was conducted entirely according to the conditions established in the ASTM D6992 standard.

Keywords: CREEP TESTING, TENSILE STRENGTH, ELONGATION, PLASTIC FILMS.

1. Introduction

In engineering practice, there is often the presence of parts with open holes[1]. The presence of such holes is most often due to the following technological reasons:

- Lightening structure
- Access to various fasteners
- Access to other parts of assembled units
- Access for repair work
- Access for electrical switches, etc.
- Functional dimensions for assembly

Parts with open holes have lower strength properties than those without holes because stress concentrators are created and cross section is reduced [2]. In solid mechanics, stress concentrators are locations in a part where the stresses are greater than the surrounding area (Fig. 1). Stress concentrators include holes, grooves, notches, scratches, etc. Stress concentrators also reduce fatigue strength.

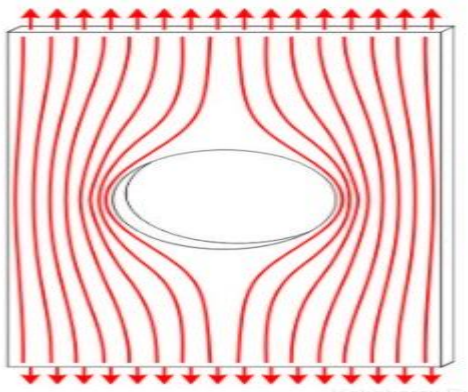


Fig.1 Internal force lines

It is known that the size and configuration of the holes play an important role in elastic properties of the material[3]. The dimensionless stress concentration factor k is used, which is equal to the maximum stress divided by the nominal stress. For round holes, the coefficient is equal to 3, i.e. the stresses along the edge of the hole are three times higher than the nominal stresses[4]. Despite the availability of CAE tools such as the boundary element method and finite element method predictive strength calculations with them through simulations are not particularly accurate[5]. A number of scientific articles study and optimize the influence of hole configuration on the tensile and compressive strength of various materials[6]. In engineering practice, the following solutions have become necessary to reduce the effect of stress concentrators:

- Optimizing holes
- Hole reinforcement
- Optimizing shapes of parts.

In plastic materials, localized plasticity occurs, while in brittle parts, stress concentrators lead to failure.

Polyethylene(PE) is the most common plastic worldwide[7]. Research into its mechanical, physical and chemical properties is very current. Polyethylene has wide application in industrial practice. Creep of materials is a slow increase in plastic deformation

caused by mechanical stresses[8]. It is an important characteristic of plastics because the process can start at room temperature[9].

2. Materials and methods

The material under the study is a PE blend, with a concentration of HDPE and LDPE 75 to 25 percent. It is characterized by low density, good chemical resistance, flexibility, easy processing by various methods, weldability, low hardness, high creep, impact resistance, excellent electrical insulator. PE is used as medical implants, packaging, pipes, bottles, ropes, etc. [10]. Products made of polyethylene are recyclable. The samples for testing have dimensions of thickness - 1.4 ± 0.01 mm, width 100 ± 1 mm, length 210 ± 1 mm. One sample has 26 open holes with a diameter of 10 ± 1 mm, and the other with 8 holes with a diameter of 20 ± 1 mm (Fig.2, Fig.3). The samples have technological holes of 8mm for installment (Fig. 4).

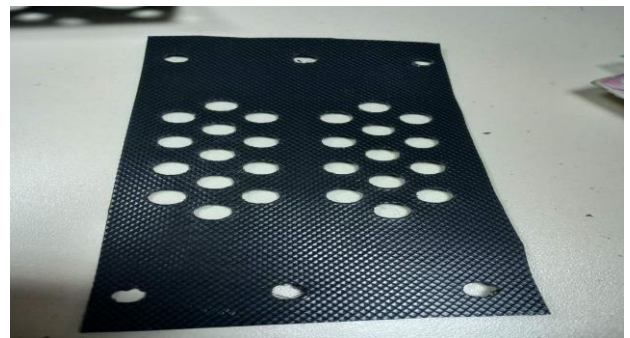


Fig.2 Sample with 26 open holes



Fig. 3. Sample with 8 holes

The area of the holes of the first and second samples is:

$$S_1 = 26(\pi \cdot r^2) = 2041 \text{ mm}^2 \quad (1)$$

$$S_2 = 8(\pi \cdot r^2) = 2512 \text{ mm}^2 \quad (2)$$

The area of the holes of the second sample is 23% larger. The samples were tested on a calibrated two-slot universal machine for creeping materials with appropriate jaws and scope. The test parameters are incremental temperatures 23,30,37,44,51,58⁰

degrees, dwell time at each step - 240 minutes - total test duration - 24 hours (fig. 5). the requirement that the test be longer than 60,000 seconds (app. 16.66 hours) has been met. The atmospheric conditions were observed according to the standard. The loading was carried out by weights of 50 kg, i.e. the force is equal to 490.5 N (Fig. 6).



Fig. 4 Installment of the samples in the chamber



Fig. 5 Parameters of the creep testing



Fig.6 Dead weight system

The test was performed on a calibrated creep machine. The test conditions specified in the standard were met. The temperature during the test was within the standard limits of $\pm 1^\circ\text{C}$. The selected incremental temperatures correspond to test conditions for heavily loaded structures. For the purposes of this article, only the elongation in mm after the test is considered, ignoring the elongation obtained at the different temperature steps. Both sockets

of the testing machine are used to ensure that both specimens are subjected to the same test conditions.

3. Results and discussion

The results of the accelerated creep test are presented in a table 1.

Table 1: Accelerated tensile creep results

Sample No	Elongation mm	Elongation %
1	14.06	6.69
2	9.06	4.31

The results show significant deformation of specimen 1, even though it is the specimen with the smaller hole area. The reason is that the linear force lines are denser in specimen 1 due to the selected hole configuration, fig. 7.



Fig. 7 Tested samples

The software of the universal creep machine records every 5 minutes and in the logs with the results there are no visible shock strains, which would compromise the test results (fig. 8) slippage or displacement of the test specimens is observed. No strain is observed in the gripping area. The strain is characterized by thinning around the horizontal axis of symmetry, forming an arc on both sides of the samples.

2024/11/13 13:09:11	13920	9060	58.1	"HOLES"	"	5.08	4.31	6.62
2024/11/13 13:14:11	13940	9060	58.1	"HOLES"	"	5.08	4.31	6.63
2024/11/13 13:19:11	13960	9060	58.1	"HOLES"	"	5.08	4.31	6.64
2024/11/13 13:24:11	14000	9060	58.0	"HOLES"	"	5.08	4.31	6.66
2024/11/13 13:29:11	14040	9060	58.0	"HOLES"	"	5.08	4.31	6.68
2024/11/13 13:34:11	14060	9060	57.9	"HOLES"	"	5.08	4.31	6.69

Fig. 8 Log file

The test results are significant, as plastics are increasingly used as a structural materials. Such tests can serve for more optimal designs of similar types of products with open holes [11].

4. Conclusions

. After testing the accelerated creep of the PE films blend with 2 different hole configurations, the following conclusions can be made:

- Sample 1, which has a smaller hole area, strains 55% more than sample 2
- The test conditions fully comply with the accelerated creep standard D6992
- No significant deviations were observed that would affect the test results
- The test results can be used for plastic films with a similar open hole configurations.

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Methods of Temperature Measurement in Heat Affected Zone during Welding

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Abstract: The metal materials are subject to severe temperature variation during the application of the welding process. The heat input and thermal shock during arc welding cause change of the base material properties in the heat affected zone. Temperature measurements and destructive tests of sample pieces, subjected to different process parameters and conditions, enable identification of minimum and maximum cooling rates, at which the material properties remain in pre-defined limits. Temperature measurements on the surface of the material in proximity to the weld seam enables cooling rate monitoring and material properties evaluation at manufacturing or site installation conditions, when the products could not be subjected to destructive tests. The requirements to the surface temperature measurement during welding and the characteristics of the different measuring devices were investigated for fitness to the purpose of cooling rate calculations, material properties evaluation, as well as for calibration of welding process simulations in volumes in close proximity to the arc welding seams.

Keywords: WELDING, COOLING RATE, TEMPERATURE MEASUREMENT

1. Introduction

The weldability of the metallic materials depends on a range of variables that affect the applicability of the method and the properties of the resulting joints. The properties of the joints of the products could not be assessed in full after the completion of manufacturing process. According to the definition in [1], welding is a “special process” and control before, during and after the completion of the works is necessary, if comprehensive quality requirements are to be met [2].

In arc welding, the increase of the temperature of the material from an initial value (T_0), to values above the liquidus is very fast, and the decrease of the temperature is slower, depending on variable conditions, process properties and material characteristics. Standard recommendations regarding the process variables [3], are issued for the most common materials, like ferritic steels [4], stainless steels [5], aluminium and aluminium alloys [6]. Weldability information is provided by the manufacturers of advanced materials [7].

The calculation of the cooling time in the temperature range from 800 °C to 500 °C, $t_{8/5}$, is possible, according to standard equations, defines by the standards [4], [8]. For example, a three-dimensional heat dissipation the equation was formulated for ferritic steels:

$$t_{8/5} = (6700 - 5 \cdot T_0) \cdot Q \cdot \left(\frac{1}{500 \cdot T_0} - \frac{1}{800 \cdot T_0} \right) \cdot F3 \quad (1)$$

where:

T_0 – initial temperature of the material,

Q – heat input, depending on the arc voltage, current and welding speed

$F3$ – seam geometrical factor.

The equations are valid under limited range of values of the essential variables, considering the thermal properties of all ferritic steels as same. The $t_{8/5}$ is calculated for the cooling of the weld metal.

While the cooling rate in the weld seem is important, the applied ferritic welding filler materials are of chemical composition and mechanical properties, that are superior to the properties of the base materials, irrespective of the cooling time. The area in risk of decreased mechanical properties is situated in the heat affected zone (HAZ), in immediate proximity to the weld seam. For materials of low thermal conductivity the cooling of the weld could not be considered as simultaneous in the whole volume of the weld seam, as the solidification of the liquid metal starts at the sides of the seam and ends at the weld central line, increasing the risk of hot cracking. When the essential variables are outside of the defined limits, the cooling time evaluation is possible on the T/t (Temperature / time) curve, built by measurements performed according to a standard method [4], within the weld seam. The applied method includes

insertion of a thermocouple in the liquid metal after the end of heat application by the welding arc. The method do not provide information for the cooling rate in the HAZ and it is not applicable in manufacturing conditions.

The standard qualification of the welding procedures [9] is based on heat input limitation. It is an indirect method of control of the cooling rate. As shown in (1), the heat input is the only variable influencing the $t_{8/5}$, assuming same thermo-mechanical characteristics of the materials and actual T_0 value, equal to the temperature of the sample pieces during the tests. The above assumptions are not always correct. As reported in [10], the thermal properties of different grade structural steels vary, depending on their chemical composition. Depending on the ambient conditions during the manufacturing process, the T_0 temperature could differ significantly to the initial temperature of the test pieces during the qualification process. Temperature measurement in defined spots in HAZ allows for direct calculation of the cooling rate and more accurate evaluation of the resulting material properties.

Surface temperature measurement in proximity to a welding arc represents a challenge, due to:

- variable thermal conductivity of the different materials surfaces,
- influence of the welding arc emissions,
- high temperature gradient, dependent on the distance to the weld fusion line,
- wide range of temperature values.

The choice of correct sensor properties is of essential importance to the measurement results.

2. Evaluation of surface temperature measurement device parameters

The temperature measurements are possible applying devices of different physical nature. Measuring devices based on changes of the volume/size of a sensitive material, semiconductor and resistive sensors, thermocouples and contactless pyrometers are widely applied in a range of technical applications.

2.1 Thermometers based on volume, length or other geometrical parameter of the sensor

This principle is by far the most widely applied for general temperature measurement, due to the simplicity and reliability of the method. The volume of gas or liquid thermo-sensitive material, or the length of a solid (or combination of solids) material is directly transferred into temperature reading. Disadvantages of the method are the long time necessary for equalization of the temperature in the whole volume of the sensitive material, especially in cases of flat surface temperature measurements. The

method is mostly used for slow processes and when significant surface of the sensitive material volume could be subjected to the temperature.

2.2 Thermistors and semiconductor temperature sensors

The resistor or semiconductor temperature sensors have a limit of temperature measurement which is lower than required for temperature measurements of post welding cooling rates.

For the thermistors, the highest measurable temperatures, depending on the type and model of the sensor, vary up to 300 °C. The semiconductor sensors have even lower maximum measurable temperature, typically up to 120 °C [11]. Semiconductor sensors are frequently employed in combination with thermocouples, in order to provide temperature data at the cold junction, required for compensation during measurements.

2.3 Resistance thermal devices (RTD)

This type of devices use the changes of the electrical resistance of the thermo-sensitive material (Pt 100) with temperature. For some models the measuring temperature range of -40 °C to 1000 °C is reported, as well as measurement spot area of 3.9 x 1.9 mm [13]. While faster than the linear expansion sensors in p. 3.1, the volume of the sensitive material should be heated in order to obtain correct temperature measurement. Electrical current should be supplied by the device to the sensor in order to measure the resistance. The self-heating effect should be considered.

2.4 Thermocouples (TC)

The thermocouples use the Seebeck effect that the junction between a set of two different metals produce emf voltage, depending on the temperature at the place of contact. The thermocouples function on the basis of the difference of the temperature between the cold and the hot end of the materials. The cold junction temperature should be known or measured, for instance by placing the cold end of the materials in ice bath, or in the modern devices, by measuring the cold end temperature by sensor of another type (for instance a semiconductor sensor), and compensating for the difference. The most-common sets of materials form thermocouple types are standardized, designated and their properties are tabulated in [14], together with the equations and constants, required to calculate the generated emf. An excerpt is presented in Table 1. The properties and rules for the compensation cables, used for connection of the thermocouples to the measuring instruments were issued in [18].

Table 1. Properties of high temperature thermocouples, according to IEC 60584-1:2013 (excerpt)

Type	Material (+ leg)/(- leg)	T range, °C, min/max	Tolerance Cl 2, °C @ T max °C	Emf, mV @ 100 / 1000 °C
J	Fe / Cu Ni	-180 to +750	± 5 @ 750	5.269 / 76.373
K	Ni Cr / Ni Al	-180 to +1350	± 9 @ 1200	4.096 / 41.276
N	Ni Cr Si / Ni Si Mg	-270 to +1300	± 9 @ 1200	2.774 / 36.256
E	Ni Cr / Cu Ni	-40 to +900	± 6.75 @ 900	6.319 / 76.373
S	Pt 10%Rh / Pt	-50 to +1750	± 4 @ 1600	0.646 / 10.506
B	Pt 30%Rh / Pt 6%Rh	+100 to +1820	± 4.25 @ 1700	0.646 / 10.506
C	W 5%Re / W 26%Re	+20 to +2300	± 1.0% (T)	1.451 / 18.257

The measuring spot diameter of the exposed thermocouples is in direct relation to the diameter of the wires. The butt welded thermocouples have measurement spot diameter, which is equal to the diameter of the wire. The beaded thermocouples tip diameter

vary up to 2x the wire diameter. The most common thermocouple diameters vary from Ø 0.1 mm to Ø 8 mm. The measuring spot area of bare exposed thermocouples of Ø 0.1 mm could be as low as 0.00785 mm².

According to thermocouple manufacturer's data [16], the maximum measuring temperatures for some types of thermocouples vary, depending on the wire diameter, as in Table 2.

Table 2. Dependence of the maximum measurement temperatures depending on TC wire diameter

Thermocouple type	Type K (NiCr/NiAl)			Type B (Pt 30%Rh / Pt 6%Rh)	
Wire diameter, mm	0.5	1.0	1.5	0.35	0.5
Max T, °C	800	1000	1100	1800	1800

The thermocouples response time depend on:

- The measuring spot contact area / diameter of the probe tip,
- The type of contact of the probe tip to the surface: direct contact of exposed thermocouple wires, grounded or ungrounded insulated thermocouple wires,
- Contact surface medium: air, water, metal to metal etc.

According to [15], the response time in air varies from 1.8 sec for 0.8 mm exposed butt welded wire thermocouples, to above 10 sec for 3.0 mm grounded thermocouples. The response times for beaded and for ungrounded thermocouples are respectively 1.5 x higher.

The response time in water for exposed butt welded wire of 1.25 mm and below is 0.25 sec.

The response time for exposed thermocouples in contact to heated surface, depend on the contact area between the two metals. Bad contact would result in long response times and in measurement errors, due to the thermal resistance of the surfaces.

Recommendation for use of thermocouples were issued by the International electrotechnical commission (IEC) [17], where the preparation procedure, placement, attachment to the measured surface, as well as for the connection to the thermometers were clarified.

2.5 Contactless thermometers

2.5.1 Contactless thermometers based on filament IR emitted radiation

One of the widely used principles of contactless temperature measurement is to pass an IR image of the heated object through an optical system, visible to the operator. The system contains a resistive filament that is heated, depending on the electrical current passing through it, to different temperature. While the filament emitted radiation wave length differs to the measured body radiation wave length, it would be visible to the operator through the optical system. By controlling the current, the wave lengths of the measured object and the filament equalize. The filament shape disappears on the same colour background of the measured object. The measuring temperatures of above 700 °C are common. Disadvantage of the principle is that is not usable for continuous recording of changing temperature values, due to the time necessary for wave length equalization.

2.5.2. Contactless thermometers, based on thermopile sensing elements

The thermopiles are IR detectors, based on multiple micro thermocouple elements, connected in series, in order to obtain highest sensitivity. The IR radiation is focused through an optical system to a matrix of thermopiles, each of these representing one pixel of the thermometer sensor. Commercial thermometers of this type offer a measuring range of -40 to +2250 °C, measuring different wave lengths, from 8-14 µm for the low temperature sensors, to 3.9 µm for the high temperature sensors [19]. The response times of the devices were between 60 and 120 ms. Temperature resolution of 0.1 °C and accuracy ± 1% of the reading are achievable.

The measuring spot size should not be confused with the pointing laser spot, which could be very small in wide range of distances between the sensor and the surface. The actual measuring spot size depends on the optical resolution of the thermo sensors. Optical resolutions Distance: Spot between 10:1 and 120:1 are common. The meaning of optical resolution of 120:1 is that the distance of the sensor to the surface spot is equal to 120 x the measuring spot size. For instance, if the measuring distance is 120 cm, the measuring spot size would be 10 mm.

The optical resolution is generally linear within a range between minimum and maximum distance to the surface. As reported in [20] the minimum measuring spot size for different types of sensors of optical resolution D:S between 9:1 and 75:1 was equal to 6.5 mm.

Challenges to the applicability of the IR contactless thermometers represent:

- The dependence of the measuring accuracy on the measured material surface emissivity in the measured wave length. The shiny metal surfaces are of low emissivity and high reflectivity, which could cause the thermo sensors to read reflected, instead of emitted radiation. In order to decrease the influence of the emissivity double wave length sensors are applied, that measure the difference between the radiation in the two wave bands;
- The proximity of the welding arc during the measurement process influence the contactless temperature measurement sensors. As demonstrated in [21], this challenge was addressed by the application of narrow band filters, passing through the wave lengths that were emitted by the measurement surface, but not the reflected waves and the arc emissions. The complex equipment allowed for high speed measurements at frequency of readings of 60 Hz.

2.6 Fiber-optical measuring systems

The fiber-optical measuring systems have the advantage, that it is not necessary to consider the emissivity of the measured surface, but instead a sensor tip with known emissivity is used. The emitted radiation, or pre-emitted radiation is conveyed via optical fiber. In such way the ambient volume does not influence the measurement in optical or electromagnetic way. The sensor principles include blackbody radiation, luminescence in crystals, refractive index phenomenon in the fiber Bragg gratings or phase shift of two coherent beams in interferometric sensors [24].

The surface measurement spot size of the fiber-optical system in the range from 0.4 mm, to volumetric systems utilizing fiber sensors with lengths of hundreds of meters were reported [25].

The response times of the blackbody radiation systems in the range between 1 ms to 100 ms, and the temperature range of 800 to 2000°C was reported [26], utilizing single crystal sapphire fiber sensor.

A disadvantage of the fiber optical systems include great complexity of the equipment, if wide temperature range is to be achieved, including combinations of several types of sensors in the different areas of the range.

3. Requirements for temperature measurement for cooling rate monitoring and calculations in HAZ during and immediately after welding

Depending on the requirements for the measurements, there are several evaluation criteria for the devices and sensors, in order to be applicable for surface temperature measurements and real time monitoring, in close proximity of the weld seam, in manufacturing conditions or at site installations.

3.1 Temperature range

The required temperature range varies, depending on the material, which would be subject to temperature measurement. The lower limit of the range could be set based on evaluation of the lowest initial material temperature. The higher limit of the range depends on the material liquidus, thermal conductivity and phase transformation temperatures.

For ferritic structural steels, operational temperatures down to -60 °C are possible and site ambient temperatures could be considered

as equal to the initial material temperature. A phase transformation from ferrite to austenite occurs at temperature range between 700 and 900 °C. The phase transformation temperature for same type of steel increases in cases of high speed heating, similar to the conditions occurring during arc welding processes [21]. The sensors range for high temperatures measurement between the liquidus and the transformation temperature could be considered as appropriate.

3.2 Accuracy and tolerance limits of the measurements

The permissible tolerance limits of the measurements time-temperature cycles related to heat application to welded structures is set in table 44 of [22], as ± 10 °C. In [23] the tolerances of the heat treatment measuring equipment were set as in [14].

3.3 Latency

The latency of the measuring system should be appropriate for the required measurement accuracy. Initial data for the cooling time for ferritic structural steel was obtained by a measurement, performed with K-type thermocouples, positioned at 3 mm from the weld seam edge, and at 11 mm from the weld seam line. The maximum temperature measured a 3 mm from the weld seam edge was 1040 °C, and at 11 mm was 480 °C, Fig. 1a.

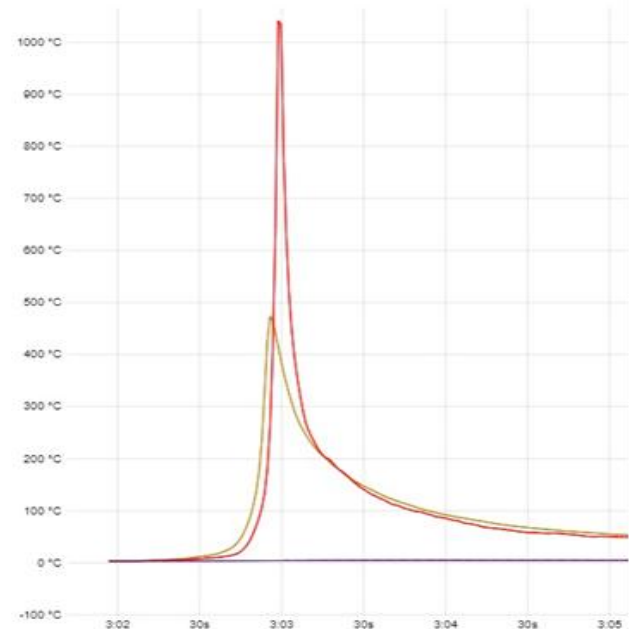


Fig. 1a. Temperature cycle measured at welding heat affected zone at distance of 3.2 mm and at distance of 11 mm from the weld seam edge, with maximum temperatures of 1040 °C and 480 °C respectively.

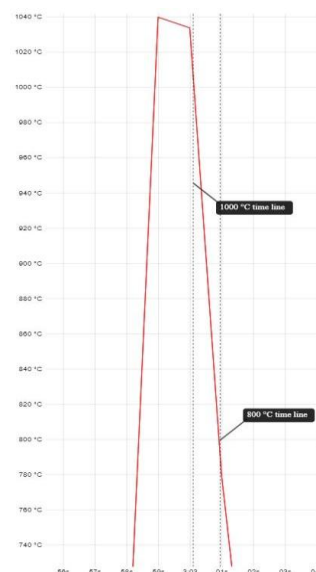


Fig. 1b. Measuring of the time of temperature decrease from 1000 °C to 800 °C

The decrease of the temperature of the material at 3 mm from the seam line, 1000 °C to 800 °C was calculated on the graphic,

by deducting the time when temperature decreased to 1000 °C to the later time, when the temperature decreased to 800 °C, to be 1 second, as on Fig. 1b.

In order to achieve measurement accuracy of ± 10 °C, in case that the temperature decrease from 1000 °C to 800 °C happened in 1 sec, measurements frequency of above 20 readings per second would be necessary. In order to fulfill the requirements of the sampling theorem, in order to read at frequency of 20 Hz, at least 2X sampling rate of the sensors would be required. In such case frequency of 40 readings of the sensor data per second would be adequate.

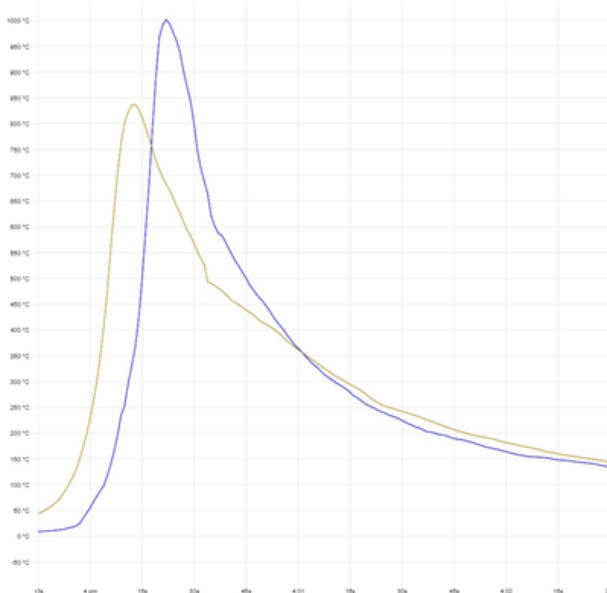


Fig. 2 Temperature cycle measured at welding heat affected zone at distance of 3.5 mm and at distance of 5.5 mm from the weld seam edge, with maximum temperatures 1000 °C and 840 °C

3.4 Measurement spot area

The measurement of the temperature occurs on area of the object surface, specific to the type of sensor that is used. The different sensors have different area resolution, depending on the size of the sensor, distance to the measuring spot, disturbances caused by movements or vibrations etc. Measuring the temperature near the seam edge require smaller measurement spot diameter, compared to measurements at a bigger distance. According Fig. 2, obtained for structural ferritic steel, the maximum temperature measured at 3.5 mm distance to the seam edge was 1000 °C. At 5.5 mm distance from the seam edge, the maximum measurement temperature was 836 °C. Comparing the data obtained during the measurements presented on Fig. 1 and Fig. 2, the temperature difference per mm distance to the weld seam edge could be calculated, as in Table 3.

The targeted measurement accuracy of 10 °C at distance to the weld seam with maximum temperature of 1000 °C could be achieved by determination of the measurement position with resolution of 1 / (133/10), mm / (°C / mm). The measurement principle and the sensor spot size should allow determination of the measurement position within a tolerance of the distance to the weld seam edge of ± 0.075 mm. In cases of measuring spot diameter, bigger than the requirement for the accuracy of the distance to the weld seam edge, the equalisation of the temperature in the sensor could be calculated. For the non-contact sensor types, the temperature equalisation could be evaluated by calculation of the filters wave length band, to pass the radiation emitted from a spot corresponding to 3 pixels of the sensor matrix. For contact sensors, RTD, TC, the temperature equalisation at the sensor element could be calculated, based on the volume of the sensitive material and the difference between the thermal conductivity of the measured material and of the sensor material. In general, the cases when the thermal conductivity of the sensor material is higher than the thermal conductivity of the measured material are preferential.

Table 3. Maximum measured temperature measured at different distances from the seam edge

Measurement Nr.	1a	2a	2b	1b
Distance, mm	3.2	3.5	5.5	11
Max temperature, °C	1040	1000	840	480
Distance (n+1) – (n), mm	0.3	2.0	5.5	
ΔT , °C	40	160	360	
ΔT to distance, °C / mm	133	80	65	

3.5 Other evaluation criteria

Influence of the external disturbances to the accuracy of the measurements. The welding arc emissions in wide spectre of radiated heat, waives and rays require comprehensive evaluation of measures for disturbance avoidance. The presence of fumes and smoke of varying consistency during the process represents an obstacle to the non-contact measurements.

The applicability of the measuring devices depend on additional factors, which influence the possibility to implement these in manufacturing or site installation conditions, including complexity of the equipment [12] and economic considerations.

4. Results

The properties of the temperature measuring devices were evaluated as of high, medium, low or insufficient compliance to the above defined requirements, as in Table 4.

Table 4. Evaluation of the correspondence of the thermometer properties to the requirements to surface temperature measurement in the HAZ during and after welding

Sensor type / criteria	Temp. range, °C	Accuracy °C	Latency, readings	Measuring spot	Other criteria
Required range	-40 to 1000 °C	± 10 °C	< 40 per sec	$\varnothing 0.1$, mm	reliability
Volumetric	High	High	Insufficient	Low	High
Semiconductor	Inadequate	High	High	Low	Medium
RTD (Pt100)	High	High	Low	Low	High
TC K, □□□	Medium	Medium	Medium	Medium	High
TC K, □□□	High	Medium	Medium	Low	High
TC N, □□□	Medium	Medium	Medium	Medium	High
TC N, □□□	High	Medium	Medium	Low	High
TC, type B	High	High	Medium	Medium	High
IR / filament	Medium	High	Insufficient	Insufficient	Low
IR / thermopile	High	High	High	Insufficient	Low
Fiber-optical	High	High	High	Medium	Low

5. Conclusions

The welding of materials with advanced-properties, achieved by precise control of the chemical composition, combined heat and mechanical treatment during manufacturing, could require additional control of the temperature cycles during the process.

Direct measurement of the temperature in the weld seam is possible by non-contact optical devices, in cases when the measurement spot of considerable size is acceptable. The accuracy is subject to various

influences. The validity and accuracy of the results is influenced by a number of factors, including material surface emissivity, reflections, weld arc emissions, humidity, and intermediate air/medium quality among others. The utilization of non-contact optical measuring devices is limited to manufacturing with high repeatability, performed in controlled conditions, and by the complexity of the measuring equipment if reliable results are to be achieved.

Direct measurement of the temperature is possible in close proximity to the weld seam, utilizing single or multiple spot thermocouple sensors, allowing monitoring of the welding thermal cycles, as well as additional process parameters including welding speed (arc travel time between sensors positioned along the seam), actual preheating temperature at process start, inter-pass temperatures, heat treatment parameters etc.

Calibration of welding process simulation models is possible by multi-spot temperature measurement, allowing evaluation, in a finite number of positions in the whole volume of the heat affected material, of different properties of the metal materials, including convection and radiation at the surface; stress versus strain; materials changes such as microstructure transformations; dissolution or precipitation; mechanical behaviour such as elasticity; instantaneous or time dependent-plasticity; strain hardening and recovery effect; thermal expansion; transformation induced plasticity [11].

Measuring of the temperature on the surface, at pre-defined distance from the weld seam line, could be utilised for an evaluation of the material properties in the welding manufacturing process. Determining of t_{8/5} limits in defined spots or distances to the weld seam edge during procedure tests and continuous monitoring of the temperature during manufacturing allows for increased control of the joint properties. The type and the design of the thermocouple sensors should be carefully reviewed due to the operation close to the properties and applicability limits of the measuring devices. Multiple sensors of different type could be applied in order to address the limitations.

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Мрежа от автономни безпилотни летателни апарати за контрол на урбанизиран район

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Резюме: През последното десетилетие безпилотните летателни апарати (БЛА) или дроновете заеха водеща роля и приложение в интелигентното земеделие, ефективната реакция при извънредни ситуации и много други. Създаването на мрежа от автономни рояци от дронове, работещи съвместно за наблюдение на урбанизиран район би помогнало за решаването на широк кръг от нови задачи. В настоящата статия се анализират възможностите и се посочват предимствата на рояците от дронове при наблюдение и инспекция на различни процеси и съоръжения в урбанизиран район.

Ключови думи: рояк дронове, проследяване на цели, комуникационни характеристики

1. Въведение

През последните години дроновете се използват за решаване на различни полезни задачи, като: инспекции [1], наблюдение и разузнаване [2], доставка на пакети [3], търсене и спасяване [4], селско стопанство [5], дистанционно наблюдение [4], транспорт [6], спешна медицинска помощ [7], военни операции [8] и много други. Дроновете са изключително удобни за работа в урбанизиран район и в критични среди, които не са лесно достъпни на хората. Те притежават значителни предимства като висока ефективност, ниски разходи, бързи и качествени резултати. За инспекция и контрол на урбанизиран район е подходящо да се използват рояци от дронове, които да са свързани с облачни услуги, така че наземният сегмент, поддържащ мисията автономна, да се актуализира с най-новата информация относно ограниченията за полета. Също така, облачните услуги подпомагат движението на рояка дронове чрез задаване на маршрута на всеки един от дроновете.

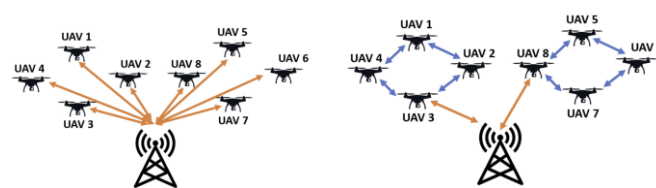
Традиционното наблюдение и инспекцията на различни процеси и инфраструктурни съоръжения в урбанизиран район се извършва посредством визуални проверки. Този метод е трудоемък, бавен и скъп, като не гарантира добри резултати за труднодостъпни зони. Основното предимство на използването на дронове за наблюдение е способността им да покриват и труднодостъпни зони. В литературата се споменават различни възможни безпилотни средства за осъществяване на наблюдения и инспекции, като: дронове с фиксирани крила [9], роторни дронове [1], автономни роботизирани дирижабли [10] и дори хибридни [11] или кооперативни решения [12].

Повишаването на ефективността за наблюдение и контрол в урбанизиран район може да се постигне с използването на рояци от дронове, които не изискват допълнителни подобрения на съществуващите комуникационни или компютърни технологии [13]. Технологиите, използвани за рояк дронове позволяват на групи дронове да се координират помежду си, като не е задължително да има пряка човешка намеса. Тези технологии могат да координират от няколко до хиляди дронове с цел съвместно изпълнение на мисии с ограничена нужда от човешка намеса и управление. Рояците дронове са по-ефективни от единичните дронове, тъй като изпълняват различни паралелни задачи и то без човешки надзор. Роякът от дронове продължава да изпълнява своята мисия дори при отпадане на някой от дроновете. Всичко това прави рояците от дронове предпочитани при изпълнение на сложни мисии особено в урбанизиран район с наличие на много сгради, дървета, превозни средства, хора и т.н.

2. Рояк дронове

Рояците дронове интегрират усъвършенствани компютърни алгоритми с локални сензорни и комуникационни технологии, за да синхронизират множество дронове за постигане на определена цел [14].

Те използват различни методи за управление, включително предварително програмирани мисии със специфични предварително дефинирани траектории на полета, централизирано управление от наземна станция или единичен контролен дрон, или разпределено управление, при което дронове комуникират и си сътрудничат въз основа на споделена информация [15]. По-напредналите методи за управление включват интелигентност на рояк, вдъхновена от колективното поведение на ята птици, както и техники за изкуствен интелект, за да научи рояците дронове да реагират на нови или неочаквани ситуации. Развитието на изкуствения интелект и машинното обучение през последните години подобри използването на многосензорни технологии за събиране на данни и вземането на решения. Въпреки бързия напредък използването на рояк дронове остава ограничено поради трудности като проследяване и отчитане на пространствено-времевата динамика на въздушна топология, както и характеристиките на мрежовата свързаност на дроновете в рояка. Свързаността може да се раздели на два класа [16], показани на фиг. 1.



(а) Централизиран модел

(б) Децентрализиран модел

Фиг. 1. Архитектури на рояци дронове с клетъчна връзка

Централизирана архитектура (фиг. 1а): при този подход клетъчната базова станция (BS) действа като команден център за насочване и управление на рояка дронове. Всеки дрон (UAV) предава информацията за наблюдение и мониторинг към базовата станция. Тази комуникационна връзка е известна с наименованието дрон към инфраструктура (U2I). В тази архитектура няма директни връзки между дроновете в рояка.

Децентрализирана архитектура (фиг. 1б): при този подход не всички дронове (UAVs) са свързани към базовата станция за управление и комуникация. В допълнение към архитектурата дрон към инфраструктура (U2I), дроновете може да комуникират директно един с друг в рамките на рояка с цел обмен на информация чрез комуникационни връзки дрон към дрон (U2U).

3. Приложение и предизвикателство на рояк дроне

Гражданските приложения на рояк дроне са разнородни и зависят от сензорите и устройствата, с които са оборудвани. Едни от най-често срещаните приложения са наблюдение на урбанизиран район, включително обекти, пътен трафик и други. Рояк дроне могат да покрият голям периметър от пространството, както и много на брой обекти. Инспекцията на стратегически обекти като далекопроводи, мостове, железопътна инфраструктура и много други е от съществено значение за правилното и безопасно функциониране на съществуващите системи. Управление при извънредни ситуации с помощта на рояк дроне помага за адекватна реакция и бърза намеса при намиране на изчезнали хора и предоставяне на спешна помощ и доставки на различни товари и апаратура по време на природни бедствия. Рояците от дроне са полезни в селското стопанство, като могат да засаждат семена, да идентифицират огнища на болести чрез наблюдение на големи площи и да прилагат различни обработки на културите като пръскане с пестициди, хвърляне на торове и др. Рояците от дроне могат да помогнат на пожарникарите да проследяват и контролират разпространението на пожари. Възможна е доставка на комуникационни и навигационни услуги, включително и интернет в критични области. Организаторите на развлекателни събития могат да правят различни шоу програми, включително забавления, алтернативни на фойерверките. Приложението на рояците от дроне тепърва ще се увеличава благодарение на развитието на изкуствения интелект.

Предизвикателствата, които стоят пред рояците от дроне също са големи и са свързани с безопасността и сигурността. Рояците дроне могат да работят с минимална човешка намеса, но човешката намеса със системите за управление може да е необходима за чувствителни мисии, като например тези, които могат да изложат хората на опасност. Настоящите разпоредби не позволяват на едно лице да управлява повече от един дрон едновременно. Забранява се също така използването на дроне в зони с ограничен достъп или за незаконни или престъпни дейности като шпиониране, кибератаки или разполагане на импровизирани експлозивни устройства.

Поверителността и киберсигурността е друго предизвикателство пред рояците дроне, събиращи информация за заобикалящата ги среда, така че трябва да се регламентират правила и протоколи за защита срещу събирането и съхранението на определена информация като снимки, видеоклипове или звукозаписи. Мерките за киберсигурност могат да помогнат да се гарантира, че дроне не са отвлечени или хакнати от злонамерени лица и не се използват за злонамерени цели.

Бързият технически напредък ще доведе до миниатюризация на хардуера, подобряване на техническите характеристики на сензорите и изчислителната мощност на използваните процесори. Развитието на различни софтуерни решения и оптимизация на работата на алгоритмите би подобрило управлението и поведението на рояк дроне. Подобряването на комуникационната свързаност ще позволи увеличаване на броя на дроновете в рояка, както и вземането на различни важни решения от рояка дроне.

Полезността и необходимостта от създаването на рояк дроне е неоспорима, но съществуващите ограничения затрудняват масовото му използване. По-съществените предизвикателства са ограниченията като малък полезен товар, сравнително малко полетно време, необходимостта от оператор (дистанционен пилот), който да управлява рояка с предавател или компютър с подходящ софтуер за управление и др.

Обикновено под рояк се разбира група от действащи лица, които заедно се координират, за да получат желан резултат. В природата се срещат множество примери за координирано поведение между един и същи вид животни или насекоми с цел

постигане на обща полезна цел. Пчелите се координират една с друга за изпълнение на задачи, критични за оцеляването на техния рояк. Стада на мигриращи птици координират ефективни модели на полет, за да постигнат тяхната миграция. По същия начин рояк от дроне координира всеки един дрон за изпълняване на желана задача или набор от задачи.

4. Анализ на комуникационните технологии в рояк дроне

Изборът на подходяща комуникационна технология в дадени безпилотни летателни системи (БЛС) зависи от избора на комуникационните модули, вида на антените, използваната платформа и вида на мрежовата архитектура. При избор на комуникационна технология трябва да се вземат предвид различни технологични параметри, обхват на действие, честотна лента, мобилност, честоти на сигнала, съвместимост, скорост и полезен товар. В таблица 1 са показани различните характеристики на безжичните протоколи [17-20], като се забелязва, че най-мощните протоколи, използвани за дроне са WiMAX и GSM/GPRS поради техните комуникационни обхвати, честотна лента, мощност на предаване и вида на цифровата модулация.

Таблица 1. Характеристики на безжични протоколи

Тип протокол	Bluetooth	Ultra wideband (UWB)	ZigBee	Wi-Fi	Wi-Max	GSM/GPRS
Честотна лента	2.4 GHz	3.1-10.6 GHz	868- 915 MHz; 2.4 GHz	2.4: 5 GHz	2.4: 5.1- 66 GHz	850- 900: 1800- 1900 MHz
Макс. капацитет	720 Kb/s	110 Mb/s	250 Kb/s	54 Mb/s	35-70 Mb/s	168 Kb/s
Разст. за комуникация	10 m	10-102 m	10- 1000 m	10-100 m	300m49 Km	2-35 Km
Мощност на предавателя	0 -10 dBm	-41.3 dBm/M Hz	-25 -0 dBm	15 - 20 dBm	23 dBm	0-39 dBm
Честотна лента	1 MHz	0.5- 7.5 GHz	0.3- 0.6 MHz; 2 MHz	25-20 MHz	20:10 MHz	200 kHz
Модулация	GFSK, CPFSK, 8-DPSK, $\pi/4$ -DQPSK	BPSK, PPM, PAM, OOK, PWM	BPSK, QPSK, OQPSK	BPSK, QPSK, OFDM, MQAM	QAM1 6/64, QPSK, BPSK, OFDM	GMSK, 8PSK
Техника на предаване	FHSS	DSUWB, MBOFDM	DSSS	MCDSSS, CCK, OFDM	OFDM, OFDM A	TDM A, DSSS

Качеството на комуникацията в тези системи може да се повлияе от различни физически ефекти като: Доплеров ефект, прекъсване на комуникацията, заглушаване на сигнала и др. Тези ефекти могат да повлияят на качеството на пакетната комуникация, но целта е да се постигне ефективна комуникация с ниска латентност и надеждна комуникация в системата.

Комуникацията на дроновете в градски условия се затруднява значително поради наличието на много радиоелектронни предаватели, генериращи електромагнитни смущения, наличието на сгради и ефекти от засенчване на комуникационните сигнали. За борба с тези нежелани ефекти се предлагат различни технологични и софтуерни решения като борба с импулсни смущения [21, 22], отчитане на дифракционни ефекти [23, 24] и много други.

5. Заключение

В настоящата статия са анализирани възможностите и се посочват предимствата на рояците от дроне при наблюдение и инспекция на урбанизиран район. Анализирани са протоколите за комуникация между дроновете за установяване на най-подходящите при реализиране на рояк дроне.

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Системи за откриване и противодействие на безпилотни летателни апарати

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Резюме: Нарастващото използване на безпилотни летателни апарати поражда опасения относно сигурността, безопасността и неприкосновеността на хората, като оказва силно влияние върху психологическите и социалните аспекти на националната сигурност. Всичко това води до необходимостта от разработване на системи за откриване и противодействие на нежелани дронове в чувствителни зони. Тези системи могат да откриват, проследяват и неутрализират неоторизирани безпилотни летателни апарати (БЛА). В статията се анализират методите и технологиите за откриване и противодействие на безпилотни летателни апарати. От спецификата на използваните технологии са посочени предимствата на всеки от методите за борба с дронове при защитата на хората и критичните инфраструктурни обекти.

Ключови думи: безпилотни летателни апарати, методи и технологии за откриване на дронове, противодействие на БЛА

1. Въведение

Бързото развитие на безпилотната летателна индустрия допринася за развитието на транспорта, отбраната, търговията и услугите. Масовото използване на безпилотни летателни апарати (БЛА) е необходимо да отговаря на определени законови изисквания и да покрива високи екологични и безопасни норми. Част от съществуващите дронове се използват за навлизане в личното пространство и събиране на снимки и информация. Те могат да се използват за нападения и атентати. Други се използват за контрабанда и трафик на наркотици и други незаконни действия. Всичко това налага дроновете да бъдат откривани от системи за наблюдение на въздушното пространство и при необходимост да бъдат неутрализирани.

Системите за защита от безпилотни летателни апарати се състоят от системи за откриване, проследяване и неутрализиране на нежеланите дронове.

За откриване на дронове се използват различни методи и технологии, притежаващи специфични предимства и недостатъци. Основните методи и технологии, използвани за откриване на дронове, са: Радарно откриване [1-5], използващо традиционна радарна система за сканиране на пространството или радар с пряко разсейване на сигнала, използващ съществуващите радиосигнали от други източници на електромагнитни вълни, както и микродоплеров радар за анализ на микродоплеровия ефект, причинен от витлата на дрона; Радиочестотно (RF) откриване [6-10], базирано на анализ на комуникационните сигнали между дрона и неговия контролер; Оптично и инфрачервено откриване [11-15], базирано на обработката на видео от камери с видима светлина и обработка на инфрачервени изображения, съдържащи информация за термичното състояние на дрона; Откриване с Lidar, използващ лазерни импулси и обработвайки техните отражения от дрона и околната среда; Лазерни виброметри, измерващи структурните вибрации на дрона; Акустично откриване [16-20], което анализира звуковите сигнали, генерирани от дрона; Аудио-визуални системи, комбиниращи аудио- и визуални данни за подобряване на точността на откриване и др.

За подобряване на надеждността и точността на откриване се предлагат методи за обединяване на данни от различни сензори. Алгоритмите за дълбоко обучение и изкуствен интелект могат да се използват за подобряване на точността на откриване на дрона. Съществуващите алгоритми за дълбоко обучение се използват за анализ на изображения и аудиоданни за откриване и класифициране на дронове. Алгоритмите с изкуствен интелект са удобни за едновременно откриване, идентификация и разпознаване на дрона.

Методите за неутрализиране на нежелани дронове са различни като изборът на метод зависи от естеството на заплахата, местоположението на инцидента и търсения ефект.

Най-често срещаните мерки за противодействие използват радиочестотно заглушаване, лазерни пушки и мрежи за прихващане на дронове.

В настоящата статия се анализират методите и технологиите за откриване и неутрализиране на безпилотни летателни апарати. Направени са изводи и препоръки и са посочени предимствата и недостатъците на различните методи за откриване и противодействие.

2. Обзор и анализ на методите за откриване на безпилотни летателни апарати

2.1. Радарно откриване

Акустичните радарни системи могат да откриват дронове, като излъчват електромагнитни вълни в пространството, които се отразяват от различни обекти и се приемат обратно от радара. След прилагане на специализирана сигнална обработка е възможно да се открие БЛА и да се измери скоростта и азимута на целта. Съвременните радарни системи могат да открият и разпознаят вида на обекта. Пасивното радарно откриване разчита на радиосигнали, излъчени от други радиоустройства.

