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MATERIALS 2026
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Rapid prototyping of a friction coupling for joining rebar

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Abstract: Friction couplings for joining rebar are widely used in civil engineering and mechanical structures, including the construction of high-rise buildings, towers, bridges, overpasses, tunnels, dams, and other infrastructure. These friction couplings are mechanical elements that provide a strong and reliable connection between the components being joined by transferring loads through the friction force generated between the contact surfaces. This force is applied by the geometry of the friction coupling and the installation-induced clamping force. The primary factor governing the proper selection and application of a friction coupling is the cross-sectional diameter of the rebar, as the friction coupling must be designed to withstand the loads encountered under service conditions. In addition, environmental influences, particularly the difference in the coefficients of thermal expansion of the joined materials, represent an important consideration. Another critical factor is the installation process itself, as the performance of the friction coupling depends directly on proper installation. Therefore, comprehensive testing of the installed friction coupling is essential to ensure its reliable performance throughout its service life. This paper presents an original design solution for the development of a friction coupling for joining Ø25 mm rebar, developed through three successive design iterations. The proposed friction coupling is intended to ensure structural integrity under static and dynamic loading, including seismic loading and vibration. An initial prototype design was developed in accordance with the applicable technical standards and validated using the Finite Element Method (FEM) to evaluate its load-bearing capacity under static loading conditions and identify potential stress concentrations. The prototype models were manufactured using the Fused Filament Fabrication (FFF) additive manufacturing process with the support of CAD/CAE systems. The friction coupling was ergonomically designed through mass and shape optimization.

KEYWORDS: FRICTION COUPLING, REBAR, CAD/CAE, ASSEMBLY, RAPID PROTOTYPING

1. Introduction

Rapid prototyping encompasses a set of techniques used to quickly produce physical models, enabling fast and cost-effective evaluation of a product's geometry and functionality without the use of complex tooling, most commonly through additive manufacturing technologies [1, 2]. The resulting prototype may be fully functional if its intended application is not particularly demanding. More frequently, however, prototypes are only partially functional because the materials used do not possess the properties required for full operational performance. Such prototypes are particularly useful for experimental investigations focused on geometric evaluation [3], for which a variety of file formats are available [4]. The application of this approach significantly reduces both product development time and manufacturing costs [5]. In this study, the rapid prototyping approach is applied to the development of a friction coupling for joining rebar.

Reinforced concrete is the most widely used construction material due to its high compressive strength; however, it exhibits relatively low tensile strength. This limitation is overcome by incorporating rebar, which also improves several other mechanical properties of the material. For easier transportation and handling, rebar is manufactured in standardized lengths, which are often insufficient for practical construction applications requiring longer reinforcing bars. One of the major challenges in civil engineering is to provide a reliable method for joining rebar, particularly for larger diameters, without compromising the structural integrity of reinforced concrete. Accordingly, the objective of this research is the development of a friction coupling for joining rebar in reinforced concrete structures. Couplings are widely used in civil engineering and mechanical structures, including the construction of high-rise buildings, towers, bridges, overpasses, tunnels, dams, and other infrastructure. These couplings are mechanical elements that provide a strong and reliable joint between the components being joined by transferring loads through the friction force generated between the contact surfaces. This force is produced by the geometry of the friction coupling and the installation-induced clamping force.

The primary factor governing the proper selection and application of a friction coupling is the cross-sectional diameter of the rebar, as the friction coupling must be designed to withstand the loads encountered under service conditions. In addition, environmental influences, particularly the difference in the coefficients of thermal expansion of the joined materials, represent an important consideration. A third important factor is the installation process itself, as the performance of the friction coupling depends directly on proper installation.

2. Prototype development

The friction coupling is designed as an assembly consisting of internally toothed jaws specifically adapted to the rebar profile and external tapered sleeves that provide the clamping action.

The rebar sections to be joined pass through the jaws. After the jaws and external tapered sleeves have been assembled, a compressive installation force is applied to produce a permanent joint by satisfying the self-locking condition. To achieve self-locking of the tapered connection, an appropriate selection of material, geometric parameters, and the surface finish of the tapered contact surfaces is required to satisfy the condition:

$$\operatorname{tg} \frac{\alpha}{2} \leq \mu \quad (1)$$

Where $\alpha/2$ is the half cone angle and μ is the coefficient of friction. This operating principle enables the friction coupling to be used with a wide range of rebar diameters and profiles.

The components are assembled on-site after the rebar has been positioned. Once installed, the friction coupling is intended to carry axial tensile loads. The principal performance characteristics of the friction coupling are its tensile load capacity and elongation, which are evaluated through tensile testing.

The initial prototype of the friction coupling for joining rebar is shown in Figure 1.

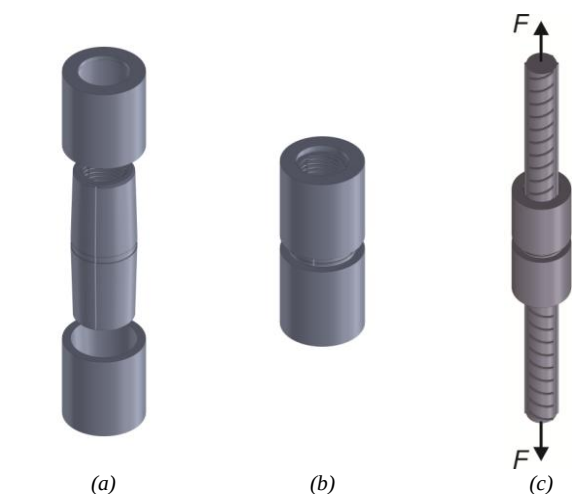


Fig. 1 Friction coupling for joining rebar
(a) Exploded view of the friction coupling; (b) Assembled friction coupling; (c) Friction coupling installed on rebar

The initial prototype was manufactured using the Fused Filament Fabrication (FFF) additive manufacturing process and is shown in Fig. 2.

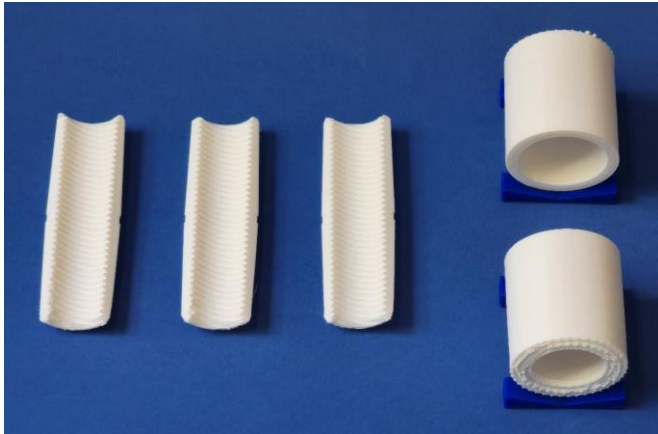


Fig. 2 First iteration of the friction coupling prototype

The main challenge arises at the contact interface between the jaws and the rebar, as the surface of the rebar exhibits complex geometry with a wide range of rib profiles and dimensional tolerances. To address this issue, the jaws of the friction coupling were designed with multi-start, large-pitch helical grooves intended to engage with the ribs of the rebar. In the first design iteration, these helical grooves were unidirectional. However, it was observed that they could not adequately accommodate rebar manufactured in different production batches, primarily because of variations in the orientation of the ribs. Therefore, in the second design iteration, the helical grooves were improved by incorporating overlapping grooves in both directions. The resulting design, shown in Fig. 3, provided significantly better conformity between the friction coupling and the rebar than the first iteration.

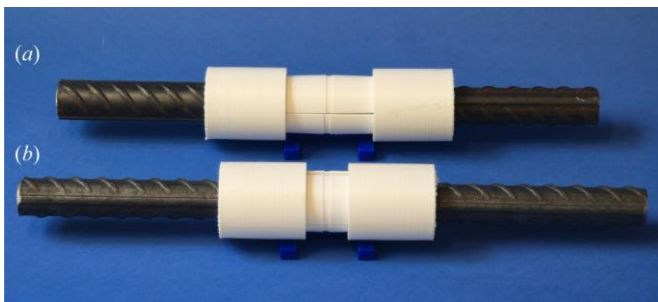


Fig. 3 Comparison of the friction coupling prototype assembly
(a) First iteration; (b) Second iteration

The second challenge was the application of the installation force under on-site conditions. To address this issue, a threaded connection was introduced between the upper and lower external tapered sleeves. This design modification enables the operator to perform the assembly and tightening procedure on-site using standard hand tools in a simple and practical manner. All three iterations of the friction coupling prototype are shown in Fig. 4.

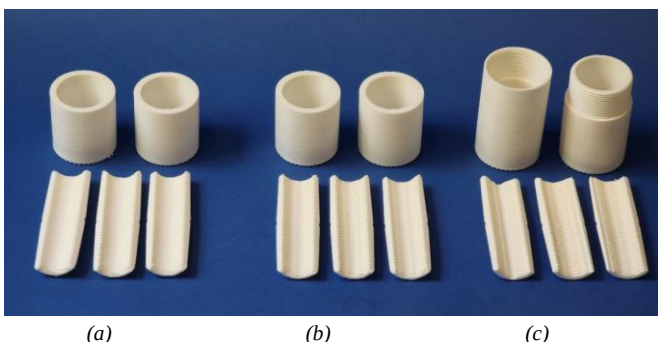


Fig. 4 Components of the three friction coupling prototype iterations
a) First iteration; (b) Second iteration; (c) Third iteration

To generate the preload force in the friction coupling, the threaded connection on the external tapered sleeves must be tightened. In order to achieve the required clamping force of $F_p = 15 \text{ kN}$, a tightening torque T_p must be applied to the specified M45 thread with a 2 mm pitch. The required tightening torque is calculated using the following equation [6]:

$$T_p = F_p \cdot \frac{d_2}{2} \cdot \tan(\varphi + \rho_n) \quad (2)$$

Where: $d_2 = 43.701 \text{ mm}$ is the pitch diameter of the thread, $\varphi = 0.83^\circ$ is the thread helix angle, $\rho_n = 13^\circ$ is the friction angle, and the tightening torque calculated using the equation above $T_p = 80.75 \text{ Nm}$.

3. Numerical simulation of the prototype

The friction coupling prototype developed in the third design iteration and manufactured from modeling materials proved to be operational. Before manufacturing a fully functional prototype from the selected metallic materials using modern CNC machining technologies, it is advisable to evaluate the CAD model in a CAE environment in order to identify potential design deficiencies.

This analysis was carried out in the *Static Structural* module of Ansys, which is based on the Finite Element Method (FEM). Since the ribs on the surface of rebar are manufactured in a wide range of profiles and dimensional tolerances according to ISO 6935-2:2019 [7], and because the interaction between the rebar ribs and the toothed jaws is highly nonlinear, these contact surfaces were approximated as smooth. Furthermore, since the preload is generated through the threaded connection of the external tapered sleeves, the threads were also simplified by representing their effect as an equivalent axial preload force.

The primary operating mechanism of the friction coupling is friction. Therefore, neglecting the interlocking effect between the rebar ribs and the toothed jaws reduces the load-carrying capacity of the coupling, representing a conservative loading scenario that may also occur in practice. The simplified geometry of the prototype is shown in Fig. 5.

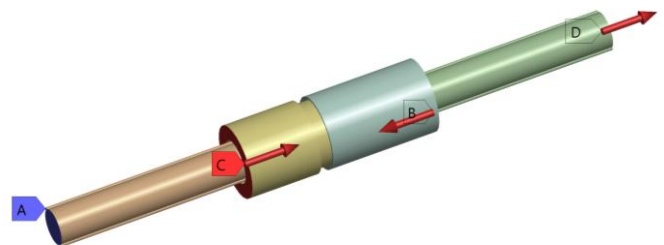


Fig. 5 Simplified geometry of the third prototype iteration with mechanical boundary conditions

One end of the rebar was defined as a *fixed support* and is denoted by A (Fig. 5). The clamping forces responsible for preloading the friction coupling were applied to the surfaces of the external tapered sleeves. These forces, denoted by B and C (Fig. 5), were each assigned a magnitude of 15 kN. To evaluate the performance of the friction coupling, the opposite end of the rebar was subjected to a tensile force, denoted by D (Fig. 5), with a magnitude of 246 kN. This value corresponds to the tensile strength of the selected Ø25 mm rebar multiplied by its cross-sectional area. This load was selected as the test load because a successful friction coupling design should withstand it without significant relative displacement of the rebar within the coupling. Under these conditions, failure is expected to occur by fracture of the rebar rather than by slippage from the friction coupling. The contacts between the surfaces in contact with the rebar were defined as frictional, with a coefficient of friction of 0.5 [8], which is representative of non-machined steel surfaces or surfaces exposed

to corrosion. The relatively high coefficient of friction partially compensates for the neglected interlocking effect between the rebar ribs and the toothed jaws.

To realistically represent the operating principle of the friction coupling, the simulation was divided into two stages. During the first stage, the friction coupling was preloaded by clamping the rebar. Only forces B and C were active, increasing continuously from zero to their final values over a period of 1 s. The stress distribution in the friction coupling at several time instants during the first stage is shown in Fig. 6.

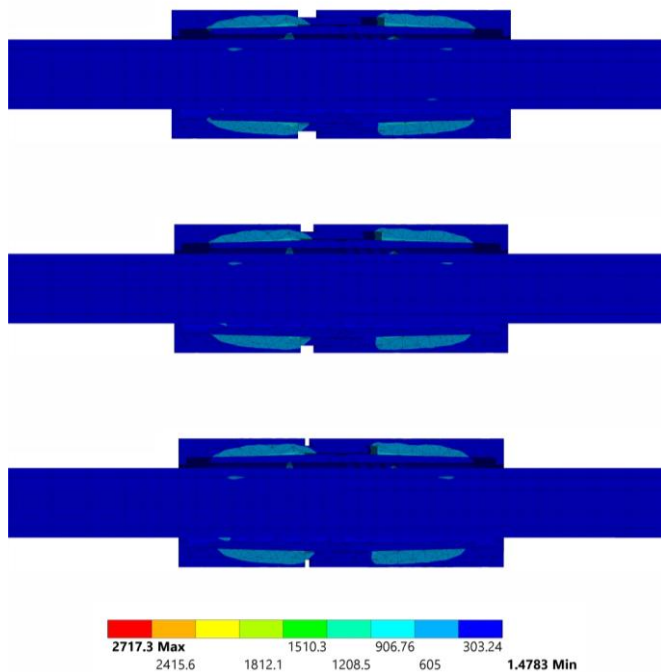


Fig. 6 Stress distribution in the friction coupling (MPa) during the first stage of the simulation at three time instants

The total deformation analysis indicates that the largest displacement occurs in the external sleeves, which is consistent with the intended operating principle of the proposed design (Fig. 7).

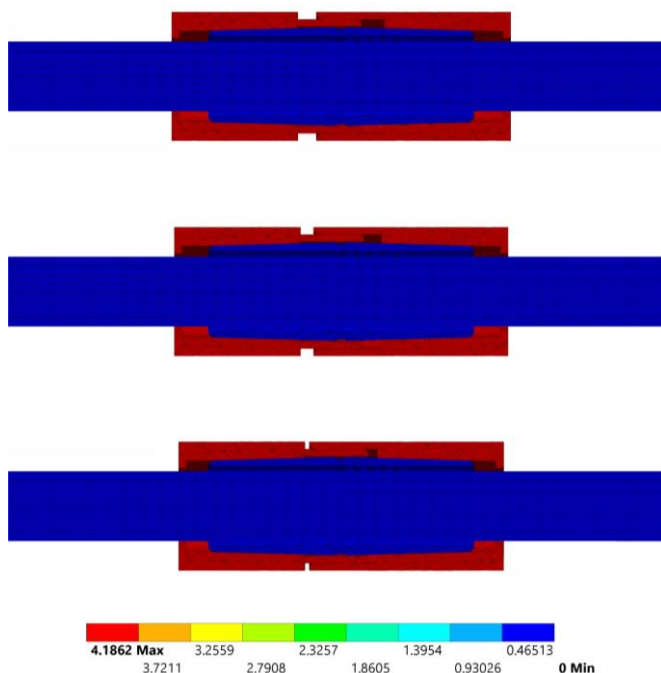


Fig. 7 Total deformation of the friction coupling (mm) during the first stage of the simulation at three time instants

Total deformation represents the magnitude of the displacement of a point (node) from its original position due to the applied loads, boundary conditions, contact interactions, and other influencing factors.

During the second stage of the simulation, forces B and C remained constant at their previously established values, while the tensile force D was applied continuously between 1 s and 2 s, reaching its maximum value at the end of this interval. The stress distribution in the friction coupling at half of the applied tensile load and at the maximum load is shown in Fig. 8.

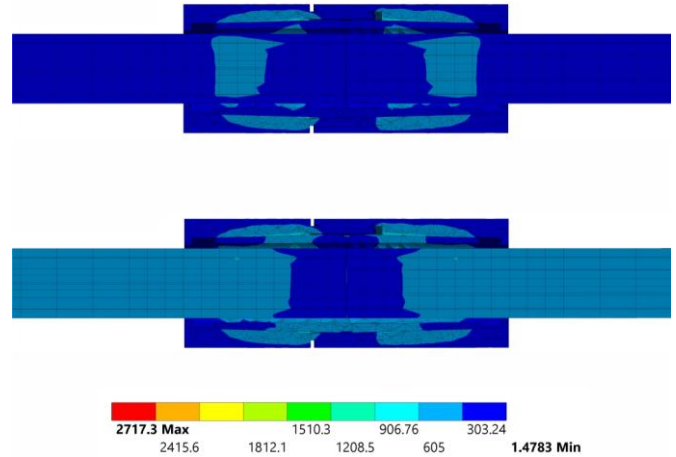


Fig. 8 Stress distribution in the friction coupling (MPa) during the second stage of the simulation at 50% of the applied tensile load and after reaching the maximum tensile load

The total deformation results indicate a slight displacement of the external tapered sleeves caused by elastic deformation, i.e., the elongation of the internal sleeve (Fig. 9).

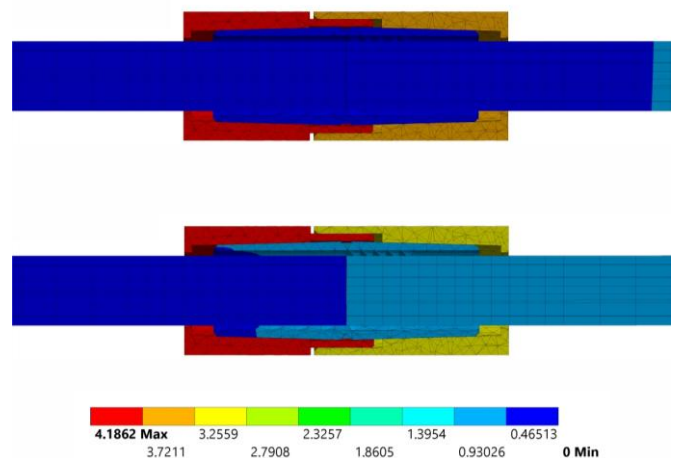


Fig. 9 Total deformation of the friction coupling (mm) during the second stage of the simulation at 50% of the applied tensile load and after reaching the maximum tensile load

To monitor the displacement of the rebar under the applied tensile force D , a *Deformation Probe* was defined on the end surface of the rebar located inside the friction coupling. The results indicate a displacement of 0.61 mm under the action of force D , which is considered acceptable.

4. Conclusion

The developed prototype, based on the design iterations presented in this study, is intended for use in civil engineering applications involving the joining of rebar. The main limitation of the initial design was related to the contact interface between the jaws and the rebar, which was successfully addressed by introducing a double-start helical groove. The second limitation

concerned the practical application of the installation force and was resolved by incorporating a threaded connection between the upper and lower external tapered sleeves. Proper installation is achieved by applying a tightening torque of $T_p = 80.75 \text{ Nm}$.

By applying the Rapid Prototyping Method, all identified design flaws were eliminated within a short development period, and in three iterations operational solution was obtained of the friction coupling. The third prototype iteration was evaluated using FEM analysis. The numerical simulation was performed in two stages. The first stage represented the installation and preloading process, while the second stage simulated the application of tensile load up to the tensile strength of the selected rebar material. Under the maximum applied load, the simulation predicted a relative displacement of the rebar with respect to the friction coupling of 0.61 mm, which is considered acceptable.

The next step of this research will involve manufacturing a fully functional prototype and conducting experimental testing under laboratory conditions. The prototype will be evaluated by determining its tensile strength and percentage elongation as indicators of joint quality. The developed friction coupling should satisfy the required performance criteria by transferring the mechanical properties of the base material, in this case Ø25 mm rebar.

The developed prototype represents a practical and innovative solution for joining rebar in a wide range of civil engineering applications. Its simple installation procedure and minimal influence on the integrity of the reinforced concrete cross-section make it a promising alternative for future construction projects, with significant potential for practical implementation.

Acknowledgement

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Green waste processing facility with parallel energy recovery

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Abstract: Today, renewable bioenergy is regarded as one of the ways to supply energy at the expense of fossil fuels. The possibility of obtaining energy from organic materials through bio-electrochemical systems has attracted considerable interest. Green waste consists of organic plant-based materials generated from grass clippings, broken branches, fallen trees, and others, whose utilization is currently not very effective. In this study, we constructed a depot based on bio-electrochemical systems for the utilization of green waste with cogeneration of energy. The facility demonstrated good performance in terms of both the degradation of organic matter and the energy produced.

Keywords: GREEN WASTE, GREEN ENERGY, WASTE MANAGEMENT, BIO-ELECTROCHEMICAL SYSTEMS

1. Introduction

The increase in the amount of municipal solid waste is a problem faced by almost all countries due to urbanization, the expansion of industrial activities, and global development. In developing countries, a large portion of municipal waste is transported directly to landfills without any intermediate treatment that could reduce the volume of solid waste. Recycling, efficient waste treatment, and source-separated collection are the main strategies for reducing emissions and the environmental impact generated by the increased production of waste [4]. Moreover, solid organic waste is a major issue leading to environmental pollution. The accumulation of large quantities of agricultural waste, animal excrement, and urban garbage are environmental problems that we must face and that are difficult to resolve [17].

Conventional methods for the disposal of organic waste, such as landfilling, composting, and incineration, are limited by low treatment efficiency and insufficient potential for bioenergy recovery. Moreover, these approaches pose risks of secondary pollution, which can potentially lead to adverse effects on soil and air quality [8]. Although the volume of waste can be rapidly reduced through incineration, this approach releases hazardous pollutants, such as fine particulate matter, creating serious risks to air quality. Landfilling, hindered by the poor biodegradability of solid organic waste, not only consumes valuable land resources but also poses a risk of groundwater contamination through leachate infiltration [14]. Furthermore, the large volumes and high transportation costs seriously compromise the economic and environmental feasibility of conventional disposal strategies.

Waste is considered to have a relatively high energy content, which is why waste-to-energy conversion is regarded as a viable alternative. However, conventional composting under aerobic conditions requires more energy for mixing and aeration, and can also lead to the formation of large amounts of leachate. Anaerobic composting, also known as anaerobic digestion, can be an alternative solution for converting solid waste into usable energy and biofuel [1].

The conversion of solid organic waste into energy represents an effective strategy for simultaneously addressing the challenges associated with sustainable energy production and waste management. Transforming organic waste into bioenergy and value-added products can reduce dependence on fossil fuels while mitigating the environmental impact linked to waste landfilling [15]. Furthermore, the recovery of nutrients from solid organic waste can partially offset the high energy demand in the production of nitrogen fertilizers and alleviate dependence on the mining of phosphorus and potassium, whose limited reserves can expose agricultural systems to market instability and geopolitical risks [16].

It has recently been established that microbial fuel cells (MFCs) are an alternative solution for generating electricity from waste without intermediate processing steps, as the anaerobic degradation within them utilizes electroactive microorganisms [2]. Microbial fuel cells (MFCs) use the metabolic activity of electroactive microorganisms to oxidize organic substrates through extracellular

electron transfer, directly converting the chemical energy contained in organic matter into electrical energy [5]. Compared to incineration and landfilling, MFC-based treatment can reduce the risk of secondary pollution, thereby enabling waste valorization through energy recovery [10].

Microbial fuel cells can be classified according to the substrate they utilize [6]. Depending on whether the substrate used is liquid or solid, liquid-phase microbial fuel cells and solid-phase microbial fuel cells are distinguished [7]. The main substance used in solid-phase microbial fuel cells is predominantly plant waste from agriculture and urban organic waste [9]. Solid-phase microbial fuel cells (SMFCs) are one of the innovations in MFC technologies that can be applied to solid organic waste [3]. They are considered to accelerate the process of anaerobic waste degradation, directly generate electrical energy, and produce mature compost from organic compounds.

Based on these considerations, the present work provides a comprehensive assessment of the technical feasibility, environmental performance, and economic sustainability of solid organic waste valorization through solid-phase microbial fuel cells. To this end, a local landfill has been constructed for the processing of green plant waste generated from mowed grass, branches, and leaf biomass, through their direct conversion into electricity and compost. Through suitable engineering solutions, a local landfill has been built, consisting of multiple interconnected solid-phase microbial fuel cells. Previous developments have demonstrated the stable operation of these systems [11, 12], as well as the production of effective compost [13]. In the present publication, an analysis has been made of the operation and energy generated by the landfill, as well as the efficiency of green waste management through the studied systems.

2. Materials and methods

Green waste processing facility

The facility for the utilization of green waste with parallel energy generation consists of two plastic containers, which serve as composters, containing stacked solid-phase microbial fuel cells. The first container has a volume of 360 liters and contains 24 solid-phase microbial fuel cells. The second container has a volume of 600 liters and contains 60 solid-phase microbial fuel cells. Depending on the electrical connection between the individual solid-phase microbial fuel cells, as well as between the two composters, different amounts of energy can be generated from the facility depending on specific needs. The facility will utilize grass clippings, leaves, branches, and other green waste from 1 decare of park area. After analyzing the operation of the facility and the generated energy, optimal planning of the storage and utilization of the electrical energy will be carried out. The resulting compost and liquid soil conditioner will be returned to the park area to nourish and improve the soil characteristics.

Construction of electrogenerative composter 1

The composter 1 is a plastic container with a volume of 360 dm³. The composter consists of 24 PVC pipes arranged vertically.

Each PVC pipe has a diameter of 110 mm and a height of 900 mm. The bottom of the pipes is closed with PVC plugs. At the bottom of the pipes are located stainless steel AISI 304 electrodes with an area of 414.48 cm^2 acting as an anode. At the top of the pipes is a second stainless steel AISI 304 electrode with an area of 414.48 cm^2 acting as a cathode. Copper wires were used as conductors in the electrical circuit. Each of the 24 tubes represents a solid-phase microbial fuel cell (SMFC) (fig. 3). The individual segments (SMFCs) were stacked in the plastic container (fig. 1).

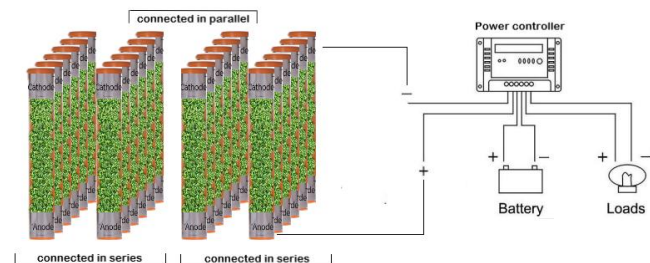


Fig. 1 The schematic diagram of construction of electrogenerative composter 1

Construction of electrogenerative composter 2

Composter 2 is a PVC container with a volume of 600 dm^3 . Inside the composter, 60 PVC pipes are arranged vertically. Each pipe has a diameter of 110 mm and a height of 100 cm. The bottom of the pipes is closed with PVC plugs. At the bottom and top end of each pipe, stainless steel electrodes are placed, serving as the anode and cathode of the solid-phase microbial fuel cell. The area of electrodes is 414.48 cm^2 . Copper wires were used as conductors in the electrical circuit. Each of the 60 pipes represents a solid-phase microbial fuel cell (SMFC). The individual segments (SMFCs) were stacked in the plastic container (fig. 2).

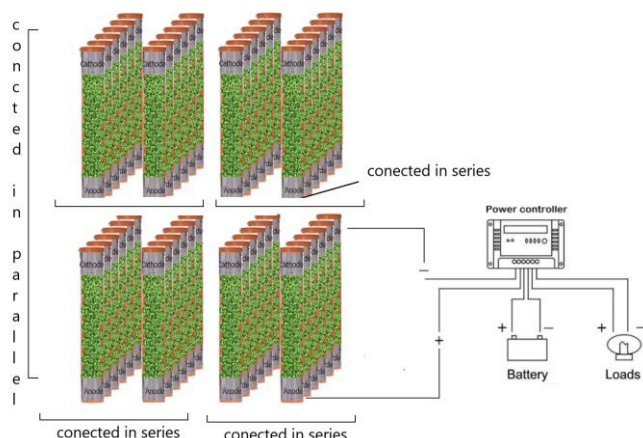


Fig. 2 The schematic diagram of construction of electrogenerative composter 2

After the construction of the two electrogenerative composters, plant biomass was placed inside the pipes and they were filled with water. The plant biomass consists mowed grass with a length of 10 - 15 centimeters. In plant biomass, ancestral species are *Cynodon dactylon*, *Capsella* and *Taraxacum*. The approximate volume of plant biomass in each tube is 8 dm^3 . The physicochemical characteristics of water was pH 7.24, Eh 253 mV and EC $69 \mu\text{S cm}^{-1}$ measured by Vernier LabQuest Mini. On Figure 3 is shown a scheme of single segment (PVC pipe) of the composter.

Electrochemical analysis

After filling with biomass and reaching a baseline voltage of the segments, the electrical parameters of the composters were measured. To determine the operation and effectiveness of the composter polarization and power curves were obtained by varying external resistances sequentially from 10, 8, 6, 4, 2, 1.5, 1.3, 1, 0.7, 0.5, 0.3 to 0.1 K Ω using MCP lab electronics BXR-04 ResistorBox.

The voltage dynamic were measured and recorded in real time with Vernier LabQuest Mini and Logger Lite software.



Fig. 3 Photo of single segment of the composter

3. Results and discussion

The electrical parameters of composter 1 were measured presented on figure 4.

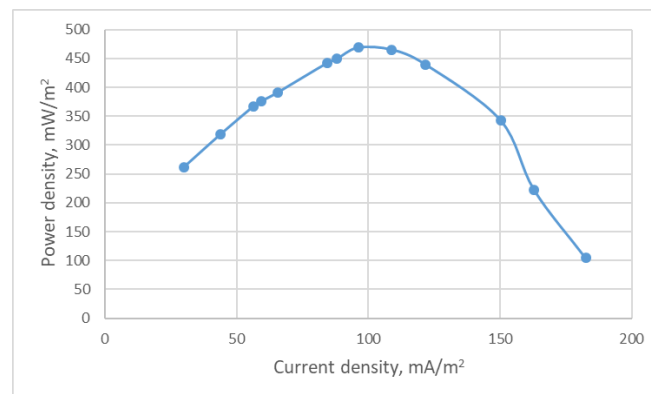


Fig. 4 Power density dynamics of composter 1

From the research conducted, it was found that the composter has the best electrical parameters when connecting the 12x12 segments in series and connecting the two pairs in parallel. With this connection the output voltage of the composter varies between 8.5 and 9 volts. A maximum power density of 474.6 mW/m^2 was recorded at a current density of 97.4 mA/m^2 and an applied resistance of 1.1 K Ω .

In order to ensure approximately equal output voltage from the two composters and to avoid 'discharging' of one of them when connected in parallel, an electrical connection configuration of 4x15 segments connected in series and parallel connection of the four groups was chosen. With this electrical connection, an output voltage of 8–8.5 volts and a maximum current of 8.5 mA were achieved.

The results from the measurements of the power density are presented in the figure 5.

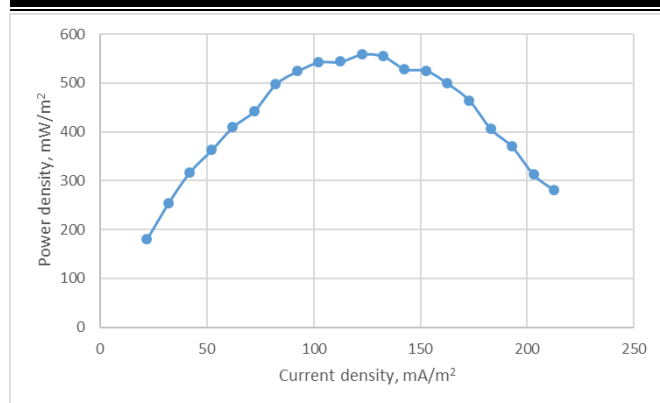


Fig. 5 Power density dynamics of composter 1

From the presented graph, it can be seen that with this connection of the individual segments, a maximum power density of 558.43 mW/m² is achieved, at a current density of 122.46 mA/m².

The constructed composters demonstrate high efficiency in the utilization of green waste. Depending on the method of utilizing the generated energy, the two composters can operate separately or connected in series or in parallel.

It is recommended to connect a Power Management System to the system, consisting of a supercapacitor and a DC-DC boost converter, which will allow increasing the voltage and stabilizing the electrical parameters of the facility. It is also possible to include a rechargeable battery with suitable parameters for storing the energy, if it is not used in a timely manner.

4. Conclusion

The development of renewable energy technologies, as well as the problems related to waste management, are key challenges for modern, highly industrialized and urbanized societies. It appears that the technology of solid-phase microbial fuel cells can successfully contribute to solving both of these problems, but steps towards specific applied developments are necessary to validate the technology. The facility for processing green waste with parallel energy generation proposed in this work is an engineering example of how the technology can be applied in practice. The constructed composters demonstrated high efficiency both in the utilization of green waste and in energy generation. Through a suitable Power Management System, the generated energy can be used in an optimal way and reduce dependence on conventional sources.

Acknowledgements

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Investigation of the effect of overcharging on the indicator diagram and heat release coefficient of a gasoline engine

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Abstract: Overcharging is one of the most effective methods for increasing the power output and improving the energy efficiency of spark-ignition internal combustion engines. It has a significant influence on the processes of mixture formation, combustion, and heat release, which necessitates a detailed experimental investigation of the engine working process. This paper presents an experimental study on the influence of supercharging on the indicator diagram and the heat release coefficient of a VW-1600 GTI spark-ignition engine equipped with a K-Jetronic mechanical fuel injection system. The appropriate operating modes, defined by engine speed and load, were selected to ensure reliable measurement and comparison of the investigated parameters under naturally aspirated and supercharged operating conditions.