Откриването на роторни дронове чрез радари за дълги разстояния посредством извличане на Helicopter Rotor Modulation (HERM) характеристики и използване на микродоплеров анализ е осъществено в [1]. За да разграничат дроновете един от друг, както и да ги разграничат от други летящи обекти, авторите използват честотите на среза. В [2] е изследван проблемът с ограничения обхват на работа на микродоплеровия ефект, получаван от лопатките на дрона, като се предлага увеличаване на обхвата на системата за откриване на дронове посредством използване на HERM характеристики, произтичащи от визуализацията на микродоплера чрез използване на дълъг прозорец на кратковременната трансформация на Фурие. Авторите предлагат да се използва техниката на логаритмично сумиране на хармониците, за да се извлекат основните честотни, съставляващи HERM характеристиките. В [3] се предлага откриване на дрон в система за проследяване, използваща малък радар с милиметрови вълни. Предложената система преобразува отразените сигнали в облак от точки, като тази техника има за цел да прави разлика между различни безпилотни цели, използвайки модели на тяхното отражение. За различаване на дронове от други цели е използван алгоритъм, комбиниращ модел на конволюционна невронна мрежа и пространствено-времеви характеристики, наречен CNN-LSTM. В [4] се анализира система за откриване на дронове, използваща доплеров радар. Авторите са използвали алгоритъм за откриване, поддържащ постоянна вероятност на лъжлива тревога (CFAR) и е разработен персонализиран модел на CNN, наречен DopplerNet по реда за разпознаване на летящи дронове

сред други обекти. В [5] е изследван радар с пряко разсейване, който използва GPS сигнал за откриване на дрон, пресичащ базовата линия на радарната система.

2.2. Радиочестотно откриване

Откриването на дроне посредством радиочестотно откриване разчита на наблюдение на комуникацията между БЛА и наземния контролер. Този метод анализира всички предавани сигнали в рамките на определен честотен диапазон, което потенциално води до голяма вероятност от фалшиви аларми, тъй като приети RF сигнали не са непременно на търсения дрон [6]. Има два основни вида пасивни RF сензори. Първият декодира комуникационния протокол за четене на данните между дрона и неговия контролер. Тук трябва да се отбележи, че декодирането на комуникационни протоколи без разрешение може да наруши правните норми и разпоредби. Вторият тип идентифицира модела на комуникационния протокол и идентифицира модела на дрона, използвайки неговия RF подпис. За да се постигне това, е необходимо използването на голям набор от данни от RF подписи с цел сравняване на открития RF подпис на дрона със съществуващите в базата данни. Едно от основните удобства на радиочестотното откриване е неговата способност за откриване на БЛА от големи разстояния, както и идентифицирането на модела на БЛА, използвайки неговия RF подпис.

В [7] е предложен алгоритъм за Wavelet Transform за извличане на подписи от RF сигнали, които са били използвани като вход към предварително обучена мрежа, наречена SqueezeNet. В [8] се предлагат алгоритъм за съвместна работа на Feature Engineering Generator (FEG) и многоканален подход за дълбоко обучение с цел откриване и класификация на БЛА чрез RF анализ. Предложеният алгоритъм използва първоначално FEG за предварителна обработка на RF сигнали чрез нормализиране на честотния компонент и намаляване на шумовия ефект чрез прилагане на филтър с пълзящо средно (moving average filter). Предварително обработените RF сигнали се подават на многоканалната техника за дълбоко обучение, като в резултат на това се получава автоматичен избор и класификация на функциите. В [9] е предложен алгоритъм за откриване на летящи БЛА чрез RF пръстови отпечатащи с помощта на дълбоко обучение и агрегиране. Архитектурата на невронната мрежа се състои от AlexNet1D с приблизително 1,1 милиона параметри и ResNet1D с близо 16 милиона параметри. Предложената версия на AlexNet1D съдържа 5 блока от 1D конволюционен слой и слой MaxPooling, последван от два напълно свързани слоя. Предложеният модел съдържа 12 броя идентични AlexNet1D.

В [10] е предложен алгоритъм за откриване на дрон посредством откриване на спектъра на RF сигнал за комуникация с дрона. Използван е CFAR алгоритъм за откриване в честотната област на сигнала.

2.3. Откриване на дроне с видеонаблюдение

Последните изследвания по въпросите за откриване на дроне с видеонаблюдение се концентрират в използването на машинното обучение (ML) и дълбокото обучение (DL). Съществуващите методи могат да се разделят на две категории: едностепенни и двустепенни откриватели. Едностепенните откриватели, известни като откриватели с един поглед, изпълняват както регионално откриване, така и директно класифициране на обекти по техните характеристики, използвайки принципите на обработка в мрежа. Такива откриватели са подходящи за откриване на обекти в реално време, тъй като могат да постигнат висока скорост на обработка. За разлика от тях, двустепенният откривател изпълнява откриването на обекти в две отделни стъпки, като първата стъпка използва мрежа за регионални наблюдения, която идентифицира няколко региона на интерес с вероятност да съдържат търсения обект. Вторият етап е свързан със задачата за класификация. Той класифицира предложените

региони, за да разпознае и локализира целта. Този алгоритъм за откриване не е оптимален за приложения в реално време.

Едно от основните предизвикателства при откриването на БЛА е, че се появяват в различни размери в изображението. Летящ надалеко БЛА може да изглежда като малък обект, който трудно се открива. Друго предизвикателство е откриването на малки БЛА на фона на други малки летящи обекти като птици. В този аспект в литературата се срещат множество алгоритми за откриване на дроне, използващи визуално базирани подходи.

В [11] е разгледан алгоритъм за откриване на злонамерени БЛА, който е базиран на визуален трансформатор (vision transformer – ViT) [12] и е предназначен за компютърно зрение. Решението дали БЛА е злонамерен, или безвреден се взема от това дали БЛА носи, или не носи полезен товар. Моделът ViT прилага преобразуването директно в части от изображението с цел откриване и класификация на обектите. В [13] е предложен модел за откриване на БЛА чрез анализиране на необичайно видеоповедение. Обработката се състои от две стъпки, първата открива наличието на аномално поведение в небето, известно още като откриване на аномалия, докато втората стъпка идентифицира дали откритата аномалия е БЛА. В [14] е предложен комбиниран DCNN метод за прогнозиране на района на търсене за последяване на БЛА. Дронът първоначално се открива с помощта на алгоритъм YOLOv4, а прогнозирането на позицията на БЛА се получава чрез BBox. В [15] е предложен алгоритъм за откриване на дроне с помощта на оптична и инфрачервена камера.

2.4. Акустично откриване на БЛА

Аудиобазирани техники за откриване са свързани с извличане на аудиохарактеристики от безпилотни летателни апарати, получени от микрофони. Сред методите, използвани за извличане на аудиохарактеристики в [16] е предложена антидрон система, базирана на акустични сигнали. Системата има за цел първо да открие летящ дрон в зони с ограничен достъп и да идентифицира по-късно дали дронът е бил разрешен в зависимост от неговия тип или модел. В [17] авторите сравняват ефективността на конволюционни невронни мрежи (CNN) с повтарящи се невронни мрежи (RNN) и конволюционни повтарящи се невронни мрежи (CRNN) относно откриването и идентификацията на дроне с помощта на акустични сигнали. В [18] е предложен аудиобазирен подход за откриване и класифициране на любителски дроне в шумна среда чрез машинно обучение. Авторите са използвали Мел честотни кепстрални коефициенти (MFCC) за извличане на характеристики, както и метод на опорни векторни машини за аудиокласификация. Моделът има за цел да класифицира звуците на любителски дрон от други звуци като птици, самолети и гръмотевични бури. За същата цел в [19] е реализиран подход на неконтролирано машинно обучение за използване на любителско откриване на дроне от акустични сигнали. Авторите са приложили спектралната плътност на мощността (power spectral density – PSD), както и средната квадратична стойност (RMS) на PSD чрез обработване на акустичните сигнали с помощта на лентови филтри. В [20] е разгледана олекотена конволюционна мрежа CNN (LWCNN) за любителско откриване на дроне с помощта на акустични сигнали. Предложеният метод има за цел първоначално преобразуване на аудиосигнали в изображения с помощта на техниката Mel Spectrum. Характеристиките, получени от модела LWCNN, са използвани впоследствие от алгоритъм за класифициране. В [21] се изследват акустични характеристики, извлечени от времевата и честотна област на звуков сигнал с цел откриване и разпознаване на безпилотни летателни апарати. От спецификата на звуковия сигнал произтича и изборът на изследваните аудиохарактеристики. Тези характеристики могат да се използват от методите за машинно и/или дълбоко обучение в изкуствена невронна мрежа за откриване на безпилотни летателни апарати.

2.5. *Откриване чрез обединяване на данни от многосензорни системи*

Авторите в [22] предлагат двустепенен подход за сливане на данни от радар и визуален сензор, който се осъществява в нивата на откриване и проследяване. Първата стъпка, наречена „Fuse-Before-Track“, се състои в съвместно използване на радарна информация и визуални откривания (осигурени от откриватели, базирани на конволюционна невронна мрежа) за премахване на ненужни радарни ехо отражения, като по този начин се подобрява премахването на смущения от земята и се ускорява радарната обработка. Във второто ниво проследяването се осъществява чрез използване на предварително извлечени (потвърдени) радарни измервания и сливането им с визуални открития с цел подобряване на точността на решението. Предложеният подход е тестван върху събрани от авторите данни по време на експериментални полетни тестове. Използвани са мощен радар и камера за дневна светлина за откриване и проследяване на малък дрон, пилотиран ръчно. В [23] авторите създават мултисензорна база данни за откриване на дронове, която включва инфрачервени и видими видео- и аудиофайлове. За да се позволи пручвания като функция на разстоянието от сензора до целта, наборът от данни е разделен на три категории (близко, средно, отдалечено). Като се има предвид, че дроновете трябва да летят във визуален обхват поради разпоредбите, най-голямото разстояние от сензор до цел за дрон в набора от данни е 200 m, а записите са направени на дневна светлина. В [24] е предложена система за наблюдение на дронове, която може да работи ден и нощ. Системата е предназначена за откриване и проследяване на дронове, като е базирана на акустичен и оптичен сензор. Тази система е наречена MUTES и комбинира множество сензорни модули, включително микрофонен масив, камера и лидар. 64-канален микрофонен масив осигурява полусферично наблюдение с високо съотношение сигнал/шум на звука, докато лидарът и видеокамерата позволяват последващо прецизно локализиране на целта. Ефективността на предложения многосензорен подход е валидиран чрез полеви експерименти.

В [25] се предлага метод за откриване и класифициране на безпилотни летателни апарати, използващ схеми за синтез на сензори. За нуждите на изследването са събрани данни чрез полети измервания на дронове с помощта на оптична камера, радар и аудиомикрофон. Използвани са и данни, получени от отворени онлайн бази данни. В първия етап на предложения метод откриването и класификацията на БЛА се извършва с помощта на моделите на конволюционната невронна мрежа (CNN), обучени отделно за оптични изображения, радарни карти и аудиоспектрограми. Резултатите от CNN са комбинирани от мултиномиалната логистична регресия за подобряване на точността на наблюдението на БЛА чрез сливане на оптични, радарни и аудиоданни. В [26] е проектирана и оценена многосензорна система за откриване на дрон. Системата използва камера с рибешко око с цел да се наблюдава по-широка част от небето и насочва другите камери към обекти, представляващи интерес. Резултатите от откриването са допълват от ADS-B (Automatic Dependent Surveillance–Broadcast) приемник, GPS приемник и радарен модул. Резултатите от интегрирането на данните от термокамерата са дали резултати подобни на видеокамерата, дори когато камерата е използвана с по-ниска резолюция. В [27] е предложен метод за откриване на дрон с помощта на микрофон и видеокамера чрез обучение на модели за дълбоко обучение, базирани на изображения и акустични характеристики. За оценяване се използват три метода: визуален, аудиобазирани и обединяване на двата метода. За постигане на по-висока производителност при откриване на дрон от дрон се използва метод за сливане на аудио- и визуални данни, получени от сензори, монтирани на дрон, който лети паралелно с търсения дрон на дистанция от 20 метра. Предложеният метод за обработка на сензорните данни използва методи за дълбоко обучение за изследване на

оптималната производителност. Конволюционна невронна мрежа се използва за обработка на акустични данни, а YOLOv5 се използва за компютърно наблюдение. В резултат на изследването е установено, че най-висока точност на откриване се получава от метода за сливане на сензорните данни.

От всичко казано дотук се вижда, че методите за сливане на данни подобряват значително откриването и класификацията на БЛА.

3. *Противодействие на безпилотни летателни апарати*

3.1. *Радиочестотно заглушаване*

Системите с радиочестотно заглушаване нарушават комуникационните и навигационните системи на дрона [28]. Чрез излъчване на мощен сигнал в честотния диапазон, използван от повечето дронове, устройството заглушава радиочестотната връзка, като не позволява на дрона да получава команди за управление от своя оператор. Също така, системата може да манипулира GPS сигнала, карайки дрона да загуби информацията за позицията си и автоматично да задейства функция за връщане към дома или кацане. Тази технология е ефективна до няколко километра и е подходяща за използване на публични събития в градски и извънградски области. Не е желателно да се използва на летища и други обекти, в които има множество радиокомуникационни системи.

3.2. *Високоенергийни лазерни системи*

Високоенергийните лазерни системи фокусират концентрирани лъчи светлина върху критичните системи на дрона като неговите двигатели, батерии или сензори и го повреждат [28]. Лазерите са много прецизни и точни и неутрализират само нежелания дрон, като не влияят на съседни електронни системи, което ги прави идеални за използване в чувствителни среди.

3.3. *Използване на мрежи и мрежови пушки*

Мрежовите пушки са предназначени да изстрелват мрежа над дрон по време на полет, като го оплитат и го карат да падне на земята [28]. Това е система за физическо улавяне на дрона. Тази система е ефективна при неутрализирането на малки и средни дронове.

3.4. *Дронове прехващачи*

Тези дронове са специализирани и могат да се използват за преследване и улавяне на нежелани дронове посредством изстрелване на мрежа или чрез използване на други средства за деактивиране по време на полет [28]. Дроновете прехващачи са особено ефективни в области с чувствителна инфраструктура.

3.5. *Киберпревземане*

Системите за киберпревземане използват уязвимости в комуникационните протоколи на дрона, за да получат контрол над дрона [28]. Тези системи позволяват да се пренасочи радиоуправлението на дрона от оператора на дрона към персонала по сигурността. Тези системи са най-модерните и усъвършенствани системи за неутрализиране на дрон.

4. *Заклучение*

Системите за откриване и противодействие на нежелани дронове могат да използват разнообразни технологии за откриване, които притежават специфични предимства и недостатъци, както и богат набор от технологии за неутрализиране на неотривизирани дронове в чувствителни зони. Използването на система за защита срещу дронове е от решаващо значение за гарантиране на обществената безопасност, защита на чувствителни места и поддържане на поверителността.

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Анализ на механичните вълнови процеси съпровождащи 122 mm артилерийски изстрел на самоходна гаубица

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Analysis of mechanical wave processes accompanying a 122 mm artillery shot from a self-propelled howitzer

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Abstract: Some of the results of the preliminary studies of the acoustic and seismic signals generated by large-caliber artillery systems are presented. A system of sensors, software and hardware provision developed for the purpose was used. The obtained results show that the parallel use of acoustic and seismic sensors allows the accurate differentiation of different artillery means.

Keywords: SEISMI, SENSORS, ACOUSTIC SENSORS, ARTILLERY SYSTEMS

1. Увод

В процеса на артилерийския изстрел протича импулсна трансформация на химичната енергия на метателния заряд в топлинна, в следствие на което разширяващите се газообразни продукти ускоряват движението на боеприпаса в цевта. При излизане от дулото част от енергията на барутните газове се разпространява под формата на звукови вълни в атмосферата. Паралелно с това при отката на дулото настъпват механични колебания на цялата артилерийска система, които се предават и разпространяват в почвата. Регистрацията и изучаването им са обект на акустиката и сеизмиката. Във военната област акустиката като метод за разузнаване се прилага от Първата световна война и непрекъснато се развива [1]. Разработването на нови типове сензори и компютърни системи за регистриране и цифрова обработка на сигналите в реално време прави акустичните средства и системи в достоверен канал за събиране на разузнавателна информация за артилерийските средства на противника [2]. Ефективна работа на звуковото разузнаване е свързана с предварителното създаване на база данни от акустични сигнатури на противниковите артилерийски средства [3,4,5].

В настоящата работа са представени резултати от предварителни изследвания с използването на комбинация от акустични и сеизмични сензори

2. Изложение

2.1. Цел на изследването

Целта на изследването е да се получат и анализират честотните характеристики на акустични и сеизмични сигнали генерирани от изстрел на 122 mm самоходна гаубица.

Особеностите свързани с настоящето изследване се определят от следните основни фактори:

- честотните характеристики на акустичните и сеизмичните сигнали получени при артилерийски изстрел зависи от масата на системата, заряда, калибър;
- типа на почвата, нейната влажност и нееднородност;
- метеорологични условия, релеф на местността, разстояние от източника.

Допълнителни експерименти биха могли да оценят реалното влияние на част от посочените фактори и да определят насоките за бъдещи детайлни изследвания.

В представения материал са изложени основни резултати и анализи на честотните характеристики на акустични и сеизмични сигнали получавани при изстрел на 122 mm самоходна гаубица.

2.2. Условия за провеждане на изследването

Експериментите са проведени на изследователски полигон, който в основната си част представлява поляна с ниска

растителност. Сензорите са разположени на 25 метра от източника на сигнал.

За регистриране на амплитудно-честотните характеристики се използва следната апаратура:

- пиезоелектрически акселерометър на фирма *Bruel & Kjaer* модел 4370 конструкция *Delta Shear®*, имащ висока чувствителност (*Uni Gain®*) 98,1 pC/g и широк линеен честотен диапазон, граница на отклонение $\pm 10\%$, 4,8 kHz, на Лаборатория по експериментална механика на ИМСТЦХА;
- малолумящ усилвател на заряд на фирма *Bruel & Kjaer* модел 2635 с честотен диапазон 0,1 Hz ÷ 200 kHz, коефициент на усилване от $-20 \div 80$ dB и режими на работа – ускорение, скорост, преместване. Входът на усилвателя на заряд се състои от операционен усилвател с капацитивна обратна връзка, което по същество се явява електронен интегратор, интегриращ постъпващия на входа електрически ток;
- хидрофон на фирма *Bruel & Kjaer* модел 8101 с честотен диапазон 1 Hz ÷ 120 kHz;
- Терц-октавен филтър модел 1614 на фирма *Bruel & Kjaer*;
- Усилвател модел 2607 на фирма *Bruel & Kjaer*;
- тестови стенд (фиг. 1), осигуряващ възможност за едновременно записване на аналогова информация от осем акустични и сеизмични сензори и нейната спектрална трансформация.

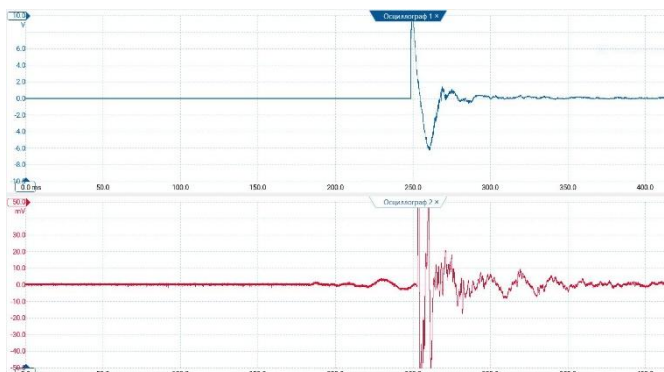


Фиг.1 Тестов стенд при провеждане на експеримента

2.3. Резултати от експериментите

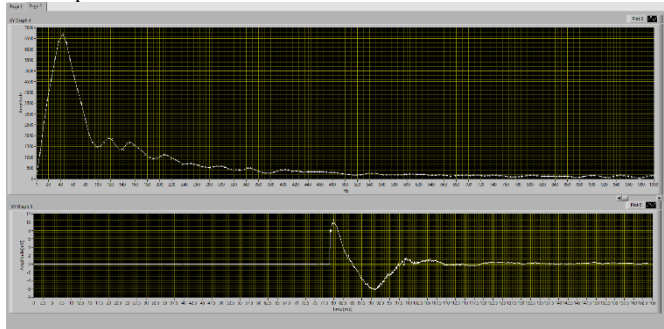
Резултатите от проведените експерименти са архивирани и са обект на анализ от екип на ИМСТЦХА в изпълнение на проект от Научния план на Института на тема «Моделиране и изследване на акустични и повърхностни сеизмични вълни създавани от антропогенни източници».

Най-характерните резултати от проведените изследвания са показани на фиг. 2. На първи канал (акустичен) на тестовия стенд (горна осцилограма), към който чрез усилвателя 2607 и терц-октавния филтър е включен един калиброван хидрофон от тип 8101. На втори канал (сеизмичен) на тестовия стенд (долна осцилограма) е включен пиезоелектрически акселерометър на фирма *Bruel & Кjaer* модел 4370 чрез усилвател на заряд модел 2635.



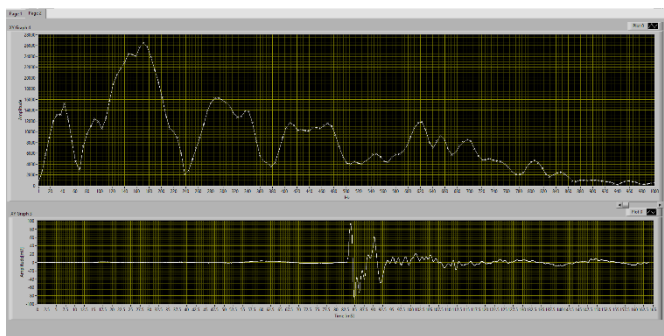
Фиг. 2. Осцилограми на акустичния (горна) и сеизмичния (долна) сигнал стробирани по време

На фиг. 3 е показан вида на акустичния сигнала генериран от изстрел на 122 mm самоходна гаубица и неговия спектър.



Фиг.3. Вид на акустичния сигнала генериран от изстрел на 122 mm самоходна гаубица и неговия спектър

На фиг.4 е показан сеизмичния сигнал и неговия спектър.



Фиг.4. Вид на сеизмичния сигнала генериран от изстрел на 122 mm самоходна гаубица и неговия спектър

2.4. Анализ на резултатите от експериментите

От представените резултати на фиг. 2 се вижда, че сеизмичния сигнал изпреварва акустичния, поради по-високата скорост на разпространение на сеизмичните вълни в почвата. От честотния спектър на акустичния сигнал – фиг.3 се наблюдават пет ясно изразени максимума в следните честотни области: първият с честота 16 Hz и амплитуда 0.5; вторият с честота 40 Hz и амплитуда 1; третият с честота 120 Hz и амплитуда 0.28; четвъртия с честота около 150 Hz и амплитуда 0.25; петия с честота 210 Hz и амплитуда 0.16.

От честотния спектър на сеизмичния сигнал – фиг.4 се наблюдават ясно изразени максимума в следните честотни области: първият с честота 42 Hz и амплитуда 0.56; вторият с честота 90 Hz и амплитуда 0.47; третият с честота 148 Hz и амплитуда 0.9; четвъртия с честота 170 Hz и амплитуда 1; петият с честота 190 Hz и амплитуда 0.8; шестият с честота 340 Hz и амплитуда 0.53 седмият с честота 410 Hz и амплитуда 0.45.

От проведената серия експерименти се наблюдава идентичност в поведението на времевите и честотните характеристики на двата сигнала, което би улеснило идентификацията на дадения обект.

3. Изводи от проведените експерименти

Изпитванията показват, че акустичните вълни генерирани при изстрел на 122 mm самоходна гаубица са силени, резки и разпространяващи се на големи разстояния, с характерни нискочестотни и средночестотни компоненти, а сеизмичните колебания са съсредоточени в честотния диапазон 40 – 410 Hz.

В съществуващите системи за артилерийско разузнаване основен информационен канал е акустичния, така че паралелното използване на сеизмични сензори би допринесло за по-точната локализация и идентификация на обектите.

Наблюдават се честотни максимума и в инфразвуковия диапазон, характеризиращ се с ниска степен на затихване, което представлява практически интерес.

4. Заключение

В настоящото изследване са представени част от резултатите, свързани с изследване на амплитудно-честотните характеристики на акустичните и сеизмичните сигнали, генерирани от артилерийски и стрелкови системи.

Демонстрирана е работоспособността и функционалната пригодност на опитната установка, включваща притежаваните от ИМСТЦХА-БАН калибровани пиезоелектрически сензори на Лабораторията по експериментална механика и разработки за целите на изследването тестов акустично-сеизмичен стенд.

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Tailoring magnetic properties and radiolabeling of magnetite-based nanoparticles for biomedical applications

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Abstract:

This study explores the synthesis, characterization, and radiolabeling of magnesium-substituted iron oxide nanoparticles (MIONs) for advanced biomedical applications, including magnetic hyperthermia, radionuclide therapy, and targeted drug delivery. Mg-doped magnetite nanoparticles ($\text{Mg}_x\text{Fe}_{3-x}\text{O}_4$) were synthesized via co-precipitation and further functionalized with citric acid (CA), oleic acid (OA), and polyethylene glycol (PEG) to enhance colloidal stability and biocompatibility. Structural analysis using X-ray diffraction (XRD) confirmed the spinel structure, while transmission electron microscopy (TEM) revealed particle sizes ranging from 4 to 20 nm, influenced by the Mg content. Surface modifications were optimized to improve dispersibility and minimize cytotoxicity.

Magnetic measurements indicated superparamagnetic behavior at room temperature and spin-glass-like properties at low temperatures. The freezing temperature (T_f) and magnetic saturation (M_s) were found to depend strongly on Mg doping levels and surface coatings. Increased Mg content led to reduced M_s due to the replacement of magnetic Fe^{3+} ions with non-magnetic Mg^{2+} ions. Surface coatings significantly influenced interparticle interactions and anisotropy energy, thus impacting the nanoparticles' versatility in applications.

Magnetic hyperthermia performance of $\text{Mg}_x\text{Fe}_{3-x}\text{O}_4$ was evaluated using a DM 100 nB AC hyperthermia device (nanoScale Biomagnetics, Spain). The intrinsic loss power (ILP) values ranged from 1.39 to 2.01 nHm^2/kg for Mg doping levels of $x = 0-0.2$, demonstrating promising heating efficiency. However, variations in chemical composition significantly affected structural, magnetic, and microstructural parameters, which in turn influenced ILP performance.

Radiolabeling with yttrium-90 (^{90}Y) was achieved using a direct adsorption method targeting surface functional groups. The labeling efficiency exceeded 98% under optimized conditions ($T = 25-60^\circ\text{C}$, incubation time of 10–60 min), as verified by ITLC-SG radiochromatography. Stability studies in physiological media and human serum showed high radiochemical purity ($\geq 96\%$) over 72 hours, indicating excellent stability, minimal radionuclide release, and reduced toxicity.

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Lightweight heavy geopolymer foam based on fayalite slag: influence of alkali concentration on cellular structure

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Abstract: The lightweight cellular building materials has driven significant interest recently due to increasing requirements of energy efficiency of the buildings. This study investigates the potential of fayalite slag, a by-product of copper production, as a precursor for lightweight geopolymer foams. Geopolymer foams were synthesized using a combination of fayalite slag and metakaolin, employing gas forming agents to achieve a porous cellular macrostructure. The influence of alkali concentration was examined on the cellular structure, physical properties and microstructure (FT-IR) of the resulting materials. The findings contribute to the development of sustainable, high-performance geopolymer materials suitable for insulation and fire-resistant applications.

Keywords: GEOPOLYMER, FAYALITE SLAG, LIGHTWEIGHT, FOAM, CELLULAR

1. Introduction

Sustainable practices in construction industry increase exploration of alternative materials that minimize environmental impact. Geopolymers, synthesized through the alkali activation of aluminosilicate sources, have emerged as a promising class of materials due to their potential for reduced carbon emissions and utilization of industrial by-products. Fayalite slag, also known as iron silicate fines, is a by-product of the copper smelting process. Fayalite slag is predominantly composed of iron silicate minerals, fayalite (Fe_2SiO_4) and magnetite. In the past considered as waste material, now fayalite slag has been promoted as valuable by-product finding application in construction sector. Fayalite slag from Aurubis, (Pirdop) has been certified as fine fraction manufactured aggregate, according EN 12620 [1]. Moreover, cement with industrially produced filler has been assessed by EPA recently.

Fayalite slags from copper smelting industry was successfully used to prepare alkali activated materials and geopolymers [2-5]. Our previous studies presented the potential of fayalite slag as raw material for geopolymer preparation [6-10]. The alkali activated fayalite slag was characterized by compressive strength up to 27 MPa [6]. The microstructural studies revealed that fayalite and magnetite stayed predominantly inert, but certain amount of ferric iron (Fe^{3+}) contributes to the structure of the geopolymer gel [6]. The addition of metakaolin to the fayalite slag significantly improved the properties and the compressive strength was measured to 101 MPa after 90 days curing at normal temperature for geopolymer pastes characterized by standard consistence [7]. Moreover, the geopolymer composed of fayalite slag and metakaolin exhibited thermal resistance up to 1150 °C, along with a further increase in compressive strength, reaching 139 MPa [8]. The geopolymer paste based on fayalite slag and metakaolin shows potential for the production of foamed materials.

The development of lightweight geopolymers with cellular structures offers advantages such as reduced density, enhanced thermal insulation, and improved acoustic properties, making them suitable for various construction applications. The characteristics of the cellular structure, including pore size distribution, connectivity, and overall porosity, are critical features of the mechanical and physical properties. The formation of a controlled porous network within the geopolymer matrix is typically achieved through methods such as chemical foaming, pre-made foams, sacrificial fillers, incorporation of lightweight aggregates, additive manufacturing, or other [11].

The most common route of preparation an inorganic geopolymer foams is by addition of foamed agent which generates gas bubbles within the fresh geopolymer gel [12]. The gas is formed due to chemical reactions that produce gas phase such as oxygen, hydrogen, carbon dioxide, ammonium or other [13, 14]. The fresh formed geopolymer foam hardens, resulting in a solid material with cellular structure that provides low density and a reduced thermal

conductivity coefficient. These properties make it well-suited for applications such as thermal and sound insulation, fireproofing, and lightweight construction elements. [15]. Geopolymer foams were synthesized by various precursors such as perlite [16], fly ash [17, 18], silica fume [19] etc. [11, 20]. To the best of our knowledge, no published data is available on geopolymer foams derived from fayalite slag produced in copper smelting plants.

This study aims to investigate the influence of varying alkali concentrations on the cellular structure and the physical properties of lightweight geopolymers synthesized from fayalite slag and metakaolin.

2. Methods and materials

2.1. Method of analysis

The chemical composition was determined by X-ray fluorescence apparatus, Rigaku Supermini 200, using 30 mm pressed tablets.

The IR spectra were analysed using a Bruker Tensor 37 FTIR spectrometer equipped with an ATR (attenuated total reflectance) accessory. The spectra were recorded in the range of 4000–600 cm^{-1} with 128 scans per measurement. The measurements were performed under controlled conditions, ensuring consistent spectral acquisition for all samples.

The density was calculated using hydrostatic weighing method. Water absorption was determined by measuring the dry specimens and then soak in water to constant mass. Density and water absorption was calculated using data from 3 specimens per series.

3.1. Materials

The geopolymer precursors in the present study were fayalite slag and metakaolin. The fayalite slag is a fine powdery material – product of flotation process from flash furnace and converters at the copper producing plant. The fayalite slag was oven dried at 80 °C to constant mass to eliminate residual moisture. The average particle size of the fayalite slag is about 20 μm [1], the absolute density was measured – 3.97 g/cm^3 . The fayalite slag consists of fayalite, magnetite and small amount of pyroxene, determined by powder X-ray diffraction in previous study [21]. Mossbauer spectroscopy revealed that fayalite slag contains also small amount of amorphous Fe^{2+} [22].

Commercial metakaolin, provided by Kaolin EAD, Bulgaria, was used in order to improve the properties of the geopolymer. The wet residue of the metakaolin was 0.40 % on 45 μm sieve. The metakaolin absolute density was measured to 2.26 g/cm^3 . The chemical analysis of the geopolymer precursors is presented at **Table 1**

Table 1. Chemical compositions of geopolymer precursors – fayalite slag and metakaolin

	SiO ₂	Al ₂ O ₃	CaO	K ₂ O	MgO	Fe ₂ O ₃	Na ₂ O	TiO ₂	Other
META-KAOLIN	53.94	43.2	0.15	0.62	0.09	1.14	0.11	0.74	0.01
FAYALITE SLAG	27.71	4.18	3.21	1.57	1.02	58.07	0.43	0.34	3.47

The activator solutions were prepared a day prior by mixing sodium water glass (molar modulus - SiO₂/Na₂O=2.98), potassium hydroxide on pellets and tap water. Five different alkali solutions were prepared with different alkali concentration. The type of the foaming agent was oxygen releasing, based on hydrogen peroxide.

2.3. Geopolymer foam synthesis

In practice, there are no standardized mix design methods for achieving specific properties in foamed concrete. Instead, trial-and-error approaches are commonly employed to develop suitable mixtures, focusing on factors such as water content, foam agent dosage, and binder content. These approaches are generally regarded as adequate for estimating the desired results.

Five series of geopolymer foams were prepared at different concentration of alkali solution and water to solid ratio. Each series was prepared by mixing fayalite slag and metakaolin in weight ratio 5:1 to obtain homogeneous dry mixture. After that activator solution was added and mixture were stirred for 90 second at planetary mixer. After 5 minutes of maturation time, equal amount of foaming agent was added to each series. The amount of oxygen gas release was calculated to about 0,14 dm³, which corresponds to 1.5 g 30% H₂O₂ per 100 g precursor. The mixture was stirred for 60 seconds more and then poured in steel moulds, covered with polyethylene and placed for 24 hours at 80 °C. The specimens were demoulded in 1 day and cured at laboratory conditions. The composition design of the five series is present at Table 2 in molar ratios. The water to solid ratio of series VFM2 was 0.15, where VFM45 was 0.14.

Table 2. Composition design of the prepared geopolymer foams in molar ratios

Series	Molar ratio		
	SiO ₂ /Al ₂ O ₃	Al ₂ O ₃ /M ₂ O	H ₂ O/M ₂ O
VFM2-1.4	5.8	1.40	12.0
VFM2-1.2	5.8	1.20	10.3
VFM2-1	5.8	1.00	8.7
VFM45-1.2	5.8	1.20	9.6
VFM45-1	5.8	1.00	8.1

3. Results and Discussion

3.1. Physical characterisation

The cross-section images of each series are presented at Figure 1. All series contain pores of varying sizes, with macropores observed as a result of pore coalescence, or bigger caverns due to entrapped air during moulding. Clearly, series VFM2-1.4 was characterised by highest porosity. It is visible that the porosity formation decreased with the decreasing the Al₂O₃/M₂O. Thus, high alkalinity affected the foaming process and hinder the pore formation. This correlation was observed at both solid to water ratio tested. The reason for hindering effect of foaming agent with the high alkalinity stays unknown.

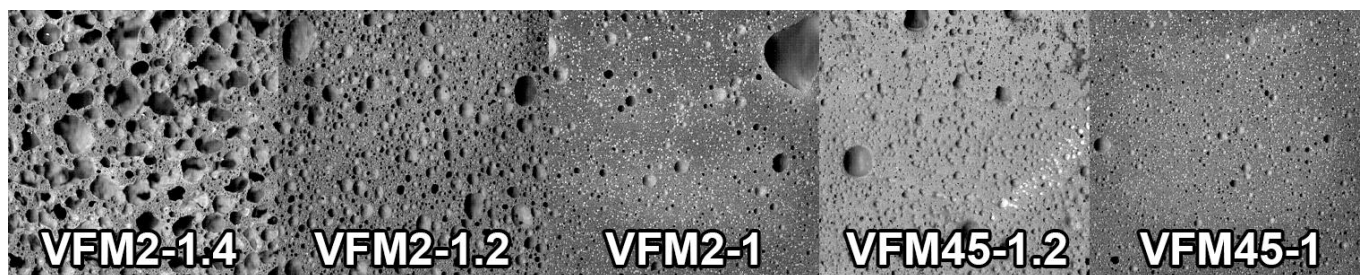
However, the lowest density of 1.09 g/cm³ was achieved at Al₂O₃/M₂O equal to 1.40. The increase of the alkalinity lead to increase of the density, hence reduction of the porosity. At fixed alkalinity (Al₂O₃/M₂O) the decrease of the water to solid ratio led to increase in the density.

The density of non-foamed geopolymer based on fayalite slag and metakaolin was 2.38 g/cm³, at absolute density of 3.59 g/cm³ and calculated relative (total) porosity of 34% [8]. Compared to the density of series VFM2-1.4, the apparent density decreased more than twice. The calculated relative porosity of series VFM2-1.4 is about 70%. Similar relative porosity was obtained with geopolymer foams: based on fly ash and rice husk ash using aluminum powder [23], or natural soil [24]. Higher relative porosity of geopolymer foamed materials were also reported: 83% with coconut ash and alumina slag [25], 83% - fly ash [17]. Despite the high relative porosity of the obtained geopolymer foams based on fayalite slag and metakaolin, the overall density remained relatively high due to the significant absolute density of the mineral phases present in the slag - fayalite (4.39 g/cm³) and magnetite (5.17 g/cm³).

The measured water absorption correlates with the change in densities: water absorption increased by the decrease of the density. Thus, highest water absorption of 26% was measured for series VFM2-1.4. The high water absorption suggest well developed interconnected open porosity.

Table 3. Physical properties (apparent density and water absorption) of the prepared geopolymer foamed material based on fayalite slag and metakaolin

Series	Physical properties	
	Density, g/cm ³	Water absorption, %
VFM2-1.4	1.09	26.0 ± 1.1
VFM2-1.2	1.51	16.2 ± 0.5
VFM2-1	2.07	14.7 ± 0.1
VFM45-1.2	1.84	13.4 ± 0.2
VFM45-1	2.11	13.1 ± 0.1

**Figure 1.** Cross images of prepared series lightweight cellular geopolymers based on fayalite slag and metakaolin. It is observed reduction in porosity at increasing the alkalinity

3.4. Microstructural characterisation

The microstructure was analysed by FTIR and all spectra are presented at Figure 2. The main band at about 1009-1015 cm^{-1} corresponds to the Si-O-T (T = Si or Al) asymmetric stretching vibration of the aluminosilicate geopolymer structure. The peak is key indicator of the polymerization degree in the geopolymer and is associated with the formation of the geopolymer gel. The position of the peak generally shifts depending of Si/Al ratio of the geopolymer gel. Since the ratio Si/Al is fixed at 5.8 band shift correlates with the reactivity of the silica and alumina. The band shift to lower wavenumbers with the decrease of the $\text{Al}_2\text{O}_3/\text{M}_2\text{O}$ ratio indicating more alumina rich structure. The decrease of the water to solid ratio (series VFM45) led to band shift to higher wavenumbers showing less incorporated alumina in the structure. This could be due to lower dissolution of the metakaolin at lower water to solid ratio.

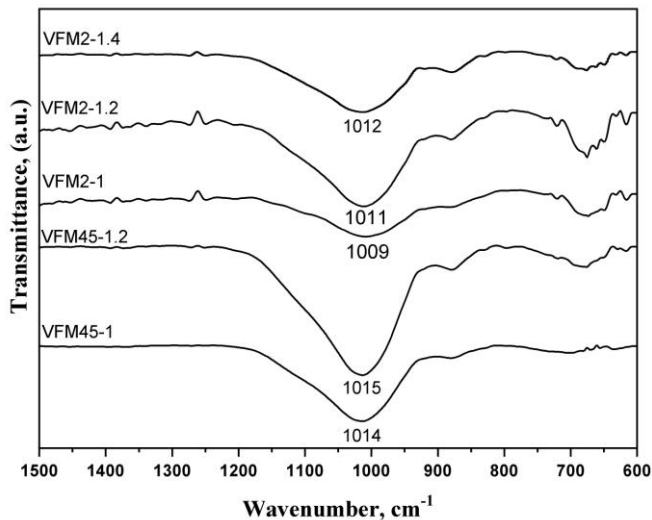


Figure 2. FTIR spectra of the prepared geopolymer foams

3.5. Potential application

The lightweight cellular geopolymers synthesized from fayalite slag and metakaolin exhibit a unique combination of low density, high thermal stability, and well-developed porosity, making them suitable for a variety of advanced applications in construction and industry. One of the most promising applications is in thermal insulation materials for buildings or industrial furnaces. The high-temperature resistance (up to 1150 °C), combined with a controlled porous structure, allows these geopolymers to function as effective thermal barriers, reducing energy consumption in heating and cooling systems. Additionally, their fire-resistant properties make them attractive for passive fire protection in critical infrastructure, such as tunnels, high-rise buildings, and industrial plants. Compared to conventional insulating materials, geopolymer foams derived from fayalite slag offer an environmentally sustainable alternative by utilizing industrial waste while maintaining excellent mechanical and thermal properties.

Beyond construction, the lightweight geopolymer foams have potential applications in sound absorption and filtration technologies. Their interconnected porosity can be optimized for noise reduction panels, especially in urban environments, transportation infrastructure, and industrial facilities where effective sound insulation is required. The porous network within the geopolymer matrix can be designed to act as an efficient medium for adsorbing pollutants, heavy metals, or particulate matter. With further research, these materials could also be explored for energy storage applications, such as in thermal energy storage or as solid electrolytes in next-generation battery technologies. The utilization of fayalite slag in these applications not only addresses waste management challenges in the metallurgical industry but also

contributes to the development of innovative, sustainable materials with multifunctional properties.

4. Conclusion

This study demonstrated the feasibility of producing lightweight cellular geopolymers from fayalite slag and metakaolin by varying alkali concentration and water-to-solid ratio. The results revealed that the alkalinity of the activator solution significantly influenced the porosity, density, and overall physical properties of the geopolymer foams. Higher alkalinity led to increased density and reduced porosity, while lower alkalinity favored the formation of a more interconnected pore network. The lowest density of 1.09 g/cm^3 and highest relative porosity of approximately 70% were achieved at an $\text{Al}_2\text{O}_3/\text{M}_2\text{O}$ ratio of 1.40.

The concept of lightweight heavy geopolymer foam may seem contradictory at first glance, yet it accurately reflects the unique characteristics of the material. Traditional lightweight materials are defined by their low density and high porosity, while heavy materials typically possess high density and mechanical strength. In the case of fayalite slag-based geopolymer foams, this paradox is resolved by the interplay between the intrinsic high density of the fayalite and magnetite phases and the engineered cellular structure formed during the foaming process. The substantial absolute density of fayalite (4.39 g/cm^3) and magnetite (5.17 g/cm^3) ensures that even highly porous specimens retain a relatively high overall density compared to conventional geopolymer foams made from materials like fly ash or perlite.

These findings highlight the potential of fayalite slag based geopolymer foams as sustainable, high-performance materials for insulation, fireproofing, and lightweight structural components. Further research could optimize pore structure control, improve mechanical performance, and expand their applicability in other fields such as energy storage and environmental remediation.

6. Acknowledgement

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Surface-modified polyethersulfon nanofiltration membranes - preparation, properties and biotechnological prospects

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Abstract: In this study, different methods for surface modification of polyethersulfone nanofiltration membranes are explored in view of improving water-ethanol separation properties. Thin copolymer film deposition as well as surface grafting of hydrophilic monomers is applied and modified membranes are characterized by using Fourier transform infrared spectroscopy, optical microscopy, atom force microscopy, etc. Surface properties of the modified membranes revealed by contact angle measurements together with specific surface area and pore distribution are evaluated and compared to those of the base membrane. The influence of structure and composition of the modifying polymer on overall membrane properties is assessed and discussed in view of their potential biotechnological application.

Keywords: NANOFILTRATION POLYETHERSULFON MEMBRANES, SURFACE COATING, PVA COPOLYMER, UV-ASSISTED SURFACE GRAFTING, N-ISOPROPYLACRYLAMIDE

1. Introduction

The use of synthetic membranes in biomedical and biotechnological applications has increased dramatically during the past few decades. Nowadays, membrane technology is widely used in the field of medical diagnostic and practice, gas separation, bioseparation, dairy, desalination of sea water, waste water treatment, and other areas [1,2]. Membrane filtration is also appropriate for application in wine technology - for example, microfiltration is used for clarification of must and wine, whereas nanofiltration, along with reverse osmosis, is used for wine and must concentration, alcohol content correction, aroma enhancement and others [3]. Wine dealcoholisation by nanofiltration is a subject of intensive investigations because this separation technique does not include high-temperature application; it consumes a low amount of energy and does not significantly affect the initial properties of wine.

Nanofiltration membranes are produced from various polymeric materials, usually a strong polymer placed on a supporting layer. Such membrane composition enables high selectivity and endurance during the nanofiltration process. Polyethersulfone is among the polymers employed to prepare nanofiltration membranes because of high mechanical and thermal stability. However, polyethersulfone is a hydrophobic polymer that makes low permeability for the membranes with an increasing trend of fouling. On the other hand, it is well known that surface properties of polymer materials are of fundamental importance in many branches of industrial applications. Performance of a membrane is strongly dependent on the surfaces properties and it is very natural that much attention is paid to the membrane surface modification. A wide variety of physical or chemical methods for membrane surface modification can be found in the literature, which can be summarized into three main approaches: physical adsorption of different components to the membrane surface; graft polymerization that chemically attaches different monomers to the membrane surface; and plasma treatment, that introduces different functional groups to the membrane surface [4].

In recent studies, conventional nanofiltration membrane NP030P has been tested for membrane separation of model water-ethanol mixtures and red wine of selected Bulgarian variety. It is found that the flux of water-ethanol mixtures is lower as compared to water. Improved separation efficiency towards ethanol and higher permeate flux is observed with increasing transmembrane pressure [5,6]. Based on the obtained results and considering modern trends in the membrane developments, a useful method for further improvement of the permeability and selectivity of the membrane would be alteration of its surface hydrophilic-lipophilic balance. The investigations are related to the project "Valorization of valuable bioactive substances and ethanol from grape products through innovative nano- and ultrafiltration membrane processes", supported by the Bulgarian National Science Fund.

This paper reports on the preparation and properties of surface-modified polyethersulfone nanofiltration membranes designed for separation of water-ethanol mixtures. Commercial flat sheet polyethersulfon nanofiltration membrane was used as a base and its surface hydrophilic-hydrophobic balance is changed by applying two different approaches: 1) surface coating with thin layer of hydrophilic polymer - partially acetalized poly(vinyl alcohol); and 2) UV-assisted surface grafting of N-isopropylacrylamide.

2. Materials and Methods

3.1. Materials

NADIR® NP030 flat sheet membranes are provided by MICRODYN-NADIR GmbH (Wiesbaden, Germany). Poly(vinyl alcohol) (PVA, approx. Mw 72000 g/mol) is purchased from Fluka Chemie GmbH. Monomer N-isopropylacrylamide, other reagents and solvents for polymer synthesis and grafting are supplied by Sigma-Aldrich (Saint Louis, MO, USA).

3.2. Synthesis of Modifying Polymer

PVA copolymer used in this work was synthesized according to procedures described elsewhere [7]. In brief, a chosen amount of acetaldehyde was added drop wise to an aqueous PVA solution at 15 °C. The reaction was let to proceed under stirring at 30 °C for four hours. The copolymer obtained was isolated by precipitation increasing the temperature of the solution to 60 °C, purified by several dissolution-precipitation cycles in deionized water, and finally isolated by freeze-drying. Chemical structure of obtained copolymer was analysed by NMR spectroscopy. Copolymer composition as determined from the integrated ¹NMR spectrum showed 10 mol. % degree of acetalization,

3.3. Methods

The Fourier transform infrared (FTIR) spectra were recorded using an IRAffinity-1 spectrophotometer (Shimadzu Company, Kyoto, Japan) equipped with an MIRacle™ ATR accessory (diamond crystal; PIKE Technologies, Madison, WI, USA) providing the depth of penetration of the IR beam into the sample of about 2 µm. All the samples were scanned over the wave number range from 4000 to 600 cm⁻¹ performing 50 scans at a resolution of 4 cm⁻¹. The NMR analyses were performed on a Bruker Avance II+ 600 NMR spectrometer (Billerica, MA, USA). The proton spectra were measured in deuterated dimethyl sulfoxide (DMSO-d₆). Clouding curve of copolymer aqueous solutions of concentration 1 wt. % was registered by means of UV-VIS spectroscopy measuring transmittance at wavelength 500 nm as a function of temperature. Cloud point temperature (T_{cp}) was calculated at transmittance level of 50%.