Keywords: OVERCHARGING, GASOLINE ENGINE, HEAT RELEASE COEFFICIENT

1. Introduction

The overcharging (boosting) is widely used in modern gasoline engines to increase the air density and improve power output. By raising the pressure and temperature of the fresh charge, boosting modifies the in cylinder thermodynamic cycle and directly affects the indicator diagram, the pressure rise rate, and the heat release characteristics. These changes influence combustion efficiency, and knock tendency. Previous studies report that elevated pressure accelerates the combustion process and increases the rate of heat release, while also altering the shape of the pressure curve [1-5].

In this context, the present study investigates the effect of overcharging on the indicator diagram and the heat release coefficient of a gasoline engine operating under nominal conditions. By comparing pressure traces, pressure rise rates, and heat release characteristics with and without boost, the work aims to provide deeper insight into the thermodynamic and combustion related consequences of overcharging. The obtained results contribute to a better working of gasoline engine with overcharging.

2. Results and Analysis

The experimental investigations were carried out on a gasoline engine VW 1600 GTI mounted on testing workbench No. 8 in Block 9 at the Technical University of Sofia.

In spark-ignition internal combustion engines (ICEs), the homogeneous air–fuel mixture, compressed to a pressure of 1.5–2.0 MPa and heated to a temperature of 700–800 K, is ignited by an electric discharge between the electrodes of the spark plug. Ignition occurs at a predefined crankshaft angle before top dead center (TDC), known as the ignition advance angle θ . The initial flame kernel heats the adjacent layers of the mixture, forming a flame front that propagates at high speed through the combustion chamber, causing a rapid rise in pressure and temperature.

The development of the combustion process in the cylinder of an ICE is most accurately described by an indicator diagram in the p – φ coordinate system, which represents the variation of the in-cylinder pressure p in the clearance volume as a function of the crankshaft rotation angle φ (CA) (Fig. 1). The same figure also shows the variations of the pressure rise rate $dp/d\varphi = f(\varphi)$ and the gas temperature in the combustion chamber $T = f(\varphi)$. The evolution of the combustion process in the cylinder of an ICE is best illustrated by the indicator diagram in the p – φ coordinate system, i.e., the variation of the in-cylinder pressure p in the clearance volume as a function of the crankshaft rotation angle φ (CA), the so-called expanded indicator diagram (Fig. 1). The same figure also presents the changes in the pressure rise rate $dp/d\varphi = f(\varphi)$ and the gas temperature in the combustion chamber $T = f(\varphi)$.

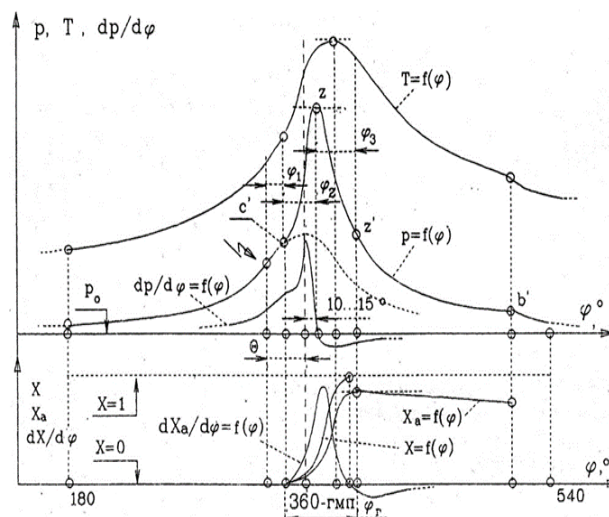


Fig.1. Dependence of the combustion process parameters of a gasoline engine on the crankshaft rotation angle φ (without overcharging).

The combustion process in gasoline spark-ignition engines is conventionally divided into three periods: (1) the ignition delay period; (2) the rapid combustion period; (3) the after-burning period.

The third period, which begins after the maximum pressure $p_{\max}$ is reached, is of particular interest. It is characterized by a significant slowdown of the combustion process, caused by the reduced flame-front propagation speed in the regions near the walls of the expanding clearance volume. This is due to increased heat transfer and reduced turbulence of the air–fuel mixture.

During this period, heat release continues, leading to an additional rise in the gas temperature. At full load, the maximum temperature reaches approximately $T_{\max} \approx 2800$ – 3100 K. For gasoline SI engines, the temperature peak occurs 10–20°CA after the pressure peak. After that, the temperature decreases sharply due to the cessation of heat release and the rapid increase in the clearance volume during expansion. In the third period, approximately 15–20% of the total heat released during combustion is produced.

The end of the third period (point z' , Fig. 1) can be determined using the law of total heat release:

$$(1) \quad X = Q_{\varphi} / Q_{\text{rui}} = f(\varphi),$$

where: X is the total heat release coefficient; $Q_{\text{cycle}} = G_{\text{cycle}} H_d$ is the amount of heat released during the combustion of the entire cycle fuel mass G_{cycle} ; Q_{φ} is the amount of heat released from the beginning of combustion up to a given crank angle φ , part of which is used to increase the internal energy of the working fluid ΔU and to perform useful work L , while the remainder is lost due to heat transfer.

The law of total heat release $X = f(\varphi)$ can be determined using a mathematical model based on the laws of mechanics and thermodynamics, using data from an actual indicator diagram. When $X \approx 1$ ($0.98 \div 0.99$), i.e. $Q_\varphi \approx Q_{\text{cycle}}$, the combustion process is considered completed.

The analysis can also be performed using the law of active heat release $X_a = f(\varphi)$, which does not require analytical accounting of heat losses. It is defined as:

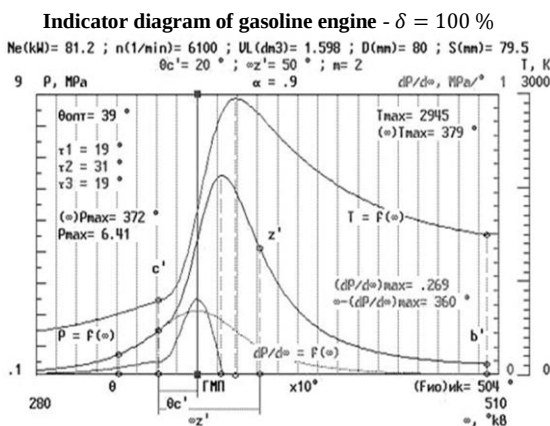
$$(2) \quad X = (Q_a)_\varphi / Q_{\text{cycle}} = \frac{\Delta U_{c'-\varphi} + \int_{c'}^{\varphi} p \cdot dV}{Q_{\text{cycle}}}$$

The maximum value of the active heat-release coefficient in gasoline engines is $X_{a \max} \approx 0.8 - 0.85$. The end of the combustion process (point z') can be approximately determined by the position of $X_{a \max}$. The rate of increase of the active heat-release coefficient $dX_a/d\varphi = f(\varphi)$ and the duration of the combustion process φ characterize the dynamics of heat release in the cylinder. The rate at which pressure increases $dp/d\varphi$ (MPa/CA) depends on the heat-release rate. The maximum pressure rise rate $(dp/d\varphi)_{\max}$ determines the dynamic loading of the crank mechanism components and is a criterion for the so-called "hardness" of engine operation. For gasoline engines, $(dp/d\varphi)_{\max} \approx 0.2 - 0.3$ MPa/CA, and they operate more smoothly compared to diesel engines [6].

In accordance with the research objective, numerical modeling was performed to study the influence of boost pressure p_k on the main parameters of the combustion process in a spark-ignition gasoline engine. The following quantities were analyzed: pressure p , temperature T , active heat-release coefficient X_a , and the rates of increase of pressure $dp/d\varphi$, temperature $dT/d\varphi$, and active heat-release coefficient $dX_a/d\varphi$ — for a gasoline engine prototype VW 1600 GTI.

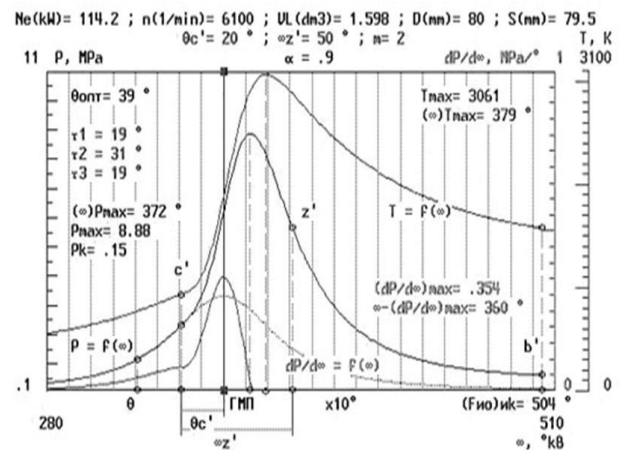
2.1. Results of the numerical investigation

In accordance with the stated objective, a numerical study was carried out to examine the influence of boost pressure p_k on the parameters of the combustion process — pressure p , temperature T , active heat-release coefficient X_a , and the rates of increase of pressure $dp/d\varphi$, temperature $dT/d\varphi$, and active heat-release coefficient $dX_a/d\varphi$ — for a VW-1600 GTI prototype engine. A specialized software product developed by [6] was used. A portion of the obtained results is shown in Fig. 1 to Fig. 4, and Table 1 presents some of their more characteristic values.



a) without overcharging

Indicator diagram of gasoline engine - $\delta = 100\%$



b) overcharging and $p_k = 0.15$ MPa pressure after compressor

Fig.2. Variation of cylinder pressure p , pressure rise rate $dp/d\varphi$, and temperature T during engine operation under nominal conditions, with and without boost, as a function of the crankshaft angle φ .

Heat release dynamics in a gasoline engine - $\delta = 100\%$
(X -heat release coefficient)

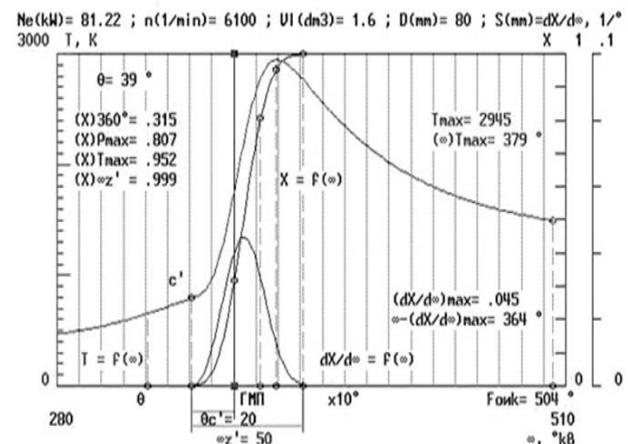


Fig.3. Shows the variation of the coefficient of total heat release X , the rate of its increase $dX/d\varphi$, and the temperature T in the cylinder during engine operation at nominal mode, as a function of the crankshaft rotation angle φ .

Table 1. The calculated performance parameters of the gasoline engine — VW-1600 GTI prototype — at $n = 6100 \text{ min}^{-1}$ and full load ($\delta = 100\%$).

Parameters	Without overcharging	Overcharging
$N_e^{\max}$, kW	81,2	114,2
G_B , kg/h	280	373
G_r , kg/h	20,8	27,7
g_e , g/kW.h	256	242
$p^{\max}$, MPa	6,41	8,88
$T^{\max}$, K	2945	3061

Heat release dynamics in a gasoline engine - $\delta = 100\%$ (X_a – active heat release coefficient)

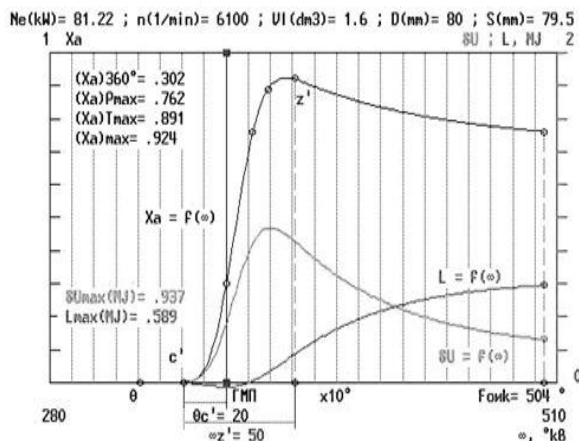


Fig.4. Presents the variation of the active heat-release coefficient X_a , the rate of its increase $dX_a/d\phi$, the indicated work L , and the change in internal energy δU in the cylinder during engine operation at nominal mode, again as a function of the crankshaft rotation angle ϕ .

The presented experimental results and tabulated data can be summarized as follows:

1. Power increase ($N_e^{\max}$)

Increasing the boost increases maximum power from 81.2 kW $\rightarrow$ 114.2 kW, which is $\approx 40\%$. This is typical for supercharging at $p_k \approx 0.15$ MPa, as higher air density allows more fuel to burn efficiently.

2. Air and fuel mass flow (G_B , G_r)

Air flow increases from 280 $\rightarrow$ 373 kg/h (+33%);

Fuel flow increases from 20.8 $\rightarrow$ 27.7 kg/h (+33%);

The proportional increase shows that the mixture composition remains controlled and combustion stays stable.

3. Specific fuel consumption (g_e)

Specific fuel consumption improves from 256 $\rightarrow$ 242 g/kW·h. This means the boosted engine becomes more efficient, producing more power per unit of fuel.

4. Maximum pressure ($p^{\max}$)

Cylinder pressure rises significantly: 6.41 $\rightarrow$ 8.88 MPa (+38%). Higher pressure increases mechanical stress on the crank mechanism and piston group — a key factor in engine durability.

5. Maximum temperature ($T^{\max}$)

Temperature increases from 2945 $\rightarrow$ 3061 K (+4%). The rise is moderate compared to pressure, indicating that the combustion process remains thermodynamically stable.

A portion of the numerical- simulation results obtained by Program Soft, which will be compared with the experimental results, is shown in Fig.4 and Table 2 presents some of their more characteristic values.

Table 2. The calculated parameters of the gasoline – $iVh = 1.588$ dm³ at $n = 3000$ min⁻¹ and full load ($\delta = 100\%$).

Parameters	Without overcharging	Overcharging $p_k = 0.16$ MPa
$N_e^{\max}$, kW	50,1	63,7
G_B , kg/h	161	208
G_r , kg/h	12	15,5
g_e , g/kW.h	239	243
$p^{\max}$, MPa	7,3	9,49
$T^{\max}$, K	2893	2900

The short technical analysis of the Fig. 4 and Table 2 showed:

1. Power Output ($N_e^{\max}$)

The power increase by boosting from 50.1 to 63.7 kW is $\approx 27\%$ at 3000 rpm. This confirms that even at medium speeds, the engine is using the increased air density.

2. Air and Fuel Mass Flow (G_B , G_r)

Air flow increases from 161 $\rightarrow$ 208 kg/h (+29%);

Fuel flow increases from 12.0 $\rightarrow$ 15.5 kg/h (+29%);

The proportional increase indicates stable mixture formation and proper fuel–air ratio control under boost.

3. Maximum Pressure ($p^{\max}$)

Cylinder pressure rises sharply: 7.30 $\rightarrow$ 9.49 MPa (+30%).

This is a major indicator of increased mechanical loading to piston crown, connecting rod and crankshaft bearings. Maximum temperature ($T^{\max}$) and specific fuel consumption (g_e) change slightly.

3.2. Comparison Between Numerical and Experimental Results

The numerical simulation results show trends that closely match the experimental observations for the VW-1600 GTI spark-ignition engine. Both datasets confirm the same fundamental effects of boost pressure on the combustion process:

- increased power output,
- higher maximum cylinder pressure,
- slightly increased maximum temperature,
- intensified heat-release dynamics,
- higher air and fuel mass flow rates.

This indicates that the mathematical model captures the essential thermodynamic and mechanical behavior of the engine under boosted operation.

3. Conclusion

- The numerical model accurately predicts the main combustion characteristics of the VW-1600 GTI engine under boosted and naturally aspirated operation.
- Quantitative differences are small and arise from real mechanical, thermal, and flow losses not fully captured by the model.
- All key trends — power increase, pressure rise, heat-release intensification — are identical in both numerical and experimental datasets.
- The model is therefore suitable for predictive analysis, optimization of boost pressure, and evaluation of combustion dynamics in spark-ignition engines.

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Furnace for the electroslag remelting of non-metallic household waste

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Abstract: The shortcomings of existing waste-processing furnaces have been analyzed, and a new furnace design for electroslag remelting and combustion of waste, developed at Akaki Tsereteli State University, is proposed. It is demonstrated that its use enables the combustion and remelting of individual waste components without causing environmental pollution from combustion products.

Keywords: waste recycling; electroslag remelting furnace; crystallizer, non-metallic materials, combustion.

1. INTRODUCTION

The processing of household, medical, and other non-metallic industrial waste is a universally recognized problem worldwide. Solving this problem inherently involves resolving two important questions: 1. Determining the scale of the problem solution; 2. Selecting the appropriate technical means, specifically, a furnace suited to the scale of waste thermal processing, or developing a better new one[1].

When determining the scale of the problem, the key factor is the volume of waste to be recycled across the entire country, in two or more regions simultaneously, or in an individual city. It is understood that the construction of factories needed to recycle certain valuable waste must be profitable, since only in this case is it possible to attract investment to address the problem. However, in this case, the population's readiness to sort household waste before disposing of it in bins is also crucial. Otherwise, it is advisable to process household waste at the city or municipal level without sorting, to prevent environmental littering. In parallel, various supportive measures will be needed to cultivate a public demand for waste sorting. In this case, it would be possible to establish a unified waste processing service on a national scale, which would reduce waste processing costs and make it feasible to create enterprises for the beneficial use of waste, for example, factories for processing paper, plastic, and other types of waste, making it profitable while simultaneously solving the problem of environmental pollution from waste.

2. METHODOLOGY

For waste thermal processing, various furnaces can be used based on currently available thermal energy sources. Among these, the most widely used are furnaces powered by liquid fuels, combustible gases, and so-called plasma furnaces.

In furnaces powered by liquid fuels and combustible gases, waste combustion is prolonged; moreover, the gases generated during the process contribute to environmental pollution. The so-called combustion residues are also significant, while the furnaces themselves have a low efficiency coefficient (less than 40%). Plasma furnaces for waste processing have a comparatively higher efficiency coefficient. However, environmental pollution from gases emitted during combustion is significant. Additionally, radiation-related environmental pollution associated with the plasma process is considerable [2]. The protective systems required to mitigate gas and radiation emissions, together with the design of the thermal energy source itself - the plasmatron - complicate plasma furnace construction and contribute to high capital costs. Operating and maintenance costs are likewise high, primarily due to the frequent replacement of plasmatron electrodes.

Within the framework of grant AR-25-2056 funded by the Shota Rustaveli National Science Foundation, we propose a new, original-design furnace for the combustion and remelting of non-metallic materials, specifically household, industrial, and medical waste, known as an electroslag remelting furnace[3]. Figure 1 illustrates the structural diagram of a variant of the so-called crystallizer, which is the section of the furnace where direct waste combustion occurs.

Its design is based on an electroslag remelting furnace for metals, consisting of a power supply, a control system, and a crystallizer. The latter is a key component in which electroslag remelting and metal crystallization occur, enabling the production of an ingot with the desired structure.

The crystallizer is also the main component of the proposed waste-processing furnace, where only melting occurs, without crystallization. However, the old name "crystallizer" has been retained in the presented furnace as well.

When current leads 9 and 10 are connected to the slag bath 11 and the power supply, the bath temperature is maintained by Joule heating resulting from the passage of current 12 through the bath. The magnitude of the temperature depends on the composition of the slag. When the slag contains only feldspar CaF_2 , the temperature of the slag bath is in the vicinity of 900°C . However, with the addition of quicklime CaO , the temperature can reach 1800°C .

3. RESULTS AND DISCUSSION

For creating an experimental waste remelting furnace, it became necessary to calculate the current required for electroslag remelting in order to select a power source for its electrical energy supply.

An analysis of the literature [5] shows that, in addition to the required capacity, one of the main requirements is that the power source should have a steep volt-ampere characteristic, while its open-circuit voltage should not exceed 40 V [6].

If the electrical resistance of the slag in the liquid state is known, then the current required for melting can be determined based on the heat released per unit [of volume/time] as the current passes through the slag bath.

$$Q_1 = 0,24I^2R \text{ kcal}, \quad (1)$$

$$\text{from which } I = \sqrt{\frac{Q}{0,24 R}} \quad (2)$$

On the other hand, if the heat capacity C , mass m , and the melting and superheating temperature T_2 of the slag bath are known, taking into account the ambient temperature T_1 , the heat required to melt a mass m of slag can be calculated [7]:

$$Q_2 = cm(T_2 - T_1) \quad (3)$$

Other factors being equal, it is implied that

$$Q_1 = Q_2 = Q \quad (4)$$

Taking into account the height h of the slag bath, the inner diameter d of the crystallizer, and the density γ_{sl} of the molten slag, expression (3) takes the form:

$$Q = \pi \frac{d^2}{4} \cdot h_{sl} \cdot \gamma_{sl} \cdot T_m \quad (5)$$

Due to the smallness of the ambient temperature T_1 compared to the melting temperature T_2 of the slag $T_2 \gg T_1$, if we neglect T_1 in expression (3), then the so-called slag-remelting current passing through the slag bath is:

$$I = \sqrt{\frac{\pi d^2 / 4 \cdot h_{sl} \gamma_{sl} C_p T_{\infty}}{0,24 R}} \quad (6)$$

Based on reference material [9], sources on the electroslag remelting of metals, as well as taking into account experimental data [4], the following data were obtained

$$d_{cr} = 25,4 \text{ cm}; \quad h_{sl} = 25,4 \text{ cm} \quad C_{sl} = 1,72 \text{ kJ/kg}; \\ \gamma_{sl} = 2700 \text{ kg/m}^3; \quad T_2 = 1773^\circ\text{K}$$

According to expression (6), the value of the calculated current is $I_{sl} \approx 300 \text{ A}$.

Taking the latter into account, for the power supply of the electroslag remelting installation for non-metallic materials, the constant-current power source VKSM-1000 [7,8] was selected,

which is used for electric arc welding, has a steep volt-ampere characteristic, and an open-circuit voltage of up to 40 V.

In the experiment conducted to evaluate the character of the combustion or slag-melting capability of waste of various compositions, a slag with a composition of 80% CaF₂ + 20% CaO was used, and the temperature of the slag bath, which we measured by means of a pyrometer, was 1200°C. The experimental data are presented in Table 1. The degree of waste combustion was assessed by observing the waste during the combustion process and by recording the combustion process. In the experiment, the criteria for evaluating the quality of the combustion process were: the nature of waste destruction, particularly whether combustion occurs on the surface of the slag or instantaneous melting into the slag occurs, and the amount of combustion products (gases) released as a result of combustion.

The specific gravity of the slag bath in the crystallizer, as well as its viscosity, is directly proportional to the bath temperature[4]. At 1200 °C, the viscosity of the slag is practically equal to that of water. Therefore, the conditions for submerging waste in it are similar to those for submerging waste in water. This is what causes the waste to sink and melt instantly in the slag.

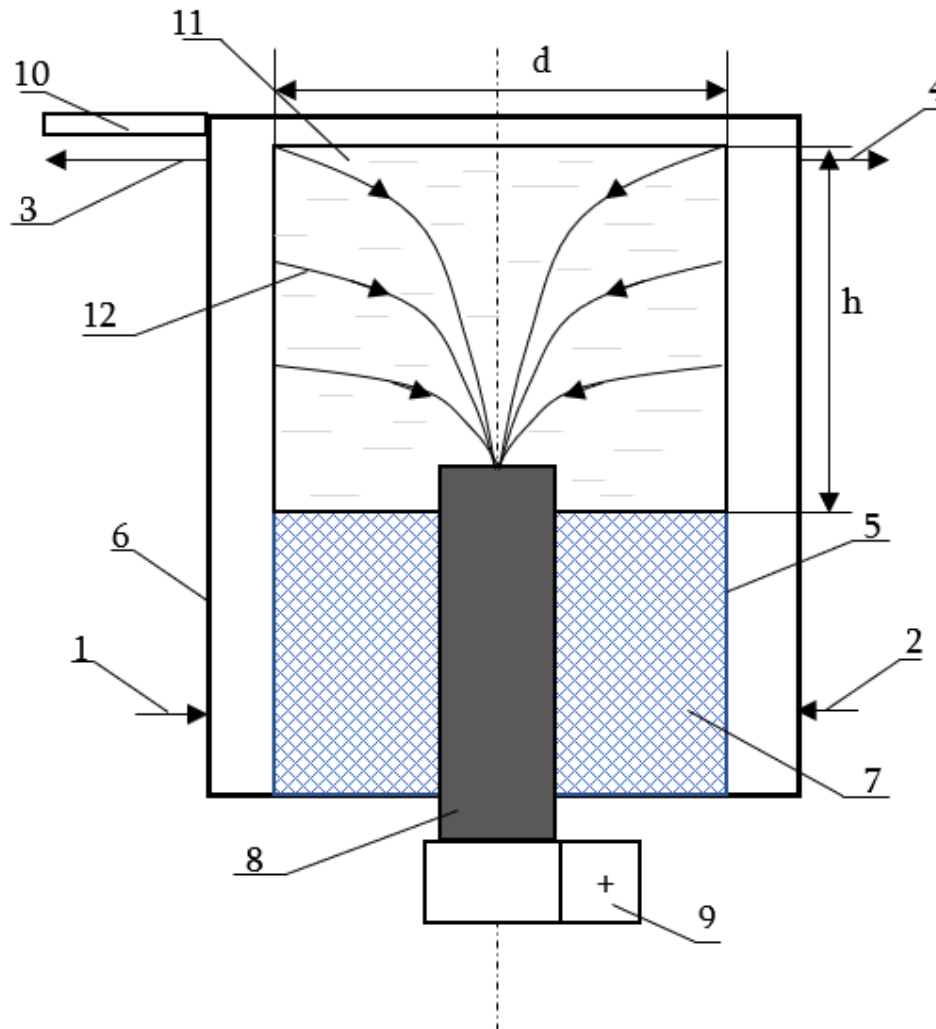


Fig.1. Structural diagram of the so-called crystallizer of the electroslag remelting furnace

1, 2 - water supply points for cooling; 3, 4 - water extraction points for cooling; 5 - inner cylindrical surface of the crystallizer; 6 - outer surface of the crystallizer; 7 - insulating and heat-resistant base; 8 - current lead of the graphite electrode; 10 - current lead to the crystallizer; 11 - molten slag bath; 12 - equivalent lines of current passing through the slag; d - diameter of the crystallizer; h - height of the slag.

According to the above table, paper existing as a separate type of waste is an exception. However, the latter, when mixed with other waste, easily sinks and instantly dissolves in the slag bath. That is, their destruction also occurs practically without residue. The separated residues from the remelting process make a minor but continuous contribution to the increase in slag bath height. They primarily occupy the upper part of the slag bath and gradually overflow out of the crystallizer

By changing the composition of the slag, it became possible, owing to the corresponding change in the resistance of the molten slag, to vary the open-circuit voltage of the power source of the electroslag remelting furnace for non-metallic materials by 40 ± 5 V, which ensured a change in current during the remelting process of up to 300 ± 10 amperes, and, correspondingly, a change in the slag bath temperature within the required range of $1773^\circ\text{K} \pm 100^\circ\text{K}$.

Table 1.: Characteristics of the waste combustion and melting process

No	Type of waste	Characteristics of the waste combustion and melting process	Note
1	Vegetables: potatoes, tomatoes, watermelons, eggplants, melons, cucumbers, beans	Instantly melted, without the release of combustion products	Mainly in small separate portions in the form of shavings
2	Paper	Initially, with combustion, accompanied by the release of a correspondingly small quantity of combustion products, after which it melted. At this point, the release of combustion products ceased completely	Crumpled
3	Plastic		In a compressed state
4	Baby hygiene diaper	Instantly melted, without the release of combustion gases	In a used state
5	Paper mixed with other wastes	Combustion with instantaneous melting, with minimal release of combustion products	With vegetable wastes
6	Plastic mixed with other wastes	Instantaneous melting, with minimal release of combustion products	With vegetable wastes

4. CONCLUSIONS

1. The proposed waste recycling furnace, as can be seen from the characteristics of the remelting process, is more promising than other types of furnaces currently used for the same purpose, including plasma furnaces.

2. In the furnace we proposed, the melting of waste is possible without combustion, which practically eliminates environmental pollution by combustion gases.

3. According to the method for calculating the current required for the electroslag remelting of non-metallic materials, the selected power source of the furnace ensures the required magnitude of the slag bath temperature and the required range of regulation

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Possibility of creating an increased-capacity gas turbine generator

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Abstract. Three different types of combustion chambers for a gas turbine generator are discussed. The advantage of a hybrid gas turbine generator with one main and four co-cell combustion chambers is presented. The data from the experiment to be conducted on it are analyzed, and the issues of selecting an electric generator for a gas turbine generator using a simplified method are shown.

Keywords: GAS TURBINE; GENERATOR; CELLS; COMBUSTION CHAMBER; PROPANE.

1. Introduction

A gas turbine generator is a heat-and-power installation in which fuel gases serve as the primary source for electricity generation. It consists of the following main components: pulse-action combustion chambers, a gas turbine, and an electric generator.

The use of pulse-action combustion chambers enables a simpler gas turbine generator design.

In gas turbine generators, the combustion chamber is the primary component under study, and research should focus on maximizing the force exerted on the gas turbine blades by the flow of combustion gases produced in the combustion chamber.

Numerous studies and patents [1], [2] have been devoted to the development of gas turbine generators for various applications to increase their efficiency and power output. Based on an analysis of these works and our own research, we have developed three combustion chamber designs that differ in both construction and operating principle.

The first design (Fig. 1) is characterized by its simplicity. It consists of a cylindrical housing with a base and a lid. A spark igniter for initiating combustion of the fuel-air mixture is mounted in the base. Its operating principle is as follows. After the combustion chamber is filled with the fuel-air mixture, the lid is opened simultaneously with ignition by the spark igniter. The resulting high-pressure combustion gases are directed toward the gas turbine blades, where they generate the torque required to drive both the turbine and the electric generator mounted on its shaft. The coordinated operation of multiple combustion chambers acting on the turbine blades maintains the generator rotor at its nominal operating speed, thereby enabling continuous electricity generation.

The second design (Fig. 2) is more complex and consists of a combustion chamber with outer and inner cells. It can operate with three gaseous fuels having different combustion characteristics, such as propane, methane, and acetylene, which may be supplied individually, in pairs, or simultaneously as the energy source for the gas turbine generator [3]. Each fuel can be ignited independently, or one fuel can be ignited first, followed by sequential or simultaneous ignition of the remaining fuels.

The third and most advanced design is a combustion chamber with one main cell and four additional cells [3]. In this configuration, the combustion gas flows generated sequentially in the main cell and the four additional cells reinforce one another, thereby increasing the power output of the gas turbine generator. The power output can be further enhanced by operating the gas turbine generator in a novel water-cooling mode.

In the hybrid operating mode, in addition to the fuel gas supplied to each cell, a pressurized water jet is injected into the main cell. The combined action of the combustion gases and the water jet on the turbine blades increases the force acting on the blades, thereby increasing the power output of the gas turbine generator.

2. Conditions for conducting the experiment

Propane was used as the fuel gas in the experiment. In the new operating mode, a water jet was used together with propane in the gas turbine generator. It was directed towards the gas turbine blades from the main cell of the combustion chamber. Simultaneously, the remaining cells were being filled with a propane-air mixture and ignited. After the cells were filled with fuel gas and before ignition, the water jet supply was cut off, along with the fuel gas supply. The water jet and the flue gas jets produced by the explosion in the cells struck the gas turbine blade almost simultaneously.

Experiments were also conducted in the same multi-chamber combustion chamber using propane fuel without water-jet injection. In this experiment, the combustion chamber was supplied with a pre-dosed propane-air mixture.

The force caused by the simultaneous action of the combustion gas and water jets, produced by the explosion of the fuel gas, on the gas turbine blade was measured using an inductive sensor [4].

Table 1.

#	Propane content in its mixture with air, %	Force acting per unit area, depending on the type of combustion chamber			
		With a single cell (kgf)	Double-cell with a common wall (kgf)	With one main cell and four additional cells (kgf)	Propane and water, when supplied in combination (kgf)
1	5	5,0	9,0	20	30
2	8	7,0	13,0	29	41
3	12	3,5	8,0	15	27
4	15	2,0	5,0	9,0	18
5	18	1,0	2,0	7,5	8,5
6	20	0,5	1,0	3,0	5,5

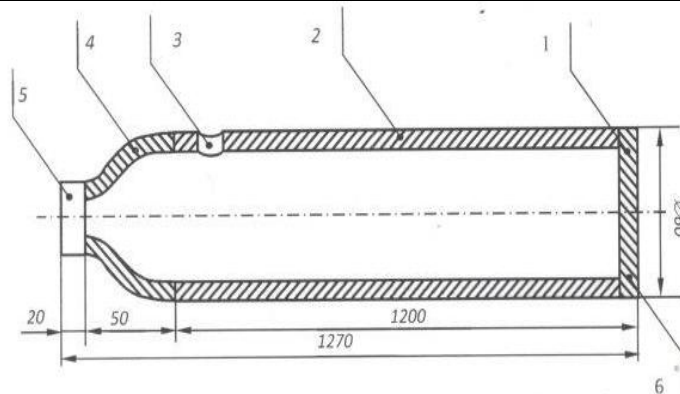


Fig.1. Layout diagram of four combustion chambers relative to the gas turbine blades

1 – combustion chamber; 2 – exhaust gas flow; 3 – gas turbine blade; 4 – gas turbine.

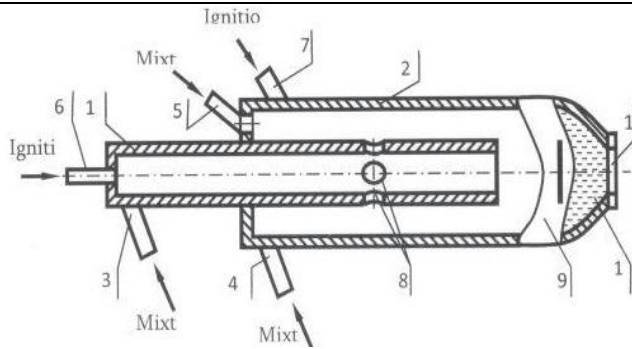


Fig. 2. Combustion chamber with cells

1 – internal combustion cell; 2 – external combustion cell; 3, 4, 5 – mixture supply valve; 6, 7 – ignition valve; 8 – chamber connecting valves; 9 – nozzle; 10 – lid; 11 – water plug.