Modified membrane surfaces were inspected using optical microscope Leica DMLP. Mechanical properties of the modified membranes were evaluated by determining stress-strain behavior of

the samples in tension on INSTRON 1185 apparatus at a speed of 100 mm.min⁻¹ according to ISO 527-3.

Contact angle measurements were applied for evaluation of the properties of the surface modified membrane derivatives. Easy Drop DSA20E KRÜSS GmbH apparatus (Hamburg, Germany) was used to determine the contact angle values. A droplet (10 µL) of water was delivered on the membrane surface and the contact angle value was assessed as an average of at least 5 measurements for each specimen. Specific surface area analysis was conducted on a Quantachrome NOVA 1200e instrument (USA). Low-temperature nitrogen adsorption was measured at -196°C and specific surface area of membranes was calculated using the BET method. The pore size and distribution was analysed using the BJH method.

3. Results and Discussion

3.1. Modification of Membrane Surface by Coating

The membrane surface can be modified by contacting with solution of modifying polymer. A thin layer of modifying polymer is left on top of the membrane after solvent evaporation. Some post-treatment (e.g. annealing or cross-linking) can also be applied.

In general, poly(vinyl acetal)s are class of polymers obtained by reaction of PVA with aldehydes, especially formaldehyde, acetaldehyde, and butyraldehyde finding advanced application as structural adhesives in the aircraft industry, as the interlayer in automotive safety glass, etc. [8]. The contents of unreacted hydroxyl groups along with the acetal rings and the molecular weight determine the polymer properties. Hydrophilicity of partially acetalized PVA is reduced while maintaining its inherent physicochemical and film-forming properties. Noteworthy, the presence of unreacted hydroxyl groups enables further chemical reactions, including chemical cross-linking.

As mentioned above, one of the approaches to membrane surface modification explored in this work comprises coating with film of hydrophilic polymer. For this purpose, partially acetalized PVA copolymer of optimal macromolecular characteristics in terms of chemical composition and hydrophilic-lipophilic balance was synthesized based on previous experimental results [9]. Schematic presentation of acetalization reaction and structure and chemical composition of obtained copolymer are shown on Fig. 1a. The increased hydrophilicity of the copolymer as compared to neat PVA is demonstrated with its clouding curve in water solution (Fig. 1b). While PVA is completely soluble in water over the entire temperature range, the copolymer aqueous solution exhibits thermoresponsive properties and turn opaque when heated above the critical temperature.

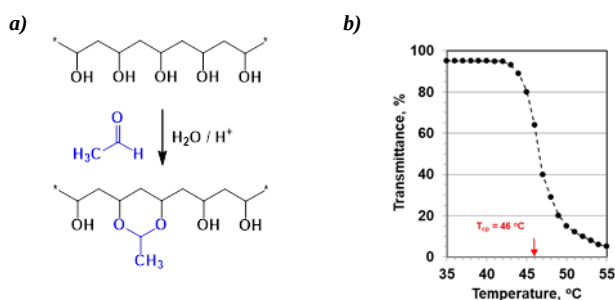


Fig. 1: a) Reaction scheme representing synthesis of modifying PVA copolymer; b) clouding curve of modifying PVA copolymer (1% aqueous solution) illustrating thermoresponsive behavior.

Copolymer solution of concentration 2.5 wt. % was prepared using water-ethanol mixed solvent (8:2 volume ratio). Immediately before modification, glutaraldehyde was added to the solution in order to crosslink coated film thus providing stability and durability of the modified membrane. Thin layer of polymer solution was evenly spread on the membrane surface and modified membrane

was dried in a desiccator at room temperature for 24 hours, washed with water, dried again in desiccator and finally annealed at 100 °C.

ATR-FTIR analysis was applied to obtain information about functional groups, identify compounds and bonds present in the membrane structure. FTIR-spectrum of top surface of the modified membrane is shown in Fig. 2 in comparison to the spectrum of original Nadir NP030P. Characteristic bands [10] for modifying PVA copolymer coated on the surface are clearly seen in the spectrum and confirm successful top surface modification.

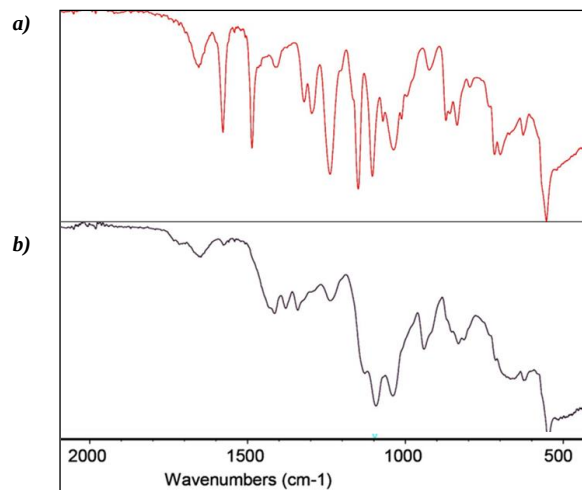


Fig.2: ATR-FTIR spectrum of surface of modified membrane (a), as compared to the spectrum of original Nadir NP030P membrane (b).

AFM analysis of the surface of modified membrane shows much smaller surface roughness compared to the original Nadir NP030P membrane (Fig. 3). This observation can be explained with excellent film-forming properties of modifying PVA copolymer and its good adhesion with selective polyethersulfon layer of original membrane. Contact angle analysis was employed to investigate the hydrophilicity and surface wettability for modified membrane as compared to the original membrane. (Fig. 3).

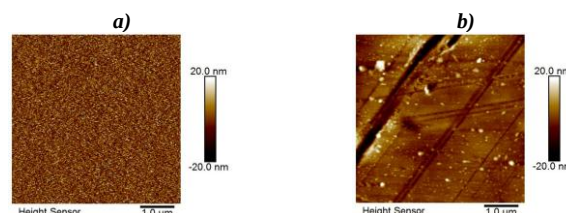


Fig.3: a) AFM analysis of surface of modified membrane; b) AFM analysis of surface of original Nadir NP030P membrane.

Table 1: Contact angle measurements

	Water contact angle [°]	Image
Modified membrane	41.3 ± 2.5	
Original Nadir NP030P membrane	66.8 ± 3.5	

Coating of polyethersulfon nanofiltration membrane with a thin cross-linked film of amphiphilic partially acetalized PVA copolymer shows significant potential for altering its surface properties to more hydrophilic. The method provides composite membranes of good mechanical properties and increased hydrophilicity with potential application in wine technology and/or separation of water-ethanol mixtures.

3.2. Modification of Membrane Surface by Grafting

UV-assisted graft polymerization is a versatile method for membrane surface treatment due to its high efficiency, simplicity, and random distribution of grafted chains near the surface without damaging the bulk membrane structure.

The mechanism of UV-assisted surface grafting of a monomer from a synthetic membrane surface is presented on Figure 4a). When UV irradiated, benzophenone (BP) molecules are excited to a singlet state and then jump to a triplet state by intersystem crossing. BP in a triplet state undergo hydrogen-abstrating reactions from substrates, providing surface radicals (R•) capable of initiating surface graft polymerization [10, 11].

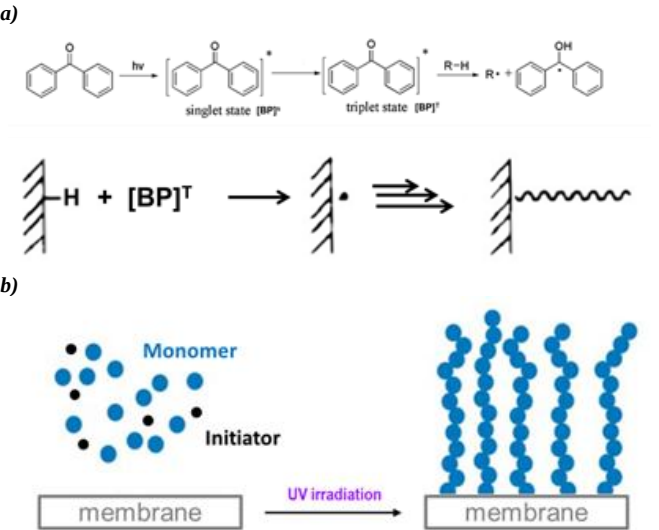


Fig. 4: a) Reaction mechanism of UV-assisted initiation of graft polymerization from membrane surface; b) Schematic illustration of the process of membrane modification.

The complete process of membrane surface modification is illustrated on the scheme. Monomer, N-isopropylacrylamide (NIPAAm) and initiator benzophenone were dissolved in isopropanol as a polymerization media. The solution was applied on the membrane surface and modification was carried out for 1 hour under UV irradiation. modified membrane was dried in a desiccator at room temperature for 24 hours, washed with water, dried again in desiccator and finally annealed at 100 °C.

The extent of modification was followed by attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR). As it is observed in FTIR spectrum of surface of PNIPAAm-grafted membrane (Fig. 5), most of the bands characteristic for selective polyethersulfone layer are overlaid or suppressed. This is expected considering the fact that the penetration of the IR beam into the sample is of about 2 μm when using ATR-FTIR method.

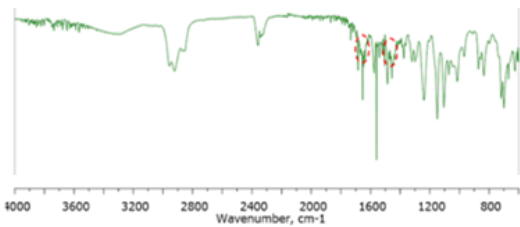


Fig.5: ATR-FTIR spectrum of surface of PNIPAAm-grafted membrane

The surface of the obtained top grafted membrane was characterized by optical microscopy as well as atom force microscopy (Fig. 6). It is clearly seen that the surface roughness of grafted sample is similar to those of original membrane.

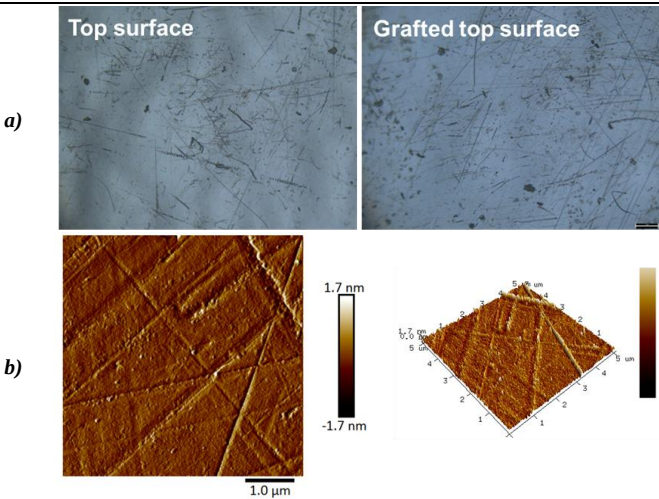


Fig.6: a) Optical microscope images (X100) of top surface of original and PNIPAAm-grafted membrane; b) AFM analysis on the surface of PNIPAAm-grafted membrane.

The result of the contact angle estimation for PNIPAAm-grafted surface is 72° as shown in Table 2. Here also the comparison with water contact angle for the original membrane indicates that the hydrophilic-hydrophobic balance is similar and even changed slightly to hydrophobic.

Table 2: Contact angle measurments

	Water contact angle [°]	Image
PNIPAAm-grafted membrane	72.0 ± 1.5	
Original Nadir NP030P membrane	66.8 ± 3.5	

However, water uptake measurements of the modified membranes confirmed that grafted PNIPAAm chains induce considerable increase of the membrane hydrophilicity (Fig. 7a). This observation can be assigned to spreading of grafted PNIPAAm chains not only on the surface, but also (or even predominantly) within the selective top layer of the membrane. Such phenomenon could also explain the results obtained from ATR-FTIR and contact angle measurements already discussed. In addition, it should be noted that grafted PNIPAAm is a temperature-responsive polymer as confirmed by clouding curve in Fig. 7b. This will open possibility to control to some extend the membrane performance by changing temperature regime during filtration experiments.

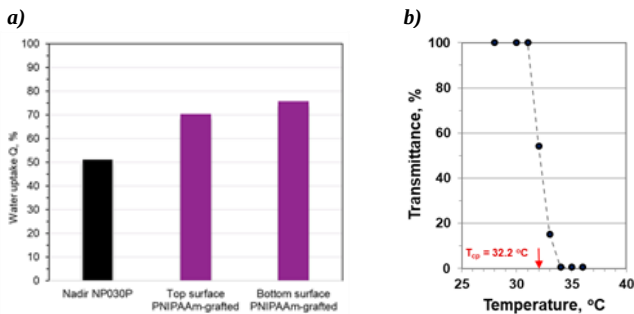


Fig. 7: a) Water uptake of PNIPAAm-grafted membrane compared to the original NP030P membrane; b) clouding curve PNIPAAm homopolymer (1% aqueous solution) illustrating thermoresponsive behavior.

4. Conclusions

The successful top surface modification of commercial polyethersulfone nanofiltration membrane was demonstrated in view of potential application in biotechnological field for separation

of water-ethanol mixtures. Amphiphilic PVA-copolymer obtained by partial acetalization was used to modify the selective top surface of the membrane by physical adsorption from aqueous solution combined with chemical cross-linking. In alternative approach, UV-assisted grafting was chosen as surface treatment method due to its high efficiency, simplicity, and distribution of grafted chains near the surface without damaging the bulk membrane structure. It was revealed that grafting of PNIPAAm chains from the membrane top surface results in considerable increase in the overall membrane hydrophilicity as proved by water uptake experiments. However, surface angle measurements do not confirmed considerable change of the hydrophilic-hydrophobic balance on the surface. Evaluation of permeation and ethanol rejection of modified membranes is currently under investigation in order to elucidate the impact of modifying polymer's chemical composition and structure on the membrane properties.

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On the tribological characterization of novel SiNb and SiW cast irons

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Abstract: In this study, the tribological behavior of new generation cast irons (SiNb and SiW) developed as an alternative to cast irons containing high silicon and molybdenum (SiMo) is investigated under dry friction conditions. All cast irons are produced by sand mold casting as Y blocks according to ASTM A536-84 standard and metallurgical characterization studies have revealed that they all have spheroidal graphite and dispersed carbides like Mo-rich M_6C , Nb-rich MC and W-rich M_6C type depending on the alloying element, within a ferritic matrix. Although no significant change is observed in the spherical morphology of graphite in cast iron matrices, a significant change is observed in the amount of graphite and image analysis studies reveal that the graphite content (area-%) in SiNb and SiW cast irons is 4,02 and 4,30, respectively, compared to SiMo cast iron (5,80). A significant change in the hardness of cast irons is also determined depending on the microstructural features; SiNb (228 ± 7 HV10) and SiW (218 ± 5 HV10) cast irons have higher hardness values compared to SiMo cast iron (192 ± 5 HV10). Cast irons and alumina ball as counterpart material are subjected to a tribological interaction for 150 m under dry friction conditions at a nominal load of 10 N and a ball sliding speed of 0.08 m/s and the findings indicate that (i) the coefficient of friction (CoF) decreases as the graphite content increases, with SiMo having the lowest CoF (0.023), followed by SiW (0.025), and SiNb showing the highest CoF (0.040), (ii) the specific wear rate increases as the hardness decreases, therefore, SiMo has the highest specific wear rate, whereas SiNb demonstrates the lowest specific wear rate and (iii) adhesive wear is the dominant wear mechanism for all ductile cast irons due to the presence of their ferritic matrix.

Keywords: CAST IRON, MICROSTRUCTURE, TRIBOLOGY, CHARACTERIZATION

1. Introduction

Nodular graphite cast irons are materials with high tensile strength, high plastic deformation ability and fatigue resistance, and with these properties they are preferably used in many industrial applications, like the automotive industry [1], especially in engine blocks, exhaust manifolds, gearboxes, turbocharger/pump components [2, 3]. It is a fact that the automotive industry needs to take many precautions (i.e. reduced CO₂ emissions) in accordance with the environmental regulations. However, it is inevitable that the systems aim to operate at higher temperatures and thus increase fuel efficiency and compression ratio [4, 5]. This brings the need for materials with high metallurgical/mechanical stability that are resistant to oxidation/corrosion, especially at higher temperatures, for engine components. Although these properties are largely achieved by stainless steels and nickel-based materials, spheroidal graphite cast irons come to the fore commercially as the mostly used materials due to their low costs [3, 6, 7].

Among spheroidal graphite cast irons, SiMo cast irons are the most widely used materials for engine components. During the production of cast iron, the effect of inoculants on graphitization tendency and high nodularity graphite formation is undeniable [8-10]. Graphitization of SiMo cast iron is promoted due to the presence of silicon in the chemical composition, and at the same time a ferritic matrix is obtained [11]. With silicon, the austenite transformation temperature can be shifted, and thus microstructural and mechanical stabilization can be achieved at higher temperatures. Although the yield strength increases due to the increase in the amount of silicon, toughness and elongation (%) decrease [12]. The matrix structure of this type of cast iron is essentially ferritic, and pearlite, graphite and Mo-rich carbides (M_6C) can also be found embedded in the matrix [13]. On the other hand, with the contribution of molybdenum, a significant increase in the high-temperature properties of this cast iron can be achieved. It is also possible to increase the oxidation resistance, creep resistance and high temperature wear resistance of SiMo cast iron by increasing its molybdenum content. It is inevitable that molybdenum will be segregated to the cell boundaries in solidification and thus boundary carbides will be formed. Although these carbides increase tensile strength, creep and corrosion resistance, they significantly reduce the plasticity of the material [6, 14, 15].

In recent years, an intense effort has been made to further improve the high temperature properties of SiMo cast iron on one hand and mechanical properties on the other. While many studies focus on the modification of SiMo cast iron with elements such as

Al and Cr, some studies focus on the development of new generation cast irons using thermodynamic approaches. Among the new generation cast irons, SiNb-xAl and SiW-xAl, having 0-4% Al by weight, are noteworthy. Compared to SiMo cast irons, the aforementioned cast irons have a higher A₁ temperature and higher oxidation resistance. Considering the mechanical properties of these materials, it is possible that elements such as Nb and W contribute to the formation of hard carbides and that the ferrite phase is hardened with the addition of aluminum [13, 14, 16, 17].

In this study, the tribological characterization of the new generation cast irons, which offer an advantage compared to SiMo cast irons in terms of high temperature use, is carried out and the effect of microstructural features on wear resistance is examined. The findings should be considered as preliminary data for the usability of new generation alloys as wear-resistant spheroidal graphite cast irons as alternatives to SiMo material in industrial platforms.

2. Materials and methods

All cast irons are produced by sand mold casting as Y blocks according to ASTM A536-84 standard. A laboratory scale induction furnace (Inductotherm, 35 kW) of 25 kg capacity is used for melting and casting is carried out from a temperature of 1560 °C. The chemical compositions of the cast irons are verified using an optical emission spectrometer (OES, Foundry Master) and the results are listed in Table 1.

Table 1: Chemical compositions (wt.%) of the studied cast irons.

Cast iron	C	Si	Mn	Mo	Nb	W
SiMo	3.51	4.15	0.40	0.86	-	-
SiNb	3.57	4.04	0.23	-	0.96	-
SiW	3.45	4.12	0.21	-	-	0.92

Samples are taken from the bulk materials, and their surfaces are etched with 3% nital after grinding and polishing. Then the solidification structures are characterized using a light microscope (LM, Olympus BX41M-LED) and quantitative analysis is carried out according to ISO 945-2, by an image analyzer (Leica Las V4.12) in order to determine the area % of graphite embedded in the solidified structure. Hardness is determined as the primary mechanical property of cast irons, and tests are carried out under 10 kgf load using a micro Vickers hardness tester (Emco-Test).

Cast irons and alumina ball as counterpart material are subjected to a tribological interaction for 150 m under dry friction conditions at a nominal load of 10 N and a ball sliding speed of 0,08 m/s. After dry friction tests, the change in the coefficient of friction for each cast iron is monitored as a function of sliding distance. Specific wear rates are calculated by determining the volume losses occurring during the tribological interaction in accordance with the ASTM G133-05 standard. The wear surfaces are characterized microscopically, and the dominant wear type for each cast iron in tribological interaction is examined.

3. Results and Discussion

Figure 1 shows the etched microstructures of the cast irons. Graphite (G), Mo-rich carbide and partial pearlite (P) phase regions are observed in the ferritic (F) matrix in the SiMo cast iron structure (Figure 1a). In the solidification structure of SiNb cast iron (Figure 1b), it is observed that graphite and pearlite exist in the ferritic matrix and that the matrix also contains Nb-rich carbide. Similar phase components are found in the solidification structure of SiW cast iron (Figure 1c), and it has been observed that W-rich carbides are dispersed in the ferritic matrix due to the presence of another carbide-forming element i.e. tungsten in the composition.

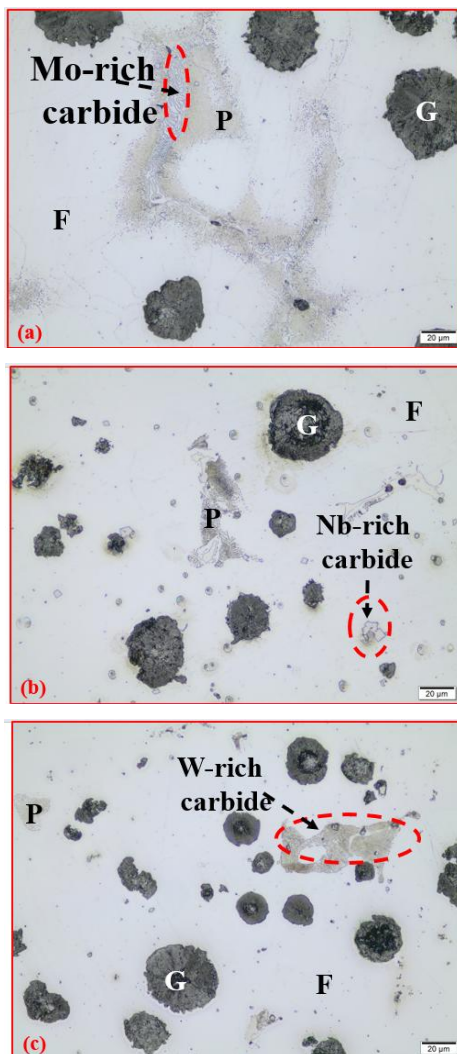


Fig. 1. LM images showing the microstructural features within the cast irons; (a) SiMo, (b) SiNb and (c) SiW.

The morphologies of the graphite found in the microstructure of such cast irons are affected by possible chemical composition changes and the morphological change can be monitored. Although no significant change is observed in the spherical morphology of graphite in cast iron matrices, a significant change is observed in the amount of graphite and image analysis studies reveal that the

graphite content (area-%) in SiNb and SiW cast irons is 4.02 and 4.30, respectively, compared to SiMo cast iron (5.80). It is inevitable that the change in the type and amount of carbide, as well as the change in the amount of graphite in the ferritic matrix, will affect the mechanical properties. Thus, a significant change in the hardness of cast irons is also observed depending on the microstructural features; SiNb (228 ± 7 HV10) and SiW (218 ± 5 HV10) cast irons have higher hardness values compared to SiMo cast iron (192 ± 5 HV10).

CoF changes as a function of sliding distance in the tribological interactions of cast irons with a hard ceramic material are given in Figure 2. Among the cast irons tested, SiNb cast iron exhibits the highest coefficient of friction (0.040) over the total sliding distance. In tribological interaction, the amount of graphite in the matrix that can provide a lubricating effect can be a determining factor in the change of CoF values. In Figure 3, the effect of the amount of graphite in the matrix on the CoF values of each cast iron in the steady state is correlated and the findings indicate that CoF decreases as the graphite content increases.

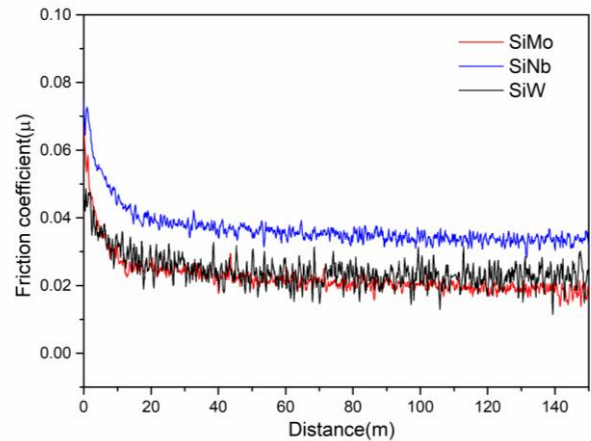


Fig. 2. COF variation for cast irons as a function of sliding distance.

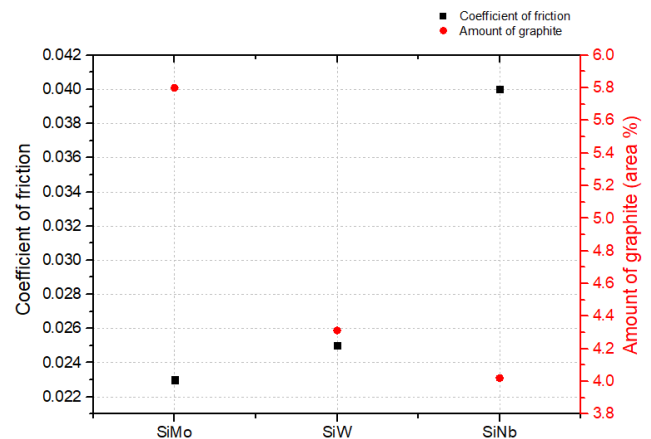


Fig. 3. Relationship between CoF and graphite content values of the studied cast irons.

In tribological interaction, hard phases like carbides that may be present in the matrix have a direct effect on material loss during wear. Primary Nb-rich carbides found in the solidification structure of SiNb cast iron are faceted hard phases that can crystallize directly from the liquid phase. These types of carbides can be harder than Mo-rich and W-rich carbides, which solidify from liquid by eutectic reaction. Although the pearlite phase with lamellar morphology in the ferritic matrix also contains carbide like cementite, primary and eutectic carbides, which have a coarse distribution within the solidified structure, are more decisive in the final hardness of the material. In Figure 4, the relationship between the hardness values of cast irons and the specific wear rates calculated after tribological tests is diagrammed. The findings indicate that there is a decrease in specific wear rates due to the

increase in material hardness. After tribological tests, LM examinations are carried out on the worn surfaces and the obtained micrographs are given in Figure 5. Worn surfaces typically indicate the presence of two tribological patterns along the sliding direction; (i) scratches due to abrasion and (ii) layers due to adhesion. Such patterns are mostly observed in ferritic matrix cast irons. It should be noted that abrasive wear occurs when the cast iron is scratched by a hard counterpart material and particles separated from the surface move as free bodies on the surface and may cause re-scratching. Although the surface roughness can be reduced under friction conditions, it is possible for cold metal to adhere to the surface due to adhesive wear. Adhesion causes a layer to form on the surface and it is also possible for the layers to undergo delamination and separate from the surface.

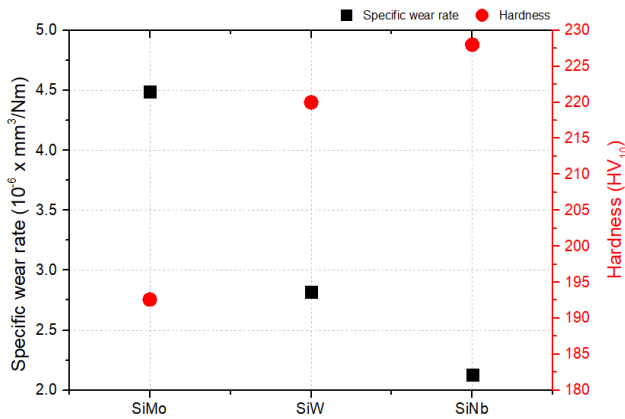


Fig. 4. Relationship between specific wear rate and matrix hardness values of the studied cast irons.

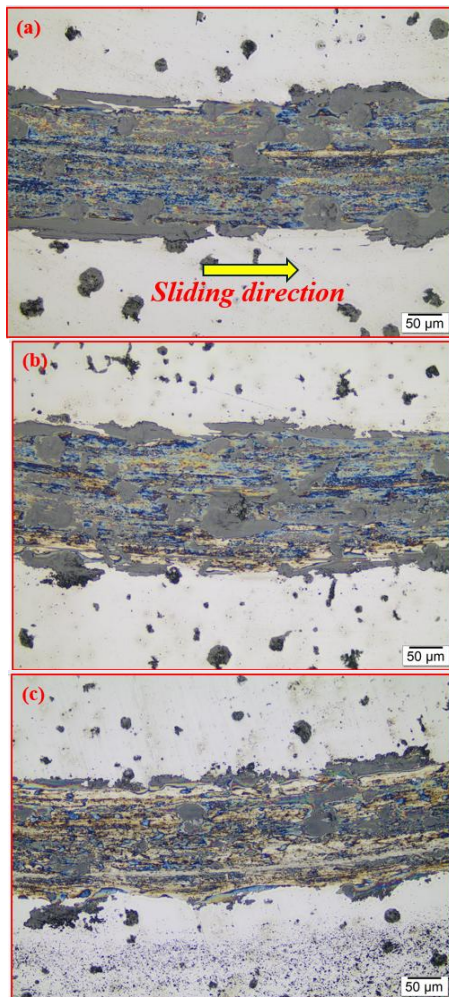


Fig. 5. LM images showing the worn surfaces of the cast irons; (a) SiMo, (b) SiNb and (c) SiW.

4. Conclusion

In this study, the tribological behavior of new generation cast irons (SiNb and SiW) developed as alternatives to cast irons containing high silicon and molybdenum (SiMo) is investigated under dry friction conditions. The findings reveal the presence of graphite phase with spheroidal morphology and pearlite phase in the ferritic matrix, together with carbides, with varying hardness and morphology, of the carbide-forming elements (i.e. Mo, Nb and W) in the chemical composition. Compared to SiMo cast iron, lower graphite amounts are detected in the solidification structure of new generation cast irons. A significant increase is observed in the CoF values of cast irons due to the decreasing amount of graphite under dry friction test conditions. Such a finding is evaluated as an indicator that high CoF values would be achieved if the lubricating effect of graphite decreases. However, the effect of matrix hardness on tribological interaction, especially on the specific wear rate, is observed quite clearly. Novel cast irons have higher hardness values and relatively lower specific wear rates compared to SiMo cast iron. Characterization studies on wear tracks indicate that adhesive wear is the dominant wear mechanism for all ductile cast irons due to the presence of their ferritic matrix.

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Force-Stroke Analysis of Metallic Materials using the Upper Bound Method

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Abstract: All plastic deformation applied to metallic materials create a metal flow. The main factors affecting metal flow are the friction conditions between the metallic material and the mold. In addition upset forces are closely related to the geometric shapes of the metallic materials that changes deformation rates. It is possible to express the formation rates with mathematical approaches in shaping metallic materials. However obtaining these mathematical approaches can be done with very complex methods. Numerical and analytical expressions are generally used in the mathematical approaches. Some of the approaches applied to obtain more accurate results are slip lines methods, finite elements analysis, finite difference methods and Boundary element methods. In this study the upper bound method was applied for metallic materials. The force-stroke diagrams were obtained by the investigation of deformation energy and friction losses. The analyzed parts were prismatic specimen with arbitrary profiles and the results obtained were supported by graphics.

Keywords: PLASTIC DEFORMATION, FORCE-STROKE ANALYSIS, UPPER BOUND METHOD

1. Introduction

Metal plastic shaping is a common process that has been done for many years. Upsetting is one of the most commonly used metal forming methods. It is based on the principle of changing the shape of the metals without changing their volume with the help of force and pressure. This application area, which is extremely important in industry and applications, can be done more easily with ductile materials that can be easily shaped. It means that the length of a stacked piece is shortened by the effect of the compressive force applied to it in the direction of its axis and the section is widened in the direction perpendicular to the direction of force. While the volume of the material does not change after upsetting, geometrical changes may occur in the material due to strain. The upsetting ratio is generally measured as the percentage reduction in length for cylindrical parts. Performing force analysis before the upsetting process positively affects the upsetting results. This prevents unexpected results and errors. In order to perform force analysis, the stamp force must be calculated depending on the upsetting rate. These calculations can be made thanks to some methods. One of these is the upper limit method, and this method can be applied to a part whose cylindrical coordinates are given. In the application of this method, stacking ratio and friction coefficients are taken as basis.

Gunasekera and Hoshino (1985) examined curvilinear profile molds to examine the extrusion of polygonal profile products. Curvilinear profile molds facilitate material flow and improve the forming process. Upper limit analysis was carried out for curvilinear profile and linear shrinking molds with 60% extrusion rate and 0.1 friction factor values and solution graphics were obtained. The results show that in curvilinear profile molds, the power component from plastic forming becomes dominant when the mold length decreases, and this force decreases suddenly when the mold length increases. In contrast, in straight shrinking dies the force component remains almost constant throughout forming. The study also revealed that the optimal pattern length of curvilinear patterns is shorter. The obtained theoretical results were confirmed by experiments with lead samples at room temperature. The result shows that dies with curvilinear profiles are more efficient and effective in extrusion processes [1].

Yang, D.Y., et al. (1986) stated in their study that in molds with inlet and outlet sections of different profiles, it is difficult to make the analytical expression of the mold surface connecting these sections in cylindrical coordinates. The study aims to solve this problem by proposing a transition function $R(\theta,0)$ and $R(\theta,L)$ that defines the input and output sections as $R(\theta,z)$, respectively. The transition function was chosen to define the input section for $z=0$ and the output section for $z=L$ and was obtained using a certain number of Fourier series. Thanks to this transition function, a new velocity field was defined and the results obtained for exit profiles

with elliptical and rectangular cross-sections were compared with experimental data using the Al 2024 material, using the upper limit method. The results showed that the surface quality of the resulting products was quite good. It was also observed in the study that the results obtained with the upper bound method were slightly higher than the experimental results. This difference is partly due to the fact that the profile with a square cross-section has a larger circumferential length of the surrounding circle than the elliptical profile, requiring more energy for shaping. This study improved the definition of the transition function and verified the results obtained by the upper bound method through experiments [2].

In this study, Narayanasamy et al. (2006) obtained the results of the transition from circular billet to circular cross-section exit profile and cosine profile patterns using the upper limit method and examined them compared to previous studies. The upper limit analysis was made by expressing the path between the particle's mold entry and exit profiles as a function in the Cartesian coordinate system, and the velocity fields were calculated through this function. This method has been used to predict plastic deformation. When the results obtained in the study were compared with the calculations of flat shrinking molds and convex circular molds under frictionless conditions, it was found that the cosine profile required lower extrusion pressure and was therefore more advantageous than other molds. In addition, calculations made with similar die profiles are presented graphically according to relative extrusion pressure and die length changes, with power amount comparisons made using a flat shrinking matrix. According to these comparisons, it has been shown that cosine transition profiles have lower amounts of power spent for plastic deformation and friction, and this power is less for each value according to the mold length [3].

Bakhshi-Jooybari, M. et al (2007) proposed a new approach combining cap and slice method to estimate strength in cold extrusion of aluminum and lead samples. In the study conducted with this method, the authors used conical molds they had previously designed for comparison purposes. The results obtained with the proposed new method were compared with experimental data and solutions obtained with the commercial finite element software. When the experimental force-stroke diagrams were examined, it was observed that the combined method was compatible with simulation and experimental results. Additionally, it was determined that when the optimum curvilinear die was used for aluminum and lead samples, the required force was lower than when the optimum conical die was used. This result indicates that the forming process requires less energy with optimum curvilinear molds compared to conical molds and confirms the accuracy of finite elements and experimental results [4].

Ebrahimi, R. (2008) and his team developed a new upper bound analysis method for tube extrusion. The authors compared the optimum die half angle obtained with numerical and analytical

solutions. They stated that at a constant friction factor value, there is an optimum angle that minimizes the power required for the forming process. They used a separate equation to calculate this optimum die angle. They found that as the friction factor increases, the die half angle also increases, but the constant friction factor does not have any effect on the dead metal zone angle. They also observed that when the extrusion rate increased, the optimum die half angle also increased. They emphasized that the effect of extrusion rate is more evident at high friction factor values [5].

Abrinia, K. and Makaremi, M. (2009) developed a new design and analysis method to model material flow with expanding square, rectangular and elliptical cross-sections. The molds they choose are designed to allow material to flow towards the lateral surfaces. They examined molds with various extrusion rates and investigated the effects of parameters such as friction effect and extrusion rate on extrusion pressure. While they used the upper limit analysis method to determine the maximum extrusion pressure, they preferred the finite element method to facilitate mold design. In their experiments to verify the theory, they demonstrated that the experimental, numerical and theoretical results were compatible and that these methods could be used for scattering patterns. Their proposed upper bound analysis method involves a kinematically new velocity field and has been used to estimate the maximum energy required for forming. In the experiments, commercially pure lead material and different mold sets were used [6].

Shu-peng C. and Zhong-jin W. (2020) represented the upset forging process of three-dimensional elliptical disks and rings was analyzed using the upper bound method. A general form of kinematically valid velocity fields is proposed with three control functions, each containing a free parameter. In this way, factors such as sideways spreading, lateral swelling in the thickness direction and movement of the neutral surface that occurred during the "upset" forging process of the ellipse rings could be taken into account. It has been shown that by choosing different control functions in the proposed general form, kinematically valid velocity fields suggested by other researchers in previous references can be obtained. By optimizing the free parameters with the simplex algorithm in the upper bound method, the forging load and the shapes of the workpiece in the deformation stages could be calculated. Calculations made for different workpiece shapes and friction conditions show that the theoretical results obtained are compatible with the experimental results in previous studies and the finite element analysis performed in this study [7].

Hamed A. (2022) has worked on a new mathematical model is developed to predict the gap closing behavior of metal rods during the plane strain extrusion process. For this purpose, an improved upper bound analysis has been carried out by applying the power balance between the intrinsic work rate, the rate of energy release across the gap, and the extrinsic force. Considering the limiting condition of the resulting equations at the gap closure threshold, a mechanical criterion was obtained to find the minimum extrusion rate required for the closure of elliptical gaps. The formulation was developed for frictionless and frictional conditions at the die-rod interface. Additionally, the process was modeled with the finite element method (FEM) and it was observed that the obtained analytical criterion and FEM modeling provided a good match in terms of minimum required extrusion rate. It is found that the minimum extrusion rate required to close the voids is solely a function of the extrusion die angle, the void aspect ratio, and the friction condition at the die-rod interface. Additionally, it has been determined that extruding the material under frictional conditions and with a die with a smaller taper angle is beneficial in closing the gaps [8].

Khanlari H. and Honarpisheh M. (2023) published a study on the upper limit (UB) analysis of the channel angular printing process was carried out by taking into account the die angle, the thickness of the entry and exit channels, the outer and inner corner radii, the die geometric properties such as the length of the exit channel and the length of the first workpiece, friction and strain

hardening of the material, under cold conditions and for a rectangular cross-section workpiece. In order to verify the results of the UB analysis, the results of the analytical equations were compared with the numerical simulation and the real process. The results revealed that decreasing the die angle, the thickness of the exit channel and the outer corner radius, and increasing the thickness of the entrance channel and the inner corner radius increased the total plastic stress and processing force. Moreover, it has been shown that the processing force increases with increasing the initial workpiece length. A very good agreement was achieved between the analytical method and the results of the numerical and experimental methods [9].

In hot and cold upsetting processes, if lubrication is very sufficient, the friction force will be close to zero. If the object on which the upsetting process is applied is a cylinder, when each part of the cylinder is divided into small elements, it is seen that the deformation between the elements is equal. Due to volume constancy, there is no volumetric change while the cylinder decreases in length.

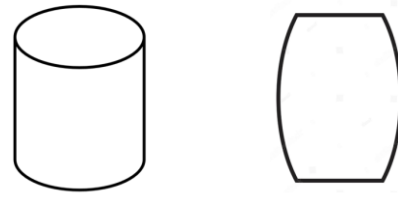


Fig. 1 Cylindrical parts before and after in the upsetting process.

2. Upper Bound Analysis Method

In order to calculate the total power consumption in the upper bound method analysis, it is necessary to take into account the total of all energy consumed. The total power consumption is obtained by calculating the power consumption of each process in which energy is consumed separately. Thus, the upper limit for total power consumption is found [10,11].

J^* : Upper bound for total power consumption

$$J^* = \sum \dot{W}_t + \sum \dot{W}_s + \sum \dot{W}_f \quad (1)$$

$\dot{W}_i$: Ideal deformation power

$$\dot{W}_i = \bar{\sigma}_m^{(l)} \int \dot{\epsilon} dV \quad (2)$$

$\dot{\epsilon}$: Effective strain velocity

$$\dot{\epsilon} = \frac{2}{\sqrt{3}} \left(\frac{1}{2} (\dot{\epsilon}_r^2 + \dot{\epsilon}_\theta^2 + \dot{\epsilon}_z^2) + \dot{\epsilon}_{r\theta}^2 + \dot{\epsilon}_{\theta z}^2 + \dot{\epsilon}_{rz}^2 \right)^{1/2} \quad (3)$$

$\bar{\sigma}_m^{(j)}$: Effective stress value

$$\bar{\sigma}_m^{(j)} = \frac{1}{\bar{\epsilon}_f^{(j)}} \int_0^{\bar{\epsilon}_f^{(j)}} \sigma d\epsilon \quad (4)$$

$\bar{\epsilon}_f^{(j)}$: Effective strain value

$$\bar{\epsilon}_f^{(j)} = \sum_{i=1}^j \ln \left(\frac{H^{(i)}}{H^{(i-1)}} \right) \quad (5)$$

$\dot{W}_f$: Friction power between the mold and the part

$$\dot{W}_f = \frac{m}{\sqrt{3}} \bar{\sigma}_m^{(l)} \int_{S_f} |\Delta V| ds \quad (6)$$

P_{avg} : Average stamp pressure

$$P_{avg} = \frac{J^*}{A_r U_0} \quad (7)$$

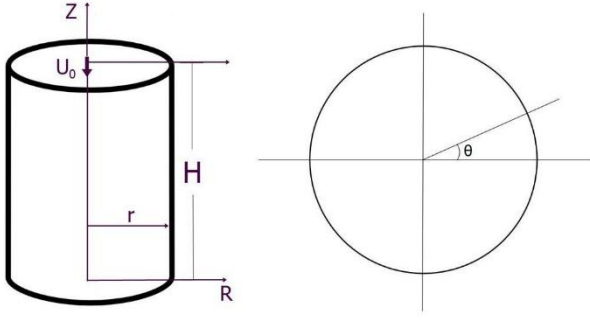


Fig. 2 Cylindrical coordinates of upsetting part.

3.1. Distribution of Kinematic Velocity Elements

The velocity elements occurring for an object whose cylindrical coordinates are given are U_z , U_θ , U_r in order and their mathematical expressions are as follows [7,8].

U_z : Axial velocity equation

$$U_z = \frac{-U_0 Z}{T} \quad (8)$$

U_θ : Tangential velocity equation

$$U_\theta = \frac{-U_0 R}{T} \omega_1(\theta) \quad (9)$$

$$\omega_1(\theta) = \frac{(a-b)}{(a+b)} D_1 \sin(2\theta) \quad (10)$$

$$\omega_1(\theta) = \frac{(a-a)}{(a+a)} D_1 \sin(2\theta) = 0 \quad (11)$$

$$U_\theta = \frac{-U_0 R}{T} \omega_1(\theta) = \frac{-U_0 R}{T} \cdot 0 = 0 \quad (12)$$

$$U_\theta = 0 \quad (13)$$

U_r : Radial velocity equation

$$U_r = \frac{U_0 R}{2T} \cdot (1 + \frac{d\omega_1(\theta)}{d\theta}) \quad (14)$$

$$U_r = \frac{U_0 R}{2T} \cdot (1 + 0) = \frac{U_0 R}{2T} \quad (15)$$

$$U_r = \frac{U_0 R}{2T} \quad (16)$$

3. Force Analysis for Cylindrical Prism

The expressions of the velocity components occurring on the material are given in the equations below:

$$U_z = \frac{-U_0 Z}{T} \quad (17)$$

$$U_\theta = \frac{-U_0 R}{T} \omega_1(\theta) \quad (18)$$

Since $b = a$ in cylindrical materials, equations are shown as follows:

$$\omega_1(\theta) = \frac{(a-b)}{(a+b)} D_1 \sin(2\theta) \quad (19)$$

$$\omega_1(\theta) = \frac{(a-a)}{(a+a)} D_1 \sin(2\theta) = 0 \quad (20)$$

$$U_\theta = \frac{-U_0 R}{T} \omega_1(\theta) = \frac{-U_0 R}{T} \cdot 0 = 0 \quad (21)$$

$$U_\theta = \frac{-U_0 R}{T} \omega_1(\theta) = \frac{-U_0 R}{T} \cdot 0 = 0 \quad (22)$$

$$U_\theta = 0 \quad (23)$$

$$U_r = \frac{U_0 R}{2T} \cdot (1 + \frac{d\omega_1(\theta)}{d\theta}) \quad (24)$$

$$U_r = \frac{U_0 R}{2T} \cdot (1 + 0) = \frac{U_0 R}{2T} \quad (25)$$

$$U_r = \frac{U_0 R}{2T} \quad (27)$$

Since the volume does not change in compressed materials, based on the principle of volume constancy, strain rates are given by the following equations.

$$\frac{\partial U_r}{\partial R} + \frac{U_r}{R} + \frac{1}{R} \cdot \frac{\partial U_\theta}{\partial \theta} + \frac{\partial U_z}{\partial Z} = 0 \quad (28)$$

$$\dot{\epsilon}_r = \frac{\partial U_r}{\partial R} \quad (29)$$

$$\dot{\epsilon}_r = \frac{\partial U_r}{\partial R} = \frac{U_0}{2T} \quad (30)$$

$$\dot{\epsilon}_r = \frac{U_0}{2T} \quad (31)$$

$$\dot{\epsilon}_\theta = \frac{U_r}{R} + \frac{1}{R} \cdot \frac{\partial U_\theta}{\partial \theta} \quad (32)$$

$$\dot{\epsilon}_\theta = \frac{U_r}{R} + \frac{1}{R} \cdot \frac{\partial U_\theta}{\partial \theta} = \frac{U_0}{2T} + \frac{1}{R} \cdot 0 = \frac{U_0}{2T} \quad (33)$$

$$\dot{\epsilon}_\theta = \frac{U_0}{2T} \quad (34)$$

$$\dot{\epsilon}_z = \frac{\partial U_z}{\partial Z} \quad (35)$$

$$\dot{\epsilon}_z = \frac{\partial U_z}{\partial Z} = -\frac{U_0}{T} \quad (36)$$

$$\dot{\epsilon}_z = -\frac{U_0}{T} \quad (37)$$

Since the total deformation force must be found by taking all equations into consideration, mathematical operations are continued as follows:

$$J^* = \sum \dot{W}_t + \sum \dot{W}_s + \sum \dot{W}_f \quad (38)$$

$\dot{W}_t$ is the ideal deformation power:

$$\dot{W}_t = \bar{\sigma}_m^{(I)} \int \dot{\epsilon} dV \quad (39)$$

$$\dot{\epsilon} = \frac{2}{\sqrt{3}} \left(\frac{1}{2} (\dot{\epsilon}_r^2 + \dot{\epsilon}_\theta^2 + \dot{\epsilon}_z^2) + \dot{\epsilon}_{r\theta}^2 + \dot{\epsilon}_{\theta z}^2 + \dot{\epsilon}_{zr}^2 \right)^{1/2} \quad (40)$$

$$\dot{W}_t = \bar{\sigma}_m^{(I)} \int \dot{\epsilon} dV = \bar{\sigma} \iiint \frac{2}{3} \left(\sqrt{\frac{1}{2} (\dot{\epsilon}_r^2 + \dot{\epsilon}_\theta^2 + \dot{\epsilon}_z^2) + \dot{\epsilon}_{r\theta}^2 + \dot{\epsilon}_{\theta z}^2 + \dot{\epsilon}_{zr}^2} \right) r dr d\theta dz \quad (41)$$

$$\dot{W}_t = \pi R^2 \bar{\sigma} U_0 \quad (42)$$

$\dot{W}_f$ indicates power consumed due to friction between die and the stamp resulting in :

$$\dot{W}_f = \frac{m}{\sqrt{3}} \bar{\sigma}_m^{(I)} \int_{S_f} |\Delta V| ds \quad (43)$$

$$\dot{W}_f = \frac{m}{\sqrt{3}} \bar{\sigma}_m^{(I)} \int_{S_f} |\Delta V| ds = \frac{m}{\sqrt{3}} \bar{\sigma} \iint \sqrt{U_r^2 + U_\theta^2} r dr d\theta \quad (44)$$

$$\dot{W}_f = 2\pi R^3 \bar{\sigma} m \frac{U_0}{3\sqrt{3}T} \quad (45)$$

$\dot{W}_s$ is the amount of power consumption due to shear:

$$\dot{W}_s = 0 \quad (46)$$

$$J^* = \dot{W}_t + \dot{W}_f = \pi R^2 \bar{\sigma} U_0 + 2\pi R^3 \bar{\sigma} m \frac{U_0}{3\sqrt{3}T} \quad (47)$$

$$J^* = \pi R^2 \bar{\sigma} U_0 \left(1 + \frac{2}{3} \frac{m}{\sqrt{3}} \frac{R}{T} \right) \quad (48)$$

$$J^* = \text{Force} * \text{Velocity} = F U_0 \quad (49)$$

$$F = \text{Area} * \text{Pressure} = \pi R^2 P_{avg} \quad (50)$$

$$J^* = \pi R^2 P_{avg} U_0 \quad (51)$$

$$J^* = \pi R^2 P_{avg} U_0 = \pi R^2 \bar{\sigma} U_0 \left(1 + \frac{2}{3} \frac{m}{\sqrt{3}} \frac{R}{T} \right) \quad (52)$$

$$\frac{P_{avg}}{\bar{\sigma}} = 1 + \frac{2}{3} \frac{m}{\sqrt{3}} \frac{R}{T} \quad (53)$$

Total upsetting force is,

$$F = \pi R^2 \bar{\sigma} \left(1 + \frac{2}{3} \frac{m}{\sqrt{3}} \frac{R}{T} \right) \quad (54)$$

4. Calculations and Results

All theoretical calculations are given in the equations shown above. In the next stage, dimensional changes will be calculated and the results will be examined. In the figure below, the main axis and spare axis lengths of the ellipse are indicated as a and b respectively.