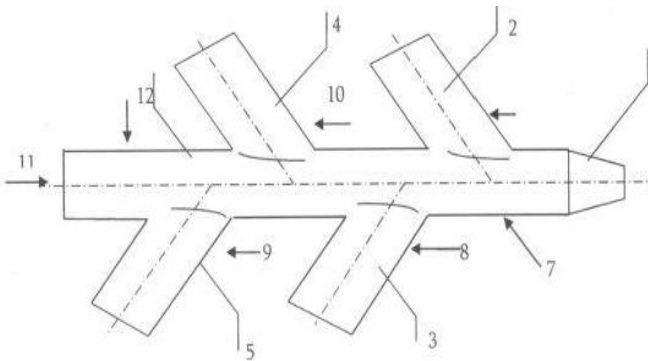


Fig. 3. Scheme of a combustion chamber with one main and four auxiliary cells.

1 – main cell; 2, 3, 4, 5 – auxiliary cells; 6 – nozzle; 7, 8, 9, 10 – fuel gas inlet valves, 11 – water jet inlet valves, 12 – spark supply valve, 13 – gas turbine valve

3. Calculations and analysis of experiments

The experimental data presented in Table 1 for different combustion chambers clearly demonstrate the advantage of the so-called combustion chamber with one main and four auxiliary cells. In this case, the maximum value of the force acting on the gas turbine blade reaches 41 kgf, whereas the corresponding value for the single-cell combustion chamber is only 7 kgf. At the same time, in all cases the propane concentration relative to air in each chamber is the same and reaches 8%. It should be noted that the combustion chambers presented in each experiment have chambers of the same volume. The difference in the forces acting on the gas turbine blade for the first three combustion chamber designs (7, 13, and 29 kgf) is attributed to the different amounts of fuel involved in the combustion process, which result from the different numbers of cells used in each design.

In the case of the combustion chamber with one main and four auxiliary cells, the significant increase in the force acting on the turbine blade (up to 29 kgf) compared to the first combustion chamber (7 kgf) should be caused, together with the increase in volume, by the mutual reinforcement of the flows generated in individual chambers, in the case of simultaneous progression of the combustion process [5].

The further increase (up to 41 kgf) in the force acting on the gas turbine blade in the combustion chamber of the gas turbine generator is caused not only by the mutual reinforcement of the combustion flows, but also by the action of the water jet directed

from the main chamber toward the gas turbine blade. Along with the increase in this force, the capacity of the electricity generated by the gas turbine generator should also increase. To determine and analyze this capacity, a simplified calculation method developed by the authors is presented.

This method is based on the general scheme adopted in engineering practice for calculating electric generators and electrical machines in general [6]. It consists in preliminarily selecting, based on practical experience and for our case, a specific generator with a power presumably suitable for the task. Based on the type of its magnetic core, dimensions, and other electrical parameters (Table 2), we determine the braking electromagnetic force F_{em} acting on the rotor when operating in the mode characteristic of the nominal regime. This force is compared with the mechanical force F_{mech} acting on the gas turbine blade, directed opposite to this force. In our case, this force is $F_{mech} = 41$ kgf.

If $F_{em} < F_{mech}$, then we consider that a generator of the required power has been correctly selected for the gas-turbine generator. For the calculation, the DC generator GSG-500 of the so-called PSG-500 converter, used in arc welding, was chosen as a similar prototype. In the converter, the drive of the electric generator is an asynchronous motor. In our case, the drive of the generator is a gas turbine, which is set into rotational motion by the flow of combustion gases produced by the explosive combustion of fuel gas in the combustion chamber.

Table 2

Specifications of the direct current (DC) generator used in the calculation

#	Generator characteristics	Units of measurement	Value
1	Rated current	Ampere	500
2	Current range	Ampere	60-500
3	Rated voltage	Volt	230
4	Operating voltage	Volt	40
5	Length of the armature magnetic core	Meter	0,8
6	External resistance of the generator	Ohm	-
7	Displacement velocity of the armature winding conductor	rpm	300
8	Number of slots on the armature	Pieces	56
9	Number of conductors per slot	Pieces	2
10	Internal resistance of the generator	Ohm	0,4
11	Type of winding	-	Loop-shaped
12	Resistance of the excitation winding	Ohm	115
13	Armature's diameter	Meter	0,6

Based on the generator's rated speed (n), the magnitude of the electromotive force (e) induced in the winding conductor can be calculated. To perform this calculation, it is also necessary to know the diameter of the generator armature's magnetic core (D) and the active length of the conductor (L). Using these parameters, the velocity of the generator armature winding conductor's displacement within the magnetic field created by the excitation winding can be calculated.

$$V = \pi \cdot D \cdot n = 3,14 \cdot 0,6 \cdot 5 = 9,4 \text{ m/s}, \quad (1)$$

where n is the number of revolutions of the armature per second

$$n = n/60 = 300/60 = 5 \text{ rev/s} \quad (2)$$

The armature magnetic core is made of electrical steel Э310 [7], for which the maximum permissible value of magnetic induction is

$$B=1,2 \text{ tesla} \quad (3)$$

The electromotive force [8] induced in one conductor of the generator armature

$$e=B \cdot L \cdot V=1,2 \cdot 0,8 \cdot 0,9=9 \text{ V} \quad (4)$$

The armature magnetic core has 56 slots.

The conductors placed in them are connected, two ends each, to two collector plates, with which two brushes of the traverse simultaneously make contact; the consumer is connected to the latter; therefore, the number of conductors in the windings is as follows:

$$n_w=z/2=56/2=28 \text{ conductors} \quad (5)$$

The total electromotive force induced in the winding

$$e_\Sigma = n_w \cdot e = 28 \cdot 9 = 252 \text{ V} \quad (6)$$

The generator's excitation current

$$I_{exc} = \frac{U_s}{e_{exc}} = \frac{230}{115} = 2 \text{ Amp.}$$

(7)

The current flowing through the generator armature magnetic core

$$I_{arm} = \frac{P_s}{U} = \frac{3500}{252} \approx 140 \text{ Amp.}$$

The retarding electromagnetic force acting on the conductors of the armature winding

$$F_{em} = B \cdot L \cdot I_{arm} = 1,2 \cdot 0,8 \cdot 140 = 13,4 \text{ newtons} \quad (8)$$

The total retarding force acting on the armature winding

$$\Sigma F_{em} = F_{em} \cdot N_w = 13,4 \cdot 28 = 375,2 \text{ newtons} \quad (9)$$

The mechanical power generated by one combustion cell

$$P_{mech} = F_{mech} \cdot V = 41 \cdot 9,4 = 385,4 \text{ kgf} \quad (10)$$

The electric generator proposed for the gas-turbine power-generating device is designed to operate at a rotational speed of $n = 3000 \text{ rpm}$

The selected generator operates at 300 rpm; therefore, the operating speed is reduced by a factor of 10 relative to the rated speed.

The retarding power at the reduced rotational speed

$$P_{em} = \frac{1}{10} (e_\Sigma \cdot I_{arm}) = \frac{1}{10} 252 \cdot 140 = 3528 \text{ watts.} \quad (11)$$

Comparing expressions (10) and (11), it follows that during the operation of the generator, the following condition for overcoming the retarding force holds:

$$[P_{mech}=385,4] > [P_{em}=352,8], \quad (12)$$

which indicates the simultaneous operation of the combustion chambers of the gas-turbine device made with the proposed combustion cells.

That is, when the proposed combustion chamber with one main and four additional cells is operating in mixed mode, in particular, when a water jet is supplied to the main cell while propane combustion is taking place under optimal conditions in all five cells, it is possible to generate a power of 3.5 kW. In this case, the generator's load current amounts to approximately 90 A.

4. Conclusions

1. Based on the analysis of gas-turbine installations, devices, and gas-turbine generators in general, a new original design of

combustion chambers with one main cell and multiple accompanying cells is proposed.

2. Experiments and calculations confirm that the original combustion chamber proposed for the gas-turbine generator makes it possible to increase the amount of electrical energy generated significantly.

3. With one combustion chamber, given proper selection of cell dimensions, the new gas-turbine generator can generate electrical energy with a power of 3.5 kW.

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Utilization of powder forging technology for the design of hypercompositional structures from powders of various heat-resistant materials

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Abstract: This study evaluates the applicability of Powder Forging for producing hypercompositional structures from heat-resistant powders based on Inconel 625, ZhS6U, and CoCrAlY alloys. One-step forging and triple forging at 1150 °C were examined to determine their influence on densification, prior particle boundary elimination, microstructural evolution, and high-temperature mechanical behaviour. Triple forging markedly improved interparticle bonding, reduced structural defects, and enabled the formation of dense composite architectures with statistically distributed constituents. The Inconel 625-ZhS6U and Inconel 625-CoCrAlY systems demonstrated high yield strength over a broad temperature range and exhibited anomalous strengthening at elevated temperatures, attributed to dynamic strain aging and phase redistribution. The results confirm that Powder Forging is a promising thermomechanical route for designing advanced heat-resistant powder composites for aerospace hot-section applications.

Keywords: POWDER FORGING, NICKEL-BASED SUPERALLOYS, STRUCTURE, STRENGTH

1. Introduction

Nickel-based superalloys are indispensable materials for key components in modern aerospace engines and industrial gas turbines due to their outstanding creep resistance, high-temperature strength, and excellent oxidation resistance. In aircraft and unmanned aerial vehicles (UAVs), the performance of the power unit is strictly determined by hot section structures, such as turbine blades, disks, and nozzles, which withstand enormous thermal and mechanical loads [1–6]. To maximize thermal efficiency and minimize fuel consumption, turbine designs aim to reduce weight while ensuring the highest possible operating temperatures.

Ni-based alloys, particularly various modifications of Inconel alloys, are widely used in aircraft engines due to their relative affordability and efficiency [6–9]. However, in the cast state, these alloys tend to develop dendritic structures and segregation zones. While conventional casting-deformation technologies are lengthy and costly, alternative approaches like additive manufacturing (AM) still require significant post-processing to eliminate low productivity and structural defects [10–12]. Among powder metallurgy methods, hot pressure processing is gaining traction because it avoids residual porosity [13–17]. Specifically, dynamic hot deformation methods allow the production of highly dense samples with a homogeneous fine-grained structure at relatively low temperatures. Furthermore, powder metallurgy opens up unique possibilities for tailoring material properties through multi-scale structural modifications. On one hand, it allows the design of advanced hybrid materials by blending different superalloy powders (e.g., combining Inconel with high-performance ZhS-type alloys) to optimize the chemical composition and γ/γ' phase balance. On the other hand, this approach facilitates the incorporation of secondary strengthening phases, such as nano-sized oxides or hard carbide particles, into the matrix. Such combined reinforcement effectively pins grain boundaries, suppresses grain growth at ultra-high temperatures, and boosts the overall high-temperature mechanical response beyond the limitations of conventional single-component alloys. [18–20].

As an alternative to AM technology, the Frantsevich Institute for Problems of Materials Science (NAS of Ukraine) has been developing a powder technology known as Direct Powder Forging (DPF) [21–25]. Within this method, the desired product shape is formed during the cold and hot deformation of powders, and the required final structure is achieved by optimal thermomechanical treatment (TMT). Generally, direct powder forging (DPF) techniques are highly effective for grain refinement and controlling phase precipitation, suppressing grain growth, and promoting the uniform distribution of strengthening phases (such as γ or γ'' precipitates). However, implementing a multiple DPF method on highly alloyed, heat-resistant superalloys often introduces complex deformation mechanisms, including localized shear banding, mechanical twinning, and dynamic recrystallization. The exact

correlation between triple forging parameters and structural homogeneity is still a subject of ongoing debate.

To address this gap, is to model the distribution of flow deformation characteristics and porosity changes during hot powder forging, alongside developing thermomechanical processing regimes for the Ni-based superalloy powder alloy. This study investigates the microstructural evolution and material behaviour at each forging stage to achieve an optimal set of mechanical properties over a wide temperature range, providing practical guidelines for optimizing industrial forging processes of advanced superalloys.

2. Materials and experimental procedure

Spherical powders of the nickel-based superalloys Inconel 625 and ZhS6U, produced by the plasma rotating electrode process (PREP), as well as powder of the cobalt-based heat-resistant alloy Co-22.3Cr-10.8Al-0.35Y (wt.%), were used in the study. The densification and deformation behavior of the Inconel 625 and ZhS6U powder alloys, as well as powder mixtures of Inconel 625 – 50 vol.% ZhS6U and Inconel 625 – 50 vol.% Co-22.3Cr-10.8Al-0.35Y, were investigated. The superalloy powders were blended in a 3D mixer for 3 h. The required amounts of the heat-resistant alloy powders were loaded into a cylindrical steel container, followed by hermetic compaction at a pressure of 450 MPa (Fig. 1).

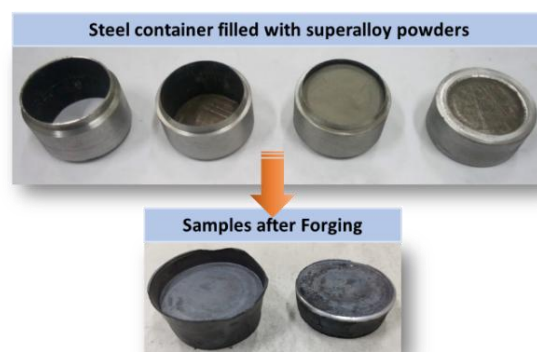


Fig. 1. Steel container with superalloy powders before and after processing by the DPF scheme.

The prepared powder-filled containers were subsequently forged using an FB1732 screw press with a nominal force of 1.6 MN and a kinetic energy of the moving parts at the end of the acceleration stroke of at least 6.18 kJ. The punch speed during compaction was 0.35 m/min.

Forging was performed in a semi-closed die with an upper flash. For the Inconel 625 and ZhS6U alloys, the densification and deformation behaviour of the powder compacts were investigated under single-pass and three-pass forging at 1150 °C. Based on the deformation data obtained for Inconel 625 and ZhS6U, thermomechanical processing of the Inconel 625 – 50 vol.% ZhS6U and Inconel 625 – 50 vol.% Co-22.3Cr-10.8Al-0.35Y powder mixtures was subsequently investigated at 1150 °C using a three-pass forging schedule (Fig.2).

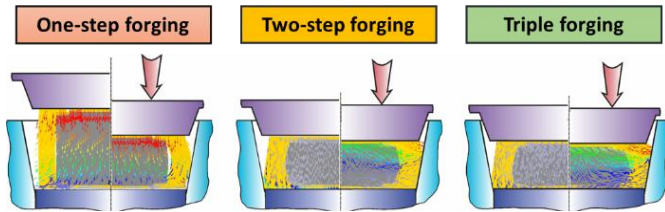


Fig. 2. Schematic of multiple forging of a heat-resistant alloy using the DPF method.

The specimens were heated by immersing them in a furnace preheated to the required temperature under an argon atmosphere and holding them at this temperature for 20 min. After forging, conical specimens with a flash diameter of 42 mm and a height of 9 mm were obtained (Fig.1). For the multiple-forging procedure, the conical specimen obtained after the first deformation step was machined to a cylindrical shape, rotated by 180°, and subjected to the second deformation step until a height of 11 mm was reached. The third and final deformation step was performed according to the same procedure. The material structure was examined using a JEOL Superprobe-733 and Tescan Vega 3 scanning electron microscopes. Compressive strength was measured on samples of 5×3×3 mm at temperatures of 20 and 750 °C using a UTM-100 universal testing machine.

3. Results and discussion

The DPF technology involves structural evolution through the localization of deformation in the region of interparticle boundaries. As a result, two important structural transformations are achieved: (I) recrystallization of interparticle boundaries in the contact regions and (II) the formation of an ultrafine-grained structure through the dynamic recrystallization (DRX) mechanism. Previous studies [24, 25] demonstrated that DRX indeed initiates in the boundary regions during a single forging operation; however, relatively high residual porosity and crack-like defects remain at the triple junctions of particles. To eliminate these structural defects, an advanced DPF technology involving three consecutive forging operations was proposed. Microstructural investigations and mechanical testing showed that interparticle fracture was eliminated after triple forging, although the yield strength decreased slightly due to grain growth. This technology also enables the fabrication of complex powder composites from different types of starting powders.

Fig. 3 presents the results of mechanical testing of ZhS6U and Inconel 625 alloys after forging at 1150 °C using one and three deformation steps. As expected, the yield strength of both alloys was higher after a one - step forging operation than after triple forging.

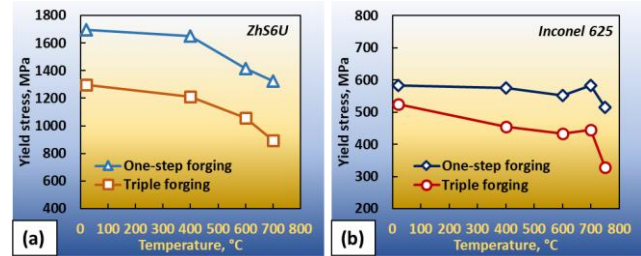


Fig. 3. Yield stress versus temperature for (a) ZhS6U and (b) Inconel 625 after different forging conditions.

Microstructural analysis shows that a large number of planar defects remain after one-step forging (Fig. 4a, 5a). Agglomerates of impurities, carbide particles, and γ' precipitates are predominantly concentrated along the prior particle boundaries (PPBs), as shown in Fig. 4a. The fracture mode for both ZhS6U and Inconel 625 is interparticle (Figs. 4b, 5b). A distinctive feature of the processing of the ZhS6U alloy is that deformation is performed below the solvus temperature, and the strengthening γ' precipitates remain within the matrix. In contrast, Inconel 625 is processed above the solvus temperature, at which the strengthening γ'' precipitates dissolve into the matrix, while the carbide particles remain. After triple forging, an increase in grain size is observed; however, the interparticle boundaries are almost completely eliminated (Figs. 4c, 5c). Repeated deformation breaks up the initial interparticle boundaries and promotes a more uniform distribution of second-phase particles throughout the matrix, although small localized clusters and some larger individual particles still remain. The fracture mode changes to transcrystalline fracture (Figs. 4d, 5d).

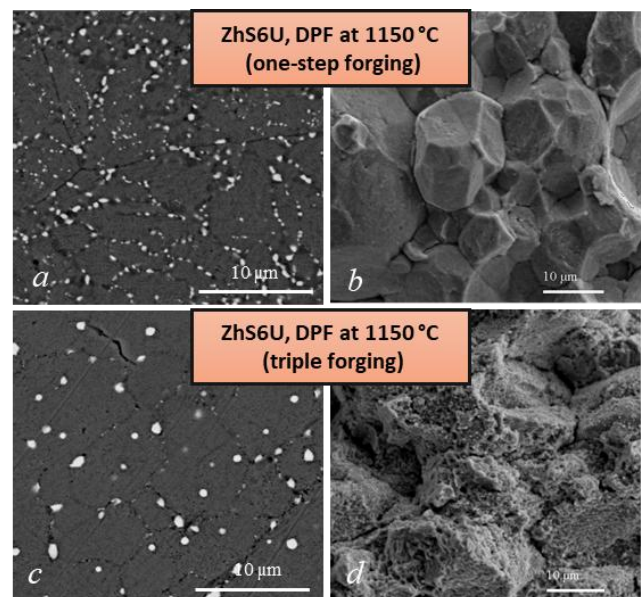


Fig. 4. Microstructure (a, c) and fracture surface (b, d) of ZhS6U powder alloy after deformation processing at a temperature of 1150 °C.

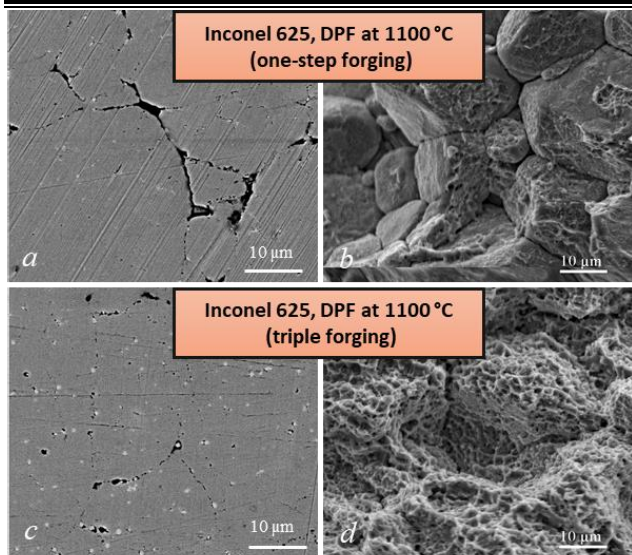


Fig. 5. Microstructure (a, c) and fracture surface (b, d) of Inconel 625 powder alloy after deformation processing at a temperature of 1150°C.

For the alloy consisting of 50% ZhS6U and 50% Inconel 625, the mechanical testing results (Fig. 6a) after triple forging demonstrated a room-temperature yield strength comparable to that of the stronger ZhS6U alloy, reaching approximately 1000 MPa. With increasing temperature to 500 °C, the yield strength decreased to approximately 700 MPa, which may be associated with the softening effects characteristic of Inconel 625. However, at higher temperatures, an anomalous temperature dependence of the yield strength was observed, with the yield strength increasing to approximately 950 MPa at 750 °C. This behaviour may be related to the dynamic strain aging (DSA) mechanism, which is characteristic of Inconel 625 in this temperature range.

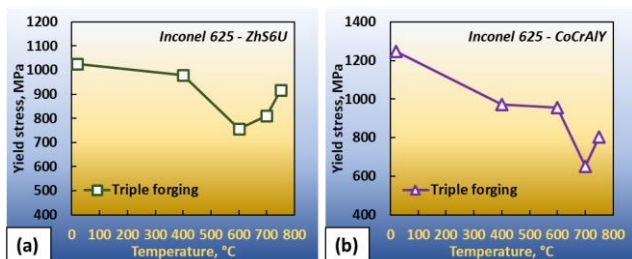


Fig. 6. Yield stress versus temperature for (a) Inconel 625-ZhS6U and (b) Inconel 625 - CoCrAlY powder alloy after different forging conditions.

No evidence of planar defects is observed at the boundaries between the Inconel 625 and ZhS6U powder particles (Fig. 7). Considering the high deformation rate of 10^2 s^{-1} , the structure does not have sufficient time to become homogenized through diffusion. The resulting alloy can be considered a hyper-composite with a statistically distributed structure and sufficient connectivity of each structural constituent. Under optimized deformation conditions, this approach enables the structure and properties to be tailored by exploiting the beneficial contributions of the individual structural constituents.

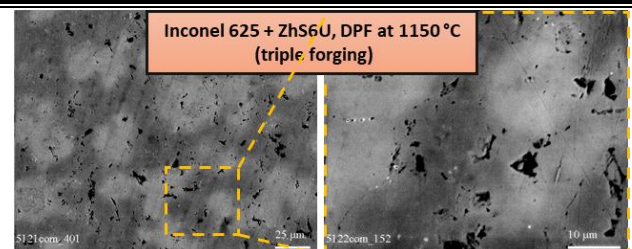


Fig. 7. Structure of Inconel 625-ZhS6U powder alloy after deformation processing at temperature 1150°C (general view and high-magnification image showing grain boundaries).

Figure 6b presents the results of mechanical testing of the Inconel 625 - 50 vol.% Co-22.3Cr-10.8Al-0.35Y powder composite. The mechanical properties are also comparable to those of the best high-temperature superalloys. At room temperature, the yield strength reaches 1200 MPa and remains at approximately 1000 MPa up to 600 °C, followed by a decrease to 650 MPa at 700 °C. However, the yield strength increases again to 800 MPa at 750 °C (Fig. 6b). This anomalous temperature dependence of the yield strength can be attributed to the characteristics of the dynamic strain aging (DSA) effect in this temperature range.

Microstructural analysis shows that, in addition to the two structural constituents characteristic of each component of the composite, a relatively wide transition zone forms between the dissimilar powder particles as a result of the diffusion of alloying elements into the interparticle contact regions (Fig. 8a). Although no crack-like defects are observed in the microstructure, the fracture path reflects the structure of the initial powder particles, apparently due to the higher brittleness of the β -phase (CoAl) formed in the interparticle region (Fig. 8b).

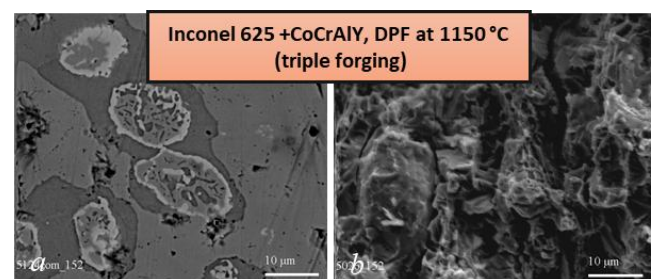


Fig. 8. Microstructure (a) and fracture surface (b) of Inconel 625-CoCrAlY powder alloy after deformation processing at a temperature of 1150°C.

Thus, DPF technology enables the development of advanced heat-resistant materials by designing hyper-composites comprising constituents of different heat-resistant powder alloys.

4. Conclusions

Implementation of multiple Direct Powder Forging (DPF) at 1150 °C successfully ensures enhanced consolidation, structural homogenization, and tailored mechanical performance in advanced powder-derived nickel-based systems. Microstructural analysis confirms that shifting from single-step to three-pass forging effectively disrupts prior particle boundaries (PPBs) and accelerates pore closure, yielding highly dense matrices with well-bonded interfaces for both the individual alloys (ZhS6U, Inconel 625) and their

blended mixtures (Inconel 625 – ZhS6U/CoCrAlY). Mechanical evaluations over a wide temperature range reveal that triple forging consistently induces a softening effect (through coarsening of the microstructure and redistribution of the strengthening phase), compared to one-step forging, resulting in increased material ductility. Furthermore, the powder composites after triple forging demonstrate distinct anomalous high-temperature strengthening peaks between 600 °C and 750°C driven by dynamic aging and phase redistribution. These findings provide critical guidelines for optimizing multi-directional hot deformation regimes to fabricate highly efficient, heat-resistant components for aerospace structures. DPF technology expands the possibilities for developing heat-resistant materials through the design of hyper-composites comprising constituents of different heat-resistant powder alloys.

5. Acknowledgements

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Tolerance treatment of sliding pairs and repeatability in assembly

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Abstract: The basic prerequisite for trouble-free assembly of moving structures is that the assembled structure has as many degrees of freedom as its function requires. In practice, we often encounter cases of statically overdetermined pairs. Designers often use them to increase the rigidity of products. This article describes a way to mitigate the consequences of problematic assembly in sliding pairs, which are often statically overdetermined. The article also addresses an important problem in assembly, which is repeated assembly accuracy, especially when designing assembly lines and automated and robotic systems.

Keywords: ASSEMBLY, KINEMATIC PAIRS, TOLERATION, REPEATED PRECISION

1. Introduction

In practice, the cases of statically redetermined couples are often encountered. The designers like using them to enhance rigidity of the product. A typical example is a ball bearing, which should theoretically have the maximum of three balls, but it has more of them in order to increase their toughness.

2. Statics in assembly

Figure 1 illustrates a hydraulic press that is statically redetermined. When the support moves on two cylinders, the couple is clearly statically redetermined, as shown in the Grübler - Valentovič equation [1, 2], according to which the number of degrees of freedom is minus two.

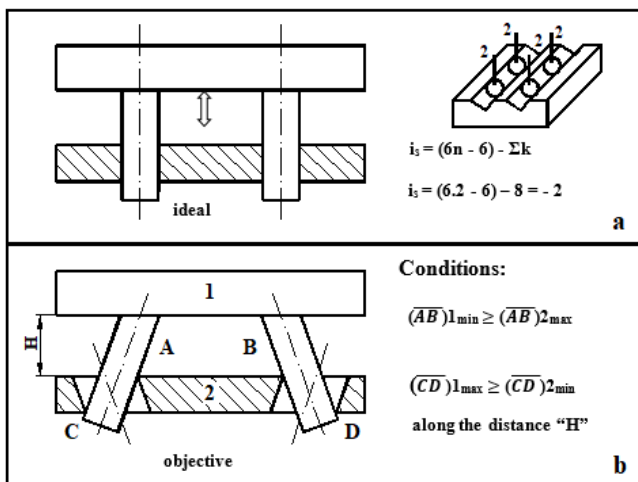


Fig.1: Toleration treatment of statically redetermined movable couples

There are more conditions for trouble-free assembly regarding the fact that the couples are not parallel but concurrent or even skewed. In such circumstances, the couple must have gaps enabling smooth assembly even despite non-parallelism of the couple, which in turn partially decreases toughness.

It is often necessary to bond two elements (Figure 1) which apparently form a statically redetermined kinematic couple.

This concerns also a flanged bond, e.g. the bond of white and black flanges with four pins (Figure 2a). It is desirable to label both parts in order to enable assembly in any case (trouble-free assembly).

Figure 3 shows a different method of mitigating the consequences of troublesome assembly of very frequent sliding couples which are generally statically redetermined. The couple depicted in Figure 3a is evidently statically redetermined; the consequences may be mitigated when first assembling the sliding workbench with rods and then connecting them to the base (Figure 3b).

Figure 3c is the best illustration of trouble-free assembly: there are two rotary-sliding couples (four-point) on one shaft, and a one-point couple on the other one (touches the left or the right side). Such a structure is statically redetermined but definite, which may assure completely trouble-free assembly.

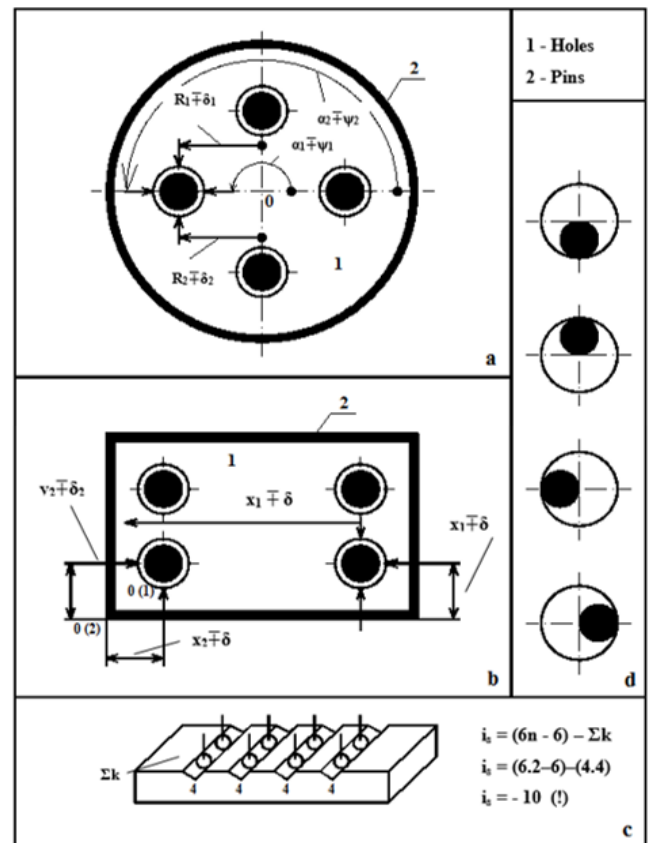


Fig.2: Assemblability of statically redetermined unmovable couples. a, b – dimension method, c – number of degrees of freedom, d – assemblability check

Figure 3 shows a different method of mitigating the consequences of troublesome assembly of very frequent sliding couples which are generally statically redetermined. The couple depicted in Figure 3a is evidently statically redetermined; the consequences may be mitigated when first assembling the sliding workbench with rods and then connecting them to the base (Figure 3b).

Figure 3c is the best illustration of trouble-free assembly: there are two rotary-sliding couples (four-point) on one shaft, and a one-point couple on the other one (touches the left or the right side). Such a structure is statically redetermined but definite, which may assure completely trouble-free assembly.

If high accuracy in the product is not needed, so called extenders of gaps can be used. The principle is shown in Figure 4.

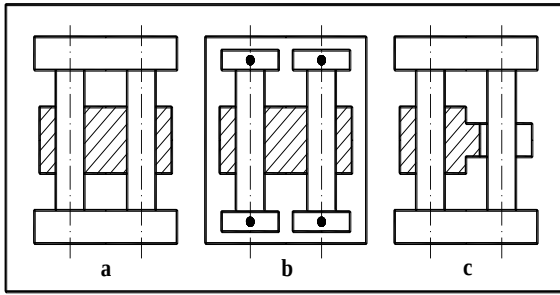


Fig.3: Assemblability of movable couples [3]. a – statically redetermined structure, b – gradual assembly of statically redetermined structure, c – statically determined structure (trouble-free assembly).

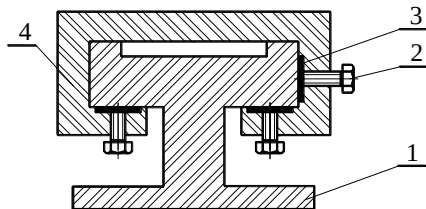


Fig.4 Assemblability of movable couples. Demands for precision will decrease when using extenders of gaps. 1 – frame, 2 – screws, 3 – extenders, 4 – bench.

3. Statics repeated precision of assembly

A very important issue in assembly is repeated accuracy, especially when constructing assembly lines (Figure 5).

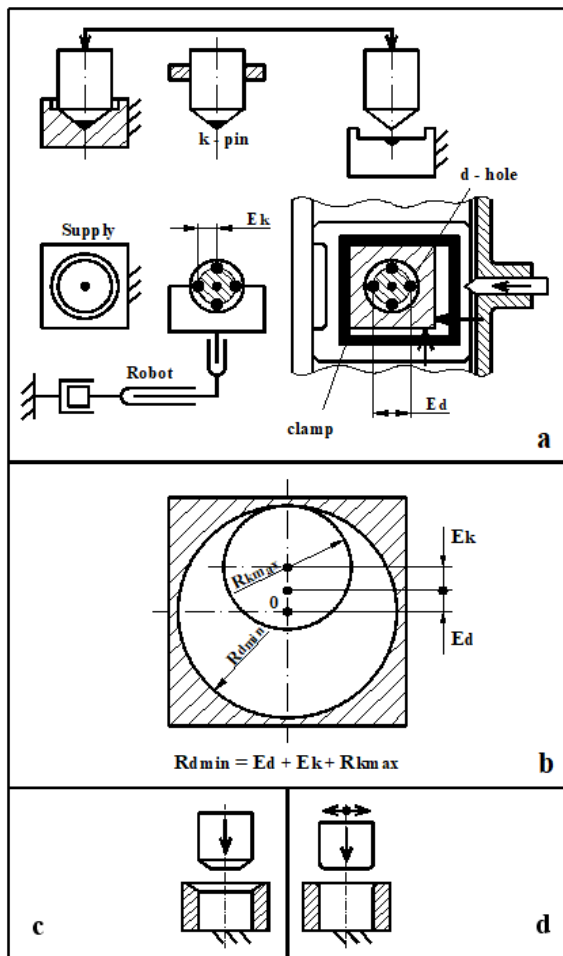


Fig.5 Repeated accuracy of the robot and clamp increases the probability of the insertion of pin into hole [4]. a – robot and clamp, b – conditions of insertion, c – chamfers improve probability of insertion, d – vibration improves probability of insertion.