The dimensions of the cylindrical part are shown as a and b before the process, and as a_1 and b_1 after the process.

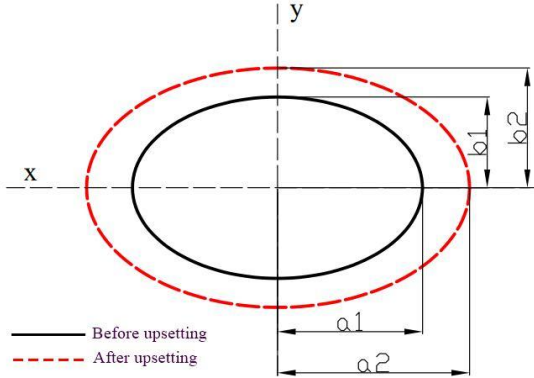


Fig. 3 Surface changes before and after upsetting on a cylindrical part.

$$\frac{a}{b} = \lambda = \frac{a_1}{b_1} = \frac{a_2}{b_2}$$

$$V = \pi \cdot a \cdot b \cdot h$$

$$V = \pi \cdot a \cdot b \cdot h = \pi \cdot a_1 \cdot b_1 \cdot h_1 = \pi \cdot a_2 \cdot b_2 \cdot h_2$$

$$b_2 = \sqrt{\frac{a_1 \cdot b_1 \cdot h_1}{h_2 \cdot \lambda}}$$

The values resulting from calculations for different upsetting rates are shown in the table below. $\dot{W}_i$ and $\dot{W}_f$ values were found separately and then the total power J^* was calculated.

For the friction and deformation power requirements that occur when upsetting is applied at different rates to a cylindrical object with a height of 20 units and a radius of 30 units, which is taken as an example to examine the results after theoretical calculations, are shown below in the graphics for two different upsetting ratios.

Table 1: Lengths obtained according to %10 upsetting rates.

a_1	b_1	h_1	h_2	b_2	a_2
30	30	20	20	30	30
30	30	20	18	31,6227766	31,6227766
30	30	20	16	33,54101966	33,54101966
30	30	20	14	35,85685828	35,85685828
30	30	20	12	38,72983346	38,72983346
30	30	20	10	42,42640687	42,42640687
30	30	20	8	47,4341649	47,4341649

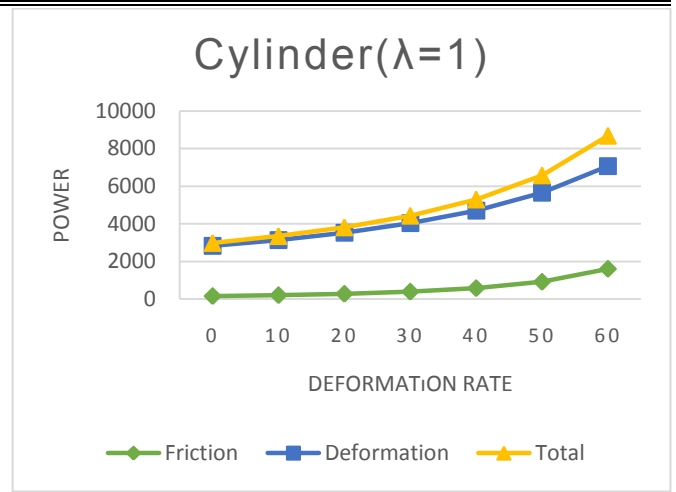


Fig. 4 Graphic for the %10 upsetting rates of cylindrical prism.

Table 2: Lengths obtained according to %5 upsetting rates.

a_1	b_1	h_1	h_2	b_2	a_2
30	30	20	20	30	30
30	30	20	19	30,77935056	30,77935056
30	30	20	18	31,6227766	31,6227766
30	30	20	17	32,53956867	32,53956867
30	30	20	16	33,54101966	33,54101966
30	30	20	15	34,64101615	34,64101615
30	30	20	14	35,85685828	35,85685828

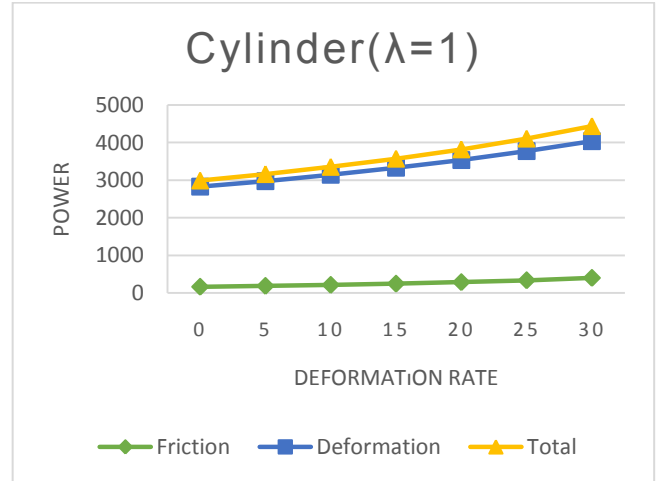


Fig. 5 Graphic for the %5 upsetting rates of cylindrical prism.

5. Results and Discussion

When all the results are examined, when upsetting is applied to a metallic object, the required power varies depending on the upsetting rate applied. In compression at high rates, more power is required for both friction and deformation. As the ratio of diameter to height increases, both amounts of power to be applied increase logarithmically. The aim of this study is to pre-calculate the appropriate conditions and the required amount of power when applying upsetting to cylindrical metals. With the results of the study, the theoretically intended numerical values were reached.

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Influence of Al_2O_3 content on the mechanical properties of sintered Al-10Cu-x Al_2O_3 composites

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Abstract: This study investigates the influence of Al_2O_3 content on the mechanical properties of sintered Al-10Cu-x Al_2O_3 ($x = 2.5, 5$, and 7.5 wt.%) composite materials, produced via powder metallurgy and subjected to quasi-static and dynamic compressive loadings. Quasi-static tests were performed at a constant strain rate of 0.003 s^{-1} , while dynamic tests were conducted at strain rates corresponding to impact velocities of approximately 10 m/s and 20 m/s . The results indicate that a higher Al_2O_3 content enhances the mechanical properties of the composite under both quasi-static and dynamic compression. The most significant improvements were observed under high strain rate impact loading, highlighting the potential of sintered Al-10Cu-x Al_2O_3 for applications in dynamic environments.

Keywords: SINTERED Al-10Cu-x Al_2O_3 COMPOSITES, POWDER METALLURGY, Al_2O_3 CONTENT, COMPRESSIVE STRENGTH

1. Introduction

Aluminium and its alloys are proven as prospective base materials for manufacturing light metal matrix composites (MMC) and powder metallurgy is a suitable technique for their production [1-4]. To further improve its properties, primary alloying elements are added to aluminium powder. Copper is believed to be the most significant due to its ability to cause higher densification and age hardening in Al-Cu alloys [2,3,5]. It is known by the literature that Al-Cu composite materials, manufactured by powder metallurgy method are widely used in the automotive and aerospace industries for cost-saving lightweight functional components [3,4,6]. The reinforcement is done mainly by ceramic particles including SiC, B₄C, TiC, WC and Al_2O_3 [1-4,7-13]. Among them, Al_2O_3 is generally used to improve the hardness, strength and wear behaviour of aluminium alloy matrix [3,4].

Some research has already been published with 2.5, 5 and 10 wt % Al_2O_3 reinforcement [10] and improvement in tensile and compressive strength of hot extruded MMC materials reinforced by alumina particles up to 5 wt.% is found. The influence of the percentage of the Al_2O_3 reinforcement, the H/D ratio of the compacted samples and the compaction pressure on the physical and mechanical properties of the composites were also investigated using different methodologies [3,4]. The experimental data showed that the best yield and compression strength are obtained at 10% Al_2O_3 , 700 MPa, and 1.25 H/D, while the maximum fracture strain is achieved at 700 MPa, 1.25 H/D, and 0% Al_2O_3 [4]. Higher Al_2O_3 content decreases relative density by increasing volumetric voids and segregation between the higher amount of reinforcement and Al-4Cu matrix, but has a positive effect on the hardness of the composites [3]. Despite the serious research that has been conducted on the mechanical characteristics of aluminium-based composites, the distinct mechanical evaluation of Al-10Cu-x Al_2O_3 composites, produced through powder metallurgy, under low and high-strain compressive loading continues to be insufficiently investigated. Current research aims to fill this knowledge gap by studying the performance of Al-10Cu-x Al_2O_3 composites under distinct loading regimes.

This study aims to evaluate the influence of Al_2O_3 content on the mechanical properties of sintered Al-10Cu-x Al_2O_3 ($x = 2.5, 5$, and 7.5 wt.%) composite materials, produced via powder metallurgy and subjected to quasi-static and dynamic compressive loadings.

2. Materials and methods

Aluminium-based alloys were produced using aluminium, copper and Al_2O_3 powders with a particle size of ≤ 325 mesh. All powders were first mixed in the desired proportions and then subjected to high-energy ball milling with a ball-to-powder ratio of 6:1. The milling process continued for 5 hours at a rotation speed of 500 rpm to achieve a uniform distribution of Cu and Al_2O_3 particles within the Al matrix. After the ball milling process, the powder

mixtures were compacted in a cylindrical steel die with a diameter of 16 mm using a hydraulic press. The resulting green bodies were sintered at 530°C for 3 hours. After sintering, the samples were cooled slowly in the furnace to minimise thermal stresses and to secure uniform microstructural development. The specimens for determining the mechanical properties are cylindrical with a diameter and height of $10 \times 15 \text{ mm}$ for quasi-static testing and $5 \times 5 \text{ mm}$ for dynamic testing. A universal servo-hydraulic testing machine Zwick Roell HA250 was used for quasi-static compression tests at a constant low strain rate of 0.003 s^{-1} . A modified Split Hopkinson Pressure Bar (SHPB) apparatus for impact testing of light materials [14], was used for the dynamic compression tests at high strain rates of about $\sim 1500 \text{ s}^{-1}$ and $\sim 3200 \text{ s}^{-1}$, corresponding to impact speeds of about 10 m/s and 20 m/s .

3. Results and discussion

When it comes to analysis of the mechanical response of certain material it is always helpful to be aware of its structure and possible changes during fabrication. According to the Al-Cu phase diagram, Al_2Cu , AlCu, Al_3Cu_4 , Al_2Cu_3 , and Al_4Cu_9 could be formed in the Al-Cu system through solid-state phase transformation [15,16,17] among which stable phases are AlCu, Al_2Cu , and Al_4Cu_9 [18]. Al_2Cu phase evolves among the first due to its lowest formation energy [16,17] and usually the increasing of Cu content results in the formation of only Al_2Cu in the fabricated composites [15] because the thermal processing time is long enough to convert AlCu to Al_2Cu . Thus, the structure of the matrix material is believed to be consistent with aluminium grains with dissolved copper surrounded by copper precipitated in the form of the Al_2Cu phase at the aluminium grain boundaries [1, 2].

In addition, alumina leads to grain refinement. If the harder alumina particles are reasonably distributed, they could increase the dislocation density of the matrix and the load is transferred from the matrix to the reinforcement/second phases. The Al_2O_3 particles distort the crystal lattice of the Al-Cu matrix, hinder the mobility of dislocations and thus strengthen the alloy [3]. The Al_2Cu phase and alumina particles are believed to be the main reason for the enhanced mechanical properties of the tested Al-10Cu-x Al_2O_3 composites

Quasi-static compression test results are presented in Fig. 1, showing the stress-strain curves for three types of powder-metallurgy-derived Al-10Cu-x Al_2O_3 materials with different Al_2O_3 content. The graphs show similar behaviour under quasi-static compression, starting with the stage of elastic compression up to the proportional limit and yielding, strain hardening with ductile deformation and non-uniform deformation at compressive strength with shear bands beginning to develop [19,20]. A decrease in the load-bearing capacity of the compacted composites occurs because of the deformation within the shear bands, followed by complete failure due to the separation of bonded particles along the shear bands [20].

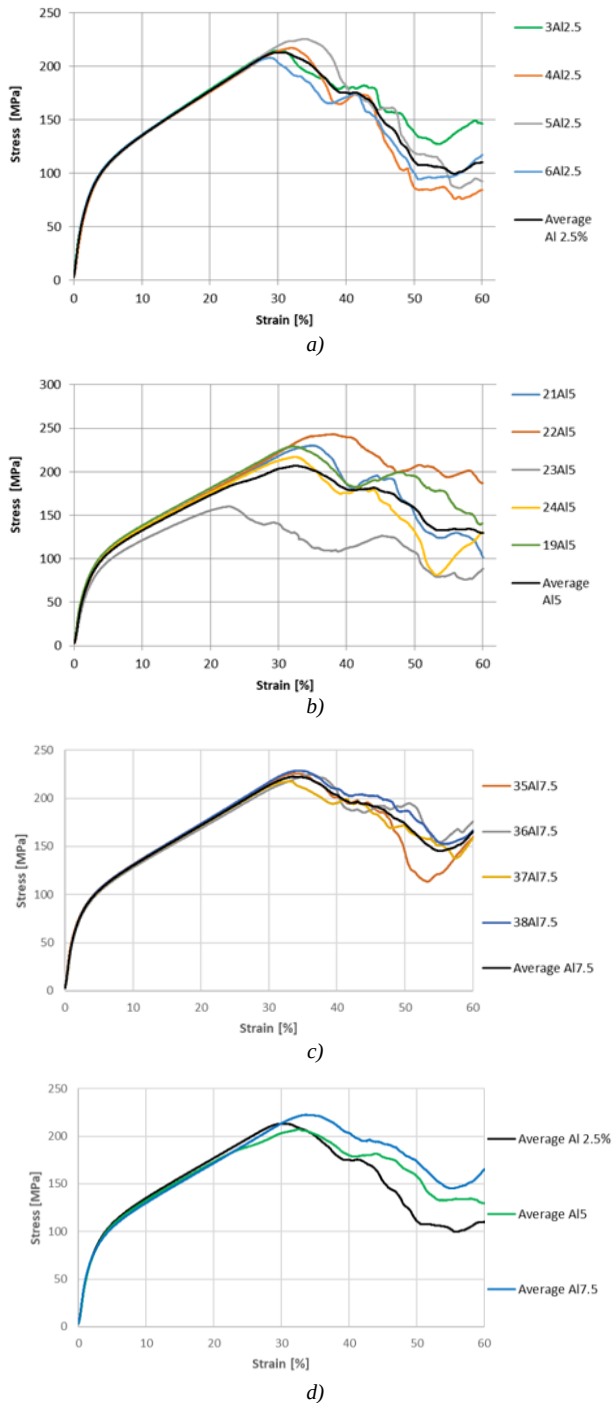


Fig. 1 Quasi-static tests result at a constant strain rate of 0.003 s^{-1} of: a) sintered Al-10Cu-2.5Al₂O₃; b) sintered Al-10Cu-5Al₂O₃; c) sintered Al-10Cu-7.5Al₂O₃; d) average curves

The stress-strain curves are smooth up to about 30% deformation in the tested specimens. The yield stress in all the specimens is almost identical and the material continues to strain harden till 30% plastic strain. The first drop of stresses is notable right after 30% strain in alloys with 2.5 wt% Al₂O₃, followed by the ones with 5 wt% Al₂O₃. Before 40% deformation all tested materials have reached their compressive strength with 7.5 wt% Al₂O₃ alloy being at the maximum. The variation graphs of elastic modulus and yield stress (fig. 2a) and compression strength and strain (fig. 2b) are obtained from the experimental data at a constant strain rate of 0.003 s^{-1} . The elastic modulus and the compression strain at the strength point of the Al-10Cu-xAl₂O₃ composites showed an upward trend when a higher content of Al₂O₃ was added. The average yield stress stays almost unaffected by alumina content. The compression strength remained constant up to 5 wt% Al₂O₃ and then raised at a concentration of 7.5 wt% Al₂O₃. It can be concluded that the higher amount of alumina particles demonstrates

improvement in the properties of the composites tested under quasi-static loadings.

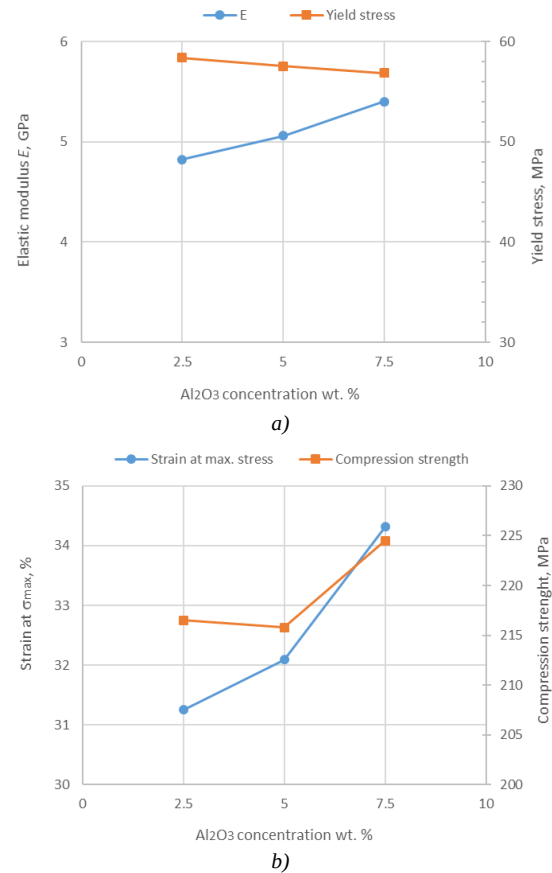


Fig. 2 Variation curves of the mechanical properties of the composites with different Al₂O₃ content, tested under quasi-static loading: a) elastic modulus and yield stress; b) compression strength and strain

The mechanical properties under all strain rates of the tested Al-10Cu-xAl₂O₃ composites, as summarised in Table 1.

Table 1: Average values of the mechanical properties of tested materials.

Alumina concentration wt%	2.5% Al ₂ O ₃	5% Al ₂ O ₃	7.5% Al ₂ O ₃
quasi-static compression at the strain rate of 0.003 s^{-1}			
Modulus of elasticity [GPa]	4.8	5.1	5.4
YS(offset=0.2%) [MPa]	58	58	57
Compressive strength [MPa]	217	216	225
Deformation ε [%]	31.3	32.1	34.3
high impact dynamic compression at the strain rate of 1500 s^{-1}			
Modulus of elasticity [GPa]	7.9	9.7	12.9
YS(offset=0.2%) [MPa]	72	82	93
Compressive strength [MPa]	328	323	338
Deformation ε [%]	44.4	45.7	45.8
high impact dynamic compression at the strain rate of 3200 s^{-1}			
Modulus of elasticity [GPa]	7.4	5.3	5.0
YS(offset=0.2%) [MPa]	84	64	71
Compressive strength [MPa]	1399	1365	1614
Deformation ε [%]	83.7	85.1	84.8

The results from dynamic compression tests are shown in Fig. 3 for moderate strain rates of 1500 s^{-1} and Fig. 4 for high strain rates of 3200 s^{-1} . All specimens tested at high strain rates exhibited a distinct elastic stage, followed by strain hardening up to the compression strength, with plenty of fluctuations in stress during the tests under dynamic compression at 10 m/s. The ultimate strength and failure strain increased with the strain rate for all tested materials. There is some difference in the failure mode – at impact speeds of 10m/s the tested alloys generally exhibit plastic failure, while in the ones tested at 20 m/s a mixed mode of splitting and smash failure is observed. The stress-strain relationships revealed

superior dynamic mechanical properties at a strain rate of 3200 s^{-1} compared to 1500 s^{-1} , although the yield strength values are quite close.

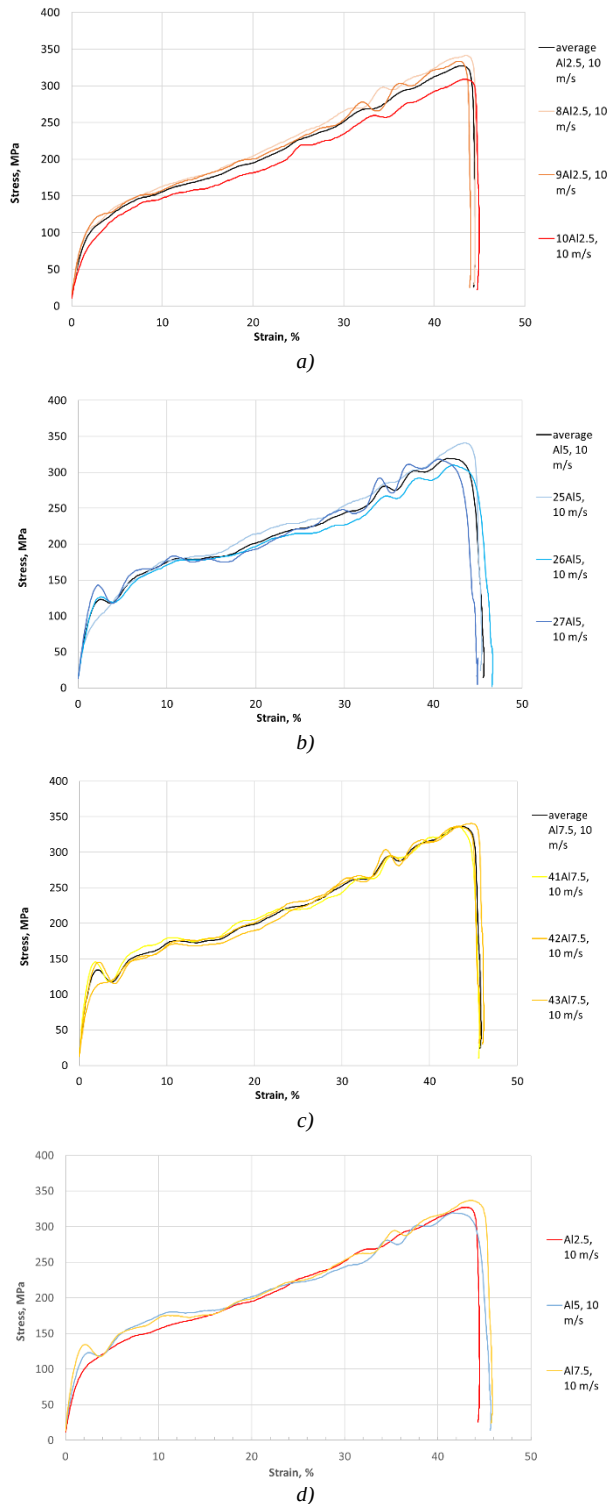


Fig. 3 SHPB dynamic test results at 10 m/s of: a) sintered Al-10Cu-2.5Al₂O₃ ; b) sintered Al-10Cu-5Al₂O₃ ; c) sintered Al-10Cu-7.5Al₂O₃ ; d) average curves

The variation graphs of elastic modulus and yield stress (fig. 5 a, b) and compression strength and strain (fig. 5 c, d) are obtained from the experimental data at a strain rate of 1500 s^{-1} and 3200 s^{-1} . At high strain rates no obvious trend in the properties is observed. It is visible that the strain is not affected by the Al₂O₃ content. The elastic modulus and yield stress are varied in very tight intervals, especially for the strain rate of 3200 s^{-1} and can hardly be attributed to the alumina content. The major positive effect of alumina content is demonstrated in compression strength for 7.5 wt% Al₂O₃, with a slight drop in the values at 5 wt% Al₂O₃.

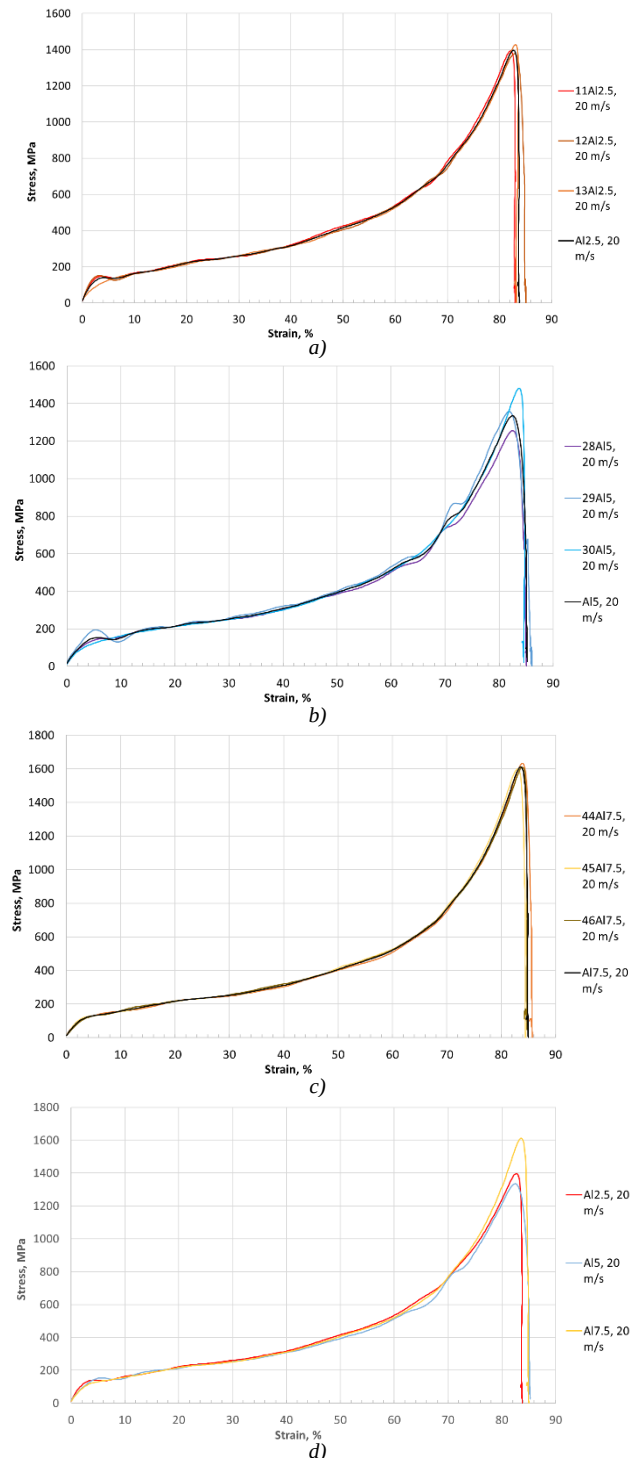


Fig. 4 SHPB dynamic test results at 20m/s of: a) sintered Al-10Cu-2.5Al₂O₃ ; b) sintered Al-10Cu-5Al₂O₃ ; c) sintered Al-10Cu-7.5Al₂O₃ ; d) average curves

The increase in compressive strength is attributed to the Al₂O₃ particles, with best results of Al-10Cu-7.5Al₂O₃ under all strain rates in the investigation. Also, it is noticed that the amount of alumina addition (up to 7,5 wt%) does not affect the plasticity of the composite at a certain strain rate. The difference in deformation is observed only when comparing the results from tests at different strain rates, with the highest results for strain rates 3200 s^{-1} . The compressive strength increases from 200-250 MPa at quasi-static rates (0.003 s^{-1}) to 1400-1600 MPa at high strain rates (3200 s^{-1}) and the strain corresponding to the maximum compressive stress is shifted from about 30-35% to 85%. The reduction in strength, observed in Al-10Cu-5Al₂O₃ is attributed to the presence of a higher amount of unbounded free particles in the Al-Cu matrix than in other tested composites [10]. The slight differences in properties

could also be due to the different densification of specimens [19], although this is not obvious from compression tests.

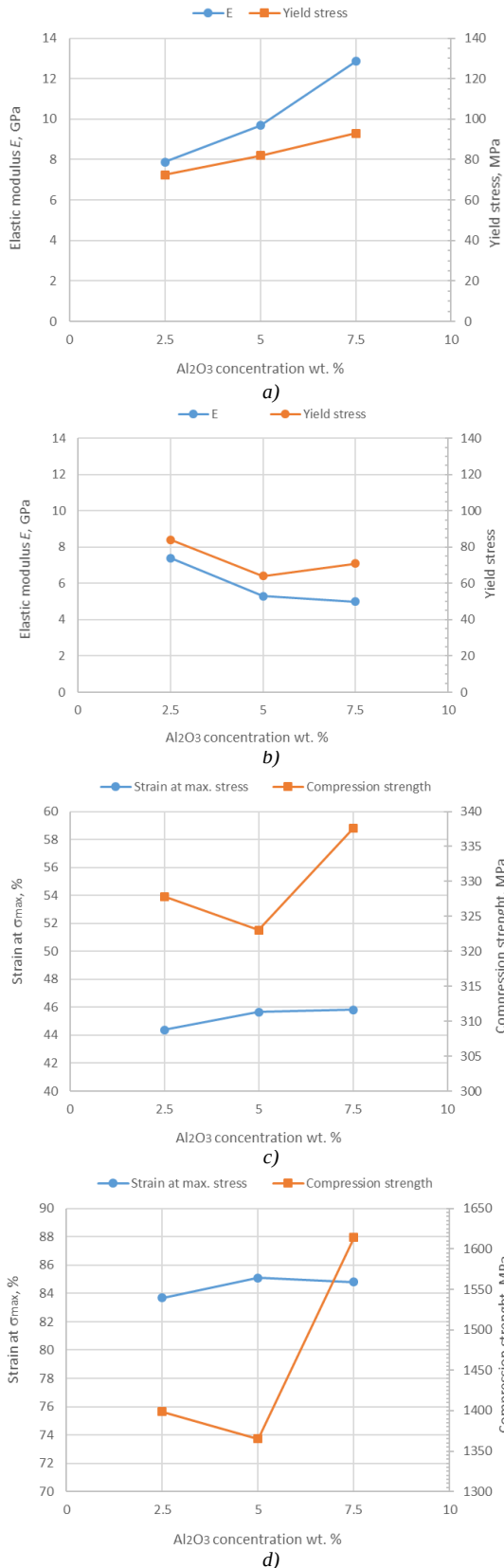


Fig. 5 Variation curves of the mechanical properties of the composites with different Al₂O₃ content: a) Elastic modulus and yield stress at 1500 s⁻¹; b) Elastic modulus and yield stress at 3200 s⁻¹; c) Strain at maximum stress and compressive strength at 1500 s⁻¹; d) Strain at maximum stress and compressive strength at 3200 s⁻¹.

4. Conclusions

The current investigation focuses on the influence of Al₂O₃ addition on the mechanical behaviour of Al-10Cu-xAl₂O₃ composites (x = 2.5, 5, and 7.5 wt.%) produced through powder metallurgy under quasi-static and dynamic compressive loading conditions. The major conclusions that could be drawn back are:

1. Al-10Cu-xAl₂O₃ composites with 2.5, 5, and 7.5 wt.% Al₂O₃ were successfully synthesised through the powder metallurgy method.
2. Increased compressive strength up to 1600 MPa and deformation up to 85% at high strain rates (3200 s⁻¹) were experimentally achieved by SHPB testing.
3. The improvement in mechanical properties is attributed to the higher Al₂O₃ content, which enhances strength and deformation capacity under dynamic loading conditions of the tested alloys.

5. Acknowledgments

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Application of thermal-derivative analysis to study phase transformation in AlSi7Mg alloy with different iron content

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Abstract: Using an increasing share of aluminum scrap (production and post-production) requires paying special attention to its impurities. Gaseous inclusions (e.g., hydrides) can be removed from the liquid alloy by refining, but the situation is worse with metallic impurities. In Al-Si alloys, one of the worst is iron, which goes into solution due to its low solubility in the solid state, at a content of over 0.4wt.%, it crystallizes in morphologically unfavorable phases, which worsen the functional properties and increase the porosity of aluminum alloys, limiting their use. The crystallization of these phases causes thermal effects that various methods can record. The paper presents studies of phase transformation, especially iron phases, using thermal-derivative analysis occurring in the AlSi7Mg alloy with different iron content. The studies were performed on Crystaldigraph NT3-8K coupled with the MLab program. It was found that the most unfavorable phase is β -Al₅FeSi, which crystallizes preeutectic (and mainly primary crystallize) dimensions of up to 1000 μ m, causing the formation of shrinkage porosities.

KEYWORDS: AL-SI CAST ALLOYS; THERMAL ANALYSIS; PHASE TRANSFORMATION; CRYSTALLIZATION; POROSITY

1. Introduction

Analysis of thermodynamic processes is proper when it is possible to record their kinetics. In industrial settings, temperature is the parameter that can be most easily controlled and determines the form of the microstructure and the physicochemical properties occurring in the material. One of the methods of the time course of change of these properties as a function of temperature is thermal analysis (TA) [1-3]. Among its many variations, e.g., thermogravimetric analysis (TGA) [4, 5], thermodilatometric analysis (TDA) [6], thermomechanical analysis (TMA) [7] or differential scanning calorimetry (DSC) [8, 9], thermal-derivative analysis (TDA) has found wide application in both laboratory and industrial research [10-12]. ATD analysis is a method that uses the visual effects of phase transformations occurring in metal alloys (usually during the transition from the liquid to the solid state), accompanied by a change in energy. As the sample cools, its temperature is lowered by the release of heat, a characteristic feature of the alloy under study and the transformations taking place in it. Due to the ease of measuring temperature, the ATD method is widely used, for example, in metallurgy, making it possible to quickly obtain much information about the alloy under study [13-16].

Such great interest in the ATD method is due to its versatile application for, among other things:

- nucleation analysis and determination of the degree of overcooling of the alloy ΔT . It can be used, for example, to assess the correctness of the modification process incident - the beginning of the cooling curve,
- indicative prediction of the chemical and structural composition of the alloy - the middle range of the crystallization curve (from $T_{liq.}$ to $T_{sol.}$),
- indicative assessment of the alloy's purity and the presence of impurities, inclusions, and gases in it - the final section of the crystallization curve. Impurities cause the formation of low-melting eutectics, which affect a significant increase in crystallization time,
- determination of the amount of heat of crystallization of alloying elements and eutectics - the entire crystallization curve,
- evaluation of phase transformations due to the separation of intermetallic phases (separate or as a component of eutectics).

In particular, the last capability of the ATD method helps determine the range of crystallization temperatures of alloying constituents, for example, in alloys where crystallization of phases that negatively affect functional properties is possible. This mainly concerns iron phases of unfavorable morphology in aluminum alloys due to the increased proportion of circulating scrap as a feedstock component.

Studies [20-22] indicate that the iron contained in Al-Si alloys, as a result of different heat removal conditions and chemical

composition, can crystallize in the form of four phases with different morphologies:

1. β -Al₅FeSi, also referred to as Al₉Fe₂Si in Group 6XX.X (Al-Mg-Si) alloys,
2. α -Al₈Fe₂Si (or α -Al₁₂Fe₃Si₂), which under thermodynamic equilibrium conditions has a hexagonal structure and is stable only in Al-Si-Fe alloys with high charge purity,
3. δ -Al₄FeSi₂ present in Al-Si alloys with more than 18 wt. % Si, and
4. γ -Al₃FeSi present at contents of more than 4 wt. % Fe and more than 16 wt. % Si.

However, the problem arises when the iron content in Al-Si alloys exceeds 0.4wt.% (for gravity castings), resulting in the crystallization of the β -Al₅FeSi phase (which has a lamellar and needle-like structure). As reported in the literature [17, 18], such a structure causes the propagation of microcracks and stress concentration, which increases the brittleness of castings, makes them more difficult to machine, reduces mechanical properties, and increases porosity. Therefore, it seems reasonable to research evaluating the crystallization of Al-Si alloys with higher iron content and analyzing the associated phase transformations. This problem is particularly relevant as the share of circulating scrap in producing Al-Si(Fe) alloy castings needs to be increased for economic and environmental reasons.

2. Purpose and scope of the research

The study aimed to analyze the crystallization process of an AlSi7Mg alloy with increasing iron content (from 0.4 to 2.0wt.%, in 0.2% increments) and its effect on microstructure and porosity.

To realize the stated purpose of the work, the scope of the research included:

- execution of melts according to the established experimental plan (Fig.1),
- chemical composition studies,
- analysis of crystallization by ATD method: recording the cooling curve and its first derivative,
- evaluation of microstructure change and porosity of AlSi7Mg alloy with different iron content.

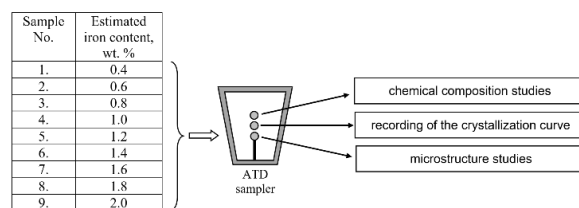


Fig. 1. Research plan for the AlSi7Mg alloy with increasing iron content.

3. Research material and methodology

The material for the study was AlSi7Mg alloy. The choice of the alloy was dictated by its wide application, both for gravity castings and pressure castings, such as rims. AlFe20 master alloy (as a substitute for scrap iron) was added to the alloy in such quantities as to achieve the assumed content of 0.4 to 2.0wt.% Fe (Fig.1). Any magnesium deficiencies were supplemented with AlMg10 master alloy (10wt.% Mg). The alloys were smelted in a Balzers VSG02/631 electric vacuum furnace (Balzers & Co GmbH, Germany). The alloys were modified with AlSr10 master alloy and refined with Rafglin-3. After reaching a temperature of about 760°C, it was cast into a standard QC-4080 Heraeus Electro-Nite sampler (Seekirchen, Austria) with a capacity of 130 cm³. The solidification temperature (T) was recorded using a NiCr-NiAl thermocouple (type K) placed on the axis of the sampler. Thermal analysis was carried out with a Crystaldigraph NT3-8K temperature recorder using MLab software (Gliwice, Poland). This set meets the requirements of both EN61010 and EN60584 for temperature measurement. The cooling curve $T=f(\tau)$ and its first derivative in time $dT/d\tau=f'(\tau)$ were recorded. For each measurement of the cooling curve, the same volume of alloy, the same coefficients for “smoothing” the first derivative, and similar melting, casting, and ambient conditions were used. From the middle part of the cross-section of the QC-4080 sampler ingot (at the temperature measurement point), samples were taken for metallographic studies performed on a MeF-2 Reichert light microscope (Wien, Austria). X-ray microanalysis was conducted on a Hitachi S-4200 scanning microscope (Tokyo, Japan) with an EDS energy dispersive X-ray spectrometer using a Thermo Noran system (System Six) equipped with a SE and BSE electron detector. X-ray studies were performed with a Philips X'Pert diffractometer, using a $\lambda_{CuK\alpha}$ - 1.54178 Å lamp supplied with 30 mA at 40 kV. The registration was made using the “step-scan” method with a step of 0.04° and a counting time of 10 seconds, in the 2 θ angle range from 20° to 140°. The gap on the incident beam was 1°, and on the deflected beam was 2°.

4. Research results and their analysis

4.1. Chemical composition test results

The results of the chemical composition of AlSi7Mg alloy with increasing iron content are shown in Table 1.

Table 1. Results of the chemical composition of AlSi7Mg (Al - balance) alloy.^{1,2}

Sample No.	Element content, wt. %									
	Si	Fe	Mg	Cu	Mn	Ni	Sn	Zn	Pb	Ti
1.	6.94	0.39	0.55	0.18	0.28	0.09	<0.01	<0.08	<0.11	0.12
2.	6.93	0.61	0.54	0.17	0.32	0.13	<0.01	<0.09	<0.10	0.13
3.	7.11	0.80	0.60	0.16	0.29	0.06	<0.02	<0.11	<0.09	0.13
4.	7.02	0.97	0.52	0.11	0.31	0.09	<0.03	<0.06	<0.10	0.12
5.	7.41	1.16	0.58	0.16	0.27	0.11	<0.02	<0.11	<0.11	0.18
6.	6.95	1.39	0.61	0.17	0.24	0.11	<0.02	<0.09	<0.10	0.17
7.	7.17	1.59	0.58	0.15	0.30	0.08	<0.03	<0.11	<0.09	0.20
8.	7.26	1.78	0.52	0.16	0.30	0.08	<0.02	<0.07	<0.10	0.15
9.	7.22	1.94	0.55	0.19	0.27	1.12	<0.02	<0.11	<0.08	0.18

¹ – averaged results from 6 measurements, after excluding the two extreme ones,

² – the sum of other elements (Na) and impurities (Ca; K) in the AlSi7Mg alloy is below 0.05.

Analyzing the results of the chemical composition of the AlSi7Mg alloy (Table 1), it can be concluded that despite the increasing iron content, the proportion of other elements has not changed and is within the limits of a typical AlSi7Mg alloy.

4.2. Results of thermal analysis using the ATD method

Representative ATD plots of AlSi7Mg alloy with different iron contents are shown in Figure 2.

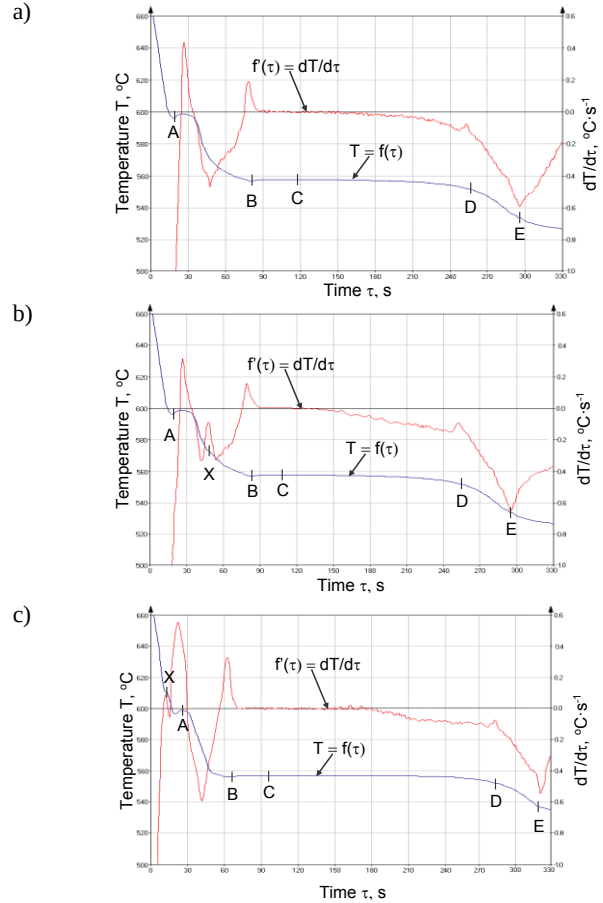


Fig. 2. Crystallization curves of AlSi7Mg alloy with different iron content: a) up to 0.49wt.%; b) from about 0.5 to 0.9wt.%; c) from about 0.91 to 1.5wt.%.

The symbols in Figure 2 stand for:

- $T=f(\tau)$ - the curve of temperature change T at time τ , - the so-called TA (Temperature Analysis) curve,
- $dT/d\tau=f'(\tau)$ - the first derivative of the temperature change over time - the so-called ATD (Analysis Temperature Derivative) curve,
- point A - temperature of onset of crystallization of dendrites of solid solution $\alpha(\text{Al}) - T_{\alpha(\text{Al})}(T_{\text{liq}})$, °C,
- A-B - range of crystallization of dendrites of solid solution $\alpha(\text{Al})$, °C,
- point B - minimum temperature (onset) of crystallization of eutectic $\alpha(\text{Al})+\beta(\text{Si})$, - $T_{\text{Emin}(\alpha+\beta)}$, °C,
- point C - average crystallization temperature of the $\alpha(\text{Al})+\beta(\text{Si})$, - $T_{\text{E}(\alpha+\beta)}$, °C,
- point X - crystallization temperature of iron-rich intermetallic phases - $T_{\text{E(Fe)}}$, °C,
- point D - crystallization temperature of magnesium-rich intermetallic phases - $T_{\text{E(Mg)}}$, °C,
- point E - temperature of the end of crystallization of EN AC-AlSi7Mg alloy (T_{sol}), °C.

Table 2 summarizes the average characteristic crystallization temperatures of AlSi7Mg alloy obtained from the ATD method (rounded to integer) for different iron contents. Based on ATD studies, the cooling curve of AlSi7Mg alloy is up to about 0.49wt.% Fe content was found to be typical of Al-Si-Mg alloys with sub-eutectic composition, i.e., dendrites of the solid solution $\alpha(\text{Al})$ -at T_{liq} , are formed first, followed by crystallization of the eutectics $\alpha(\text{Al})+\beta(\text{Si})$ and $\alpha(\text{Al})+(\text{Mg}_2\text{Si})+\beta(\text{Si})$, at about 0.5 to about 0.9wt.% Fe, an exothermic effect from crystallization of iron-rich phases was found. It occurs in the temperature range from about 574°C to about 590°C, which is before the crystallization of the $\alpha(\text{Al})+\beta(\text{Si})$ eutectic (about 558°C) and the triple eutectic containing the Mg_2Si phase (about 550°C). Crystallization of these eutectics continues to the temperature of T_{sol} . (from about 530 to 520°C). Iron content in the AlSi7Mg alloy up to more than 0.9wt.% causes

primary crystallization of the iron phases (before crystallization of the dendrites of the solid solution α), accompanied by an exothermic effect at temperatures above 590°C. Further increasing the proportion of iron results in an even more significant increase in $T_{E(Fe)}$ temperatures to as high as 640°C for 2.0wt.% Fe content. Increasing the proportion of iron and the resulting increase in the $T_{E(Fe)}$ temperature value changes the microstructure of the AlSi7Mg alloy.

Table 2. Characteristic crystallization temperatures of AlSi7Mg alloy from ATD analysis¹.

Sample No.	Name of alloy and share of iron, wt. %	Characteristic crystallization temperatures, °C					
		A	B	C	X	D	E
		$T_{liq.}$	$T_{Emin(\alpha+\beta)}$	$T_{E(\alpha+\beta)}$	$T_{E(Fe)}$	$T_{E(Mg)}$	$T_{sol.}$
1.	AlSi7Mg+0.4	590	555	558	---	551	532
2.	AlSi7Mg+0.6	590	556	558	---	550	530
3.	AlSi7Mg+0.8	592	555	557	574	551	530
4.	AlSi7Mg+1.0	595	556	559	579	551	525
5.	AlSi7Mg+1.2	596	554	557	591	551	522
6.	AlSi7Mg+1.4	600	556	557	619	550	522
7.	AlSi7Mg+1.6	603	556	558	624	550	520
8.	AlSi7Mg+1.8	604	555	558	633	550	520
9.	AlSi7Mg+2.0	606	556	558	640	551	520

¹ – the symbols in the table are the same as in Figure 2.

4.3. Microstructure test results

Figure 3 shows representative microstructures of AlSi7Mg alloys with different iron contents. Figure 4 shows X-ray scattered energy spectra and micro-area chemical composition, and Figure 5 shows the X-ray diffractogram.

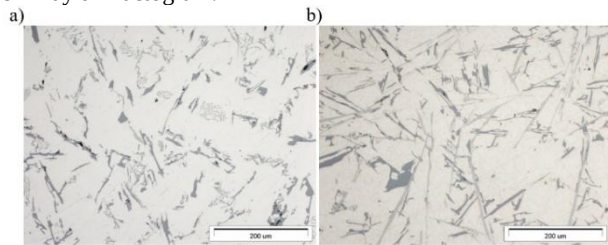


Fig. 3. Representative microstructures of AlSi7Mg alloy with different iron content: a) up to 0.49wt.%; b) from about 0.5 to 0.9wt.%.

The study of the microstructure of the AlSi7Mg alloy shows that, in addition to the traditional components, i.e., the dendrites of the $\alpha(Al)$ solid solution, the silicon crystals included in the $\alpha(Al)+\beta(Si)$ eutectic and the Mg_2Si phase included in the $\alpha(Al)+Mg_2Si+\beta(Si)$ eutectic, the $\beta-Al_5FeSi$ phase was identified. Its structure resembles a “slab” with sharp edges, which on the surface of the metallographic specimen is visible as a “needle-like” separation with pointed corners. The privileged direction of propagation to one side causes the $\beta-Al_5FeSi$ phase, especially at maximum iron content, to be often cracked [19].

It can be seen from Figure 6 that long precipitations of $\beta-Al_5FeSi$ phases (from 600 μm to even 1000 μm - at contents of more than 0.9wt.% Fe) are privileged sites for the formation of porosity, affecting the reduction of interdendritic permeability, thus blocking (inhibiting) the free flow of liquid melt during cooling. The moving crystallization front “does not keep up” with the filling with the liquid metal of the sites “closed” by $\beta-Fe$ phases, causing the formation of shrinkage pores in the vicinity of $\beta-Al_5FeSi$ phases. The presence of voids causes local stresses, which transform into micro- and macro-cracks during the casting operation. This is because igneous separations of $\beta-Fe$ phases oriented with the longer side perpendicular to the given load undergo decohesion, while those arranged in parallel - crack along their arms and undergo fragmentation. Thus, it is reasonable to believe that the $\beta-Al_5FeSi$ phases appear in the microstructure of the AlSi7Mg alloy after exceeding about 0.5wt.% Fe represents the nuclei of pores and/or shrinkage microcracks, increasing the porosity of the castings and reducing the feed capacity by reducing the flow of liquid melt between the branched dendrites of the $\alpha(Al)$ solid solution. This is in line with the view presented in studies [20, 21], which show that these voids are sites of microcrack initiation.

Observations of the microstructure of the AlSi7Mg alloy also revealed numerous clusters of shrinkage-type porosity, examples of which are shown in Figure 6.

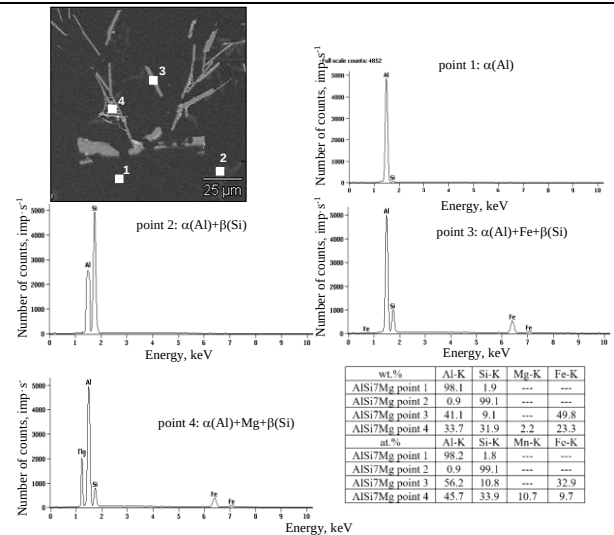


Fig. 4. Microstructure and scattered energy X-ray spectra and chemical composition in micro areas of AlSi7Mg alloy.

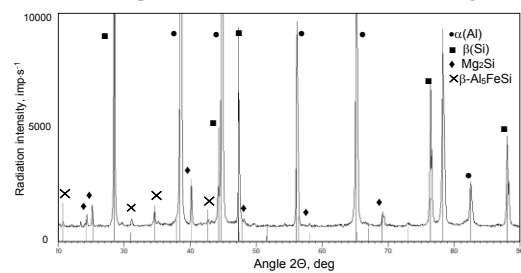


Fig. 5. XRD X-ray of AlSi7Mg alloy with about 0.9wt.% Fe.

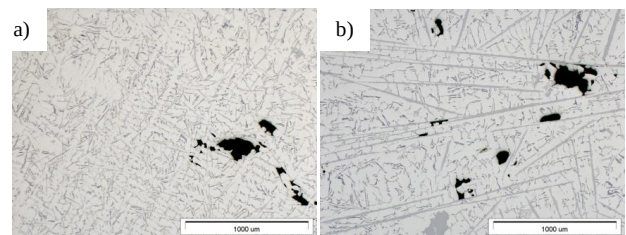


Fig.6. Microstructure of AlSi7Mg alloy with different iron content: a) up to 0.49wt.%; b) from about 0.5 to 0.9wt.%.

5. Summary and conclusions

Based on ATD studies, it can be concluded that the crystallization of AlSi7Mg alloy with increasing iron content proceeds in the following stages:

- for contents up to about 0.45wt.% Fe:
 - Stage 1: start of the crystallization process of dendrites of the solid solution $\alpha(Al)$ - temperature $T_{liq.}$,
 - Stage 2: nucleation and crystallization of the double eutectic $\alpha(Al)+\beta(Si)$ - temperature $T_{E(\alpha+\beta)}$,
 - Stage 3: nucleation and crystallization of multicomponent eutectics $\alpha(Al)+Al_xFe_ySi_z+\beta(Si)$ and triple $\alpha(Al)+Mg_2Si+\beta(Si)$ - $T_{E(Fe)}$ and $T_{E(Mg)}$ temperatures,
 - Stage 4: end of alloy crystallization process – $T_{sol.}$ temperature.
- for contents from about 0.46 to 0.9wt.% Fe:
 - Stage 1: start of solid solution dendrite crystallization $\alpha(Al)$ - $T_{liq.}$,
 - Stage 2: nucleation and pre-neutectic crystallization of $\beta-Al_5FeSi$ - $T_{E(Fe)}$ phase,
 - Stage 3: dual eutectic crystallization of $\alpha(Al)+\beta(Si)$,
 - Further crystallization stages unchanged.
- for contents of more than 0.9wt.% Fe:
 - Stage 1: nucleation and primary crystallization of the phase $\beta-Al_5FeSi$ - $T_{E(Fe)}$,
 - Stage 2: crystallization of solid solution dendrites $\alpha(Al)$ - $T_{\alpha(Al)}$,
 - Stage 3: crystallization of the double eutectic $\alpha(Al)+\beta(Si)$ and further stages without change.

Based on microscopic studies, it can be concluded that in the AlSi7Mg alloy, there are dendrites of solid solution $\alpha(\text{Al})$, which constitute the matrix of the alloy, silicon crystals $\beta(\text{Si})$, which are part of the double eutectic $\alpha(\text{Al})+\beta(\text{Si})$, Mg_2Si phase - as a component of the eutectic $\alpha(\text{Al})+\text{Mg}_2\text{Si}+\beta(\text{Si})$, and separations of the $\beta\text{-Al}_5\text{FeSi}$ phase with lamellar morphology (on the surface of the needle-shaped deposit). Based on ATD, microstructure studies, and literature review [17, 20, 21], it was concluded that the essential criteria determining the formation of a particular type of iron phase include the alloy's iron content and its cooling rate. Phases that crystallize before the formation of the $\alpha(\text{Al})$ solid solution dendrites growing freely in the metallic liquid, or those that form at the same time as the dendritic network (but in the remaining liquid), tend to grow much more significantly ($\beta\text{-Fe}$ phase) than those that form later - during or after the formation of the $\alpha(\text{Al})+\beta(\text{Si})$ eutectic and the $\alpha(\text{Al})+\text{Mg}_2\text{Si}+\beta(\text{Si})$ triple eutectic. The increase in the size of the $\beta\text{-Al}_5\text{FeSi}$ phase, due to the rise in the iron content of the AlSi7Mg alloy, also directly correlates with its silicon concentration. According to research results [20, 21], there is a close correlation between the silicon content and the resulting iron content, which may be acceptable before crystallization (mainly primary crystallization) of the $\beta\text{-Al}_5\text{FeSi}$ phases begins. The iron level is referred to as the so-called "critical" level and follows the relationship [21]:

$$Fe_{kryt} = 0.075 \cdot (\text{wt. \% Fe}) - 0.05 \quad (1)$$

According to this relationship, the Fe_{kryt} for Al-Si alloys with about 7 wt. % Si is about 0.47 wt. % Fe, which is consistent with the results of ATD and microstructure studies of AlSi7Mg alloy presented here. The consequence of neutralizing the morphologically and dimensionally unfavorable $\beta\text{-Al}_5\text{FeSi}$ phases in Al-Si alloys is the introduction of elements that, due to their physicochemical properties, tend to change the unfavorable form of iron phases. Studies [18, 19, 22-27] show that manganese, chromium, and cobalt are additives. Research into the effects of these elements on the crystallization of iron phases, microstructure, and properties of Al-Si alloys is ongoing.

Based on the study, the following conclusions were made:

1. In AlSi7Mg alloy with content up to about 0.45wt.% Fe, crystallizing iron phases are included in multicomponent eutectics with little effect on the microstructure.
2. The content of about 0.5 to about 0.9wt.%Fe causes a change in the crystallization order of the AlSi7Mg alloy. The $\beta\text{-Al}_5\text{FeSi}$ phase separations crystallize before the eutectic $\alpha(\text{Al})+\beta(\text{Si})$, in the temperature range from about 574 to about 590°C. Their average length ranges from about 500 μm to 600 μm .
3. At a content of more than 0.9wt.% Fe, the $\beta\text{-Al}_5\text{FeSi}$ phases crystallize initially in the temperature range from about 590 to as much as 640°C. Their average length ranges from 600 μm to 1000 μm . The free-growing lamellar-ligneous $\beta\text{-Fe}$ phases block interdendritic channels and impede liquid melt flow, causing increased casting porosity.

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Investigation of Bi_2Te_3 single crystal doped with Se obtained using Bridgman method

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Abstract: Researches in this paper included characterization of bismuth telluride single crystal doped with selenium. In order to study the effect of Se doping on the thermoelectric properties of $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ single crystal, an ingot was prepared by Bridgman method. The $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ single crystal in 11 mm × 80 mm size was grown.

The obtained empirical formula does not deviate from the given compound formula. Bulk sample was characterized by Seebeck coefficient (S) as a function of temperature in the range of 40 - 320°C by a homemade impedance meter.

KEYWORDS: SINGLE CRYSTAL, SEMICONDUCTOR, BRIDGMANN METHOD, DOPING, THERMOELECTRIC

1. Introduction

Due to good ability to directly convert heat into electricity and vice versa, thermoelectric (TE) materials have garnered significant attention.

A parameter that evaluates the quality of thermoelectric materials, thermoelectric figure of merit, Z , is determined by the dimensionless value, ZT , [1 - 4] which is defined as:

$$ZT = \frac{S^2 \cdot \sigma \cdot T}{k} = \frac{S^2 \cdot T}{k \cdot \rho} = \frac{S^2 \cdot T}{(k_e + k_l) \cdot \rho}$$

where: S - Seebeck coefficient, σ - electrical conductivity, k - thermal conductivity, T - absolute temperature, ρ - electrical resistance.

Bi_2Te_3 -based alloys have excellent thermoelectric performance at room temperature. Thermoelectric transport properties of doped Bi_2Te_3 have been examined for a long time [5-11].

The most effective composition of n-type low-temperature thermoelectric materials used in thermoelectric coolers and generators in the Bi_2Te_3 - Bi_2Se_3 system was found in the interval up to 33.3% mol Bi_2Se_3 [12-14]. Compounds of bismuth and tellurium have a narrow energy gap, which at room temperature is $E_g = 0.15$ eV.

Doping is an effective strategy for enhancing thermoelectric properties.

The aim of the present study was to obtain bulk single crystal growth by spontaneous nucleation and characterized it by Seebeck coefficient (S).

2. Experimental

Single crystal ingot of Bi_2Te_3 doped with selenium was synthesized using the Bridgman method at the maximum temperature of about 700°C. High purity elements (5 N) were used as the source material. Tellurium (Sigma – Aldrich, 99.999%), bismuth (Sigma – Aldrich, 99.999%) and selenium (Alfa Aesar, 99.999%) were taken in a certain proportion.

The temperature gradient was 2°C/mm in the zone of heating and 5°C/mm in the zone of cooling. The ingot was grown at the speed of 2.2 mm/h.

Measurements of thermoelectric properties of single crystal was carried out by home-made impedance meter based on the "Lagrange ΔT method" [15] with temperature range from 40 to 320°C. Home-made impedance meter simultaneously measured all three thermoelectric parameters, Seebeck coefficient, conductivity and resistivity. The measurement process was performed in vacuum of 10^{-2} to 10^{-3} Pa on sample with a height of about 11 mm and a diameter of 80 mm and took at least 15 hours.

The measurement principle is such that the sample in a vacuum is heated on the hot side to a temperature determined by the measurement program. Once this temperature is reached, the heater is turned off, and the measurements begin. These measurements are taken during the cooling process of the sample at one-minute intervals until the sample cools down below 40°C. The entire process is automated, and the measurement results are

recorded on a computer. It is essential that the upper and lower surfaces of the sample are plane-parallel to ensure optimal contact between the sample and the hot and cold sides of the system.

3. Results and Discussion

Bismuth, tellurium and selenium are merged in a stoichiometric ratio of 2:3, namely with given compound formula of $\text{Bi}_2\text{Te}_{2.7}\text{Se}_3$.

The results of thermoelectric property measurements in the temperature range from 40 to 320°C for a bulk n-type single crystal are given in Figure 1.

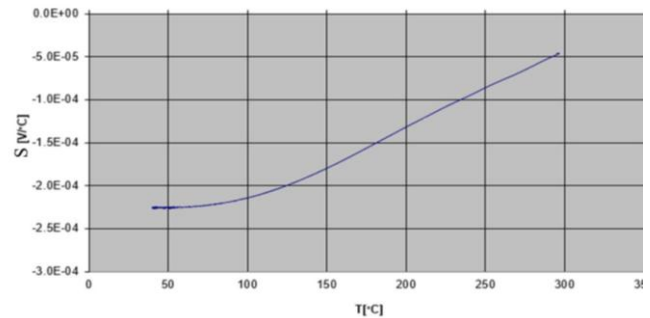


Fig. 1. Temperature dependence of Seebeck coefficient for sample $\text{Bi}_2\text{Te}_{2.7}\text{Se}_3$

Fig. 1. shows the temperature dependence of Seebeck coefficient (S) of sample. A TE characteristic was measured to define quality. These properties strongly depend on carrier concentration, mobility, crystal structure and defects in the crystal structure.

The sample showed value of Seebeck coefficient of $-187 \mu\text{V}^\circ\text{C}^{-1}$ at 40°C to $-163 \mu\text{V}^\circ\text{C}^{-1}$ at 292°C. Seebeck coefficient had a negative sign over the whole investigated temperature range which indicates n type of conduction (major conductivity carriers are electrons).

4. Conclusion

Possibility of producing high quality $\text{Bi}_2\text{Te}_{2.7}\text{Se}_3$ single crystal using the Bridgman method was examined. Single crystal was successfully prepared. The obtained empirical formula does not deviate from the given compound formula.

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Advanced characterization methods of titanium alloy

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Abstract: In recent decades, increasing research in materials science and biomedical engineering has contributed to significant progress in biomedical metallic materials, some of which are titanium materials. The titanium used in the production of biomedical materials is usually alloyed with other elements such as niobium, molybdenum, copper and zirconium. Titanium alloys have become one of the most successful materials for biomedical applications, especially in orthopaedics and dentistry, due to their excellent biological, physical and mechanical properties. This article gives an overview of advanced characterization methods for titanium alloys such as light and electron microscopy, X-ray diffraction, energy dispersive spectrometry, differential scanning calorimetry, differential thermal analysis, thermogravimetry and dynamic mechanical analysis. The article provides of the current status of the development of biomedical titanium alloys and use of advanced characterization methods, in the development of Ti-Cu alloys.

Keywords: ADVANCED CHARACTERIZATION, BIOMEDICAL MATERIALS, TITANIUM ALLOYS

1. Introduction

Materials that are used and adapted for medical purposes are biomaterials. Biomaterials can be bioactive, i.e. they are used for an interactive purpose, such as hip or knee implants. However, biomaterials also have a useful function, such as in heart valves. They are also used daily in surgery, dentistry and drug delivery. If we have to define the term biomaterial, we say: "A biomaterial is a natural or artificial material that consists in whole or in part of a living structure or biomedical device that performs, replaces or enhances a natural function" [1]. When it comes to metals that can be used as biomedical, it is also titanium. Titanium is a hard, shiny and light metal. As a metal, titanium has a high strength-to-density ratio and a high yield strength [2]. The mechanical properties combined with its low density, biocompatibility and non-toxicity make titanium alloys good biomaterials compared to some competing alloys such as SS 316 L and CoCr alloys. Despite the high cost of titanium and titanium alloys, they continue to be used in a number of industries [3]. In general, titanium and its alloys are excellent biomedical materials [4].

2. Application of Ti alloys

The use of titanium and its alloys has increased worldwide in recent years in high-value markets such as aerospace, marine, automotive and biomedical, power generation, heat exchangers, etc. Titanium alloys owe their popularity to their superior characteristics and properties. These include a high strength-to-density ratio, also known as high strength-to-weight ratio or specific strength, and high corrosion resistance. Titanium alloys are used in the marine industry to prevent corrosion from seawater or the environment. They are also used in the aerospace industry to protect the hull from corrosion and thermal damage caused by air friction at hyper- and supersonic speeds [4]. Ti and its alloys are widely used for medical devices and implants in dentistry and orthopedics due to their excellent bone regeneration capacity, mechanical properties and corrosion resistance. Although Ti and its alloys are widely used for biomedical applications, there are still issues that need to be resolved to meet clinical applications. For example, osseointegration with the surrounding bone tissue has been highlighted as a major problem in the initial phase of implant placement. In recent years, much attention has been paid to the development of new Ti alloys and composites with modified processes such as advanced casting methods, powder metallurgy and artificial intelligence to produce dental and orthopedic implants with lower elastic moduli while maintaining higher strength to improve osseointegration. On the other hand, some researchers have focused on modifying the surface of titanium-based medical devices and implants to promote faster bone growth [5]. Titanium is the most common choice in orthopedics for components that are subject to high mechanical demands, such as hip, shoulder, ankle and knee joints, where polymeric materials would not achieve the required strength. As they are osseointegrated over time, titanium implants provide good secondary fixation [6]. Despite much scientific interest and numerous attempts to solve some of the problems encountered with titanium implants, titanium has proven unsuitable for components that are subject to wear, such as the femoral

component in knee arthroplasty or the femoral head in hip arthroplasty, etc. In dentistry, titanium is used for dental screws, prostheses and instruments [7]. Titanium alloys can withstand the mechanical forces that occur during chewing and other oral functions because they have a good balance between strength and durability. This ensures that dental implants can successfully replace natural teeth and function for a long time [8,9]. Constant pH changes that occur with the release of aggressive chemical substances from food and drink, as well as tartar or plaque, can greatly reduce the chances of clinical success and the lifespan of such a biomaterial. Spinal implants where titanium or titanium alloys can be used include rods, artificial discs and wires used to correct spinal deformities in scoliosis. In cardiovascular devices, titanium and its alloys are used as the main components of stents and heart valves [7].

The titanium-based dental alloys that are used alongside the familiar Ti-6Al-4V include Ti-Ag, Ti-Mo, Ti-Nb and Ti-Cu alloys. Implants made from these alloys can have excellent antibacterial properties thanks to the slow release of silver or copper ions in the body [10]. As titanium implants are prone to bacterial infections, which in extreme cases require painful and expensive revision surgery, one of the newer solutions to this problem is the use of copper as an antibacterial agent in the titanium alloy system. The addition of copper provides an excellent antibacterial effect, but the mechanisms are not yet sufficiently understood as these are newer alloys that have been increasingly studied in recent years [11]. Copper is essential in the human body for the maintenance of metabolic processes and daily organ stability [12]. It is also an important component of connective tissue and nerve sheaths. It is a synthetic element of a variety of proteins and enzymes, such as superoxide dismutase, cytochrome c oxidase, dopamine, intrinsic copper blue plasma protein and β -hydroxylase. Copper is also a component of red blood cells and is involved in glucose metabolism and synthesis. It is also involved in the release of proteins and enzymes. Copper is also used as an alloying element to regulate the rate of decomposition. It can therefore be concluded that the role of copper will be very important in the development of these new Ti-Cu alloys [13]. Recent research has shown Ti-Cu alloys with favorable antibacterial properties to be promising materials for the manufacture of medical implants [14]. It has been proven that Ti-Cu alloys used for dental implants can inhibit bone resorption caused by bacterial infections and the formation of biofilms in implants well without compromising corrosion resistance [11]. Alloys with a high copper content of up to 25 % by weight have good biocompatibility and excellent antibacterial properties, while increasing the copper content in Ti-Cu alloys leads to a deterioration in ductility. Ti-2Cu alloys have the best ductility (25-28 %), while Ti-Cu alloys with a copper content of 5-25 % by weight are characterized by significantly poorer ductility. Poor ductility is a serious problem in many clinical applications. Previous studies have shown that excessive intake of copper can cause various stomach problems as well as nausea and even tissue damage. For these reasons, the use of Ti-Cu alloys with a copper content of more than 6 % by weight in various biomedical

applications may be associated with certain risks. As this is still relatively new research, the literature is very limited [15].

3. Description of advanced characterization methods

This article gives an overview of some advanced methods for the investigation of titanium alloys, such as light and electron microscopy (SEM, TEM), X-ray diffraction (XRD), energy dispersive spectrometry (EDS), differential scanning calorimetry (DSC), differential thermal analysis (DTA), thermogravimetry (TG) and dynamic mechanical analysis (DMA), with an emphasis on the characterization of Ti-Cu alloys.

The principle of light microscopy is very simple. It includes any type of microscopic work that provides information about a sample illuminated by visible light [16]. A light microscope consists of a system of two lenses for magnifying small objects, one of which is called the objective, which produces an image of the object in the central image plane. This image can be viewed with a second lens, the eyepiece, to provide additional magnification [17]. The most common and general form of light microscopy is brightfield microscopy, in which the light passes directly through or is reflected by the specimen [18]. The use of light microscopy has some limitations. One disadvantage is the particle size, the lower limit of which is 1 μm . However, there are also devices that can determine up to 0.3 μm . Another disadvantage of this microscope is the low image contrast when using a video camera [19].

Scanning electron microscopy is one of the most common methods of visualizing the microstructure and morphology of materials that can operate from low to high magnification using secondary electrons (SE) and backscattered electrons (BE). SEM analysis can provide information about the surface topography, crystal structure, and chemical composition [20]. In this microscope, a beam of low-energy electrons is focused on the material and scans the surface of the sample. When the beam reaches and penetrates the material, various interactions occur, resulting in the emission of photons and electrons from or near the sample surface. The received signals generated by the interaction between electrons and sample are captured by different types of detectors to produce an image, depending on the SEM mode used [21].

The methods of energy dispersive spectrometry (EDS) is often used in conjunction with scanning electron microscopy (SEM) to analyse the elemental composition of materials [22]. This technique enables the elemental analysis of the sample and thus the identification of the elements present and their distribution. When the sample is bombarded with an electron beam, characteristic X-rays are detected. The energy and intensity of these X-rays are determined to identify the elements present in the sample and their relative concentration. When an electron beam interacts with the atoms of a sample, the atoms can be excited and eject electrons from their inner shells, creating a gap in the electron shell which is then filled by an electron from a higher energy level, releasing energy in the form of an X-ray photon. The energy of the emitted X-ray is characteristic of the element that emitted it, so that the element can be identified. The X-rays emitted by the sample are detected by an energy-dispersive X-ray detector. The detector produces a spectrum of X-ray emissions that can be used to identify the elements present in the sample and their relative concentrations. EDS provides valuable information about the distribution of elements and the chemical composition of a material at it is therefore an important tool for the analysis and characterization of materials and is therefore widely used in engineering and materials science to determine the composition of materials, including ceramics, polymers and metals [2].

Transmission electron microscopy (TEM) is a method in which an electron beam is passed through a thin sample to obtain an image [23]. This technique enables high-resolution visualization of the atomic arrangement of a material and its microstructure. Imaging with this type of microscope offers a much higher resolution than light-based imaging techniques, as the sample is illuminated with electrons rather than light. The thickness of the sample, i.e. the amount of material through which the electron beam must pass, and

the material of the sample affect the amplitude and phase changes of the transmitted beam, which ultimately results in the image contrast. Using TEM it is possible to analyse high-resolution images with diameters of just 0.2 nm [24]. It operates at 200 kV and is equipped with a high-resolution camera to capture the atomic structure of the samples. The lens used is an objective lens, which is located above the sample and whose task is to focus the electron beam onto the sample and capture the scattered electrons that pass through the sample itself. The objective lens is usually a magnetic lens. A magnetic lens usually consists of one or more magnetic coils. Depending on the atomic structure and composition of the material, the electrons interact with the atoms of the sample and are either scattered or absorbed. The scattered electrons are then collected by a detector and used to create an image of the sample. The images obtained by TEM analysis can provide information about the atomic structure, crystallographic orientation and defects of the sample with a resolution down to the sub-Angstrom range [25].

X-ray diffraction analysis (XRD) is a powerful method for characterizing crystalline materials. It provides information on the phases, structures, crystal texture and other structural parameters such as average grain size, crystal deformation and defects, and crystallinity [26,27]. X-ray diffraction peaks are generated by the constructive interference of a monochromatic X-ray beam scattered at specific angles from each set of lattice planes in the sample. The intensity of the peaks is determined by the distribution of atoms within the lattice. An X-ray diffraction pattern is a fingerprint of the periodic arrangement of atoms in a given material [27]. The diffraction pattern is a simple sum of the diffraction patterns of the individual phases [5].

Differential scanning calorimetry (DSC) is a method used to determine changes in the structural properties of a sample as a function of time and temperature, that is, it is used to study the thermal transitions that occur in materials during heating/cooling [3,28]. It provides essential information about the structure, from stability to structural changes. It consists of calculating the energy required to increase the temperature of the sample in relation to the reference material [4]. DSC can also be used to analyze liquids and solids. Two types of DSC are used, heat flow DSC and power compensated DSC, depending on the mechanism of operation. In heat flow DSC, the sample vessel and the empty (reference) vessel are placed on a thermoelectric disk enclosed in a furnace that is heated by transferring heat to the vessels through the disk. The temperature difference between the two vessels is measured by thermocouples, which is then converted into a heat flow signal through a further calibration procedure. In power compensated DSC, separate furnaces hold the sample and reference vessel at the same temperature. The difference in heat power between the sample and the reference vessel is determined and plotted as a function of temperature or time. Endothermic events are conventionally displayed as rising signals in power compensated DSC and as falling signals in heat flow DSC. In order to avoid reactions with the atmosphere, nitrogen or helium gases are used to purge the instrument [3].

Differential thermal analysis (DTA) is used to determine thermodynamic properties that are important for understanding the behavior of materials at different heating and cooling rates, in an inert, reducing or oxidizing atmosphere or in different gaseous atmospheres. This analysis encompasses a group of techniques in which the physical properties of a substance are measured under a controlled temperature program. It is basically based on the principle that a substance, after heating, undergoes reaction and phase changes that involve the emission or absorption of heat. The sample to be analyzed is placed with a control (of known properties) and an inert material, and the changes in phase and temperature are recorded. By comparing the DTA curves obtained from the unknown with the curves of the known elements, the substance is identified [29].

Thermogravimetric analysis (TGA) is a method in which the mass of a substance or sample is measured as a function of temperature at a constant heating rate or time while the sample is

subjected to a controlled temperature program in a controlled atmosphere. Here, the sample is usually heated at a constant heating rate. The sample is placed on a high-precision thermobalance inside a furnace. The mass of the sample is then continuously and precisely monitored during the heating or cooling process. The TGA consists of a sample pan supported by a precision balance. The results of the TGA measurement are displayed as a TGA curve in which mass or mass percentage is plotted against temperature or time. The choice of temperature program will depend on the type of sample being analyzed. It is also very important to provide a controlled environment to minimize oxidation or other unwanted reactions [30].

Dynamic mechanical analysis (DMA) is a method that complements the information obtained from differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) and measures the properties of materials when they are deformed under periodic stress. DMA is the preferred method for measuring the glass transition temperature and other minor changes in the phase or structure of a material. This method determines the elastic modulus, viscous modulus, and damping coefficient as a function of temperature, frequency, or time. The results are usually presented in the form of a graph [31].

4. Ti-Cu alloys

As Ti-Cu alloys are still poorly investigated, they have become increasingly interesting to scientists as new biomedical alloys in recent years. All Ti-Cu alloys must be subjected to various analyses and characterization methods before they can be used, some of which are mentioned in this article.

Hayama et al [32] investigated cast Ti-Cu alloys with a copper content of 5, 7.1 and 15.0 wt. %. Using advanced characterization methods, they obtained the following results. SEM images of Ti-5Cu and Ti-7.1Cu alloys after rapid cooling showed a martensite microstructure, and after heat treatment and slow cooling a eutectoid microstructure, while in the Ti-15Cu alloy the Ti₂Cu phase was formed. TEM micrographs of the Ti-7.1Cu alloy showed a low volume fraction of martensite microstructure and a eutectoid microstructure after heating, and the Ti₂Cu phase was visible after slow cooling. The DSC analysis revealed an exothermic transformation at 575 °C during the first heating cycle, which can be attributed to phase decomposition. The second heating cycle to which the same alloy was subjected did not result in an exothermic reaction near 575 °C, suggesting that this type of transformation could be caused by an irreversible metastable phase transformation involving the complete decomposition of the martensitic phase and indicating its low stability at high temperatures. Hayama et al. found that the microstructure of the as-cast alloy samples exhibited a martensitic structure, while the heat-treated samples consisted of α/α' and Ti₂Cu intermetallic phases formed during eutectoid transformation. The results obtained suggest that the β -phase cannot be stabilized at low temperatures after rapid cooling and that higher cooling rates are required to stabilize this phase. It was also found that a higher Cu content increases the volume fraction of the eutectoid structure and thus the amount of intermetallic Ti₂Cu phase [32].

Kikuchi et al [33] investigated a Ti-Cu alloy with five different concentrations of copper (0.5, 1, 2, 5 and 10 wt. %). Each of these alloys was produced by melting in a furnace. The alloys were then analysed. The microstructural analysis using SEM of the tested showed Ti-0.5Cu alloy shows a needle-like α -Ti structure, Ti-1Cu and Ti-2Cu were identical, α -Ti formed on the surfaces of the previously formed β -grains, while Ti-5Cu showed a hypoeutectoid structure. This structure includes primary precipitates of α -Ti and lamellae of α -Ti and Ti₂Cu phases that essentially transformed from β -Ti at the eutectoid temperature. The alloy with 10 wt. % Cu showed a eutectoid microstructure comprising primary precipitates of α -Ti and eutectoids, i.e. lamellae of α -Ti and Ti₂Cu transformed from β -Ti at the eutectoid temperature. XRD analysis of the investigated Ti-5Cu and Ti-10Cu alloys revealed two peaks corresponding to reflections from Ti₂Cu, while all other peaks were from α -Ti, while only peaks corresponding to α -Ti were detected in

the Ti-2Cu alloys. SEM analysis showed cracks in the alloys with 1, 5 and 10 wt. % Cu, while the cracks in the Ti-10Cu alloy were smaller. From this, Kikuchi et al. concluded that these alloys have satisfactory properties for use in prostheses and bridge frames in dentistry [33].

Pina et al [34] investigated titanium alloys with a copper content of 3, 7.1 and 12 wt. %. The alloys were produced by conventional powder metallurgy using the technique of mixing elemental powders and then characterize. Firstly, the microstructural analysis using SEM revealed that the Ti-3Cu and Ti-7.1Cu alloys have a hypoeutectoid structure with an increase in the amount of eutectoid at the grain boundary due to the increase in the copper content in the alloy, while in the Ti-12Cu alloy the structure is close to eutectoid with α -Ti phases. In all cases, pores were visible in the samples regardless of the copper content. The porosity decreased with increasing copper content in the alloys. XRD analysis of the Ti-xCu alloys showed that the volume fraction of the Ti₂Cu phase increased with increasing copper content in the alloys, which was consistent with the data obtained by SEM analysis of the samples. Using characterization methods, Pina et al. found that the alloys analysed have potential for use in dental medicine from the results obtained [34].

Akbarpour et al [35] investigated a new Ti-50Cu alloy that was mechanically alloyed and sintered in a planetary mill. The alloy was analysed by XRD after different grinding times, and after a grinding time of 5 hours, the samples showed very sharp diffraction peaks of crystalline Cu and Ti with a small shift in peak positions due to the formation of a solid solution during grinding. Due to the higher solubility of the Ti atoms in the Cu lattice, the shifts of the Cu peaks were larger than the shifts of the Ti peaks. After 15 hours of grinding, only a broadening of the Cu and Ti peaks and a significant decrease in their intensity was observed, while in samples milled for 30 hours, the disappearance of most of the individual Cu and Ti peaks was observed, but TiCu peaks with lower intensity were visible. XRD analysis showed that the intermetallic phase had a nanocrystalline structure. Using TEM analysis, the average size of the synthesized TiCu phase was determined to be 8.0 ± 1.1 nm by the intercept method. For all samples, XRD analysis after sintering showed the formation of primary Ti₂Cu₃ and TiCu phases as well as secondary CuTi₂ and Cu₄Ti phases. It was also found that with increasing grinding time, the proportion of Ti₂Cu₃ phase in the samples increased, while the proportion of TiCu phase decreased. It was concluded that as the grinding time increased, the proportion of TiCu phase in the sintered samples decreased while the proportion of Ti₂Cu₃ phase increased. Akbarpour et al. concluded that intermetallic Ti-Cu alloys can be a very good substitute for expensive biomedical implants [35].

Ukabhai et al [36] investigated cast Ti-Cu alloys. The alloys tested had different chemical compositions: Ti-20Cu, Ti-33Cu, Ti-40Cu and Ti-50Cu. The XRD analysis of the Ti-33Cu alloy confirmed the presence of the TiCu phase, which was also visible in the SEM analysis. In the Ti-20Cu sample, the α -Ti and Ti₂Cu phases were detectable. The results of the DSC analysis for the Ti-40Cu alloy clearly revealed a main peak, which showed that the formation of Ti₂Cu was congruent, meaning that the reaction involving (β Ti) and Ti₂Cu was eutectic and not peritectic, which was the reason why no clear eutectic structure was observed in Ti-20Cu. The temperature curves determined by DSC analysis showed that only one peak persists in the Ti-33Cu alloy, indicating that the formation of Ti₂Cu is congruent and that an L(Ti)+Ti₂Cu reaction occurs. It was also confirmed that the eutectic points are much closer to Ti₂Cu than to Ti. No Ti₃Cu phase was found [36].

Urban et al [37] where investigate the Ti₇₅Cu₂₅ obtained by mechanical alloying, after which certain analyses were performed. Various analyses were then carried out. The results of the SEM analysis of the tested alloy during the different stages of the grinding process showed particles with irregular and dendritic shapes belonging to titanium and copper, respectively. After 1 hour of grinding, the titanium and copper particles were crushed and plastically deformed by the impact of the balls. After 20 hours of grinding, the particles were hardened and fragmented due to their

brittleness. With increasing grinding time, the particle sizes continued to decrease up to a grinding time of 60 hours. The results of the DTA analysis of the alloys during different grinding phases showed narrow peaks with high intensity. As the grinding time increased, the crystal peaks began to broaden as a result of the reduction in particle size and the intensities of the elemental crystal diffraction peaks decreased. The diffraction pattern after 20 hours of grinding showed only a broad diffuse peak with a maximum at $2\theta = 40^\circ$, indicating that the elemental powder mixture had transformed into a nanocrystalline and/or amorphous phase, while the diffraction patterns of powders ground for a longer time did not show any significant change. The results of the TEM analysis of the alloy milled for 15 hours showed the presence of a nanocrystalline phase, while in the alloy milled for 60 hours, the crystalline and nanocrystalline phases completely disappeared and transformed into an amorphous phase. Urbani et al. concluded that the $\text{Ti}_{75}\text{Cu}_{25}$ alloy can be used in dentistry and medicine [37].

Wang et al [38] investigated Ti-xCu alloys with different copper contents of 3, 5 and 7 wt. %. The alloys were produced by arc melting and then analysed. DSC analysis of the samples of Ti-3Cu and Ti-5Cu alloys revealed two endothermic peaks associated with the eutectoid transformation $\alpha + \text{Ti}_2\text{Cu} \rightarrow \alpha\text{Ti} + \beta\text{-Ti}$ and the β transus, $\alpha\text{-Ti} + \beta\text{-Ti} \rightarrow \beta\text{-Ti}$. The copper content of the Ti-7Cu sample was close to the eutectoid copper content. The XRD diagrams of the Ti-xCu alloys ($x = 3, 5$ and 7) showed diffraction peaks of the $\alpha\text{-Ti}$ phase and slight peaks of the Ti_2Cu intermetallic phase, and no peaks of $\beta\text{-Ti}$ were found. In addition, an increase in the intensity of the diffraction peaks of the Ti_2Cu phases was observed in all the alloys analysed. Microstructural analysis of the alloys showed that each Ti-Cu alloy showed fine precipitates identified as Ti_2Cu phase dispersed in the $\alpha\text{-Ti}$ matrix. SEM analysis of the Ti-5Cu alloy revealed the presence of precipitates with a size of 200–800 nm that were uniformly distributed in the matrix, while EDS analysis revealed the presence of the Ti_2Cu phase, which had a similar morphology as observed with an optical microscope. TEM analysis of the same alloy together with EDS and SEM analysis revealed that the microstructure was mainly composed of a lamellar, secondarily precipitated $\alpha\text{-Ti}$ phase separated by Ti_2Cu phase particles, indicating that the primary $\beta\text{-Ti}$ phase was completely transformed during the eutectoid transformation $\beta\text{-Ti} \rightarrow \alpha\text{-Ti} + \text{Ti}_2\text{Cu}$ in the cooling process. Through this research, Wang et al. concluded that the Ti-5Cu alloy with the microstructure of $\alpha\text{-Ti} + \text{Ti}_2\text{Cu}$ has great potential for clinical application as dental implants [38].

4. Conclusion

Based on research to date, titanium alloys are emerging as one of the most successful alloys for biomedical applications. This review describes the advanced characterization methods used in the development of titanium alloys. It can be concluded that further efforts should be made to obtain new Ti-Cu alloys with improved properties and characteristics for biomedical applications in order to reduce the number of implant failures and the number of repeat surgeries.

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Phase transformations in titanium biomedical materials

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Abstract: New types of materials have been developed for years, including titanium-based alloys, which have the potential for various applications. Due to the combination of their very good mechanical properties with outstanding corrosion resistance and excellent biocompatibility, titanium alloys are developing into materials that can be used in aerospace, automotive, energy systems and especially in medicine. A fundamental understanding of the phase transformations that occur at high temperatures in all these cases, especially during cooling from elevated temperatures, is necessary to achieve optimal mechanical properties of titanium alloys. It is known that the mechanical properties of titanium alloys depend on a significant extent upon the microstructure. Therefore, it is very important to understand the nature of the phase transformations that occur under different heat treatment conditions the leads to microstructure development of titanium alloys microstructure. The aim of this article is to review the current state of knowledge and previous research and to point out some of the most interesting phase transformations of titanium alloys.

Keywords: TITANIUM ALLOY, BIOMEDICAL MATERIALS, PHASE TRANSFORMATIONS, MICROSTRUCTURE

1. Introduction

About 70-80% of the different types of implants are made of metallic biomaterials. Metallic biomaterials are extremely important for the reconstruction of damaged tissue, especially hard tissue, which is associated with an improvement in the patient's quality of life. Considering the fact that the world population is ageing and the elderly population is at greater risk of hard tissue failure, the demand for metallic biomaterials has been increasing rapidly for years. Research has shown that the mechanical and biological biocompatibility of metallic biomaterials needs to be greatly improved in order to reduce the number of failed operations. The biofunctionality of metallic biomaterials also needs to be improved, which is the goal of future research [1]. Depending on the specific application, biomedical materials can be used in different parts of the human body, e.g. as intravascular stents, heart valves, heart simulators and replacement implants in knees, hips, elbows, shoulders and also in dentistry. In the elderly population, implants are most commonly used for hip replacement. These diseases worsen over time and lead to a gradual deterioration of the mechanical properties of the bone due to the loss of its self-healing function and excessive loading [2].

Metals have been considered promising materials in various industries for years [3]. Today, many metals are known that are characterized by good properties and possibilities for various applications. Some of the metal studied are iron, aluminium, nickel and titanium. In addition to their good properties, iron alloys also have some disadvantages, such as their large mass. Steel decomposes slowly and has a higher stiffness than bone, but is highly biocompatible. The decomposition of steel occurs through the mechanism of corrosion, which occurs when a metal sample comes into contact with human body fluids through electrochemical dissolution [4,5]. The porous structure of steel is one way to improve corrosion in these alloys [6]. Another known metal is aluminium. The potential toxicity of aluminium alloys has been the subject of debate throughout history. When aluminium ions are released in significant quantities, they can have neurotoxic effects and contribute to diseases such as Alzheimer's, multiple sclerosis and dementia in Parkinson's patients [7]. Second popular metal is nickel, which is often used as alloying element for stainless steel. Today, however, nickel is considered a high-risk element due to incompatibilities. For this reason, nickel is avoided as far as possible as an additive for metallic biomaterials [1]. For these reasons, researchers are looking for other, more favorable materials to solve these problems [8]. One metal that has recently attracted the interest of more and more scientists and researchers is titanium [9].

Titanium is the fourth most abundant structural metal in the earth's crust, behind only iron, aluminium and magnesium [10]. Pure or elemental titanium has a relatively low density and a low modulus of elasticity as well as low thermal conductivity. Its properties also include high reactivity with various elements as well

as good strength and good corrosion resistance. Tab. 1 shows selected properties of titanium compared to some competing metals such as iron, aluminium and nickel [9]. In order to obtain alloys that are suitable for biomedical purposes, copper is increasingly being added to titanium as an alloying element [11].

Copper is an essential trace element in the human body. It not only has strong antibacterial properties, but also offers good ductility and hardness. The addition of copper gives the alloys improved corrosion resistance, which is a significant advantage for these alloys. The antibacterial effect of Ti-Cu alloys depends largely on the copper content. Too high a copper content can cause various problems, including cytotoxicity. It can therefore be said that the copper content plays an important role in the development of new biomedical alloys [11].

Table 1: Physical properties of titanium compared to certain metals [9].

Metals	Ti	Fe	Al	Ni
Density (g/cm ³)	4.5	7.9	2.7	8.9
Elastic modulus (GPa)	115	215	72	200
Melting point (°C)	1670	1538	660	1455
Thermal conductivity (W/mK)	15-22	68-80	221-247	72-92
Corrosion resistance	high+	low	high	medium
Reactivity with oxygen	high+	low	high	low

2. Crystal structures of titanium

Pure titanium can crystallize in different crystal structures in a wide temperature range. A complete transformation from one crystal structure to another, where the "transition temperature" is the corresponding transformation temperature, is called an allotropic transformation [12]. Titanium can occur in the form of two crystallographic structures (Fig. 1), namely: α -phase, which has a hexagonal close-packed (hcp) crystal structure, and β -phase, which has a cubic volume-centered (bcc) crystal structure.

At 883 °C, titanium undergoes a temperature transformation from a hexagonal close-packed to a cubic volume-centered structure, which remains stable up to the melting point, i.e. a temperature transformation from the α to the β phase takes place [13,14].

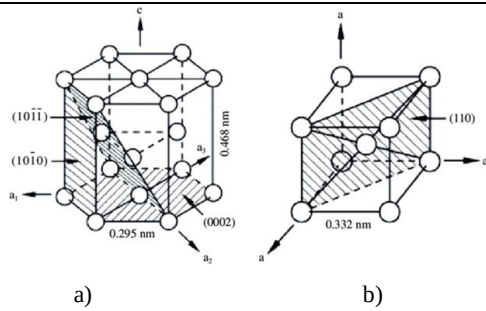


Fig. 1 Crystal structures of a) α (hcp) and b) β (bcc) titanium [15].

Fig. 1 a shows the hexagonal close-packed crystal structure at low temperatures together with the three most densely packed lattice planes and the lattice parameters shown at room temperature. The resulting ratio $c/a = 1.587$ for pure α -Ti is less than the ideal ratio for the hcp structure with $c/a = 1.633$. Fig. 1 b shows the unit cell of the bcc β phase. The figure shows one variant of the most densely packed $\{110\}$ lattice planes as well as the lattice parameter of pure β -Ti at a temperature of 900 °C [16]. When alloying elements are added to titanium that increase the stability of the α phase at higher temperatures, they are called α stabilizers, while on the other hand, β stabilizers can be added that increase the stability of the β phase at lower temperatures. Neutral elements, on the other hand, have no influence on the transformation temperature [17]. Fig. 2 shows different categories of titanium phase diagrams formed with different elements. Ultimately, each element added to pure titanium will determine the mechanical properties by affecting the stability of the α and β phases [18].

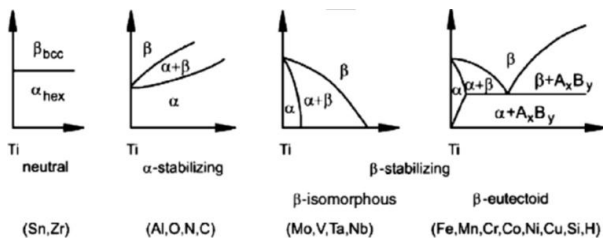


Fig. 2 Different phase diagrams formed with different elements [18].

α –stabilizers

α –stabilizers are both substitutional and interstitial alloying elements. Substitutional α stabilizers are elements such as germanium, aluminum and gallium while interstitial α stabilizers are nitrogen, oxygen and carbon. Of the above substitutional alloying elements, aluminum is the most widely used, since it has a high solubility in the α and β phases. Aluminum also reduces the density of the alloy. The addition of aluminum in titanium alloys is limited to 5-6 wt. % since with increasing aluminum content, the Ti-3Al (α_2) phase will form, while at about 5 wt. % aluminum, a two-phase region $\alpha + \text{Ti-3Al}$ is formed. The formation of the Ti-3Al (α_2) phase makes titanium alloys brittle. Although their solubility is much lower than aluminum and oxygen, other substitutional alloying elements are rare earth elements such as scandium, yttrium and lanthanum. On the other hand, oxygen, carbon and nitrogen are considered extremely strong α -stabilizers. Carbon has a much lower solubility in the α phase than oxygen or nitrogen. These alloying elements tend to harden the alloy, but also reduce the ductility of titanium alloys. Commercially pure titanium at room temperature consists primarily of the α -phase. As alloying elements are added to titanium, they change the amount of each phase present and the temperature of the β transition [19].