Inaccuracy of the robot inserting the pin into the hole may cause the inaccuracy of the hole location in the gripper, which may prevent a successful insertion of the pin into the hole (Figure 5a).

This may be due to the reasons described below [4]. The first reason for the failure of assembly is that a robot collecting pins from the palette has a certain repetitive accuracy (uncertainty) of positioning, i.e. when inserting the pins, their spikes are not positioned to the same point (position), but make a circle of eccentricity “ E_k ” (Figure 5a).

Another reason for the failure of assembly is that the center of the hole is not in the same place with respect to the frame of the machine, but may be in a different place in each clamp, which is due to inaccuracy of the gripper and clamp, and sometimes also due to inaccurate contours of the part in the clamp (Figure 5a).

The issue poses a problem since the two causes may be combined (inaccuracies may increase by mutual summation) and it can be easily proved (Figure 5b) that to insert a pin by the radius R_{kmax} into the hole, the radius of the hole must be:

$$R_{dmin} = E_d + E_k + R_{kmax} \text{ [mm]} \quad (1)$$

where:

R_{dmin} – minimum radius of opening [mm],

E_d – eccentricity of opening [mm],

E_k – eccentricity of pin [mm],

R_{kmax} – maximum radius of pin [mm].

From experience it is known that such assembly requires a disproportionately large gap between the pin and the hole. This can be eliminated by using the maximum chamfer of both components (Figure 5c) and then trying to insert the pin into the hole by utilising flexibility.

Another option is a little more complicated: the pin is placed next to the hole and then oscillatory motions are performed until success is achieved, under some pressure, to capture the pin into the hole (Figure 5d).

4. Conclusion

Online references will be linked to their original source, only if possible. To enable this linking extra care should be taken when preparing reference lists.

The above-mentioned suggests that the shorter and of better quality the line, the lower the values of E_k and E_d . As illustrated in Figure 5b, assembly will be trouble-free under the following condition:

$$R_{dmin} = E_d + E_k + R_{kmax} \text{ [mm]} \quad (2)$$

or for the diameters:

$$(\varnothing d_{min})/2 = E_d + E_k + (\varnothing k_{max})/2$$

$$\varnothing d_{min} = 2E_d + 2E_k + \varnothing k_{max} \text{ [mm]} \quad (3)$$

After supplying particular values, e.g. $\varnothing k_{max} = 40.1$ mm, $E_k = 0.2$ mm, $E_d = 0.1$ mm:

$$\varnothing d_{min} = 0.2 + 0.4 + 40.1 = 40.7 \text{ [mm]}$$

Such a large gap (0.7 mm) cannot be accepted; the diameter of the hole will be therefore reduced. From experience it is known that the gaps between pin and hole will be disproportionately large in such assembly.

The gap can be eliminated by using the maximum chamfer of both parts (Figure 5c) and then applying flexibility to insert the pin into the hole.

Another option is a little more complicated: the pin is placed next to the hole and then oscillatory motions are performed until

success is achieved, under some pressure, to capture the pin into the hole (Figure 5d).

The machine to carry out such operations must be fitted with artificial sight and feeling. Such systems are very complex.

5. Acknowledgements

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Investigation of the causes of corrosion of drinking water supply steel pipes

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Abstract: Galvanized steel pipes have long been widely used in drinking water supply systems due to the zinc coating that protects the steel from corrosion. However, during operation, especially when hot water is supplied, the zinc protective layer gradually breaks down, especially if there are galvanic pores in the pipeline or stray electric currents are generated in the pipeline. If the zinc coating is damaged, the steel base begins to corrode, rust forms, the pipe wall thickness decreases and the water quality deteriorate.

In addition to internal corrosion, a significant problem is external pipe corrosion caused by condensation forming on the surface of cold-water pipes. When the pipe surface temperature is below the dew point of the ambient air, moisture accumulates on it. Long-term wetting of the surface, especially if the zinc coating or protective paint layer is damaged, creates favorable conditions for the development of atmospheric corrosion. The corrosion process is further accelerated by poor indoor ventilation, high relative humidity or incorrectly calculated insulation layer thickness.

The article will examine two corrosion situations of steel pipes for drinking water supply using metallographic, water parameter studies, and stray current measurements.

Keywords: DRINKING WATER, MICROSTRUCTURE, STEEL PIPES, CORROSION.

1. Introduction

The continued reliance on metallic materials across industrial, energy, transportation, biomedical, and infrastructural sectors underscores the persistent importance of understanding and mitigating degradation phenomena that compromise structural integrity and operational reliability. Corrosion, wear, and fatigue remain among the most pervasive and costly challenges associated with metallic systems, particularly as modern applications demand higher performance under increasingly aggressive environmental and mechanical conditions. The major form of material degradation is corrosion of metallic materials [1, 2].

Over the past four years, the authors of this article have investigated five cases of corrosion of drinking water supply pipelines and identified possible causes. The cases of pipeline corrosion were recorded in Vilnius, the capital of Lithuania, in apartment buildings and corporate buildings designed approximately 5-10 years ago. In this article, we will examine one of these cases.

The research site is a building in Vilnius, the capital of Lithuania, where cases of advanced corrosion of pipelines have been recorded (Fig. 1). The Customer provided a cut section of the chilled water (according to the Customer, up to approximately +7°C) pipeline with obvious areas of corrosion damage and samples of the building's hot and cold water. The cold-water sample was taken from the building's cooling pipeline; the hot water sample was taken from the kitchen tap.



Fig. 1. Corrosion-damaged section of cold-water piping in a building

The problem is that the cooling system's water supply pipes (+7°C) are rusting, while the return (warmed) water pipes remain intact.

The purpose of the research is to investigate the possible causes of corrosion of the drinking water supply pipeline.

2. Methodology

The microstructure of the steel of pipes was analyzed, as well as the welds of the connections of pipes segments. The influence of possible stray electric currents and contact of the material with moisture was examined, too.

Equipment used for experimental studies:

- Portable computer oscilloscope Picoscope 2204.
- Portable meter BK Precision LCR Meter 880.
- Laptop Dell Latitude E6420 with installed manufacturer-provided software for the oscilloscope Picoscope 2404.
- Equipment and materials for preparing samples for metallographic examination (microsections).
- Microscope Nikon Eclipse LV100ND with objectives: Nikon L Plan 2.5x/0.075, Nikon TU Plan Fluor 5x/0.15, Nikon TU Plan Fluor 10x/0.30, Nikon TU Plan Fluor 20x/0.45 and Nikon TU Plan Fluor 100x/0.90.

The chemical composition and other properties of hot and cold-water samples were examined in an accredited laboratory (see Appendices). The samples were set as “drinking water” type in the laboratory.

Sample preparation and testing methods:

- Testing of the chemical composition and other properties of water samples in an accredited laboratory according to the standards: LST ISO 6059:1998, LST ISO 6059:1998/P:2008, LST ISO 6058:1998, LST ISO 6058:1998/P:2008, LST EN ISO 10304-1:2009, LST EN ISO 10523:2012 (Except p. 8), LST EN 27888:1999 and LST EN ISO 9963-1:1999 (Except p. 8.1).
- Preparation of steel pipe microsections according to the standard ASTM E3-11:2017 [3] (Fig. 2).
- Analysis of the obtained measurement results based on technical literature, scientific articles (see literature list) and standards: EN ISO 14713-1:2017 and ISO 4967:2013 [4, 5].



Fig. 2. Samples prepared for optical microstructural analysis: pipe parts and welds

3. Results and Discussion

3.1. Examination of the effect of stray electric currents on the corrosion of cold-water supply pipeline in a building

The measurements were performed on 2026-05-04, using the BK Precision LCR Meter 880 device for measuring the resistance, inductance and capacitance of connecting cables, a portable computer oscilloscope Picoscope 2204, a DELL Latitude E6420 laptop with installed software for controlling the Picoscope 2204 oscilloscope and processing the results. To measure the voltages between different parts of the pipeline, the pipeline parts were connected according to the scheme shown in Figure 3, a.

First, the resistance measurements of the connected cables were performed at the pipeline installation site, selecting a measurement frequency of 1000 Hz. After measuring the resistances of the connecting cables, the oscilloscope was connected and voltage measurements were performed according to the scheme provided.

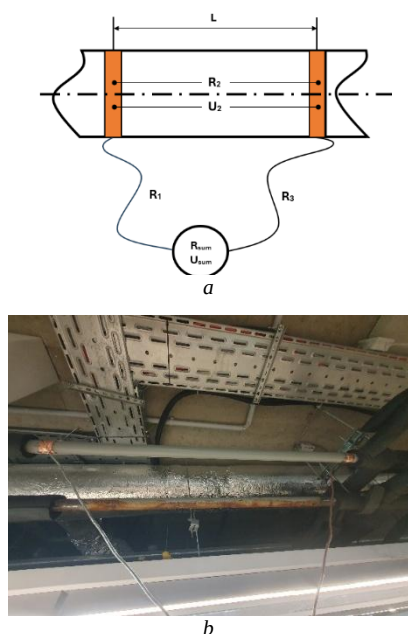


Fig. 3. Voltage measurement in a cold-water pipeline: a – electrical circuit connection diagram; b – voltage measurement in a cold-water pipeline, after connecting the circuit according to the diagram a

A random result of an alternating voltage measurement is recorded (Fig. 4). Figure 4 shows the dependence of the voltage magnitude (y axis) over time (x axis). The time scale starts from 0, and the 1st cell corresponds to 5 seconds. The alternating voltage scale is shown in the interval from -50mV to +50mV, the 1st cell corresponds to 10mV. The measured signal (blue color) is like noise. Part of the signal is on the negative voltage side; part is on the positive. This proves that an alternating voltage current flows through the pipeline. The generated voltage is not of constant magnitude, it jumps from the minimum amplitude value (Fig. 4, black dashed lines) and the maximum (the highest peaks on the positive and negative sides of the vertical axis).

Characteristics of the examination data provided:

- Measurement type and time – units are hour and minute, respectively.
- $U_{\min}$ – minimum amplitude value of the measured signal.
 $U_{\max}$ – maximum amplitude value of the measured signal
 ΔU – difference between these values.
- R_{sum} – Measured resistance of the electrical circuit.
- f – the most frequently recurring frequency determined during the spectrum analysis of the measured signal.
- I_{peak} – calculated short-term current strength of the measured signal.

- U_{RMS} – RMS voltage magnitude of the measured signal.
- I_{RMS} – Calculated RMS current strength of the measured signal.

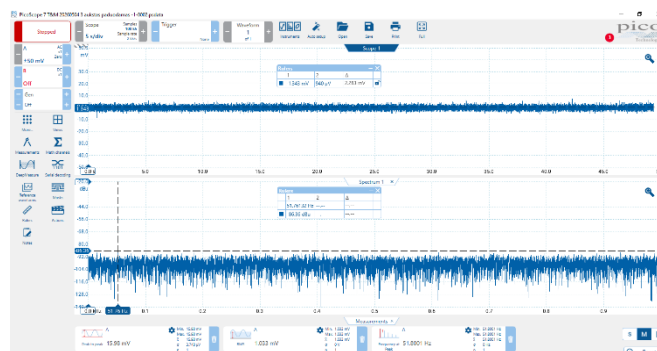


Fig. 4. A voltage measurement lasting approximately 50 seconds was recorded when the Picoscope 2204 portable computer oscilloscope was connected to the cold-water supply pipeline, maintaining 1m between the connection poles

The measurement results were carried out in equal sections (about 1 meter between the connection poles), both the supply (abbreviation – “P”) and the return (abbreviation – “G”) pipelines on different floors of the building 2 (two measurements), 3 (two measurements) and 5 (one measurement). During the measurement, the values of the electrical signals were also included, when one pole was connected to the supply pipeline, and the other to the return (Abbreviation – “PG”).

The cold-water pipeline was suspended and covered with thermal insulation materials inside the building, in the ceiling. The cold-water pipeline structure has screw connections, welded joints and other connecting sleeves and transitions from metal pipeline to polymer. After processing the measurement results, considering the electrical signal noises generated by the circuit and eliminating them from the measurement curves, it was determined that the RMS voltage of the alternating electric current in the cold-water pipeline is about 0.83-1.14 mV.

On the second floor of the building, the values of the electric current I_{peak} and I_{RMS} of the supply and return pipelines were calculated by applying Ohm's law and, accordingly, subtracting the average values of unwanted noise from the $U_{\max}$ and U_{RMS} signals. The calculated short-term I_{peak} current values ranged from 0.07 A to 0.08 A. Their duration is about 1 ms, and the density is 1-3 times per 45-second measurement period. This current value affects the melting of the metal due to the electrochemical phenomena that occur. The calculated long-term I_{RMS} current values range from 0.005 A to 0.007 A. Based on Faraday's laws and the prevailing calculated RMS electric currents, electrochemical phenomena occur but considering the strength of the calculated electric current in milliamperes and the applied frequency - the influence on the melting of the metal will be extremely slow and practically insignificant.

The same results were obtained and on the third and fifth floors.

In the hot water supply pipeline, it was observed that the prevailing electric field frequencies were unstable, and the alternating electric current I_{peak} reached up to 0.73 A, and I_{RMS} – about 0.018 mA. The peak repetition rate was intense during the 45-second measurement period.

3.2. Results of the study of the chemical composition of hot and cold water and the influence of water on corrosion of pipes

According to scientific literature, metal corrosion can be promoted by the concentration of oxygen dissolved in water, pH, water hardness, HCO_3 , Cl^- , SO_4 ion concentration, temperature (temperature is especially influential in closed systems, then

corrosion in steel can reach about 0.25 mm/year at 40°C, and at 80°C – ~0.5 mm/year [6, 7]) and other factors.

The chemical composition and other properties of the relevant components of hot and cold-water samples were studied in an accredited laboratory. The data from the chemical test protocols are presented in Table 1. This study determined the amounts and properties of chemical elements and compounds that may affect or promote pipeline corrosion.

Table 1. Results of water sample testing

Analyte	Result		Units	Allowed up to (HN 24:2017)
	Hot water	Cold water		
Magnesium	15	16	mg/l	Not regulated
Calcium	70	10	mg/l	Not regulated
Total hardness	2.32	0.90	mmol/l	Not regulated
Chloride	23	9.9	mg/l	250.0 mg/l
pH	7.7	8.9	pH when T = 20.3°C	6.5 – 9.5
Specific electrical conductivity	479	137	μS/cm, when T = 24.8°C	2500 μS/cm, when T = 20°C
Total alkalinity	4.36	1.13	mmol/l	Not regulated
Hydrocarbonate	266	69	mg/l	Not regulated

After determining the amounts of chemical elements and compounds that may affect metal corrosion and other properties of hot water, calculations were performed according to various methodologies found in the literature [8-14] to find out what properties the water flowing through the pipeline under study has (results are presented on Table 2). The chemical properties of water are described by such parameters as: water hardness (Ca and Mg in water), water alkalinity (HCO₃ and CO₃ in water), Langelier index (determining whether the water is chemically balanced, or promotes corrosion or lime formation), Ryznar stability index (determining whether the supplied water promotes corrosion or lime formation), Puckorius stability index (determining whether there is a tendency to lime formation or corrosion) and Larson index (determining whether corrosion is promoted) (Table 3).

Table 2. The results of the calculations performed according to the methodologies established by other scientists [8-14]

Parameter	Result	Tendency of pipe steel to corrosion when exposed to water of its current composition
HOT WATER		
Water hardness, mg CaCO ₃ /l	236.33 (hard)	-
Water alkalinity, mg CaCO ₃ /l	219.21 (strongly alkaline)	-
Corrosive media Langelier index (LSI)	0.91	No
Ryznar stability index (RSI)	5.89	No
Puckorius stability index (PSI)	5.62	No
Larson index (LaI)	0.70	No
COLD WATER		
Water hardness, mg CaCO ₃ /l	90.74 (medium hard)	-
Water alkalinity, mg CaCO ₃ /l	60.80 (moderately alkaline)	-
Corrosive media Langelier index (LSI)	-0.14	Yes
Ryznar stability index (RSI)	9.18	Yes
Puckorius stability index (PSI)	10.93	Yes
Larson index (LaI)	2.3	Yes

Although the chemical analysis of cold water shows that water is aggressive to steel pipes, when the sample was cut lengthwise, no

internal corrosion sites were visible, although the outer surface of the pipes was damaged by corrosion (Fig. 5). The cuts were made in the most likely places in the pipeline - at welds or when the direction of water flow changes.

It can be stated that the intense surface corrosion was caused by the external operating conditions of the pipeline, and the chemical composition of the water had no influence on it.