β -stabilizers

Since during heating at a temperature of about 883 °C α transforms into a β structure, this transformation temperature can be raised or lowered depending on the type and amount of impurities

or additions of different alloys. β -stabilizers are transition (Fig. 2) metals and are divided into two categories: β -isomorphous and β -eutectoid. β -isomorphous stabilizers have complete solid solubility with β Ti and some of them are vanadium, niobium and molybdenum. Elements such as tantalum and tungsten are rarely used due to their density. Among the β -eutectoid stabilizers commonly used for alloying with titanium are silicon, iron, chromium, while nickel and manganese have very limited use. Hydrogen is an element that has a low eutectoid temperature of 300°C and forms a β eutectoid, i.e. it stabilizes the β phase. Chromium is limited to 5 wt. % because otherwise an undesirable TiCr_2 intermetallic compound will form. Similar to chromium, the amount of iron is also limited to 5.5 % by weight. On the other hand, silicon is a common alloying element for titanium alloys at high temperatures [12]. Elements such as silicon and iron increase the strength and reduce the ductility of titanium products. A well-known and useful parameter for a given titanium alloy composition, which characterizes the stability of the β phase, is the molybdenum equivalent ($[\text{Mo}]_{\text{eq}}$). It is a quantitative measure of the combined effects of β phase stabilizing elements, α phase stabilizing elements, and neutral elements contained in titanium alloys on β phase stability [18]. In addition to the mentioned α and β stabilizers, there are also some neutral elements such as, for example, tin, zirconium, silicon used for some purpose other than the enhancement of α and β phases such as strengthening or processing [20,21]. The mentioned elements are added to metastable alloys of the β Ti phase because they are considered to change the kinetics of the hexagonal isothermal formation of the ω -phase during aging, which in turn stabilizes the bcc β -phase. Some studies have shown that the addition of zirconium or tin to binary Ti-V alloys reduces the volume fraction of the isothermal ω -phase and suppresses its nucleation in sufficiently high concentrations. Similar results were observed in the same study only for the addition of zirconium in binary Ti-Mo alloys [22].

3. Classification of titanium alloys

Ti alloys are categorised into three basic types depending on their phase composition, namely:

- α -Ti alloys,
- $\alpha+\beta$ Ti alloys,
- β -Ti alloys.

There are two main groups of α -Ti alloys, namely super- α and near- α -Ti alloys. Super- α alloys contain a large amount of α -stabilising alloying elements, more than 5 wt. %. These alloys consist entirely of α -Ti grains. Another type of these alloys, the near- α alloys, contain a large amount of α -stabilisers with a lower amount of β -stabilising elements of less than 2 wt. %. The microstructure of these alloys consists of a small volume fraction of β -Ti grains distributed between a much larger volume fraction of α -Ti grains. They are characterized by better hardness properties compared to super α -Ti alloys [23]. Nearly α -Ti titanium alloys are highly valued for their low density, exceptional strength and corrosion resistance. They are suitable for applications in the aerospace, automotive and medical technology industries. They also have good resistance to high temperatures and are therefore suitable for use in extreme temperature conditions. Alloys close to α -Ti offer an excellent weight-to-strength ratio, enabling the production of lightweight components while maintaining structural integrity. This is one of the key benefits in aerospace applications for improved fuel efficiency and performance even at temperatures up to 540 °C [24]. When using these alloys at elevated temperatures from 300 °C to 650 °C, interactions between α -Ti alloys and salts present in the atmosphere above the ocean can lead to limitations in application and a reduction in service life. An adjustment of the types and contents of certain alloying elements is necessary to improve the corrosion and mechanical properties of α -Ti alloys. Alloying elements such as molybdenum and copper have been added to develop high-temperature titanium with high heat resistance and good oxidation behavior up to 600 °C [25]. They are generally used in non-structural applications where good corrosion resistance and

low strength are required [22]. The main hardening mechanism of α -Ti alloys is the formation of solid solutions, texture hardening and precipitation hardening through the formation of the α_2 phase [20]. The modulus of elasticity and yield strength of α -Ti alloys are between 102 and 104 GPa and 170 and 485 MPa respectively. The maximum tensile strength is in the range of 240 to 550 MPa. α -type Ti alloys are not used as implants due to their low mechanical strength at room temperature. Nevertheless, commercially pure titanium belongs to the first generation of biomedical materials that were used as biomaterials for dental and other medical applications [26].

Ti $\alpha+\beta$ alloys contain between 4 and 16 % β stabilisers [27]. These are two-phase alloys that contain a mixture of both phases. They are most commonly used for applications that require good toughness, higher strength and excellent corrosion resistance [22]. At room temperature, $\alpha+\beta$ alloys usually contain 10-20 % β phase [28]. Nearly β -alloys, also known as metastable β -alloys, contain a small amount of α -stabilisers and 10 to 15 % of β -stabilisers. β -stabilisers promote the retention of the β -phase in a metastable state at room temperature. The alloys can be heat treated by ageing, resulting in the deposition of a very fine α -phase dispersed in the β -matrix [27]. In addition to all the properties mentioned above, these alloys with $\alpha+\beta$ also exhibit the ability for rapid osseointegration in the body [29]. The combination of higher strength and density of 4.5 g/cm³ and phase stability results in excellent specific properties that extend up to high temperatures of around 600 °C [30]. The best-known alloy is the biomedical Ti-6Al-4V, which accounts for 50 % of titanium production. In the Ti-6Al-4V alloy, vanadium is a stabiliser of the β -phase. The alloy had good mechanical properties, but it was later discovered that aluminium and vanadium are not biocompatible and cause irritation and various diseases [14]. The first generations of alloys without harmful effects on the human body were alloys containing non-toxic components such as molybdenum, niobium and iron [20,26].

β -Ti alloys are one of the most versatile groups of materials in terms of their microstructure, mechanical properties and processing. These alloys include stable β -, metastable β - and β -rich α/β -alloys. They offer an alternative to α/β alloys due to the better heat treatment options. They have a wide range of strength-to-weight ratios as well as some other good properties such as a low modulus of elasticity, reasonable biocompatibility and high corrosion and wear resistance. Compared to α/β alloys, β -Ti alloys have better fatigue resistance [31,32].

Compared to $\alpha+\beta$ alloys, these alloys contain larger amounts of β stabilisers such as zirconium and tantalum, molybdenum and smaller amounts of α stabilisers without the formation of intermetallic phases [20]. To improve their strength, they can be heat-treated at temperatures between 450 and 650 °C due to the partial transformation of the β -phase into the α -phase. Increasing the β -phase content in β -type Ti alloys can improve some properties such as plasticity, toughness and heat treatability and, on the other hand, reduce the modulus of elasticity. Compared to $\alpha+\beta$ -type Ti alloys, these alloys have better biocompatibility and are considered to be more corrosion resistant in the human body [26]. Most β -type Ti alloys have a higher density than other types of Ti alloys due to the higher content of alloying elements such as niobium, molybdenum, tantalum and zirconium [33]. These elements are quite rare, which leads to higher raw material costs for alloy production. Some of the β -Ti alloys used are Ti-15V-3Cr-3Al-3Sn, Ti-15Mo-2.7Nb-3Al-0.2Si, Ti-3Al-8V-6Cr-4Mo-4Zr and Ti-10V-2Fe-3Al alloys [34]. Due to certain difficulties in the production of β -Ti alloys, researchers have focused on the development of new, cheaper alloys consisting of alloying elements such as manganese, chromium, iron and tin, which are cheaper but also have good mechanical properties for biomedical applications [35]. An important prerequisite for the use of these alloys in biomedicine is their non-toxicity. β -Ti alloys can be used as viable alternatives in the manufacture of dental implants. Due to their high strength and low density, these alloys are excellent structural materials for use in airplanes. The choice of alloy constituents and the desired properties of titanium alloys are controlled by application-specific

design criteria [32]. These alloys can be used in the construction of power plants, but also in the automotive industry and in the manufacture of sports equipment [36].

4. Phase transformations of Ti-Cu alloys

Cast Ti-Cu alloys have been studied for many years [37]. To date, many studies have been carried out on the binary Ti-Cu phase diagram and on the possibilities of different titanium and copper contents in the alloys. The Ti-Cu binary phase diagram shown in Fig. 3 shows the possibility of obtaining multiple intermetallic phases, while Tab. 2 contains the crystal structure data of the Ti-Cu alloy, which is discussed below [37,38,39].

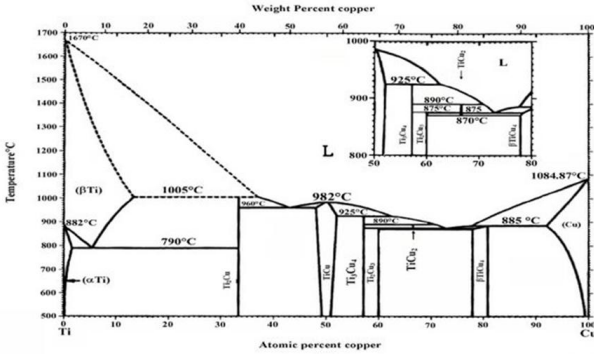


Fig 3: Ti-Cu binary phase diagram [37,38].

Table 2: Data on the crystal structure of Cu-Ti alloy[39].

Phase	Composition, wt% Cu	Pearson symbol	Space group
(α Ti)	0 to 2.1	hp2	P63/mmc
(β Ti)	0 to 17.2	cl2	Im3m
Ti2Cu	39.9	tl6	I4/mmm
TiCu	55 to 59	tP4	P4/nmm
Ti3Cu4	63.9	tl14	I4/mmm
Ti2Cu3	67	tp10	P4/nmm
TiCu2	72.7	oC12	Amm2
TiCu4	83 to 84.9	oP20	Pnma
α TiCu4	-83 to 84.9	tl10	I4/m
(Cu)	94 to 100	cF4	Fm3m
Metastable phases			
TiCu3		oP8	Pmnm
B''		tP2	P4/mmm

The most interesting point of the Ti-Cu diagram is that in previous studies, two different versions were published about the stability of different intermetallic compounds on both the Cu-rich side and the Ti-rich side. Some researchers spoke of the existence of Ti₃Cu and others of the existence of Ti₂Cu as a stable equilibrium phase.

In 1912, the first study on the Ti-Cu binary alloy system was conducted, and the results showed large impurities in the alloy, so the results were inconclusive. In addition, in 1939, Laves and Wallbaum first identified the intermetallic compounds Ti₂Cu and TiCu₃ in the binary phase diagram of Ti-Cu, and over the years of research, four new intermetallic compounds Ti₂Cu, TiCu, Ti₂Cu₃ and TiCu were identified through various heat treatments [40].

Canale et al [40] proved the existence of the Ti₃Cu phase as a stable phase together with six previously identified stable intermetallic compounds when investigating Ti-Cu alloys with a Ti content of more than 75 at [40].

Qin et al [41] demonstrated in their studies that the Ti₂Cu phase forms in Ti-5Cu, which contains a small amount of the α' phase at the melting boundaries, resulting in lower corrosion resistance. The heat treatment process used in this study eliminates the detrimental effect of the α' phase, and the Ti₂Cu phase is refined at different cooling rates, which improves the corrosion resistance. Heat-treated Ti-5Cu samples were found to have similar corrosion properties to

pure CP-Ti. The slow cooling rate resulted in a larger gap between the Ti₂Cu phases in the microstructure. This ultimately leads to a higher corrosion resistance [41].

Zhu et al [42] proved that α -Cu₄Ti, Cu₃Ti, Cu₃Ti₂ and Cu₄Ti₃ are metastable phases, while β -Cu₄Ti, CuTi and CuTi₂ are stable phases at 0 K by calculating the enthalpy of formation of Ti-Cu alloys. They also found that the CuTi phase has the highest hardness, stability and brittleness among all Cu-Ti intermetallics [42].

In their investigations, Zhang et al [43] demonstrated that the Ti₂Cu phase forms in the Ti-Cu alloys investigated, which plays a key role in the antibacterial mechanism. The antibacterial effect of the Ti-Cu alloy was strongly proportional to the Cu content and Ti₂Cu phase on the surface, and high Cu and Ti₂Cu content contributed to higher strength and strong antibacterial effect [43].

Kikuchi et al [44] demonstrated that three structures are present in Ti-Cu alloys with up to 10 % Cu: α -Ti+Ti₂Cu, hypoeutectoid and hypereutectoid structure. An Ti₂Cu interface was found to form a eutectoid structure with α -Ti. No β -Ti primary phase was found. The eutectoid reaction of this alloy is relatively fast. The microstructures of Ti-Cu alloys with 1, 2 and 5 % Cu were similar to those of α -Ti. The solubility of Cu in the α -Ti phase decreases with decreasing eutectoid temperature, while the tensile strength increases with increasing copper concentration in the alloy [44].

Liu et al [45] proved in their investigations that the binary Ti-Cu alloy has a eutectoid structure at 7.0 %. A Ti₂Cu interface was found in the titanium-rich region. Alloys with a near-eutectoid structure should have lower ductility and higher strength than cp-Ti. The same study also found that Ti-Cu alloys with up to 10.0 % Cu have better tensile strength but lower ductility, while Ti-5Cu alloys have up to 2 times higher strength than cp-Ti. Studies have shown that Ti alloys with 5 and 10 % Cu have significantly higher corrosion resistance than cp-Ti [45,46].

Hayama et al [47] in their study of the Ti-Cu alloy, concluded that the β -phase cannot be stabilised after rapid cooling at low temperatures and that higher cooling rates are required for its stabilisation. The investigated Ti-5Cu alloy exhibited a martensitic microstructure. They also found that a higher copper content in the alloy increased the proportion of the intermetallic Ti₂Cu phase [47].

The results of previous studies suggest that the Ti₃Cu phase does not form during the equilibrium process and therefore does not exist as an equilibrium compound in the equilibrium phase diagram of Ti-Cu alloys.

5. Conclusion

Driven by developments and major discoveries in biomedicine in recent years, the demand for titanium and its alloys is increasing rapidly. Thanks to favourable properties such as biocompatibility, Ti-Cu alloys have become increasingly popular as biomedical materials. This article provides an overview of phase transformations in biomedical titanium materials, with a focus on Ti-Cu alloys.

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Study on the Failure of Heat Exchanger Tubes in the Chemical Industry

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Abstract: In the chemical industry, heat exchangers play a critical role in maintaining process efficiency. However, these systems are prone to frequent failures, often necessitating extensive maintenance or repairs, which in turn adversely affect the overall economic performance of industrial operations. The chemical industry commonly operates in highly corrosive environments, making corrosion the predominant failure mechanism for heat exchangers. Key factors contributing to corrosion-related failures include defects in fabrication and welding, suboptimal material selection, improper water flow or chemistry management, and the presence of corrosive species in water. This study investigates the corrosion behaviour of heat exchangers under various corrosive conditions, utilizing COMSOL Multiphysics for simulation and analysis.

Keywords: HEAT EXCHANGER, STEEL CORROSION, STRESS CRACKING CORROSION, COMSOL MULTIPHYSICS, FAILURE ANALYSIS

1. Introduction

Heat transfer devices or heat exchangers, which provide heat transfer between 2 or more fluids, are standard equipment in the chemical industry. Thus, there is no technological installation of at least medium complexity in which this type of device does not exist [2]. The most common type of heat exchanger in chemical industry applications is the shell-and-tube heat exchanger (Fig.1).

Heat exchanger tubes in the chemical industry are prone to failure due to operating conditions. The primary causes of failures in heat exchangers include:

- **Corrosion** is a major problem, accelerated by the presence of corrosive substances (H_2O , CO_2 , chloride-rich environments). Hydrogen sulfide (H_2S) and low-velocity water can lead to electrochemical corrosion and perforation of the tubes [1]. Localized corrosion is caused by the synergistic effect of H_2S and CO_2 , forming corrosion cells [5]. The most common are pitting corrosion [6,8], crevice corrosion, and stress corrosion cracking (SCC). SCC in heat exchangers is a complex phenomenon influenced by material properties, environmental conditions, and stress factors [7].
- **Erosion-corrosion** is induced by disturbed flow and chemical composition of water, particularly in environments rich in chlorides [9]
- **Thermal stress:** Temperature gradients between the shell and tube fluids can cause thermal stress, especially at the tube-to-tube junction or near baffles [9], leading to failure. For example, copper and steel behave differently under stress, exhibiting the lowest strain rates but offering better safety factors [6]. Figure 2a,b shows the variation of temperature along the length of the exchanger (COMSOL Multiphysics simulation) [4].

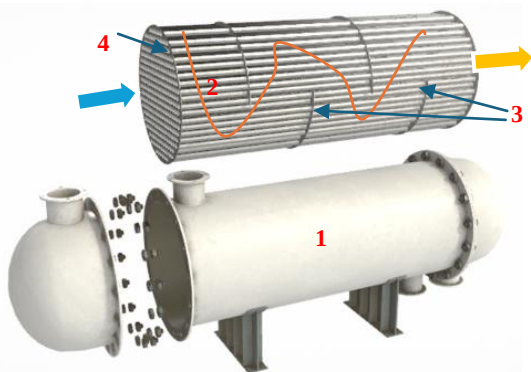


Fig. 1 Basic structure of a heat exchanger: 1-shell, 2-tubes, 3-baffles, 4-plate [3]

- **Mechanical failure:** Fatigue, creep, and vibration are mechanical problems that can lead to the failure of heat exchanger tubes. High-cycle fatigue and vibration (induced by fluid flow) can cause cracking and eventual failure if not properly managed [5]. In Figure

2c (COMSOL Multiphysics simulation), it can be observed that areas of baffles are mechanically stressed during operation [4]

- **Fouling and Clogging:** The deposition of salts and other materials on tube surfaces can lead to blockages and reduced heat transfer efficiency, causing tube failure [2,5].

- **Design and manufacturing defects:** Poor design and manufacturing can lead to premature failure. Weld defects or cracks in tube-to-tube sheet metal weld joints can lead to failures [10].

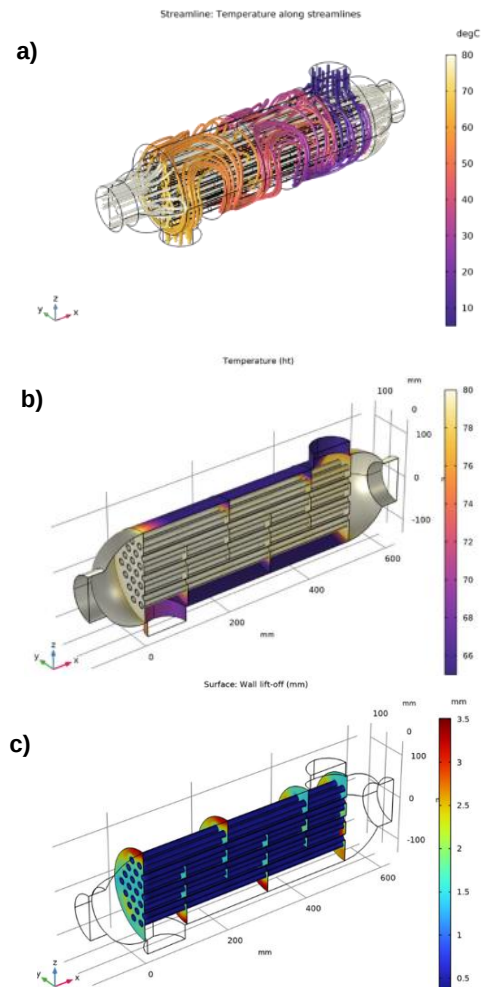


Fig. 2 Variation of temperature: (a) in the intertubular space; (b) along the shell; (c) lift-off wall [4]

The choice of materials for heat exchanger tubes is crucial. For instance, brass heat transfer equipment can be applied to specific occasions requiring high heat transfer capacity, corrosion resistance, and efficiency [2]. The materials commonly used for heat exchanger tubes in the chemical industry include carbon steel, copper, brass, stainless steel, and copper-nickel alloys [2,9].

In this context, this paper aims to investigate the corrosion behaviour of heat exchangers under various corrosive conditions, utilizing COMSOL Multiphysics for simulation and analysis.

2. COMSOL Multiphysics corrosion simulation

For the simulations, stainless steel (SS) was chosen, and as an electrolyte, a solution containing 0.5M HCl. The corrosion behavior of the SS was analyzed at 30°C and 90°C. Using COMSOL Multiphysics, SS crevice corrosion and pitting corrosion were studied (the geometries are presented in Fig. 3) [11,12]. The results are presented in Fig. 4 and Fig. 5.

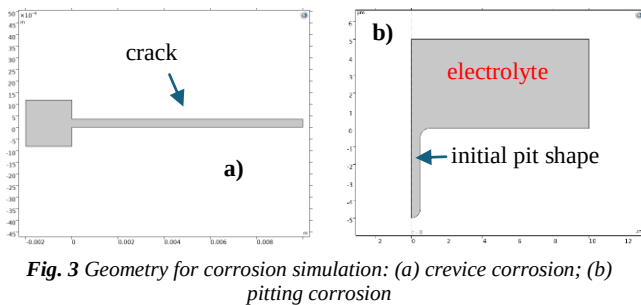


Fig. 3 Geometry for corrosion simulation: (a) crevice corrosion; (b) pitting corrosion

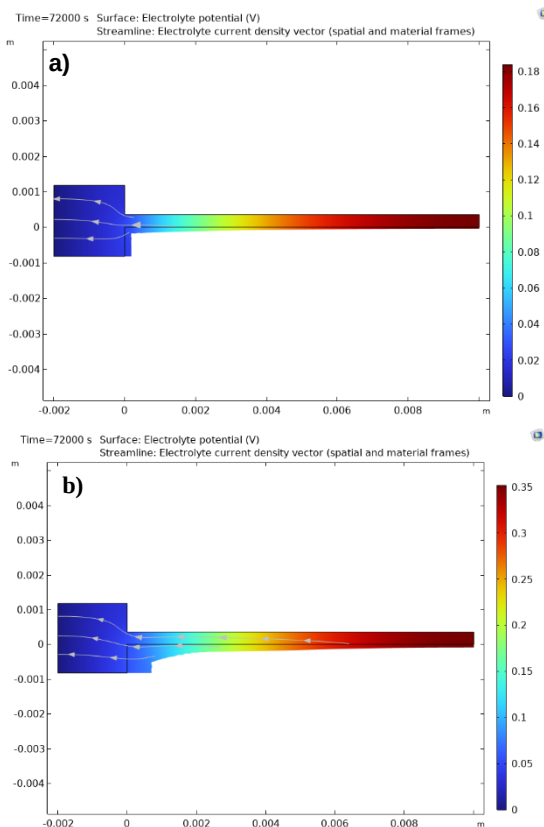


Fig. 4 Crevice corrosion for stainless steel: (a) 0.5M HCl at 30°C; (b) 0.5M HCl at 90°C; surface: electrolyte potential [V]; streamline: electrolyte current density vector

In Fig. 4 it can be seen that after 50 days, in both cases, the part of the surface outside the crack is severely deformed, although SS is resistant to corrosion. Also, as expected, the increase in temperature accelerated corrosion.

In Fig. 5a it is observed that after 10 days, a growth of the pit and Fig. 5 shows that in time (30 days), a pronounced convex pit formed. This is probably because the pH gradually decreases as the pit grows, accelerating its growth.

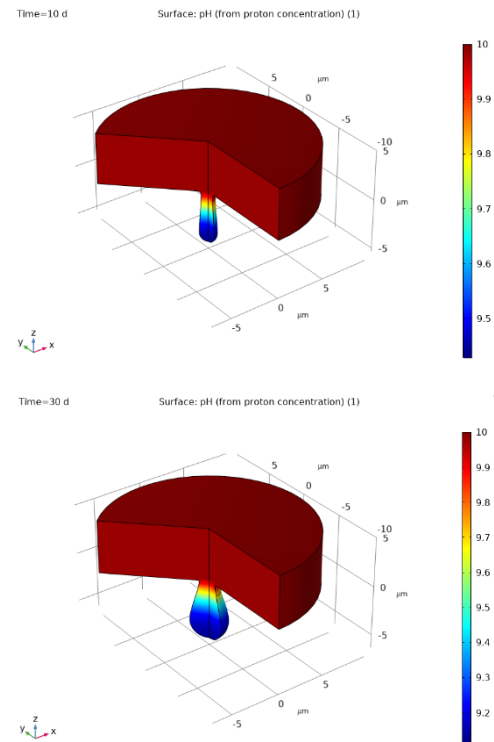


Fig. 5 Pitting corrosion for stainless steel, 0.5M HCl at 90°C: pH and pit after (a) 10 days (b) 30 days

3. Conclusions

Failure of heat exchanger tubes within the chemical industry is attributable to several mechanisms, including material fatigue, corrosive interactions, and incompatibility between materials and operating environments. Such failures can critically undermine the operational efficiency of chemical processes, posing risks of significant economic repercussions. Mitigation strategies, such as comprehensive failure analysis methodologies and meticulous material selection practices, are essential to minimize these risks and enhance the reliability and longevity of heat exchanger systems. A thorough understanding of failure mechanisms, coupled with the implementation of effective preventive measures, can increase both the reliability and the operational lifespan of heat exchanger tubes within the chemical industry.

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Investigation of hardness and tensile strength ratio of a polyethylene blend

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Abstract: In this paper, the results of Shore D hardness and tensile strength according to ISO 10319 were investigated. The results were processed statistically by creating a trend line and regression analysis in order to study in details the existing relationship

Keywords: HARDNESS,TENSILE STRENGHT,TRENDLINE,MECHANICAL PROPERTIES

1. Introduction

Mechanical properties are characteristics that determine how the material reacts to external forces. The main mechanical properties are: hardness, strength, stiffness, creep resistance, fatigue resistance, etc. The possibility of determining mechanical properties such as tensile strength, yield strength and fatigue by hardness is well known and often used in engineering practice. Hardness is the property of a material to resist plastic deformation, a property that only applies to the surface layer[1].Tensile strength is the maximum stress that a specimen can withstand. The hardness-tensile strength relationship is used because hardness testing is non-destructive, experimental data are obtained quickly,there is no need to manufacture standardized specimens, and the test is simple[2]. The equation for the relationship between hardness and tensile strength is:

$$UTS=H.k \quad (1)$$

The range of the coefficient k are well known for a number of materials such as brass, steel, aluminum alloys, etc.Polymer blends are like alloys mixing of two polymers aims to obtain a new material with different properties. Thermoplastic blends such as PE are of wide interest due to the large variety of PE-HDPE, LDPE, etc. PE blending is a common industrial practice[3].

2. Materials and methods

The material under study is a PE blend, with a concentration of HDPE and LDPE 75 to 25 percent. Polyethylene is a thermoplastic plastic with an amorphous-crystalline structure. It is obtained by polymerization with high (50 MPa), medium (3-7 MPa) and low pressure[4]. At room temperature, PE is a hard and elastic polymer with high chemical resistance.

The Shore durometer is used to measure the hardness of polymers (fig.1)[5]. Higher values indicate higher resistance to penetration, and lower values indicate softer material. There are different Shore measurement scales[6].Shore measurement is performed in dimensionless unit [7].For the purpose of the article, the D scale was used, which is intended for hard plastics. The durometer was calibrated, reference blocks were used before the tests.



Fig. 1 Shore D(durometer) tester

The maximum tensile strength measurements were performed on a calibrated universal mechanical testing machine (fig.2). The scope and jaws correspond to the test conditions of the test. The strain gauge is capable of applying a maximum force of 10 kN.



Fig. 2 Universal tensile machine

16 samples were made with dimensions of thickness 1.4 ± 1 mm, width 200 ± 1 mm. 4 hardness tests were made from each sample, after which the values were averaged.

Based on the experimental results, a trendline and a linear regression equation were created. The trendline shows the direction of the relationship, and the regression equation shows the stochastic values of the process. The regression equation includes points interpolated by the graph of the linear equation

3. Results and discussion

The results of the hardness measurement are shown in a table (tab. 1), (fig 3).The values are averaged to 3 decimal places. The requirements of the Shore hardness testing standard ISO 7619 are met.

Table 1: Hardness Shore D results

Sample	Hardness Shore D				Average
1	49.4	52.5	49.6	49.9	50.35
2	52.2	49.7	55.1	53.7	52.675
3	50.9	50.4	48.9	51.2	50.35
4	54.8	52	53.5	49.5	52.45
5	49.8	48.6	48.2	49.3	48.975
6	55.2	55.4	51	52.6	53.55
7	49.7	50.9	51.7	51.9	51.05
8	50.2	49.3	47.3	49.1	48.975
9	48.8	46.8	50.8	48.7	48.775
10	58.1	52.8	55.3	52.7	54.725
11	47.8	49.3	47.8	48.7	48.4
12	58.1	46.8	50.8	48.7	51.1
13	47	48.2	50.2	53.6	49.75
14	51	52.9	50.4	50.2	51.125
15	47.8	48.9	51.3	49.9	49.475
16	49.8	51.6	51.9	52.2	51.375



Fig.3 Test result sample 10, testing No 3.

The results of the average values of the measured hardness and maximum tensile strength are presented in a table, tab. 2.

Table 2: Hardness and ultimate tensile force results

Sample No	Hardness	Ultimate tensile force (kN)
1	50.35	4.91
2	52.675	5.05
3	50.35	4.82
4	52.45	4.9
5	48.975	4.81
6	53.55	5.01
7	51.05	4.85
8	48.975	4.93
9	48.775	4.87
10	54.725	4.99
11	48.4	4.79
12	51.1	4.81
13	49.75	4.93
14	51.125	4.97
15	49.475	4.89
16	51.375	4.92

Based on the results, a scatter plot is presented and a trendline is constructed (fig.4)[8]. The hardness is chosen as the independent variable, and the maximum tensile force as the dependent variable.

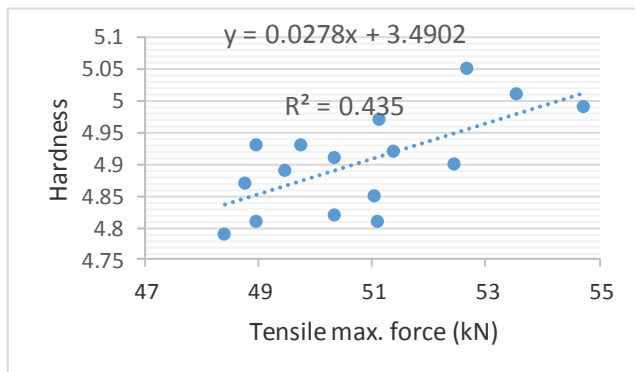


Fig.4 Scatter plot and trendline

The coefficient of determination (R^2) shows how well an independent variable in a statistical model predicts the dependent variable[9]. R^2 results between 0.25 and 0.5 indicate a moderate relationship between the variables. R^2 is in the range between 0 and

1. Based on the experimental results, a linear regression model was created, shown in tab. 3 and tab. 4

Table 3 : Characteristics of the regression model

	Df	SS	MS	F	Significance F
Regression	1	21.60182	21.60182	10.81151	0.005389
Residual	14	27.97255	1.998039		
Total	15	49.57438			

Significance F is less than 0.05, which means that the null hypothesis is rejected and that the equation is statistically significant.

Table 4 Characteristics of the regression model

	Coefficients	Standard Error	t Stat	P-value
Intercept	-26.0263	23.37341	-1.1135	0.284247
Tensile max. F	15.67266	4.766499	3.288086	0.005389

The regression equation has the form:

$$y = 15.67x - 26.03 \quad (2)$$

The analysis of the coefficients, P-value shows that the coefficient a is significant, and the coefficient b is insignificant. The residuals and standardized residuals are shown in table 5

Table 5: Residuals and standard residuals

Observation	Predicted Hardness	Residuals	Standard Residuals
1	50.9265	-0.5765	-0.42216
2	53.12067	-0.44567	-0.32636
3	49.51596	0.83404	0.610754
4	50.76977	1.680227	1.230404
5	49.35923	-0.38423	-0.28137
6	52.49377	1.056234	0.773464
7	49.98614	1.06386	0.779048
8	51.23995	-2.26495	-1.65859
9	50.29959	-1.52459	-1.11644
10	52.18031	2.544688	1.863434
11	49.04578	-0.64578	-0.47289
12	49.35923	1.740766	1.274736
13	51.23995	-1.48995	-1.09107
14	51.86686	-0.74186	-0.54325
15	50.61305	-1.13805	-0.83337
16	51.08323	0.291774	0.213661

Residuals are a term in statistics that indicates the difference between an observed value and a predicted value[10]. They are also known as model error. It is noted that 14 out of 16 residual values are within ± 2 and only 2 are out of the range ± 2 but in the range of ± 3 . The results are within the accuracy of the Shore D hardness measurement. Standard residuals deviation is the standard deviation of residual values, or the difference between a set of observed and predicted values. The standard residuals indicate how useful the model is. All standard residuals are within ± 2 [11]. These values indicate the adequacy of the model.

4. Conclusions

Based on the experimental results and the created mathematical models, the following conclusions can be drawn:

- The range and distribution of the results for hardness and tensile strength do not show significant deviations.
- No discrepancies were observed during the tests
- The created trendline shows the trend of the results
- The Significance F value rejects the null hypothesis and proves that the model is statistically significant.
- The R2 value showed a moderate strong relationship between the results
- The analysis of the residuals and standardized residuals proves that there are no critical deviations from the created model.

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Structure and properties of magnetostrict materials of the Fe-Ga AND Fe-Ga-Al systems

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Abstract. The structure and some properties of magnetostrictive powder materials of the Fe-Ga and Fe-(Ga+Al) systems, obtained by sintering at a temperature of 1150 °C in a neutral Ar environment for 1 h, were studied. The structure and phase composition of the obtained materials were studied. Mechanical tests of the samples were carried out, corrosion resistance was determined and magnetostrictive properties of samples of the composition Fe-Ga and Fe-1.7Al-19.3Ga were evaluated as the most promising for use as magnetostrictive materials. An increase in hardness and level of compressive strength of the material of the composition Fe-1.7Al-19.3Ga compared to materials of the composition Fe-21.4Ga was revealed. In terms of corrosion resistance, these materials are corrosion-resistant materials (3-4 points on a 10-point scale of corrosion resistance). The highest level of magnetostriction is possessed by materials of the dual composition Fe 21.5 wt.%Ga (>210 ppm). The material with the composition Fe 19.3 wt. % Ga-1.75 wt. % Al, which had a lower level of compressive elasticity, also differed in a lower value of magnetostriction (<130 ppm). The level of magnetostriction of the studied materials is within the requirements for commercial deformed Fe-Ga alloys (galfenols).

KEYWORDS: FE, AL, GA, MICROSTRUCTURE, PHASE FORMATION, MICROHARDNESS, MECHANICAL CHARACTERISTICS IN COMPRESSION

1. Introduction

The ternary system Al-Fe-Ga is currently little studied, although alloys of binary composition based on components of this system are widely used in technology and still have significant potential for creating new functional materials. Active development of developments in this direction began due to the high values of magnetic characteristics of these materials in the area of existence of solid solutions based on iron (20...30 wt. % Ga) [1]. At the same time, binary alloys of the Fe-Al system also turned out to be possible candidates for devices that use magnetomechanical properties of materials [2] and this has formed a wide demand for such alloys in various industries [3,4]. Since each of these metals has the ability to increase the functional properties of Fe, further study of the ternary system Fe-Ga-Al has become relevant.

In [5,6], the absence of an additive effect is indicated when iron is doped simultaneously with aluminum and gallium and the significant influence of the phase composition and features of structure formation on the properties of ternary alloys is noted.

In this regard, the aim of the work is to study the features of structure formation and phase formation in the Fe-Al-Ga system in the temperature range above 650-800 °C, provided that Fe and Al powder components and a Fe-Ga alloy are used.

2. Materials and research methods

Iron and aluminum powders) and gallium in ingots were used as starting components. Gallium was introduced into a mixture of iron and aluminum powders as part of a ligature with an equiatomic composition of Fe – 44.9 wt.% Ga (50 at.% Ga), which was previously obtained according to the technological scheme presented in [7]. The composition of the mixtures was selected in order to investigate the competing influence of Al and Ga on the structure and phase composition of ternary alloys of the Fe-Al-Ga system in the temperature range of the existence of solid solutions based on α -Fe. 7 batches of mixtures of different compositions were produced, which had either only two components, namely Fe-17.5 wt.% Al (composition No. 1) and Fe-21.4 wt.% Ga, (composition No. 7), or all three components with a gradual replacement of Al by Ga with a “step” of 1.75 wt.% (compositions No. 2 to 6) (Table 1).

The main sintering temperature was 1150±5 °C, at which the samples were kept for 1 h. Samples of powdered Fe, without alloying impurities, were subjected to heat treatment together with the test samples in the same chamber.

The phase composition of the obtained alloys was determined by X-ray phase (XRF) and local X-ray microspectral (XMS) analyses. Diffractograms were taken on a DRON-3M spectrometer in the spot mode in FeK α -1 radiation. XMS was performed using a JEOL microscope from CAMECA (France).

For studies of magnetic properties, the method of indirect magnetostriction estimation was used using specially designed equipment for monitoring the dynamics of material remagnetization processes in an alternating magnetic field.

Table 1. Composition of mixtures for sample preparation

Mixture No.	Concentration of elements in the mixture					
	Fe		Al		Ga	
	% wt.	% at.	% wt.	% at.	% wt.	% at.
1	82,5	70	17,5	30	0	0
2	82,1	70,5	15,8	28	2,1	1,5
3	81,3	72,7	12,3	22,7	6,4	4,6
4	80,5	75,1	8,75	16,9	10,7	8,0
5	79,7	77,7	5,25	10,6	15	11,7
6	79,0	80,5	1,75	3,7	19,3	15,8
7	78,5	82,1	0	0	21,5	17,9

3. Research results

Sintered samples had visual signs of volume increase to varying degrees with a tendency to decrease the effect of “swelling” of ternary composition samples with increasing Ga content in the material. This is possibly due to the high diffusion mobility of Ga and a change in the speed and direction of diffusion flows in the system. Fig. 2 presents the most typical sections of the microstructure of some samples with the designation of individual places of PCMA, and Table 2 shows the results of quantitative analysis on these sections.

In the structure of the Fe-17.5Al binary composition sample, the main component, which occupies up to 90% vol. of the surface of the section, is a Fe-based phase. This sample is characterized by significant porosity, therefore it includes C and O in significant quantities (Table 2). According to X-ray diffraction, the main phase in the structure of these materials is a solid solution based on Fe, in which Al and Ga are dissolved in appropriate proportions, in addition, locally separate phase separations are recorded, containing, in addition to the main metals, carbon and oxygen in significant quantities (up to 40...50% at.). However, it should be noted that in materials of ternary composition with the maximum amount of Ga (Fe-1.7Al-19.3Ga) and double composition Fe-21.5Ga, carbon is not detected. The material of double composition Fe-21.5Ga (No. 7) contains the main phase based on Fe of a fairly homogeneous composition, the remaining area of the section is occupied by pores (4-6% vol.), oxygen is present in a small amount.

The microhardness of the α -Fe-based solid solution decreases with the gradual substitution of Al atoms by Ga atoms in its composition (Fig. 3). The microhardness of the pure Fe sample is significantly lower than the range of changes of this parameter in the samples of dual and ternary composition of comparable porosity

This effect is expected, since the dissolution of many metals in α -Fe is accompanied by its strengthening [8].

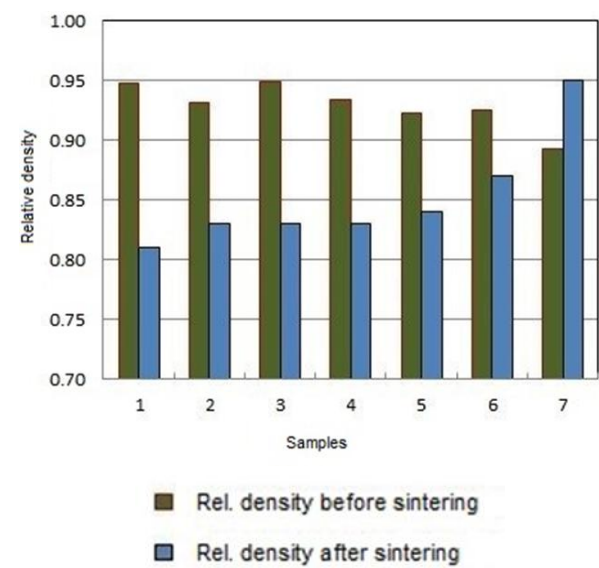


Fig. 1. Relative density of sintered samples depending on their composition (% wt.): 1- Fe-17.5Al; 2-Fe-15.7Al-2.1Ga; 3-Fe-12.2Al-6.4Ga; 4-Fe-8.7Al-10.7Ga; 5-Fe-5.3Al-15Ga; 6-Fe-1.7Al-19.3Ga; 7-Fe-21.5G.

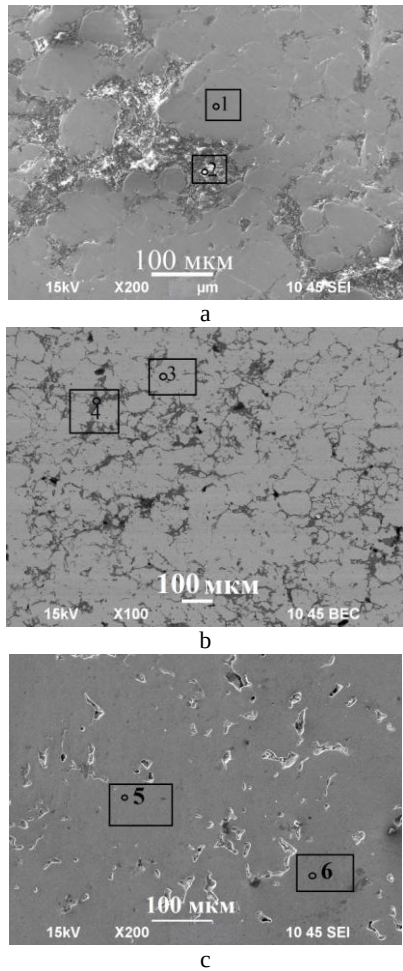


Fig. 2 Electron microscopic image of the microstructure of sintered samples of composition Fe-17.5 Al (a), Fe-1.7Al-19.3Ga (b) and Fe-21.5Ga (c) (photo in secondary electron mode).

Determination of the hardness of the samples by Brinell due to the high sensitivity of this characteristic to the porosity of the samples turned out to be somewhat lower than the results obtained when measuring the microhardness by Vickers. Thus, the Brinell

hardness of samples of double and triple compositions No. 1 to 5, which had a porosity of 16-19%, could not be determined correctly due to significant deformation of the indenter imprint. For samples from the material Fe-1.7Al-19.3Ga (No. 6) and for samples from pure Fe, which have comparable porosity values (10-13%), this indicator varies within 83...93NV, which is somewhat lower than the values obtained when measuring the microhardness (see Fig. 4). At the same time, the material of the Fe-21.5Ga composition, which has minimal porosity after sintering (4-6%), is characterized by a hardness of 119...123 HB, and these values are in good agreement with the data obtained as a result of measuring the microhardness according to Vickers.

Table 2. Elemental composition of phases in the structure of samples determined by the RSMA method

Com-position (% wt.), (Point No)*	C	O	Fe	Al	Ga
	%, at.	%, at.	%, at.	%, at.	%, at.
Fe-17,5Al (1)	-	4,40	69,79	25,81	-
(2)	57,29	21,95	16,92	3,84	-
Fe-1,7Al-19,3Ga (3)	-	3,32	76,48	3,23	16,96
(4)	-	3,20	76,02	3,52	17,26
Fe-21,5Ga (5)	-	2,90	78,36	-	18,74
(6)	-	3,00	78,03	-	18,97

See Fig. 2 for the location of the "points"

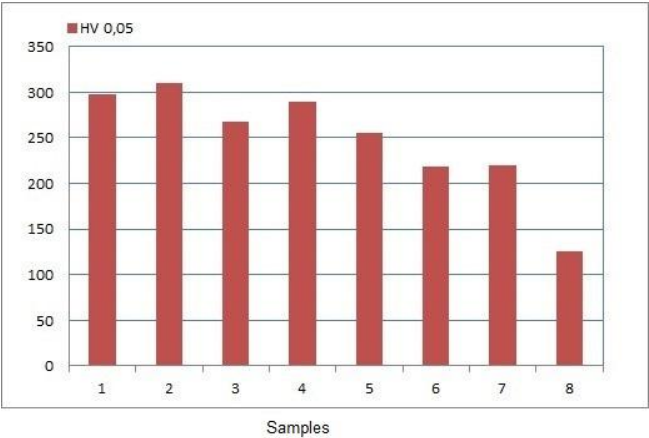


Fig. 3 - Microhardness of solid solution based on α -Fe in the structure of samples depending on their composition (% wt.): 1- Fe-17.5Al; 2 - Fe-15.7Al-2.1Ga; 3 - Fe-12.2Al-6.4Ga; 4 - Fe-8.7Al-10.7Ga; 5 - Fe-5.3Al-15Ga; 6 - Fe-1.7Al-19.3Ga; 7 - Fe-21.5Ga; 8 - Fe.

Samples of compositions No. 6 and 7, ha the greatest densification after sintering, were subjected to mechanical and magnetostrictive tests together with samples of pure Fe for comparison.

Mechanical compression tests of the samples showed that for materials of the ternary composition Fe-1.7Al-19.3Ga the ultimate strength reaches values of 680 MPa, while for the binary alloy Fe-21.4Ga and pure iron, fracture does not occur under test conditions, since both materials are characterized by high plasticity and do not fracture, reaching significant deformation under load. (Table 3). The increase in the strength of the material of the ternary composition Fe-1.7Al-19.3Ga, as well as the increase in its hardness compared to pure iron and a binary alloy with aluminum, indicates the additive effect of strengthening the solid solution based on Fe when simultaneously dissolving the elements Al and Ga in it. Sufficiently high mechanical characteristics obtained during testing

indicate the potential for further significant strengthening of these materials in the event of their additional densification. The obtained strength level of the ternary alloy Fe-1.7Al-19.3Ga is comparable to the strength of single crystals of Fe (20 25 %)Ga, which are positioned in the class of magnetostrictive materials as materials of increased strength [1].

Table 3. Porosity and mechanical characteristics of sintered samples depending on their composition

Samples material compo-sition (% wt.)	Density, % of theor.	Mechanical characteristics in compression tests		
		σ_{02} , MPa	σ_B , MPa	ε , %
Fe-19,3Ga-1,7Al (№6)	0,87	294±10	680±50	27
Fe-21,4Ga (№7)	0,95	379±20	>2083	82
Fe	0,90	336±10	>2760	75

The corrosion resistance of these samples was tested in a neutral aqueous solution of 3% NaCl (PH≈7) for 141 hours. The samples were periodically immersed in the solutions for 30 minutes, followed by removal and exposure to air for 15 minutes. It is believed that this mode prevents the development of stagnant phenomena at the interface of the sample surface with the solution, which inhibit oxidation processes. The temperature of the solution was 40±5 °C, air - 38±5 °C. After testing, the samples underwent visual inspection to determine the nature of corrosion, and then they were dried and weighed. After weighing, the samples were subjected to mechanical cleaning to remove the remains of corrosion products on their surface, followed by ultrasonic treatment in an ethyl alcohol solution for 3-5 minutes, and drying in a vacuum drying oven for 30 minutes.

The quantitative assessment of the mass loss of the material during corrosion was calculated using the negative corrosion index K and the parameter K- is a basic value that is directly used in the calculations of the depth P, which characterizes the reduction in the thickness of the material due to corrosion and is expressed in linear units per unit of time.

The test results showed that the samples of both compositions in terms of corrosion resistance belong to corrosion-resistant materials (3-4 points on a 10-point scale of corrosion resistance according to DSTU 13819-68). It was established that the corrosion products mainly contain Fe(OH)₃.

To study the magnetostrictive properties, the increase in the relative elongation of the samples from the intensity of the alternating magnetic field was evaluated (Fig. 4). These quantitative estimates agree quite well with the results of magnetostriction studies using standard methods, and the graphs of the dependence of magnetostriction on the magnetic field strength are similar to such dependences obtained as a result of measurements using the strain gauge method in a constant magnetic field.

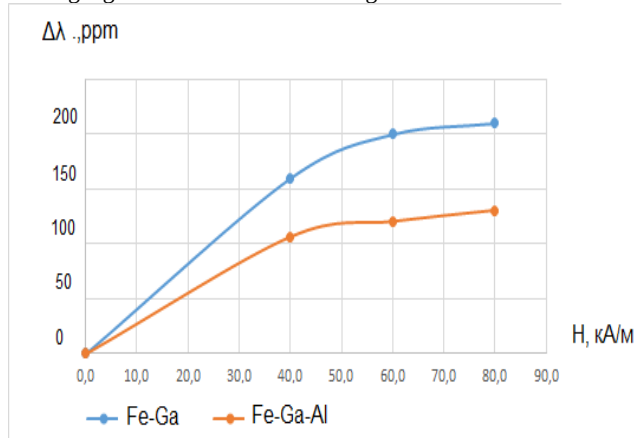


Fig. 4 Dependences of the increase in the relative elongation of samples on the intensity of the alternating magnetic field

It was established that the highest level of magnetostriction is possessed by materials of the dual composition (wt.%): Fe-21,5Ga (>210 ppm). The material of the composition Fe-19,3Ga-1,75Al, which had a lower level of compressive elasticity, was also distinguished by a lower value of magnetostriction (>130 ppm). The level of magnetostriction of the studied materials is within the requirements for commercial deformed Fe-Ga alloys (galenols).

Conclusions

The structure and some properties of magnetostrictive materials of the systems Fe-21,4Ga and (78...82)Fe-(Ga+Al), obtained by sintering at a temperature of 1150 oC in a neutral Ar environment for 1 h, were studied. Mechanical tests of the samples showed an increase in hardness and level of compressive strength of the material of the ternary composition Fe-1,7Al-19,3Ga compared to the materials of the binary composition Fe-21,4Ga and pure Fe. In terms of corrosion resistance, these materials belong to corrosion-resistant materials (3-4 points on a 10-point scale of corrosion resistance).

It was established that the highest level of magnetostriction is possessed by materials of the binary composition Fe-21,5Ga (>210 ppm). The material of the composition Fe-19,3Ga-1,75Al, which had a lower level of compressive elasticity, also had a lower magnetostriction value (>130 ppm). The magnetostriction level of the studied materials is within the requirements for commercial deformed Fe-Ga alloys (galfenols).

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Effect of technological parameters on the structure and physico-mechanical properties of Fe-FeCr800 system composite

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Abstract: The article investigates the influence of technology and technological parameters of manufacturing on the structure, phase composition and physical and mechanical properties of the Fe-FeCr800 composite system. It was established that the determining factor in phase and structure formation is the manufacturing technology, while vacuum sintering and hot forging have their own optimal technological parameters. At the same time, hot forging makes it possible to obtain a composite with a higher microhardness of structural components due to a change in the content of component components. The results of the research also showed that the preheating time (for 20 min.), as well as thermomechanical treatment, is sufficient for the phase formation process, in particular, with the release of carboboride phases of the type Me_3CB and $Me_3(CB)_2$, while the densification processes are intensified, which makes it possible to obtain a material with lower residual porosity. Analysis of mechanical tests showed that vacuum sintering makes it possible to obtain composites with higher mechanical properties due to the active interaction between the components of the composite. However, high-temperature annealing after hot deformation will allow for a composite with high mechanical properties.

KEYWORDS: COMPOSITE MATERIALS, CARBIDE STEEL, HOT FORGING, MECHANICAL PROPERTIES, HARDNESS.

Introduction

Ensuring an increase in the service life of components and mechanisms requires the development of materials with the highest possible physical, mechanical and functional properties. At the moment, carbide steels have shown their effectiveness and are used in components and mechanisms to increase the service life of the unit as a whole. Every year, the search for new component compositions of the material becomes more and more difficult, since a lot of research has already been conducted on the development of carbide steels with different component compositions. However, all attention was paid to classical methods of powder metallurgy. Therefore, changing the technology for obtaining a composite will make it possible to obtain a material with other functional properties. Over the years of research, powder metallurgy methods have already proven their superiority over foundry production, since sintered materials are distinguished by a homogeneous structure and low anisotropy of properties. However, classical technological schemes of powder metallurgy, which include the preparation of the charge, pressing and sintering of the material, do not always ensure the production of an absolutely compact material (especially with a high content of carbide components). As a result, there is a need to apply technological schemes based on the use of high-temperature sintering in the presence of a liquid phase, static hot pressing or electric discharge sintering, etc. [1–4].

To obtain and compact the material, hot forging of porous blanks has proven to be one of the most effective processes of powder metallurgy [5–9]. Hot plastic deformation allows obtaining semi-finished products of both simple and complex shapes with low residual porosity, low defectivity and high material uniformity. In addition to high density, hot plastic deformation allows obtaining a fine-grained structure without additional heat treatment [10].

A significant number of publications are devoted to the production of chromium carbide steels by powder metallurgy methods [11–14], but there are almost no publications on their production by methods of metal processing by pressure (including hot forging). Therefore, the purpose of this work is to establish the influence of the production technology and technological parameters of production on the structure, phase composition and physical and mechanical properties of the composite of the Fe-FeCr800 system.

Experimental Procedures

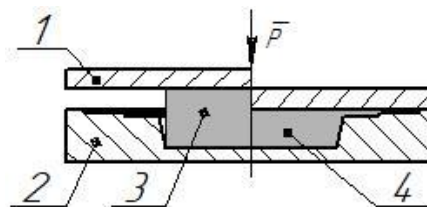
The starting materials were iron powder, high-carbon ferrochrome FCr800 and titanium boride powder. Titanium boride was introduced with the aim of forming, during subsequent thermal synthesis, the corresponding boride phases, which increase the hardness and wear resistance of the resulting materials [15]. High-carbon ferrochrome powder was obtained from lump FKh800 by mechanical grinding in a jaw crusher and subsequent grinding in a

ball mill. Powder mixtures were prepared by wet grinding-mixing in a ball mill in an alcohol environment, the composition of the mixtures is given in Table 1.

Table 1 - Content of components of the initial mixtures of Fe-FeCr800-TiB₂ composites

№	Element content, (% wt.)		
	Fe	FeCr800	TiB ₂
1	65	35	-
2	64,25	35	0,74
3	63,5	35	1,5

The samples were formed in a closed mold at a pressure of 800 MPa. Then the samples were prepared at home by different methods: 1 - sintering in a vacuum electric furnace at 1100–1250 °C with an isothermal holding time of 30 min.; 2 - hot forging in an open die, the scheme of which is presented in Figure 1, for hot deformation the workpieces were preheated in a furnace in an argon environment to a temperature of 1100°C and 1200°C for 20 min..



1- striker; 2 - matrix; 3 - blank; 4 - forging;

Figure 1 - Hot forming deformation

The hardness of AMKM was determined using the Novotest TC-BRV device using the Rockwell method (HRA scale). Bending tests of samples were performed using a three-point scheme on the RM 102M machine using a special device, on the plate of which two prisms were installed, on which a sample measuring 6x6x42 mm was placed. The load on the sample was applied in the middle of its length. The distance between the prisms was 35 mm. The microhardness of the structural components was determined using the PMT-3 device at room temperature and a load of 10 - 25 g, the holding time was 30 s. The measurement area should be twice the diagonal of the impressions. The measurements were repeated 10 - 30 times. The microstructure and phase composition of the sintered samples were studied using optical ("Olympus LX-70") and electron (TESCAN VEGA) microscopes. Local micro-X-ray spectral analysis was performed on an MS-46 microprobe.

Results and discussion

To establish the optimal conditions for obtaining chromium carbide steels, studies were conducted on the influence of sintering temperature and technological parameters of hot forging on the structure and properties of composites. From the given Fig. 2 it is

seen that samples of different compositions obtained by hot forging have significantly higher density values than composites obtained by sintering in vacuum. This dependence can be explained by the fact that in the process of hot forging, densification processes are intensified due to the application of additional mechanical energy.

During sintering in vacuum at temperatures of 1100-1150 °C, shrinkage occurs mainly as a result of solid-phase sintering, but the alloys do not reach high shrinkage and density, which is explained by the low intensity of diffusion processes, as well as the inhibition of the compaction of the samples by the carbide framework. Therefore, to intensify the compaction process of the samples, it is necessary to increase the sintering temperature to 1200-1250 °C, since as a result of reaching the eutectic temperature, a liquid phase appears and the mechanism of liquid-phase sintering is implemented.

Thus, the hot forging method without the use of a vacuum environment allows you to obtain high-density composites compared to sintered samples. Accordingly, the density of the forged materials is slightly higher $6.92 \div 7.32 \text{ g/cm}^3$ compared to the sintered $6.44 \div 7.1 \text{ g/cm}^3$.

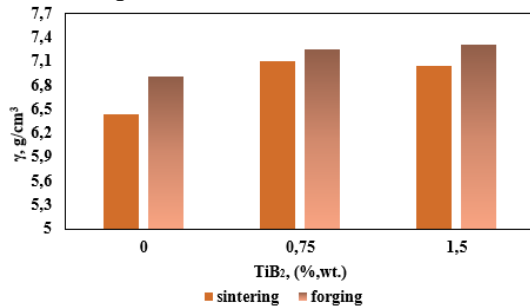
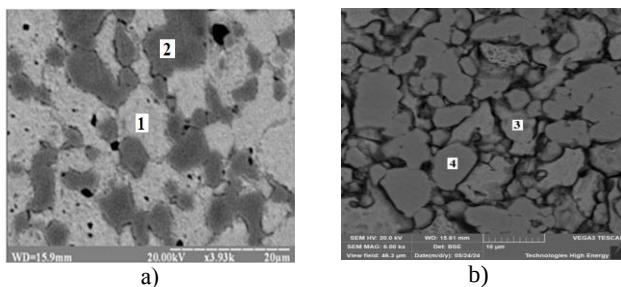


Fig. 2 – Effect of titanium boride content on the density of sintered and hot forged composites at optimal temperatures

The microstructure was investigated and the distribution of elements in the metal and carbide phases of composites obtained by different methods was determined. Fig. 3 shows the microstructures of the sintered (Fig. 3, a) in a vacuum environment at 1250 °C and forged at a preheating temperature of 1200 °C with a holding time of 20 min. (Fig. 3, b) composite of the composition Fe-35FeCr800. Regardless of the method of obtaining the composite, their structure remains heterogeneous, consisting of an elastic-plastic matrix and carbide phases located in it. Namely, the light gray phase is an iron-chromium matrix of the composite, the gray phase is a complex iron-chromium carbide of the M_3C type.



Spec trum No.	Fe	Cr	C	Carbide phase type	Microhardnes s, GPa
1	82,75	17,07	0,17		2,8
2	19,78	73,13	7,08	M_3C	10,5
3	88,84	10,93	0,22		3,69
4	22,26	68,79	8,94	Me_7C_3	11,5

Fig. 3 – SEM and EDS analysis of the Fe-35FeCr800 composite (% wt.) obtained by vacuum sintering (a) and hot forging (b)

According to the analysis conducted using an energy dispersive microanalyzer, the elemental composition of the matrix and carbide phases of the forged composite differs from the sample

obtained by vacuum sintering (Fig. 3). From the table in Fig. 3, after hot forging, the microhardness of the phases in the Fe-35(% wt.) FeCr800 composite slightly increased, the matrix phase - $2.8 \div 3.69 \text{ GPa}$ and the carbide phase - $10.5 \div 11.5 \text{ GPa}$, which is associated with the redistribution of chemical elements in the structural components of the studied material.

Fig. 4 shows the microstructure of hot forging composites of different compositions obtained at different preheating temperatures. The structure, chemical composition and size of the phases are affected by the content of titanium boride in the charge and the temperature of hot deformation. As can be seen from Fig. 4, at a titanium boride content of 0.75 (% wt.) the composite structure consists of a matrix, iron-chromium carbide (light gray phase) and carboboride (dark gray phase). At the specified titanium boride content, the formation of the 3rd phase (dark gray) of the Me_3CB carboboride is clearly recorded already at a heating temperature of 1100°C. Increasing the preheating temperature to 1200°C for this composition leads to grain growth. The composite doped with 0.75 (% wt.) of titanium boride obtained at a preheating temperature of 1100°C differs from other hot-forged composites in its fine-grained structure (Fig. 4, d). The composite with a composition of 1.5 (% wt.) of titanium boride after thermomechanical treatment has a coarser-grained structure, especially at a temperature of 1200°C (Fig. 4, c). The structure of this sample consists of a matrix, iron-chromium carbide and carboboride. However, the amount of carboboride in the structure of this composite varies significantly depending on the heating temperature. At 1100°C, carboboride of the type $Me_3(CB)_2$ is fixed on the structure in a small amount (Fig. 4, b), but with an increase in the heating temperature to 1200°C, its amount increases and is clearly displayed in the form of dark gray phases (Fig. 4, c).

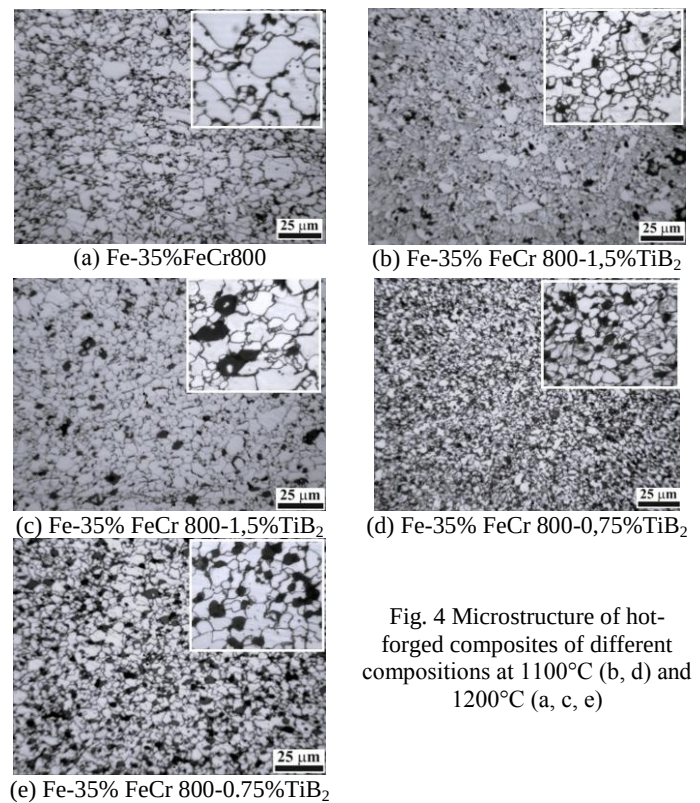


Fig. 4 Microstructure of hot-forged composites of different compositions at 1100°C (b, d) and 1200°C (a, c, e)

One of the advantages of hot forging samples, in contrast to those obtained in the process of vacuum sintering, is that after deformation of samples doped with titanium boride, the grains of the matrix and carbide components of the composites were crushed. Also, hot forging allows you to obtain a composite of higher density even without prior sintering in a vacuum. Also, the preheating time (for 20 min.) before deformation and the applied energy during impact loading are sufficient to undergo the phase formation

process, in particular, with the separation of carboboride phases such as Me_3CB and $\text{Me}_3(\text{CB})_2$.

Metallographic studies and local micro-X-ray spectral analyses of materials based on the Fe-35% (wt.) FeCr800 – TiB_2 system obtained by hot forging showed (Fig. 5) that a microheterogeneous, multiphase microstructure of the composite is formed, containing complex iron-chromium carbides Me_3C_2 and complex carboborides of the $\text{Me}_3(\text{CB})_2$ type.

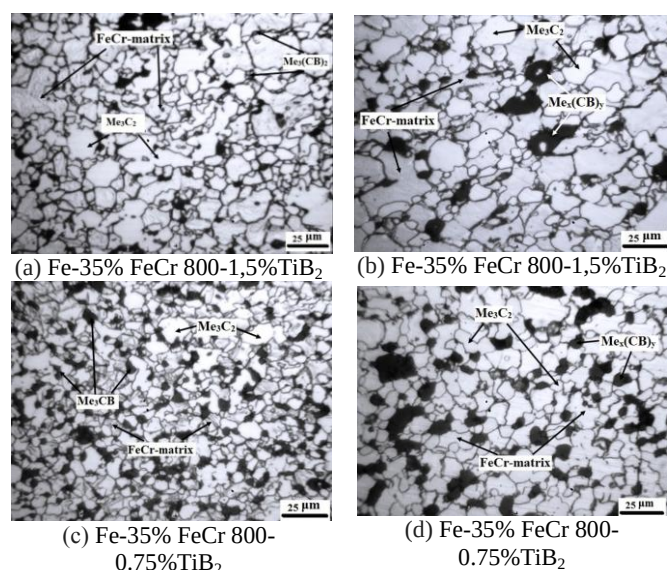
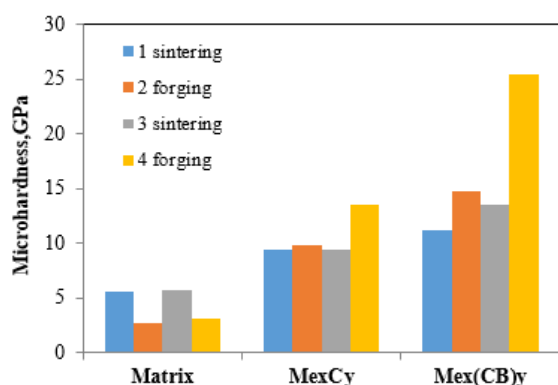


Fig. 5 Micro-X-ray spectral analysis of titanium boride-doped composites at different preheating temperatures of 1100°C (a, b) and 1200°C (c, d)

The microhardness of the structural components of the composites doped with titanium boride was also investigated (Fig. 6). As can be seen from the graph, the microhardness values of the carboboride phases of hot-forged and vacuum-sintered composites are significantly different. For example, in the composite of the composition Fe-35%FeCr800-0.75% TiB_2 obtained at a sintering temperature of 1250 °C, the microhardness value of the carboboride phase is 12.5 GPa, and in the composite Fe-35%FeCr800-1.5 % TiB_2 - 13.7 GPa, which is significantly less than in the hot-forging samples. This is explained by the difference in the elemental composition of the corresponding phase of the hot-forging and sintered composites.



1,2 - Fe-35%FeCr800-0.75% TiB_2 ; 3,4 - Fe-35%FeCr800-1.5% TiB_2
Fig. 6. Microhardness of structural components of the composites

The hardness of sintered and hot forging samples is shown in Fig. 7. A comparison of the obtained values showed that hot forging allows obtaining composites with almost the same hardness as sintered ones. However, the hot-forged composite of the composition Fe-35%FeCr800-0.75% TiB_2 stands out, the hardness of which increased from 75 to 79 HRA compared to the sintered

one, which is associated with the crushing of the structure after hot deformation.

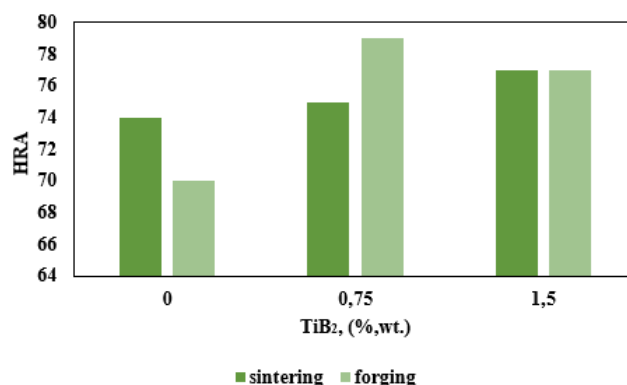


Fig. 7. Hardness of composites obtained with optimal technological parameters

Conclusions

1 The influence of the technology for obtaining carbidosteel of the Fe-FeCr800 system on its structure and properties was studied. It was shown that sintering in vacuum and hot forging allows to obtain composites with a homogeneous structure. At the same time, hot forging provides a finer microstructure compared to sintered composites, as well as a different ratio of components in the structural components, which leads to an increase in their microhardness.

2. The results of microstructural analysis showed that the preheating time (for 20 min.) before deformation and the applied energy during impact loading are sufficient to undergo the phase formation process, in particular, with the separation of carboboride phases of the type Me_3CB and $\text{Me}_3(\text{CB})_2$.

3. The results of the studies showed that the hardness of hot forging composites was relatively unchanged compared to sintered ones.

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Investigation on maximum tensile force and Shore D hardness of 3D printed samples of Polyamide PA6-CF

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Abstract: In this article, the maximum tensile strength and hardness of 3D printed samples of Nylon CF15 Carbon were investigated. 20 test specimens were produced with dimensions according to the ASTM D 638 standard for tensile strength and 3 specimens for Shore D hardness testing. The experimental results were processed statistically.

Keywords: TENSILE STRENGTH, 3D PRINTING, HARDNESS

1. Introduction

3D printing technology is the creation of a three-dimensional object from a 3D model by adding material [1]. The technology has evolved from a limited technology for rapid prototyping to being increasingly used in modern industry [2]. 3D printing finds applications in a number of sectors - space industries, dental and medical industries, mechanical engineering, automotive industry, etc. [3]. A number of materials can be used in 3D printing such as ceramics, metals and composites, but the technology is mostly used for thermoplastic plastics. There are different technologies for 3D printing - Stereolithography (SLA), Fused Deposition Modeling (FDM), PolyJet, etc. The use of 3D printing for responsible applications increases the requirements for parts. The object of study of plastics are the mechanical, physical and chemical properties, with particular interest in mechanical properties, because to a large extent industrial application of 3D printed parts depends on their behavior under force loads.

Mechanical properties characterize the behavior of materials under external load, as well as determine the scope of application and serve to assess the expected service life. Mechanical properties serve to classify all types of materials and the production technology [4]. They are not constant and depend on different conditions such as temperature, type of load, etc. Very often there is a large variation in results tested under the same conditions. This makes the study of mechanical properties very relevant. The main mechanical properties are hardness, plasticity, strength, stiffness, elasticity, fatigue strength, etc..

Ultimate tensile strength (UTS) is the maximum stress that the specimen can withstand before breaking [5]. For brittle materials, the value is close to the yield strength. The test is applicable to alloys, ceramics, plastics, rubbers and other materials. The determination of the UTS is key for materials used for constructions. The essence of the test is to measure the maximum tensile strength and relative elongation. The equipment for testing the maximum tensile strength is universal tensile machine, load cells, controller or indicator, suitable jaws for fixing specimens.

Hardness is defined as the resistance to penetration by a harder indenter. It is a key engineering parameter in design of devices, machines, tools, machines and systems. Various standardized methods are used to determine hardness. The characteristic is used to evaluate all types of materials - alloys, ceramics, elastomers, etc. The results of hardness tests also provide information about other strength characteristics of materials. An important advantage of hardness testing methods is that they are non-destructive,

Carbon fiber reinforced plastics are extremely light and strong materials that contain carbon fibers. They are characterized by a high strength-to-weight ratio, rigidity, high durability, corrosion resistance. They find wide application - shipbuilding, automotive, aerospace applications, civil engineering, sports goods, etc.

2. Materials and methods

Polyamide PA6-CF carbon is a high-temperature material. Carbon fibers significantly increase the strength characteristics of the material. It is characterized by low linear shrinkage, chemical

resistance, high creep resistance. It is used for tooling, prototyping, industrial modeling, etc.

The Shore durometer is used to test hardness, primarily for plastics [6]. Different scales are used for softer and harder materials. The Shore hardness test is performed in dimensionless units. According to ASTM D2240, 12 scales are distinguished. For hard plastics, the Shore D method is used, the indenting foot is hardened steel rod 1.1 mm – 1.4 mm diameter, with a 30° conical point, 0.1 mm radius tip. The test range is from 0-100. The Shore D test is characterized by high accuracy. The Shore durometer used is calibrated.

The maximum tensile force test was performed on a calibrated universal mechanical testing machine. The gripping jaws are tapered to prevent slipping during the test. A graphical interface displays the stress strain graph.

For the tensile strength test, 20 specimens with a standardized shape according to ASTM D638 and a thickness of 6 mm were used (fig.1).

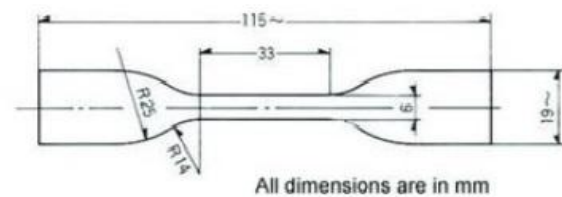


Fig. 1 Tensile strength specimen dimensions

Three hardness specimens with dimensions of 40x40x6 mm were produced. fig.2

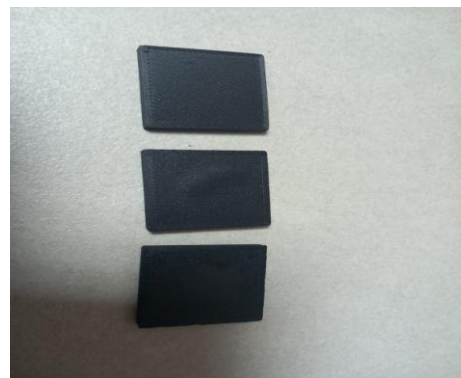


Fig 2. Hardness Shore D test specimens

The minimum number of attempts per sample according to the standard is 5, for greater accuracy 8 attempts per sample were made. The distance between the tests is at least 6mm. The parameters for 3D printing are as follows:

- Layer height-0.275 mm
- 3D printing speed-350 mm/s
- Bed temperature-105°

- Nozzle-0.6 mm
- Infill-100%
- Flow-86%

3. Results and discussion

The results of the test for maximum tensile force are presented in tab.1

Tab.1 Maximum tensile force experimental results

Sample	1	2	3	4	5	6	7
$F_{\max}$ kN	1.38	1.31	1.28	1.34	1.3	1.25	1.34
Sample	8	9	10	11	12	13	14
$F_{\max}$ kN	1.3	1.27	1.33	1.35	1.31	1.38	1.35
Sample	15	16	17	18	19	20	
$F_{\max}$ kN	1.29	1.26	1.3	1.33	1.35	1.36	

No deviations were noted during the test, as well as breakage in uncharacteristic areas, fig.3. The statistical processing of the data is presented in Table 2.



Fig.3 Test specimens after the tests

Tab.2 Statistical summary ultimate tensile force

Summary	
Mean	1.319
Standard Error	0.008488
Median	1.32
Mode	1.3
Standard Deviation	0.037961
Sample Variance	0.001441
Kurtosis	-0.83696
Skewness	-0.13239
Range	0.13
Minimum	1.25
Maximum	1.38
Sum	26.38
Count	20

From the obtained statistical processing, it is noticeable that the mean, median and mode are extremely close in values, which means that the data are symmetrical. Low values of standard deviation and

sample variance are also observed. The negative value of kurtosis indicates a flat distribution. The obtained value of skewness in the range -0.5 and 0.5 also indicates that the values are symmetrical. A diagram of the obtained distribution is shown in Fig. 4.

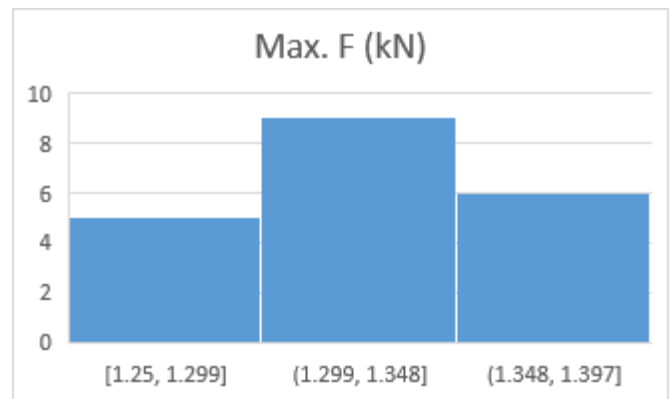


Fig.4 Results distribution.

Hardness results are presented in table 3 and statistical summary is presented in table 4.

Tab.3 Hardness results

Sample 1	Sample 2	Sample 3
82	81.6	82.2
81.5	82	82.2
82.7	81.1	83.3
80.3	83	83.2
82.6	81.7	81.8
82.2	81.9	82.2
83.6	83.1	83
82.2	81.7	82.5

Tab. 4. Statistical summary hardness results

Summary	
Mean	82.23333
Standard Error	0.154834
Median	82.2
Mode	82.2
Standard Deviation	0.758526
Sample Variance	0.575362
Kurtosis	0.533763
Skewness	-0.36801
Range	3.3
Minimum	80.3
Maximum	83.6
Sum	1973.6
Count	24

The statistical summary is similar to the study of maximum tensile force with the exception of kurtosis. At a value above 0.5 the distribution is moderately skewed, fig.5

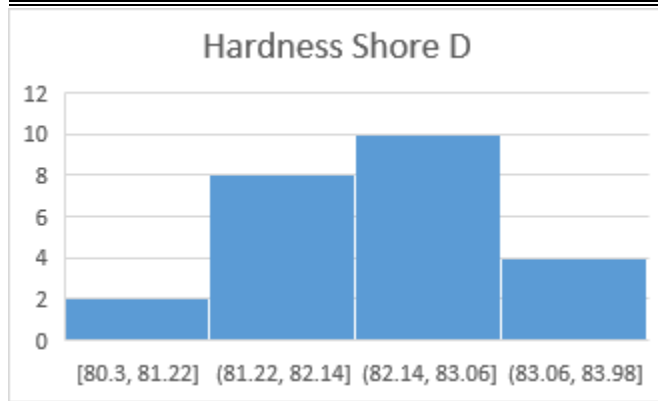


Fig.5 Distribution Shore D hardness results

4. Conclusions

From the studied results and the subsequent statistical processing, the following conclusions can be drawn:

1. The study of the maximum tensile strength of Polyamide PA6 CF showed a range in the interval 1.25-1.38 kN.
2. The study of the hardness of Polyamide PA6 CF according to Shore D showed a range in the interval 80.3-83.6
3. The data of both mechanical strength tests are symmetrical, which means that no deviations were obtained in the tests
4. The distribution of the results for maximum tensile force is flat, and for hardness moderately skewed.

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Luminescent properties of sol-gel synthesized ZrO_2 and Sm_2O_3 coatings on glass

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Abstract: Thin coatings of zirconia oxide (ZrO_2) and samarium oxide (Sm_2O_3) on glass have been prepared by using sol-gel method and dip coating technique. Materials were characterized in detail by X-ray diffraction. The luminescent properties at room temperature were also investigated. Excitation and emission spectra of ZrO_2 coating consists of broad bands with maximums at 318 and 365 nm respectively.

The photoluminescence measurements of samarium coating suggest that can be efficiently excited with 700 nm and consist of emission bands centered at 567, 603, 639 and 707 nm, consistent with characteristic Sm^{3+} transitions. The CIE (Commission Internationale de L'Eclairage) chromatic coordinates, color purity and color correlated temperature were determined.

Keywords: LUMINESCENCE, SOL GEL, COATINGS, SOLAR CELLS, PHOTOVOLTAICS, ZrO_2 , Sm_2O_3

1. Introduction

The development of new luminescent materials in recent decades has led to significant interest in rare earth ions embedded in oxide matrices. These materials find wide application in optoelectronics, displays, laser technologies, biosensors and other high-tech areas [1-5]. Thin-film optical coatings based on metal oxides are also widely used in optoelectronics, photonics and sensor technologies. Among them, zirconium dioxide (ZrO_2) is one of the most studied oxide materials due to its exceptional mechanical, thermal and optical properties [3]. It has three main crystal modifications: monoclinic (m- ZrO_2), tetragonal (t- ZrO_2) and cubic (c- ZrO_2). Doping with rare earth elements, such as samarium (Sm^{3+}), can stabilize the desired crystal phase. [4]. The incorporation of Sm_2O_3 into ZrO_2 leads to changes in the spectral characteristics of the material and improve the luminescence properties.

The sol-gel method has been established as an effective technology for the synthesis of thin films of ZrO_2 , as it allows control over the morphology, chemical composition and phase transitions of the material [2]. This approach allows homogeneous dispersion of rare earth ions in the matrix, which is essential for achieving effective luminescence. This method makes it possible to introduce Sm^{3+} ions into the structure of ZrO_2 , which leads to the appearance of a characteristic orange-red luminescence associated with the electronic transitions of Sm^{3+} [7,8,9]. For the synthesis of $\text{ZrO}_2:\text{Sm}^{3+}$ thin films, precursors such as zirconium alkoxide ($\text{Zr}(\text{OC}_2\text{H}_5)_4$) and samarium nitrate ($\text{Sm}(\text{NO}_3)_3$) are commonly used. The incorporation of Sm^{3+} ions into the ZrO_2 structure leads to the formation of a stable tetragonal phase at lower temperatures, which favors the luminescent properties [3]. Valladares-Barrera et al. [10] studied the synthesis of rare earth oxide powders by two sol-gel methods – using chloride and nitrate precursors. Their results show that the choice of precursors significantly affects the morphology and spectral properties of the final material.

Luminescent properties of Sm^{3+} in ZrO_2 matrix are well known for their characteristic electronic transitions, especially those from $^4\text{G}_{5/2}$ to various low-lying energy levels, which lead to emission in the orange-red region of the spectrum [8]. Upon excitation of Sm^{3+} ions with UV or blue light, strong emission lines are observed around 560–620 nm, which are useful for a number of applications such as phosphor materials for LEDs, sensors and laser media [7]. The phase composition of ZrO_2 has a significant impact on the luminescent properties of Sm^{3+} . Li et al. [7] demonstrated that Sm^{3+} -doped ZrO_2 exhibits the highest luminescence intensity when stabilized in the tetragonal form. Doping reduces the energy of defects in the crystal structure, improving the quantum yield of luminescence.

Applications and future prospects: Thin films of $\text{ZrO}_2:\text{Sm}^{3+}$ are extremely attractive for various technological applications, including: • Optoelectronics – The luminescent properties of Sm^{3+} make these materials suitable for emitting devices and laser media [6]. • Sensor technologies – The sensitivity of Sm^{3+} ions to changes in the environment makes them suitable for the development of gas sensors and biosensors [9]. • Phosphor materials for LED lighting – Mixtures of ZrO_2 and Sm_2O_3 can

be used as stable phosphors with high resistance to thermal and chemical degradation [8]. Ongoing research in this area focuses on the optimization of synthesis parameters and the improvement of the quantum yield of luminescence. Future developments are expected to include further engineering of the crystal structure and modification of the chemical composition of the matrix [9].

Samarium-containing materials have recently been investigated. A borosilicate glass doped with samarium (Sm) was studied, designed and prepared by the melt cooling method to obtain controlled and tunable red emission for further applications of rare earth (RE) doped glass. The influence of Sm_2O_3 concentration on the structure, composition and luminescent properties of the samples was studied. It was shown that the samples have a glassy non-crystalline nature and high transparency in the visible light region. The characteristic transitions of Sm^{3+} ion, assigned to $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$ (J=5, 7, 9, 11), which realize the red-orange emission, were found [11]. The spectroscopic and structural characteristics of different concentrations of strontium-doped lead-zinc phospho-tellurite glasses with Sm_2O_3 with molar composition $x\% \text{Sm}_2\text{O}_3 + 5\text{SrO} + (15-x) \text{PbO} + 20 \text{ZnO} + 50 \text{P}_2\text{O}_5 + 10 \text{TeO}_2$ (where $x = 0.25, 0.5, 0.75, 1 \text{ mol}\%$) are reported. The most intense excitation band around 400 nm was chosen as the excitation wavelength to investigate the luminescence properties. The luminescent spectra show the most intense emission band at 598 nm, referring to the $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$ transition, which is responsible for the appearance of a reddish-orange color [12]. Phosphate glasses with the chemical composition $(42-x/2) \text{P}_2\text{O}_5-(42-x/2)\text{Na}_2\text{O}-15\text{ZnO}-x\text{Sm}_2\text{O}_3$ (where $x = 0.5, 1, 1.5 \text{ mol}\%$) were synthesized by the melt cooling method. The influence of Sm^{3+} doping on the optical properties of the phosphate glasses was studied. The photoluminescence (PL) spectra showed three Sm^{3+} transitions: $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{5/2}$ (600nm), $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$ (564nm) and $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{9/2}$ (645nm). The dependence of the PL intensity on the concentration was studied. The quenching of the PL intensity occurs at Sm^{3+} concentrations above 1% [13].

The aim of the study is to determine the luminescence of coatings containing ZrO_2 and Sm_2O_3 on the glass of photovoltaic panels when light causes excitation of electrons in these coatings. These coatings can be designed to improve the efficiency of the panel by reducing reflections or increasing light absorption, as they increase the efficiency of light conversion into electricity.

The present work is focused on the study of the luminescent properties of thin-film coatings of ZrO_2 and Sm_2O_3 , synthesized by the sol-gel method and deposited on glass substrates. The main focuses are the luminescent properties of the obtained films, with the aim of evaluating their potential applications in optical devices and fluorescent sensors.

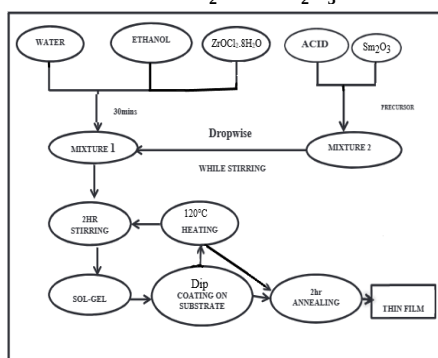
2. Experimental procedures

All compounds were supplied as analytical grades and were used without further modification. Thin film coatings were performed on slides from AGC Glass Europe. $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$, Sm_2O_3 , 99.9% ethanol ($\text{C}_2\text{H}_5\text{OH}$), all purchased from Alfa Aesar Germany. The stabilizer is nitric acid / HNO_3 /.

Preparation of the glass substrate. Before being cleaned, the slides were cut to size (20 mm x 70 mm x 2 mm). Substrate cleaning is important prior to the deposition process, as this allows the cleaning and removal of submicron particles from the substrate surface. This is decisive for the quality of the adhesive film on the base. The glass substrates were cleaned/washed in 99.9% ethyl alcohol for 15 minutes and acetone for 15 minutes and then dried for 30 minutes at 120°C. Cleaning the glass substrates improves the adhesion of the coating material to the substrate and prevents contamination of the formed coating. The scheme presents the production technology.

Sol-gel technology was applied. $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ was dissolved in H_2O and absolute alcohol - so that a 0.01 - 0.02 molar solution was obtained. After 30 minutes, Sm_2O_3 was added. Sm_2O_3 was added to it slowly with vigorous stirring. The ratio was 93.46 wt.% ZrO_2 and 6.54 wt.% Sm_2O_3 . During the hydrolysis, nitric acid was added or stirred until the solution became clear, i.e. until it was completely dissolved. Dip Coating technology was applied to cover the glass sample with the resulting solution. The sample was treated at 400°C with a delay of 15 minutes.

Schematics of the process of obtaining a thin film of sol gel with ZrO_2 and Sm_2O_3



3. Results and Discussion

Photoluminescence (PL) emission spectra at room temperature for all coatings were measured with a Spectrofluorometer FluoroLog3-22, 2014 (Horiba JobinYvon, Longjumeau, France).

The photoluminescence excitation (PLE) spectrum of the deposited ZrO_2 coating is displayed in fig. 1. All data were obtained at room temperature by monitoring the most intensive characteristic emission of ZrO_2 ions at 365 nm wavelength. From the figure it can be observed that the PLE spectrum consists of broad continuous band ranging from 275 to 350 nm wavelength with a maximum at approximately 318 nm. The 318 nm peak is the most intense among all the other peaks, making it the ideal excitation wavelength for recording the emission spectrum.

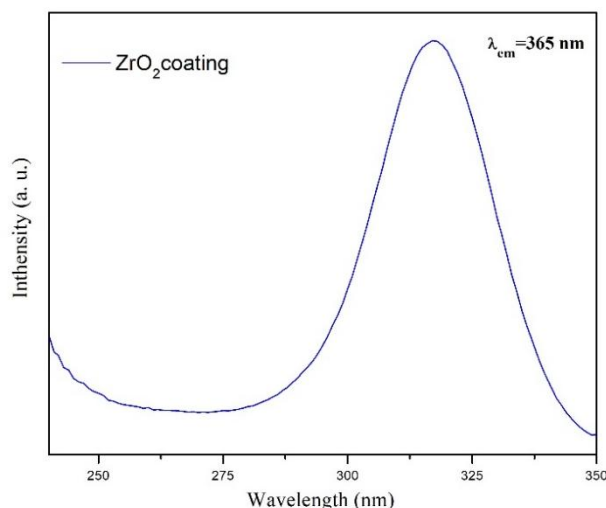


Figure 1. Excitation spectrum of deposited ZrO_2 coating

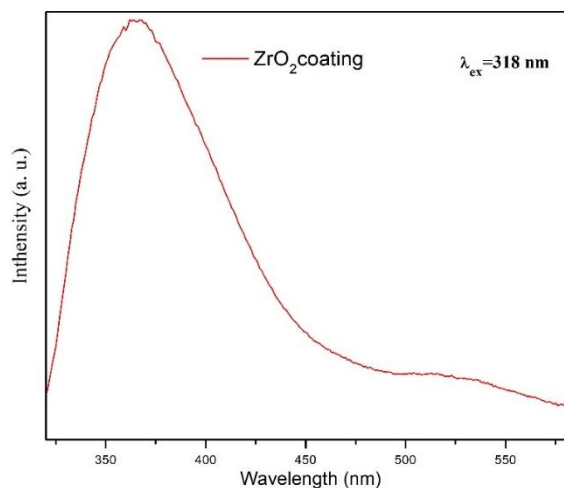


Figure 2. Emission spectrum of deposited ZrO_2 coating

The photoluminescence emission (PL) spectrum of the deposited ZrO_2 coating is displayed in fig. 2, recorded under excitation at 318 nm. As is shown as curve, the PL spectrum consists of a broad band centered at about 365 nm.

Up to now, the origin of ZrO_2 luminescence is not clear and three kinds of different mechanisms have been proposed to explain its blue-green luminescence - an impurity luminescence center model, intrinsic self-trapped excitons and a structure-defect model. [P. Iaconi, D. Lapraz, R. Caruba: Phys. Status Solidi A 50, 275 (1978); M. Garc'ia-Hipolito, R. Martinez, O. Alvarez-Fregoso: J. Luminesc. 93, 9 (2001); V.A. Emeline, N. Serpone: Chem. Phys. Lett. 345, 105 (2001) 20 W.C. Hsieh, C.S. Su: J. Phys. D: Appl. Phys. 27, 1763 (1994); Liang J, Deng Z, Jiang X, Li F and Li Y: Inorg. Chem. 41 3602 (2002)]. At present, the generally recognized model is the structure-defect one, suggesting that the luminescence of ZrO_2 arises from singly ionized oxygen-vacancy defects (F^+ centers) and we assign the emission of the synthesized zirconia coating to the existence of oxygen vacancies.

The presence of oxygen vacancies in ZrO_2 coatings can lead to the formation of new energy levels within the band gap. Additionally, the fact that the emission spectrum is formed by one broad band indicates the presence of a recombination band inside the ZrO_2 matrix band gap.

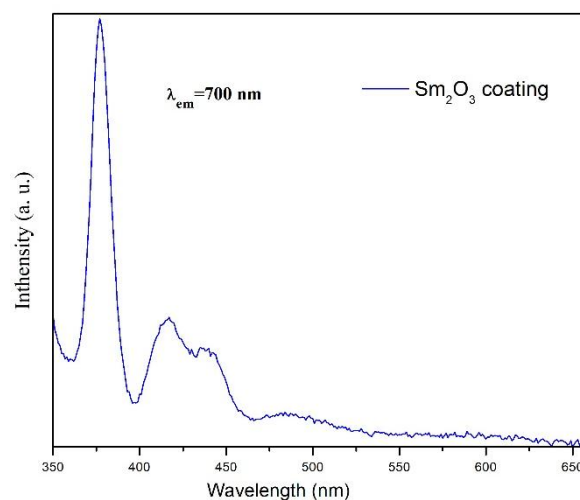


Figure 3. Excitation spectrum of deposited Sm_2O_3 coating

The optical properties of samarium containing coating were further studied. The excitation spectrum of Sm_2O_3 , shown at figure 3, was captured in the 350–650 nm wavelength range and is recorded at room temperature, monitoring the wavelength at 700 nm. Several peaks are observed in the low energy excitation

spectrum at 377, 417 and 440 nm, corresponding to the Sm^{3+} f-f forbidden transitions from $^6\text{H}_{5/2}$ to $^6\text{P}_{7/2}$, $^6\text{P}_{3/2}$ and $^6\text{G}_{9/2}$ respectively. It is noticed that the intensity of the f-f transition at 377 nm is most prominent among all the other peaks; thus, this transition is chosen for the measurement of the emission spectrum. This finding also indicates that the as-prepared phosphor can be efficiently excited by UV (~ 400 nm) LED (Light emitting diode) chips.

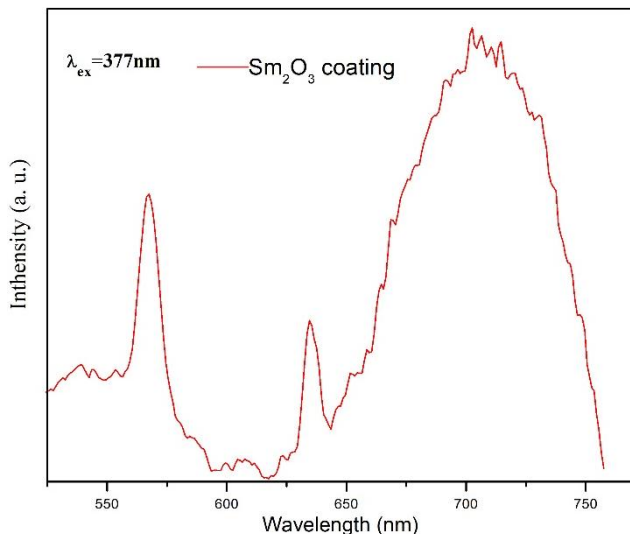


Figure 4. Emission spectrum of deposited Sm_2O_3 coating

The emission spectrum of Sm_2O_3 coating is obtained by monitoring the $^6\text{H}_{5/2} \rightarrow ^6\text{P}_{7/2}$ transition at 377 nm (fig. 4). Four groups of emission lines, approximately ranging from 500 nm to 770 nm appeared in the spectrum. They are attributed to the characteristic intra-configurational f- transition of Sm^{3+} $^5\text{G}_{5/2} \rightarrow ^7\text{H}_J$, where $J = 5/2, 7/2, 9/2$ and $11/2$ at 567 nm, 603, 639 nm and 707 nm, respectively [W.T. Carnall, P.R. Fields, K. Rajnak J. Chem. Phys., 49 (1968), p. 4424; W.T. Carnall, G.L. Goodman, K. Rajnak, R.S. Rana J. Chem. Phys., 90 (1989), p. 3443].

Among the Sm^{3+} transitions, the first one at 567nm ($^4\text{G}_{5/2} \rightarrow ^6\text{H}_{5/2}$) is a magnetic-dipole (MD) transition, which is insensitive to the coordination environment around the active ion, the second at 603 nm ($^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$) is a partly magnetic and partly forced electric-dipole (ED) transition, and the third at 639nm ($^4\text{G}_{5/2} \rightarrow ^6\text{H}_{9/2}$) is purely ED transition, which is sensitive to the changes in the Sm^{3+} surroundings [V. Singh, S. Watanabe, T.K.G. Rao, J.F.D. Chubaci, H.-Y. Kwak J. Non-cryst. Solids, 356 (2010), p.1185]. As can be seen, the first one at 567nm ($^4\text{G}_{5/2} \rightarrow ^6\text{H}_{5/2}$) is with the strongest intensity. The ratio between the MD and ED emission can reveal the degree of asymmetry in the local environment around the Sm^{3+} for various Sm^{3+} -doped compounds [Z. Xia, D. Chen J. Am. Ceram. Soc., 93 (2010), p. 1397]

The asymmetric nature is more prominent when the intensity of the ED transition is higher. The present study shows that the $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{9/2}$ transition (639 nm) of the Sm^{3+} ions has relatively lower emission intensity than the $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{5/2}$ transition (567 nm), which describes the symmetric nature of the host matrix investigated.

CIE coordinates

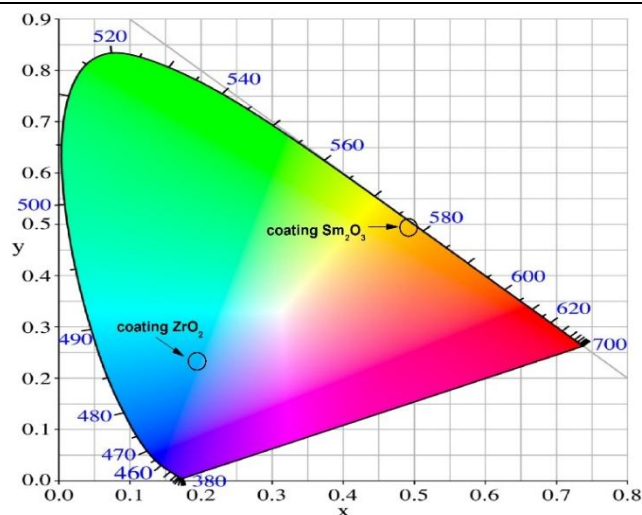


Figure 5. CIE chromaticity diagram of coatings of zirconia oxide (ZrO_2) and samarium oxide (Sm_2O_3).

In order to estimate the actual emitted color, the Commission International de l'Eclairage (CIE) 1931 chromaticity diagram was used [Smith, T.; Guild, J. The CIE colorimetric standards and their use. *Trans. Opt. Soc.* 1931, 33, 73.]. The chromaticity coordinates were calculated from the emission spectra by using color calculator software SpectraChroma (CIE coordinate calculator) [Paolini, T.B. SpectraChroma (Version 1.0.1) [Computer Software]. 2021. Available online: <https://zenodo.org/records/4906590> (accessed on 7 June 2021).]. The values of the chromaticity coordinates of the ZrO_2 coating correspond to the blue color of emission (0.192:0.245) and Sm_2O_3 coating has orange color of emission (0.489:0.506).

The color purity of the synthesized samples can also be calculated via chromaticity coordinates of CIE 1931 diagram. The following expression was used:

$$\text{Color purity} = \frac{\sqrt{(x-x_{ee})^2 + (y-y_{ee})^2}}{\sqrt{(x_d-x_{ee})^2 + (y_d-y_{ee})^2}} \cdot 100\%, \text{ [book: Schubert, E. F. (2006). Light-emitting diodes, Cambridge University press, p. 300]}$$

where (x,y) represents the color coordinates of the synthesized glasses; (x_{ee}, y_{ee}) is the illuminant point of the 1931 CIE Standard Source C with color coordinates (0.3101, 0.3162); (x_d, y_d) are the chromaticity coordinates of the dominant wavelength point, which were obtained by extending the straight line between the point of (x, y) and the illuminant point (x_{ee}, y_{ee}) to the other side of the chromaticity diagram. Generally, it is accepted that 100 % is for pure color and 0% is for white light.

The established values for the color purity of ZrO_2 and Sm_2O_3 coatings were 52% and 96%, respectively. This result indicates that samarium coatings are characterized with very-high color purity.

The color correlated temperature (CCT) was calculated by the McCamy empirical formula:

$$CCT = -449n^3 + 3525n^2 - 6823n + 5520.33$$

(McCamy, Calvin S. Correlated color temperature as an explicit function of chromaticity coordinates. *Color Research & Application*, 1992, 17.2: 142-144),

where $n = (x - x_e)/(y - y_e)$ is the reciprocal slope, ($x_e = 0.332$, $y_e = 0.186$) are epicenter of convergence and x and y are the chromaticity coordinates. The phosphors with CCT values below 3200 K are generally considered as a warm light source, while those with values above 4000 K, as a cold light source [McCamy, Calvin S. Correlated color temperature as an explicit function of chromaticity coordinates. *Color Research & Application*, 1992, 17.2: 142-144]. The calculated CCT value of Sm_2O_3 coatings is 2964.92 K and it can be considered as warm light emitting materials.

4. Conclusion:

In summary, novel thin coatings of zirconia oxide (ZrO_2) and samarium oxide (Sm_2O_3) on glass have been prepared by using sol-gel method and dip coating technique.

Luminescent study revealed that the excitation and emission spectra of the obtained ZrO_2 coating consists of broad bands with maximums at 318 and 365 nm respectively. The photoluminescence measurements of samarium coating suggest that can be efficiently excited with 700 nm and consist of emission bands centered at 567, 603, 639 and 707 nm, consistent with characteristic Sm^{3+} transitions.

The values of the chromaticity coordinate points of the Sm_2O_3 and ZrO_2 coatings are located in the orange and blue region, respectively. The Sm_2O_3 coating is characterized by a high color purity of 96%.

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Ensuring Clean Waterways: The Essential Role of Neutralization in Treating Technological Wastewater

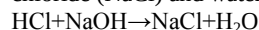
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Abstract: The treatment of technological wastewater through a neutralization process is essential for adjusting the chemical properties of the water. This method aims to ensure that the wastewater meets established discharge limits, thereby protecting aquatic ecosystems and improving water quality in local waterways. Effectively managing process wastewater from large industrial facilities, such as copper rolling mills, presents a significant challenge that requires attention. In this paper, we present experimental testing of the neutralization process for technological wastewater from copper rolling mills, conducted at laboratory levels. Experimental research has shown that the neutralization process involves adding 10% Ca(OH)₂ until a pH of 8.5 is achieved. The duration of the neutralization process is 30 minutes. During this time, a flocculant called Settafloc AP-17 is added at a concentration of 0.033%, specifically 6 ml per liter. This method effectively meets the water quality standards outlined in the Regulation on Limit Values of Pollutant Emissions into Water and the deadlines for compliance.

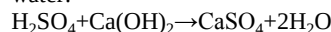
KEYWORDS: WASTEWATER, NEUTRALIZATION, CLEAN WATER

1. Introduction

Neutralization is a fundamental process in industrial wastewater treatment, aiming to adjust the pH of effluents to meet environmental discharge standards and protect aquatic ecosystems. This process involves chemical reactions between acids and bases, resulting in the formation of water and salts, thereby stabilizing the pH of the treated water. The neutralization process is based on acid-base reactions. For instance, when hydrochloric acid (HCl) reacts with sodium hydroxide (NaOH), the reaction produces sodium chloride (NaCl) and water (H₂O) [1]:



Similarly, sulfuric acid (H₂SO₄) can be neutralized by calcium hydroxide (Ca(OH)₂), forming calcium sulfate (CaSO₄) and water:



These reactions are exothermic and require careful control to prevent excessive heat generation. Effective neutralization requires careful consideration of several factors [2]:

- **Chemical Selection:** Choosing appropriate neutralizing agents (e.g., lime, sodium hydroxide, sulfuric acid) based on wastewater composition and desired outcomes.
- **Dosage Control:** Accurate dosing ensures complete neutralization without overcorrection, which could lead to secondary contamination.
- **Safety Measures:** Handling and mixing of chemicals should be conducted with caution to prevent hazardous reactions.
- **Sludge Management:** Neutralization can produce precipitates that require proper handling and disposal.

In summary, neutralization is a critical component of industrial wastewater treatment, facilitating the safe and compliant discharge of effluents. Its application across various industries underscores its importance in environmental protection and sustainable industrial practices [3].

2. Experimental

A real sample of wastewater from copper rolling mills was used for neutralization. The initial pH of this water was 5.7. Calcium hydroxide Ca(OH)₂, with a concentration of 25 g/L, was used as the neutralizing agent. The neutralization process was conducted in multiple stages to achieve different pH values, depending on the chemical characteristics of the wastewater and in compliance with legal regulations. The experiments were carried out with 1 L samples. A lime milk solution of Ca(OH)₂ was used to reach target pH levels of 8, 8.5, and 9. In a batch reactor, a specific amount of the neutralizing agent was continuously added to the wastewater sample. The stirring was performed using a magnetic stirrer at a constant speed of 400 rpm to prevent precipitation. Once the solution reached the desired pH levels of 8, 8.5, or 9, each solution

was filtered through vacuum filtration to separate the solid phase from the liquid phase.

3. Results and Discussion

3.1. Examination of the effect of pH value and amount of flocculant on the neutralization process

The influence of pH value and amount of flocculant on the neutralization process was tested on the sample. The sample had the following chemical composition: Cu= 139 mg/L; Zn= 7.5 mg/L. During the experiments, a chemical analysis of these two elements was performed, because their values exceeded the concentrations permitted for discharge into the river. The initial pH value of the sample was 5.7.

Neutralization to pH 8

A sample volume of 1 liter was neutralized to a pH of 8 using a 10% calcium hydroxide (Ca(OH)₂) solution. The initial pH of the solution was 5.7. To reach a pH of 8, 1.2 milliliters of the 10% Ca(OH)₂ was consumed. Following this, 6 milliliters of a 0.033% flocculant were added to the solution. Samples for chemical analysis were collected at two-time intervals: after 30 minutes and 60 minutes. Each of these samples received an additional 6 milliliters of flocculant. Furthermore, another sample was taken after a total of 12 milliliters of flocculant had been added to the solution at the 60-minute mark. Chemical analysis yielded the following results:

Table1. Chemical analysis of solution after neutralization to pH 8

Time/flocculant	Cu	Zn
30 minutes+6 ml flocculant	0.18	0.15
60 minutes+6 ml flocculant	0.18	0.17
60 minutes+12 ml flocculant	0.2	0.18

Neutralization to pH 8.5

A 1 L sample was neutralized to pH 8.5 with 10% Ca(OH)₂. The initial pH value of the solution was 5.7. Consumption of 10% Ca(OH)₂ up to pH 8.5 was 1.3 ml. Samples for chemical analysis were taken after 30 and 60 minutes, when 6 ml of flocculant was added to these samples, and a sample was also taken after a total of 12 ml of flocculant was added to the solution after 60 minutes. Chemical analysis showed the following results:

Table 2. Chemical analysis of solution after neutralization to pH 8,5

Time/flocculant	Cu	Zn
30 minutes+6 ml flocculant	0,17	0,059
60 minutes+6 ml flocculant	0,15	0,055
60 minutes+12 ml flocculant	0,16	0,061

Neutralization to pH 9

A sample volume of 1 L was neutralized to pH 9 with 10% Ca(OH)_2 . The initial pH value of the solution was 5.7. Consumption of 10% Ca(OH)_2 to pH 9 was 1.5 ml. Samples for chemical analysis were taken after 30 and 60 minutes, when 6 ml of flocculant was added to these samples, and a sample was also taken after a total of 12 ml of flocculant was added to the solution after 60 minutes. Chemical analysis showed the following results:

Table 3. Chemical analysis of solution after neutralization to pH 9

Time/flocculant	Cu	Zn
30 minutes+6 ml flocculant	0.11	0.012
60 minutes+6 ml flocculant	0.19	0.031
60 minutes+12 ml flocculant	0.14	0.023

3.2. Neutralization of flowing water

I - EXPERIMENT - A composite sample was formed from samples of flowing water delivered from the copper rolling mill.

Chemical analysis showed the following chemical composition of the composite sample:

Cu = 7.4 mg/L; Zn = 3.9 mg/L

A composite sample of flowing water with a volume of 1 L was neutralized to pH 8.5 with 10% Ca(OH)_2 . The initial pH value of the solution was 7.9. Consumption of 10% Ca(OH)_2 up to pH 8.5 was 0.1 ml. 6 ml of flocculant was added to the solution. Samples for chemical analysis were taken after 30 minutes and showed the following results:

After 30 minutes: Cu = 0.33 mg/L; Zn = 0.44 mg/L

II – EXPERIMENT – A sample of flowing water delivered from the copper rolling mill.

Chemical analysis showed the following chemical composition of this sample:

Cu = 8.08 mg/L; Zn = 12.4 mg/L; Cr = 0.843 mg/L

A sample of flowing water with a volume of 1 L was neutralized to pH 8.5 with 10% Ca(OH)_2 . The initial pH value of the solution was 7.83. Consumption of 10% Ca(OH)_2 up to pH 8.5 was 0.1 ml. 6 ml of flocculant was added to the solution. Samples for chemical analysis were taken after 30 minutes and showed the following results:

After 30 minutes: Cu = 0.1 mg/L; Zn = 0.15 mg/L; Cr = 0.46 mg/L.

**Figure 1.** Neutralization of flowing water to pH8.5

III – EXPERIMENT – A sample of flowing water delivered from the Sevojno copper rolling mill.

Chemical analysis showed the following chemical composition of this sample:

Cu = 0.65 mg/L; Zn = 5.3 mg/L; Cr = 0.14 mg/L

A sample of flowing water with a volume of 1 L was neutralized to pH 8.5 with 10% Ca(OH)_2 . The initial pH value of the solution was 7.66. Consumption of 10% Ca(OH)_2 up to pH 8.5 was 0.1 ml. 6 ml of flocculant was added to the solution. Samples for chemical analysis were taken after 30 minutes and showed the following results:

After 30 minutes: Cu = 0.15 mg/L; Zn = 0.33 mg/L; Cr = 0.13 mg/L.

Based on the obtained results, it can be seen that by neutralizing the flowing waters to pH 8.5 and adding flocculants, metal concentrations that meet the values for their discharge into the river have been achieved.

4. Conclusion

Based on the conducted experimental tests of the neutralization process on the enlarged (pilot) new i the following conclusions can be formed from the systematized results obtained:

1. Neutralization of running water - is done by adding 10% Ca(OH)_2 until pH8.5 is reached, the duration of the neutralization process is 30 minutes. During the neutralization process it adds flocculant Settafloc AP-17 (0.033%) in the amount of 6 ml/L. In this way it is achieved satisfactory quality of neutralized waters, in accordance with legal regulations.

Acknowledgement

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Methods for surface modification of polyethersulfone nanofiltration membranes

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Abstract: *Nanofiltration is emerged as an effective technique owing to its low footprint and cost-effectiveness and can be used in separation of divalent or multivalent ions, as well as low molecular weight organics, for example water-ethanol mixtures. On the other hand, the performance properties of a membrane such as selectivity and flux can be enhanced via surface modification and functionalization.*

In this study, different methods for surface modification of polyethersulfone nanofiltration membranes are explored in view of improving water-ethanol separation properties. Thin copolymer film deposition as well as surface grafting of hydrophilic monomers are applied and modified membranes are characterized by using Fourier transform infrared spectroscopy, optical microscopy, atom force microscopy, contact angle measurements, etc. The influence of copolymer structure and composition on overall properties of the modified membranes is demonstrated and discussed in view of their potential application in water-ethanol separation technologies.

Keywords: NANOFILTRATION POLYETHERSULFON MEMBRANES, UV-ASSISTED SURFACE GRAFTING, SURFACE COATING, FTIR, AFM, WATER UPTAKE

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The impact of AI on entrepreneurship education

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Abstract: The AI's role in life has increased significantly since the initial introduction of ChatGPT. This trend has drawn the attention of scholars to numerous topics and the entrepreneurship education is one of them. There is already research on the potential of AI in entrepreneurship education and the current paper aims at identifying benefits of AI for entrepreneurship education alongside with potential risks and challenges of its application. The results show that AI can contribute to more personalized education with real environment simulations. Alongside it can improve the assessment. However, challenges like data security, overdependence on AI and AI biases, need to be addressed. The findings identify the need for future examination of specific cases and may contribute to the development of regulations, ethical consideration and development of practices in implementing AI in entrepreneurship education.

KEYWORDS: ENTREPRENEURSHIP EDUCATION, ARTIFICIAL INTELLIGENCE (AI), ACADEMIC ENTREPRENEURSHIP

1. Introduction

The generative artificial intelligence (AI) has become a factor of development for numerous areas of life and the education is certainly one of them. In the current paper we draw attention to two interrelated topics which determine the dynamics of economics. It is inevitably affecting the functioning of market economy by its contribution to promoting the benefits of entrepreneur initiative [1]. Recent years the entrepreneurship education has transformed both its content and methodological approaches. The new realities set by the development of technologies and unpredicted events like the COVID-19 pandemic require the introduction of new methods to teach entrepreneurship both to specialists and nonspecialists [2]. Technological development sets a new framework which allows an improved interaction between teachers and students, more flexibility and engagement in the educational process [3]. AI has the potential to provide a new level and new dimensions in the education on entrepreneurship and its impact on entrepreneurial learning and pedagogical methods needs to be explored.

The research object is entrepreneurship education. **The focus** is the potential of AI to transform entrepreneurship education. **This paper aims** to identify benefits of AI for entrepreneurship education alongside with potential risks and challenges of its application.

The current study is performed in the context in a scientific project "Academic Entrepreneurship Roadmap", funded by Erasmus+ Programme, Action type KA220-HED - Cooperation partnerships in higher education, 2023. It aims to thoroughly examine the benefits of entrepreneurship education from a variety of angles, with a focus on students, faculty, and business professionals. The latter context determines that the current examination of the AI's role in entrepreneurship education uses a limited range of **research methods**, namely desk research and review of current studies on the topic. **The authors' standpoint** is that the AI provides more tools for entrepreneurship education but also leads to various challenges and risk.

The paper structure follows the main purpose and aims of the research, i.e., after the introduction, the second part presents a literature review based on current research in the field of AI in entrepreneurship education. The third section focuses on the research methodology. The latter serves as the study's framework and as the foundation for the fourth section, which presents the empirical findings. The paper concludes with conclusions and recommendations for future research on the implementation of AI in entrepreneurship education and its consequences.

2. Literature Review

2.1. AI Transformation in Education

AI has been transforming the education in numerous ways. The latter include automating instructional tasks, analyzing learning data, and implementation of more personalized teaching approaches [4]. In this regard some researchers conclude that the implementation of AI can make learning more student-centered and

equitable [4]. Thus, in general AI contributes to more personalized and adaptive learning, but also helps in assessments of the students.

Opportunities for More Personalized and Adaptive Learning

AI provides experience that meets the individual student needs. Through machine learning algorithms and data analytics, AI systems analyze learners' strengths, weaknesses, and preferences and has the potential to adapt everything – the instructions, the content, etc. [5]. For example, such platforms can modify task difficulty in real time to keep students challenged without demotivating them [6]. Such approach allows students to set learning goals, enhances competence through personalized challenges, and strengthens the connection with the students by using such AI tools [7]. There are studies, which show such systems have the potential to improve academic performance compared to traditional methods, which is empirically tested [5].

Furthermore, AI makes the learners more engaged through real-time feedback mechanisms. For example, voice and facial recognition technologies check student participation and even their emotional states during lessons. Thus, the educators can adjust their teaching strategies at the same moment [8]. If a student appears disengaged, the system might propose to introduce more interactive content to awake interest. Such adaptational approaches are related to neuroscience and educational psychology. They aim to ensure that the methods are in line with needs of the student and the acquisition of knowledge. Thus, it ensures that the interaction between learners and teachers is effective [9].

AI Application in Assessments Process

The application of AI for assessment can rely on several tools. They can automate grading and provide feedback to students. This can be performed by using machine learning models to grade essays or generate adaptive quizzes. The latter can reduce tutors' workload and improve the consistency of assessment.

Such approach for assessments is transforming the education by providing a lot of benefits and innovative applications. One of them is the ability to make the learning experiences more personalized by adaptive testing. Such approach is based on developing question with a difficulty and content, adapted to the individual student needs. The studies show that it improves the engagement and performance of students. [10]

Furthermore, automated systems (including AI) assist in evaluation of assignments and essays, where they can be useful in ensuring more consistency and accuracy. Thus, the human biases in grading can be reduced [11]. Thus, the automation reduces the time spend and focus more on making the education process more effective. The real-time feedback is another benefit of the AI introduction in assessment process. AI can provide immediate feedback on the students' performance and receive more focused instructions for further improvements. [10] Alongside, the educators are able to provide more targeted advice for student's future work [12].

The application of AI in the assessment process provides a more personalized, efficient, and effective learning. The latter leads to higher level of material acquisition and the ability to practical application of the acquired knowledge [13].

2.2. The Benefits of AI for Entrepreneurship Education

The standard approaches in entrepreneurship education have emphasized on experiential learning through creation of business plans, competitions and case studies. The introduction of information and communication technologies (ICTs) creates new teaching-learning models. Furthermore, AI represents a brand-new level by providing the opportunity to create an adaptive curriculum that corresponds to the individual needs of the learner. Chen et al. (2021) identify three evolutionary phases in teaching entrepreneurship - passive knowledge transfer (pre-2010), interactive digital tools (2010–2020), and AI personalization (post-2020).[14]

The AI has also an innovative effect on the entrepreneurship education. It fosters creative problem solving and thus develops the innovative thinking and creativity. Furthermore, it creates environment for experiential learning and more intensive collaboration among students. From the point of view of skills acquisition AI helps in the identification of various market trends and consumer preferences, assisting students in their business ideas [15]. It also provides support for future entrepreneurs to test their decision-making skills in a risk-free simulation [16]. Furthermore, AI contributes to creation of knowledge-sharing environment and collective creativity, which involves many perspectives and improves the innovation in its earliest stage [17]. However, such introduction of AI requires development of AI literacy of tutors and alongside following any ethical considerations and their latest changes [15] [16].

2.3. Potential of Specific AI Tools for Entrepreneurship Education

Generative AI

Generative AI tools, such as ChatGPT, can be utilized in various ways. On one hand they are very useful in generation of ideas and creating an interactive learning environment. Furthermore, the students can test their business concepts, simulating customer interactions, and even refine pitches through generation feedback on it. At first glance this may sound like the students do not need to be creative and think anymore, but studies show that generative AI improves their ability to critically evaluate business ideas. AI becomes a kind of tutor for them and stimulates their creativity, bridging theoretical knowledge with its practical application [18] [19].

Machine Learning & Big Data

Machine learning and big data learning support future entrepreneurs in proving them with advanced knowledge and skills to identify market trends, assess opportunities, and optimize strategies. They can identify consumer preferences and potential gaps supporting their decision making [20] alongside allowing them to allocate resources efficiently and mitigate risks [21].

Chatbots and Virtual Assistants

Such tools assist the students in understanding better the business planning by automating competitor analysis, financial modeling, and customer persona development. They can provide them with access to industry benchmarks and regulatory requirements. Thus, they reduce research time while improving accuracy. [22]

Business Simulations and Gamification

Platforms with AI, which create gamified business environments, provide the future entrepreneurs with an environment in which very realistic scenarios for virtual companies are created. In these platforms may be incorporated various challenges like

supply chain disruptions, business negotiations, problem-solving, etc. There are also gamification perspectives, which are about tracking of the progress, rewards, etc. They are proven to increase interest, engagement and dedication. [23]

Pedagogical Support

There are AI systems which main purpose is to be a kind of pedagogical partners (for example Edthena). They can provide feedback on a proposed business plan and teaching methodologies. They can analyze the submissions of students as well. Furthermore, these tools can identify gaps and even recommend resources, supporting the learning process [24].

2.4. Risks and Challenges before the Application of AI in Entrepreneurship Education

Although AI has great potential in improving entrepreneurship education, several challenges must be taken into consideration. The integration of artificial intelligence (AI) in entrepreneurship education presents significant opportunities but also comes with substantial risks and challenges.

The most common concern about the introduction of AI is the potential loss of the analytical skills of humans to make decisions. This risk comes from AI-driven tools making predictive analysis and business simulations. This may lead to high level of reliance on technology compromising their critical thinking and problem-solving abilities [25]. It also reduces the role of person of entrepreneur by limiting the nuances of decision making related to the very specifics of the situations and entrepreneurs' intuition [26].

Alongside, there are issues related to the algorithms. AIs are "educated" based on historical data, but it can contain biases, which of course will affect the recommendations and learning outcomes [27]. Such risks present a high threat to the decision making of the future entrepreneurs if they fully rely on AI for market opportunities, funding decisions, or prediction of customer behavior.

Data privacy is another challenge in the use of AI in entrepreneurship education. The AIs require a lot of personal data to be able to give back personalized answers. These issues are related to the data protection regulations [28]. Thus, universities and educators must take into consideration the data policies to safeguard student information while using AI.

Last, but not the least any institutional resistance to AI application is challenge to education. Some of the educators lack the expertise to use AI as one of their teaching tools [26]. This requires expertise development to improve AI literacy among tutors, ensuring that the AI will add to the educational approach and not substitute it.

3. Methodology of the Research

The current study uses desk research based on a literature review of the studies focused on the implementation of AI in entrepreneurship education.

At the first stage we made a list of various studies on the topics. This includes most recent works (from last five years) alongside older works, which are relevant to the current state-of-art. Next, we reviewed the statements and conclusion to identify similarities in the benefits of AI, but we also searched for challenges and risks in its implementation. Finally, we summarized the information under topics and subtopics, represented in the structure of the paper.

It should be underlined that the current work does not cover all the studies in the examined area and thus does not state that these are all benefits and risks in AI application in entrepreneurship education.

4. Discussion and Results

AI's role in entrepreneurial competency development is inevitable. It contributes to critical thinking, creativity, and business planning [29] [30]. It can be applied to enhance teaching strategies and learning outcomes by using AI-driven tutoring and student engagement [31] [32]. It has been proven that the use of deep learning improves student motivation and psychological readiness [33].

Alongside there are some challenges and Ethical considerations, which need to be taken into consideration. It may increase dependence on AI and lead to underestimation of human intuition in entrepreneurship. More efforts are necessary in insuring data privacy, bias in AI models, and ethical dilemmas [34].

These have led us to the understanding that AI should not be solely utilized but there is a need for AI-human collaboration in entrepreneurship education [35].

3. Conclusion

The integration of AI in entrepreneurship education represents a significant transformation in how future entrepreneurs are educated, trained and assessed. The current paper synthesized some research on the utilization of AI's and its role in educational strategies, curriculum development and the assessment. The findings show that AI tools such generative AI, machine learning, etc. contribute to easier opportunity recognition, simulation of real business environment and personalization of the learning process. However, there are serious concerns such as ethical dilemmas, algorithmic biases, and the risk of over-reliance on automated systems, which require certain measures.

The study provides a framework on benefits and challenges but further investigation on various aspects is required to ensure a human-centric approach to technological integration in entrepreneurial training. Future research should focus on case studies, AI's long-term impact on entrepreneurial success (as far as there is data) and the emerging trends in AI-driven business incubators and accelerator programs.

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Increasing the competitiveness of business organizations through reducing energy consumption and effective energy management

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Abstract: Energy efficiency is an important part of the EU's ambition to transition to a carbon-neutral economy by 2050. Greater energy efficiency will be needed in the future if the EU is to achieve this goal. All sectors of the economy have the potential to contribute to energy efficiency. Consistent energy management helps organisations realise untapped energy efficiency potential. In this way, they will benefit from cost savings and make a significant contribution to protecting the environment and climate by reducing harmful emissions and carbon footprints. This study aims to analyse the potential opportunities for businesses to realise savings, gain competitive advantages and achieve sustainable development and a positive image.

Keywords: ENERGY CONSUMPTION, ENERGY INTENSITY, ENERGY EFFICIENCY, ENERGY MANAGEMENT, COMPETITIVENESS.

1. Introduction

Organizations of various types and sizes are increasingly seeking to reduce their energy consumption, driven by the need to:

- reduce their costs;
- meet legislative or self-imposed carbon targets;
- reduce their dependence on fossil fuels;
- improve their reputation as socially responsible organizations;
- gain competitive advantages and achieve sustainable development.

At the same time, governments are increasingly seeking to limit greenhouse gas emissions from citizens and industries by introducing legislative measures to encourage carbon footprint reduction. Among the many indicators for efficient energy management defined by the EU, some of the most indicative are primary and final energy consumption and the energy intensity and efficiency of the economy. They are the subject of analysis in this study.

2. Analysis of energy consumption and energy efficiency of businesses in Bulgaria

In the context of the climate and energy crisis, saving energy is more important than ever. The European Union has set a target of reducing energy consumption by at least 11.7% by 2030 compared to 2020 levels.

Primary energy consumption

Primary energy consumption is the total amount of energy used by a country, region or system before it is converted or transformed into other forms. It covers energy extracted from natural sources that has not yet been processed for end use. This includes raw energy resources such as coal, natural gas, oil, renewable sources (wind, solar, water, biomass) and nuclear fuel. Primary energy is measured before it is converted into electricity, heat or other forms of secondary energy for end users. This indicator is an important indicator of a country's industrial development and economic activity. Fig. 1. shows the primary energy consumption in Bulgaria for the period 2018 ÷ 2022. After the decline in consumption during the Covid pandemic, a trend of increase in primary energy consumption is observed. [2], [3]

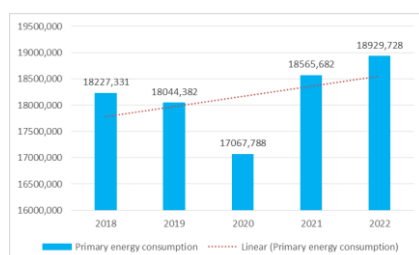


Fig. 1 Primary energy consumption in Bulgaria (thousand tons of oil equivalent - ttoe)

Fig. 2. shows the primary energy consumption in EU countries for the period 2012 ÷ 2022. Six EU Member States are experiencing growth in consumption and need to take urgent measures to reduce primary energy consumption. These are Poland, Bulgaria, Lithuania, Ireland, Hungary and Croatia. [3], [6]

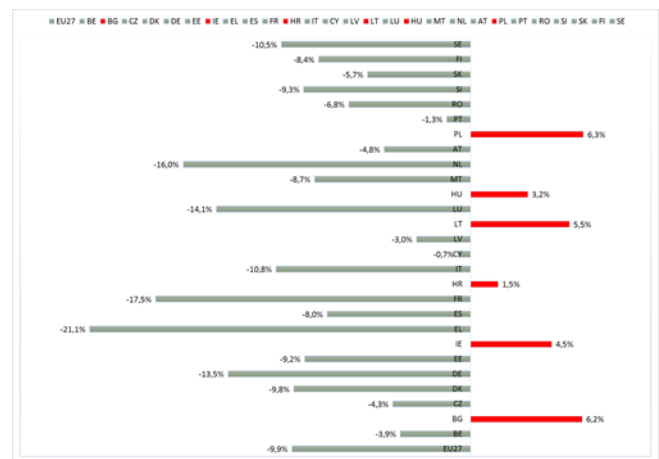


Fig. 2 Primary energy consumption (EU 2012-2022)

Final energy consumption

Final energy consumption represents the energy that is used directly by end users – households, industry, transport, agriculture and services. This is the amount of energy that reaches them after all transformations, transfers and losses within the energy system. It shows how efficiently and sustainably energy is used in different sectors and is a key indicator for understanding the real impact of the energy sector on the economy and the environment. Fig. 3 shows the final energy consumption for the period 2018 ÷ 2022 in Bulgaria by sector.[4], [6]

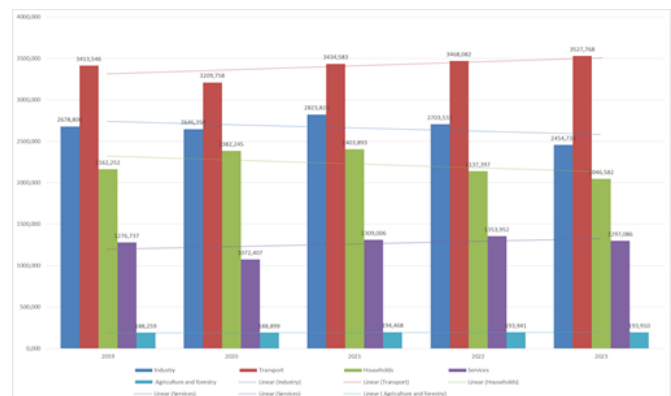


Fig. 3 Final energy consumption by sector in Bulgaria (ttoe)

The analysis shows that transport and industry dominate final energy consumption. The same trend is observed in the analysis of data for the EU countries in general. [7]

Fig. 4 shows the final energy consumption in Bulgaria by type of resources for 2023. It reports a decline in all types of energy resources and energy, except for oil, which remain unchanged.

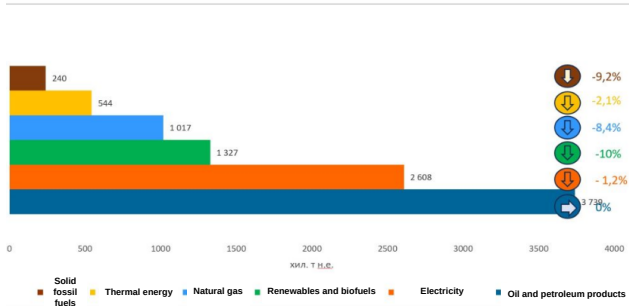


Fig. 4. Final energy consumption in Bulgaria by type of resources for 2023.[9]

Fig. 5 shows the final energy consumption of the EU countries for the period 2012 ÷ 2022. For the analyzed period, an increase in final energy consumption is reported in Malta, Poland, Ireland, Hungary, Lithuania, Bulgaria, Romania, Portugal, Croatia, Cyprus and the Czech Republic. In all other EU Member States, a permanent decrease in final energy consumption is observed.[4]

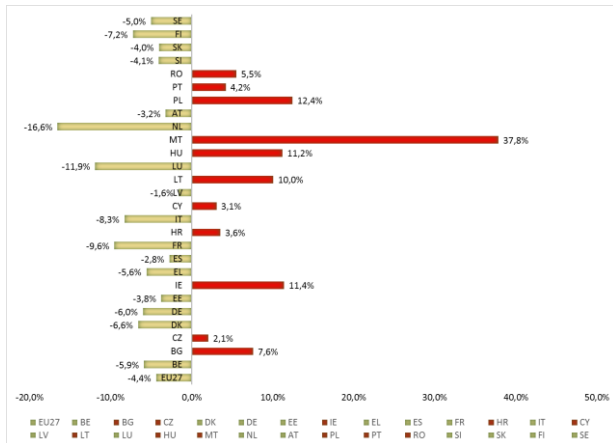


Fig. 5 Final energy consumption (EU 2012-2022)

Energy intensity and energy efficiency

Energy intensity is an indicator that measures the amount of energy needed to generate one unit of gross domestic product (GDP) of a country or economy. It reflects the efficiency of the use of energy resources in economic activity. Lower energy intensity indicates a more efficient use of energy to create economic value, which is usually associated with higher energy efficiency and sustainable development.[8]

Energy efficiency is the degree to which energy is used optimally to perform a particular activity or achieve a given result. It measures the ability to extract maximum benefit from the energy resources used, while minimizing losses. Higher energy efficiency means that less energy is needed to perform the same task, which leads to resource savings, cost reductions and reduced negative environmental impacts. Energy efficiency can include the use of energy-saving technologies, better insulation of buildings and optimization of production processes.[8]

Energy balance is a systematic record of the energy inputs and outputs within a given system, such as a country, region or enterprise, over a specified period of time. It includes information on the production, import, export, transformation, transmission, losses and final consumption of various energy resources (such as coal, natural gas, oil, electricity and renewable sources). An energy

balance is an important tool for analysis and planning, as it shows how energy is distributed and used in the economy. It helps to identify opportunities for improving energy efficiency, sustainability and dependence on imported energy resources

The energy balance serves as a natural basis for calculating various indicators of energy consumption and energy efficiency. One of the key indicators that attracts the attention of economists and energy analysts is the amount of energy needed to produce one euro of GDP, known as energy intensity. A lower value of this indicator means more efficient use of energy to generate one euro of GDP. Improvements in energy intensity are due to various factors, such as the transition from an industrial to a service-based economy, the introduction of more efficient production methods, the reduction of energy-intensive activities, the closure of inefficient enterprises and the use of energy-saving technologies. According to analyses and calculations for 2023, Bulgaria's energy intensity is about 0.303 ttoe per 1000 euros of GDP (2015 = 100). In 24 years, this indicator has decreased by more than half – this is a serious success considering the unprecedentedly high levels from which the country is starting. Bulgaria is the only country in the EU whose energy intensity was above 0.600 ttoe per 1000 euro GDP in year 2000. The economy is restructuring, but this indicator remains far from the EU average, which for 2022 is 0.098 ttoe per 1000 euro GDP. [5]

Fig. 6 shows the energy intensity of the Bulgarian economy during the period 2018 ÷ 2022..

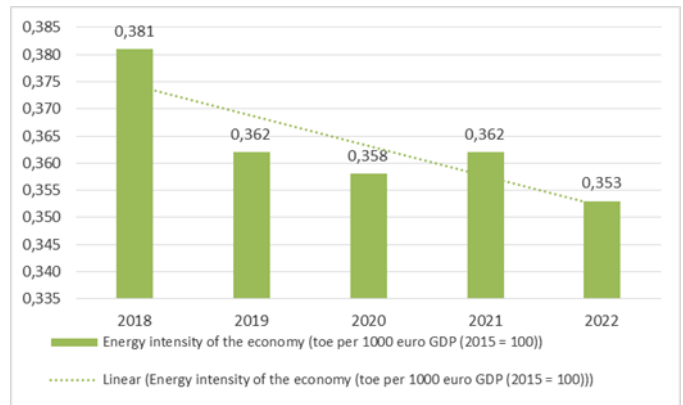


Fig. 6. Energy intensity of the Bulgarian economy (ttoe per 1000 euros of GDP) (2015 = 100)

The ratio between final and primary energy consumption reflects how much of the energy input has reached the end user after transformation and transmission. For the first time, this indicator reaches 57%. The main factor behind this improvement is the reduction of losses in transformation processes, mainly due to the lower use of coal for electricity generation. The same indicator for the EU in 2022 is 75%.

3. Efficient energy management

Energy saving and cost reduction are closely linked, especially when the organization has a certified energy management system according to ISO 50001. The efficient use of energy resources is among the top priorities for every company. The implementation of appropriate measures is aimed at constantly improving the energy efficiency and performance of the organization, which at the same time contributes to climate protection, as well as to:

- cost savings through efficient energy policy;
- transparent and optimized energy flows;
- improved energy balance and reduced harmful emissions;
- use of subsidies or tax breaks, where available.

Each Member State has individual targets for reducing energy consumption or energy intensity. These targets are set at national level, taking into account the economic and structural characteristics of the respective country. In this way, the countries

contribute to achieving the overall EU target in the field of energy efficiency.

The country's national target is set in the Integrated Energy and Climate Plan of the Republic of Bulgaria 2021-2030.[10]

According to this plan in 2030, Bulgaria plans to achieve a reduction in primary energy consumption by 27.89% and a reduction in final energy consumption by 31.67%. In terms of the absolute level of energy consumption for 2030, Bulgaria has set a target of 17,466 ktoe of primary energy consumption and 10,318 ktoe of final energy consumption.[10]

Based on the average annual final energy consumption for the period 2016 - 2018, the energy savings to be achieved in the period 2021-2030 and, accordingly, the cumulative energy savings target to be achieved by December 31, 2030 have been calculated. These values are presented in Table 1.[10]

Table 1: Annual energy savings in final energy consumption for the period 2021-2030, ktoe [9].

Year	2021	2022	2023	2024	2025	2026	2027	2028	2029	2030	TOTAL cumulative savings for the period 2021-2030
2021	69.2										69.2
2022	69.2	69.2									138.4
2023	69.2	69.2	74.05								212.45
2024	69.2	69.2	74.05	74.05							286.5
2025	69.2	69.2	74.05	74.05	92.0						378.5
2026	69.2	69.2	74.05	74.05	92.0	92.0					470.5
2027	69.2	69.2	74.05	74.05	92.0	92.0	92.0				562.5
2028	69.2	69.2	74.05	74.05	92.0	92.0	92.0	92.0			654.5
2029	69.2	69.2	74.05	74.05	92.0	92.0	92.0	92.0	92.0		746.5
2030	69.2	69.2	74.05	74.05	92.0	92.0	92.0	92.0	92.0	92.0	838.5
											4357.55

Bulgaria takes advantage of the opportunity provided by Directive (EU) 2018/2002 to gradually increase the target (from 0.7% to 0.92%), with the aim of achieving a cumulative value of 4,357.55 ktoe by 2030. [9], [11]

4. Opportunities to increase the competitiveness of business organizations by reducing energy consumption and implementing effective energy management

Increasing the competitiveness of business organizations by reducing energy consumption and implementing effective energy management can be achieved through:

1. Energy efficiency

- *Investments in energy-saving technologies:* Replacing old and energy-inefficient machines with new ones that have lower energy consumption.
- *Improving energy insulation:* Reducing energy losses through quality insulation of buildings, windows and ventilation systems.
- *Using energy-saving lighting:* Switching to LED lighting or other energy-efficient solutions.

2. Energy management

Energy management is an organized process that involves collecting and analyzing energy consumption data, identifying savings opportunities, and implementing improvement measures. This approach allows organizations to optimize the use of energy resources, reduce costs, and minimize environmental impact through:

- *Creating an energy management strategy:* Developing a long-term plan to reduce energy consumption, including monitoring and cost analysis.
- *Implementing energy standards:* Certification to international standards such as ISO 50001, which helps to structure and optimize energy management processes.
- *Using monitoring and analysis software:* Installing energy monitoring systems that allow for real-time tracking and analysis.

4.1. Importance of energy management

Economic benefits:

Energy management reduces energy costs by using resources more efficiently and reducing losses. This leads to lower energy bills and increased competitiveness of organizations.

Environmental benefits:

Optimizing energy consumption reduces greenhouse gas emissions and environmental impact. This is important for achieving environmental sustainability and conserving natural resources. Energy management is a key tool in efforts to combat climate change and protect the environment. It combines economic and environmental benefits that work together to create a greener and more sustainable world.

Productivity improvement:

Improving energy efficiency leads to increased efficiency of production processes and reduced equipment maintenance time. Energy management identifies inefficiencies in systems and suggests solutions to eliminate them, such as implementing more advanced technologies or automation.

By monitoring and predicting energy needs, unwanted production interruptions related to energy problems are prevented. More reliable energy management ensures stability in the work process and reduces deviations from quality standards.

Energy management often involves the integration of advanced technologies such as IoT, artificial intelligence, and automated control systems that improve performance.

Social benefits:

The funds released can be invested in social initiatives such as education, healthcare and sports, contributing to the general well-being of society. Energy management helps reduce emissions of harmful substances such as carbon dioxide, sulphur dioxide and nitrogen oxides, which improves air quality. Reduced energy consumption and the use of clean energy contribute to a healthy environment, reducing the risk of diseases and other health problems.

Energy management is a critical process for sustainable development and efficient use of energy resources. Through a systematic approach that includes data collection, analysis, planning, implementation, management and monitoring, organizations can achieve significant economic and environmental benefits. Energy management software solutions provide powerful tools for optimizing and controlling energy systems, leading to increased efficiency and sustainability.

Energy management applications in various areas, such as photovoltaic power plants, energy storage systems and electric vehicle charging stations, demonstrate the broad potential for improving energy efficiency. Investing in energy management technologies and systems not only reduces costs, but also supports environmental sustainability and social well-being. Energy management is the key to the future of effective resource management and sustainable development, providing the necessary tools and knowledge to address the challenges of the modern world.

Good results can be obtained by implementing an Energy Management System (EMS) in accordance with the ISO 50001:2018 standard.[1]

4.2. Benefits of energy management

Global competition, clean energy and carbon emission reduction regulations, and the integration of the European electricity market are just some of the factors that are increasing the demand for energy efficiency among industrial companies across Europe.

Accurate and regular reporting of energy consumption is an important first step in the process of continuous improvement of energy performance. Energy consumption analysis is extremely important for the organization, in order to accurately determine energy performance and identify areas with potential for energy savings, which lead to justified and effective measures to increase energy efficiency.

Companies in various industries see energy management as an opportunity to reduce operating costs. Despite the differences in the characteristics and specifics of production in individual companies, there are common advantages of implementing an energy management system that are applicable to most businesses:

1. Reducing electricity costs

- Increase productivity and competitiveness;
- Lower energy bills lead to increased financial stability;
- Regular review of energy flows and costs;
- Increased planning certainty;
- Derivation of energy efficiency measures through the obtained key energy indicators;
- Climate and environmental protection;
- Maintaining a good information environment for employees.

2. Renewable energy sources

- *Installation of renewable energy systems:* Solar panels, wind turbines or geothermal systems to produce clean energy.
- *Green energy purchasing:* Purchasing energy from suppliers that use renewable sources.

Implementing renewable energy helps companies meet regulatory requirements to reduce carbon emissions. Using renewable energy ensures that the production process remains economically viable and environmentally safe in the long term. Managing renewable energy positions companies for success in the global green economy.

3. Process optimization

- *Automation:* Integration of automated systems for control of the production process and energy consumption.
- *Energy rehabilitation:* Repair and optimization of systems and processes to reduce energy losses.

4. Staff training

- Conducting training for employees to raise awareness about the efficient use of energy.
- Promoting a culture of energy efficiency in the organization.

Creating a culture of energy efficiency can motivate employees to actively participate in optimization processes, contributing ideas and solutions. Greater engagement leads to a better work environment, increases staff satisfaction and reduces turnover. Such a culture encourages employees to seek creative solutions to improve processes, which leads to higher productivity and competitiveness.

5. Financial and tax incentives

- Taking advantage of subsidy and grant programs related to energy efficiency investments.
- Cost reduction through tax relief for environmental investments.

Tax incentives for energy efficiency free up financial resources that can be redirected to other important areas such as innovation, marketing or expansion of production. By providing incentives for the introduction of energy-efficient technologies, companies can recover their investment faster and continue with new investments. Financial and tax incentives for energy efficiency help companies meet increasingly stringent carbon regulations and environmental standards.

Investments in energy efficiency lead to a permanent reduction in energy costs, which ensures long-term financial stability. They allow companies to reduce costs, improve their reputation and meet global requirements, making them more competitive in both local and international markets.

6. Energy waste management

- Recycling and reusing waste energy.
- Converting waste heat into useful energy through technologies such as cogeneration.

Energy waste management reduces greenhouse gas emissions, which meets the requirements of sustainable development and improves the environmental reputation of the company. Reducing energy waste helps companies comply with environmental standards and avoid sanctions. Companies that effectively manage

energy waste are perceived as environmentally responsible and innovative, which attracts customers, investors and partners. Reducing energy waste leads to permanent savings that strengthen the financial stability of the company.

7. Sustainability: Smaller environmental footprint and compliance with regulatory requirements. Sustainability should not just be a goal, it should be at the heart of all processes and systems of the enterprise. From reducing the carbon footprint to optimizing energy use, we all need to be responsible and contribute with our actions to achieving sustainability goals.

8. Improving image: Organizations that implement sustainable practices are perceived better by customers and partners.

9. Innovations: Process improvement leads to the introduction of new technologies and increased productivity. Energy-efficient machines and equipment are often more innovative, more reliable and faster, which increases productivity.

5. Conclusion

Through a strategic approach to energy management, businesses can not only reduce their costs, but also improve their competitiveness by meeting the demands of the modern market. Efficient use of energy leads to real benefits: reducing resource consumption and the associated reduction in energy costs provide a significant competitive advantage. In addition, energy efficiency stimulates new business models, innovative technologies and services that can provide additional market advantages.

Energy efficiency is not only a measure for reducing costs, but also a strategic tool for increasing market share, improving sustainability and building a positive reputation of the organization. This makes businesses more competitive in the long term. Companies that demonstrate a commitment to sustainable development through energy efficiency are perceived better by customers, investors and partners.

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