Table 3. Limitations of Langelier, Ryznar, Puckorius and Larson indices [8-14]

Value	The influence of water composition on pipe corrosion
Langelier index	
LSI < 0	Corrosion susceptibility
LSI = 0	Chemical balance
LSI > 0	Scaling susceptibility
Ryznar index	
RSI > 9	Extremely severe corrosion
7.5 < RSI	Severe corrosion
7.0 < RSI	Significant corrosion
6.0 < RSI	Slight scaling or corrosion
5.0 < RSI	Light scaling
4.0 < RSI	Heavy scaling
Puckorius index	
PSI > 6.5	Corrosion susceptibility
4.5 < PSI < 6.5	Optimal index range
PSI < 4.5	Scaling susceptibility
Larson index	
LaI > 1.2	Severe corrosion
0.8 < LaI < 1.2	Significant corrosion
PSI < 4.5	Does not affect the occurrence of corrosion

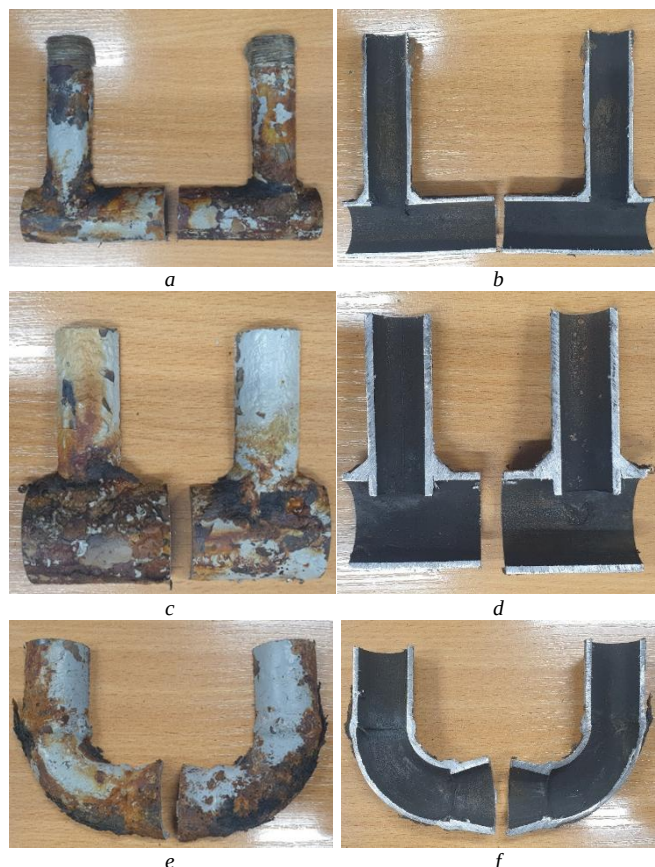


Fig. 4. Corrosion-damaged parts of cold-water pipelines: a, c, e – external surface; b, d, f – internal surface

3.3. The influence of galvanic couples on pipeline corrosion

In the pipeline under study, several metals and alloys are involved in the corrosion process – iron and copper alloys (brass or bronze) (Fig.5).

Two metals, connected to each other and surrounded by an electrolyte (water), form a galvanic couple (an electric current begins to flow between them) [15]. One of them will begin to dissolve (become the anode) at the expense of the other metal (become the cathode). The one whose electrode potential is more negative will become the anode. In the iron-copper pair, iron (the basis of steel and cast-iron alloys) will dissolve, since its potential is more positive. The influence of the galvanic couple on the occurrence of corrosion is easily observed in Fig. 5, where copper alloy components (valve, transition, etc.) are connected to a steel pipe. Corrosion has severely damaged the steel (iron alloy) pipe.

Standard EN ISO 14713-1 states that a bimetallic (two different metal alloys) connection is possible, but on condition that the structural elements are isolated from each other, i.e. do not come into contact. Bimetallic corrosion is particularly harmful in flowing water. The standard provides levels of bimetallic corrosion depending on the operating environment: a (weakest), b, c and d (most intense). In a couple of steel pipes with copper in fresh water, corrosion is assessed from b to c, i.e. from medium to quite intense.

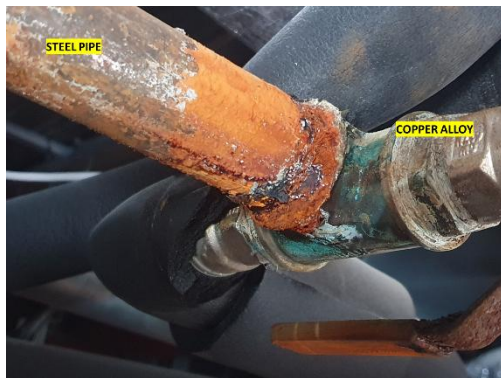


Fig. 5. Corrosion in the presence of a galvanic couple of dissimilar metals

3.4. Metallographic examination of steel pipe specimens used for cold water supply

Samples for metallographic examination were cut from the serviced pipe. The selected locations for the samples were weld seams, corrosion-damaged areas and the least corrosion-damaged areas.

The samples were mounted in resin, ground and polished. The polished samples were examined for non-metallic inclusions. Non-metallic inclusions were found to be 1-2 score (according to ISO 4967 [5]) (Fig. 6), and most of the oxides were found. In the mentioned standard, the cleanest steel from non-metallic inclusions is marked with a score of 1. Steel heavily contaminated with non-metallic inclusions (oxides, sulfides, silicates, etc.) is less resistant to corrosion, with poorer mechanical properties. In this case, the steel is sufficiently clean from non-metallic inclusions, only a small amount of fine spherical oxides was observed.

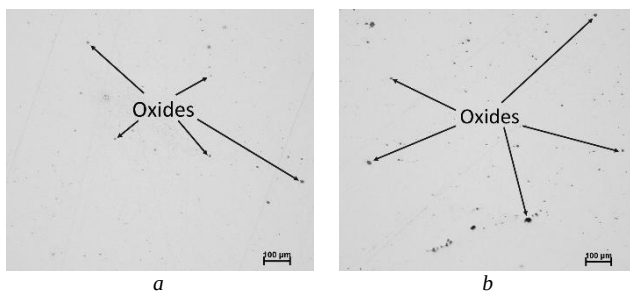


Fig. 6. Non-metallic inclusions were detected in the microstructure of the tested pipe samples: a, b – oxides, small black spherical particles

The polished samples were etched with a 3% HNO_3 solution in ethyl alcohol to reveal the metal structure and the heat-affected zone during welding. The microstructure of the weld seam of the etched sample is presented in Fig. 7. No defects were observed in the metal layers of the weld seams made during pipe production, nor were

there any signs of corrosion. When installing the pipeline in the building, individual pipe parts were connected by welding. In the part of the pipeline provided by the Customer, no corrosion damage was observed in the weld seams of the joints (Fig. 7, a, b, c). It should be noted that weld seams are by their nature more prone to corrosion due to the higher density of defects in the metal structure (which is inevitable and natural) and the possibility of non-metallic compounds entering during welding.

No corrosion-damaged areas were observed on the inner surfaces of the pipes, or only very minor sites were found. Meanwhile, a few developing corrosion-damaged sites were found on the outer surface under the paint film (Fig. 7 d, e, f).

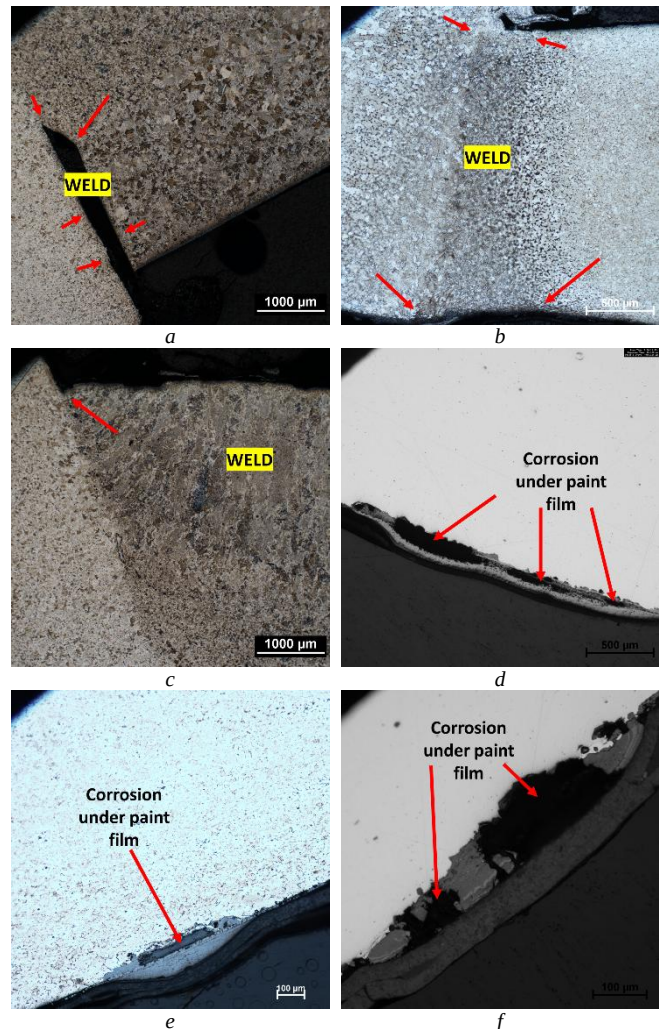


Fig. 7. Metallographic examination of samples cut from an operational pipeline: a, b, c – no corrosion was observed in the welds (arrows indicate the most likely places for corrosion to occur), d, e and f – corrosion sites under the paint film on the outer surface of the pipeline

4. Conclusions

- The calculated electric currents I_{peak} and IRMS of the supply and return pipelines in the building have an impact on the course of electrochemical phenomena, however, considering the strength of the calculated electric currents (mA), the applied frequency, and the prevailing voltages (mV), the impact on metal dissolution is extremely slow and practically insignificant.

- The calculated Langelier, Ryznar, Puckorius and Larson indices showed that there is a favorable environment for corrosion development in cold water pipelines, but no corrosion foci were observed on the inner pipe walls. Therefore, in this case, the corrosion of the cold-water pipeline was not caused by the chemical composition of the flowing water.

- Galvanic couples consisting of iron and copper alloys were found in the pipeline in service. This occurred when copper alloy

and steel structural components were joined together. The joining of copper components to steel causes moderate/severe corrosion. If it is necessary to join dissimilar metals, structural elements made of them must be insulated to avoid direct contact. Such recommendations are given in EN ISO 14713-1.

- When examining pipe samples metallographically, the number of non-metallic inclusions in the steel structure is small (1-2 score). No corrosion foci were detected at the roots of the pipe welds, but obvious corrosion was observed under the paint film.

- It was observed that the outer surface of the pipes under the thermal insulation was damp. It is unclear whether this was due to the condensation process or water leakage due to corrosion. However, it is recommended to assess the thickness of the thermal insulation layer considering the operating conditions. The methodology for calculating the thickness of the thermal insulation can be found in the EN ISO 12241:2022 standard. It is also recommended to check the thermal insulation for possible defects: unglued seams, cracks, poorly connected couplings, non-hermetic ends, slow air entry, insufficiently sealed tees, transitions, valves, etc., compressed thermal insulation due to brackets, etc.

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Concept of a Remote CNC Teaching Classroom for External Students in Manufacturing Technology Education

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Abstract: The transition toward distance and blended learning in engineering education has exposed a persistent gap in the delivery of practically oriented courses, particularly in the field of CNC programming, where hands-on interaction with machine tools is traditionally considered essential for developing professional competence. This paper addresses the lack of practical training in CNC programming within distance and blended learning formats and presents a proposed solution based on the establishment of a dedicated classroom equipped with CNC milling routers and online camera access, supplemented by multimedia teaching tools and a web portal for remote interaction and assessment. The proposed approach is positioned relative to three established families of methods for supporting practical CNC education, CNC simulation software, extended reality (XR) environments, and router-based teaching machines for CAM courses with the aim of identifying the relative advantages and limitations of each approach in terms of educational value, cost, scalability, and fidelity to real machining conditions. Particular attention is given to the integration of multi-layer automated evaluation mechanisms that support objective, efficient, and immediate feedback on student performance in CNC programming tasks. The presented concept is explicitly framed as a design-stage proposal and contributes to the modernization of higher education in computer-aided manufacturing technologies, providing a structured basis for subsequent pilot implementations and empirical evaluation in technically oriented study programs.

Keywords: DISTANCE AND BLENDED LEARNING, CNC PROGRAMMING, COMPUTER-AIDED MANUFACTURING

1. Introduction

Distance and blended learning formats have become an integral part of engineering education, driven by increasing student diversity, the need for flexible study arrangements, and the broader digitalization of higher education. In manufacturing-oriented programs, this shift raises specific challenges because many learning outcomes are tightly coupled to hands-on laboratory experience and direct interaction with physical equipment. Computer numerical control (CNC) programming and machining represent a prime example: beyond mastering G-code syntax and process planning, students must develop an understanding of machine setup, workholding, tool selection, process monitoring and safety, all of which traditionally rely on repeated practice at real machine tools.

To accommodate larger and more heterogeneous cohorts, universities and vocational institutions have increasingly complemented traditional laboratory teaching with simulation-based CNC training. Modern CNC simulators provide virtual control panels, graphical toolpath visualization, and three-dimensional representation of machining processes, allowing students to experiment with programs and process parameters in a safe, cost-effective environment [1]. These tools support distance learning scenarios by enabling learners to work on programming tasks without access to a physical machine, and they have demonstrated benefits in terms of accelerated skill acquisition and reduced risk of collisions and tool breakage during early training phases. At the same time, simulation environments inherently abstract away many sensory and contextual aspects of machining, such as chip formation, vibration, acoustic feedback, and real tool wear, which are important for developing professional judgement and tacit knowledge.

Extended reality (XR) technologies [2], virtual reality (VR) [3], augmented reality (AR), and mixed reality (MR) have gained attention as potential enablers of immersive CNC training, particularly in the context of Industry 4.0 and technical and vocational education and training (TVET) [4]. Systematic reviews of XR applications for CNC machine training indicate that VR and AR can enhance safety, support spatial understanding of machine setup, and reduce reliance on physical machines and expert mentors by allowing trainees to rehearse operations in virtual environments [5,6]. Experimental work on immersive VR error-management training for CNC milling setup shows that such environments can improve error identification and correction, increase learner engagement, and boost confidence compared to conventional

video-based instruction. In the broader TVET context, AI-ready VR machine workshops are proposed as a means to modernize CNC machining education and align pedagogical practice with digital manufacturing paradigms [4].

In parallel, router-based teaching machines have been designed and implemented as cost-effective platforms for computer-aided manufacturing (CAM) courses, providing students with access to three-axis CNC milling in an educational setting. Such systems demonstrate that CNC routers can serve as practical enablers of machining education, but they are typically used in conventional face-to-face laboratory formats and are not systematically integrated into distance-learning infrastructures or accompanied by automatic assessment mechanisms.

Despite these promising developments in simulation, XR and based teaching machines, there remains a gap between flexible remote access and authentic exposure to real machining conditions in regular university courses. Simulation software, although highly scalable and relatively inexpensive, does not provide direct experience with the mechanical behaviour of machine tools, workpiece setup and clamping, or the practical aspects of coolant, chip evacuation and tool life management. XR environments depend on specialized hardware and content development and are still limited in their ability to reproduce haptic feedback and process signals at engineering-relevant fidelity. Existing machines implementations, while valuable for on-campus teaching, do not address the specific constraints of external cohorts nor the need for integrated online assessment.

Against this backdrop, remotely accessible physical laboratories have emerged as a complementary approach in engineering education, enabling students to observe and sometimes control real equipment via networked interfaces and audiovisual streams. In CNC machining, pandemic-era initiatives showed that live camera feeds, screen sharing and interactive communication can provide realistic exposure to machine operation for geographically dispersed learners, but these arrangements were often ad hoc and temporary. Systematic designs of camera-instrumented CNC classrooms that are embedded into institutional distance-learning infrastructure, and that combine remote observation with structured automated assessment, are still rarely documented.

The present paper addresses this gap by proposing a concept for a specialized remote CNC teaching classroom built around educational three-axis milling routers, multi-camera online access, and a custom web portal with multi-layer automated assessment for

CNC programming tasks. The concept is developed for a bachelor-level course in programming of CNC machine tools for external students and is positioned relative to three established families of approaches: CNC simulation software, XR-based CNC training, and router-based teaching machines used in on-campus CAM courses. In contrast to prior router machines implementations, the proposed design explicitly targets distance and part-time cohorts, integrates remote video observation into the course workflow, and couples it with a structured automatic evaluation scheme.

The central research question is: “How can a remote CNC teaching classroom based on educational routers, online video transmission, and automated assessment support practically oriented CNC programming education for external students, while minimizing, but not eliminating the need for on-site laboratory exercises?”

The paper answers this question at the level of concept and design: it lays out the course context, the proposed hardware and software architecture, a five-stage workflow from home programming to on-site production-machine work, and a multi-layer assessment scheme, and it discusses anticipated benefits, limitations and future developments. The work is explicitly positioned as a design-stage proposal; pilot implementations and empirical evaluation are identified as future work rather than claimed as completed.

2. Background and related work

The teaching of CNC programming in manufacturing technology curricula has long relied on intensive laboratory practice at industrial machine tools, where students learn to translate drawings and CAD models into executable programs and observe the immediate consequences of their programming decisions on the machining process. Simulation tools and CNC training software have been widely introduced to support this process, enabling safe experimentation with G-code, toolpaths, and process parameters on standard PCs. These environments help reduce the risk of tool and machine damage in early training phases and allow for the repetition of typical programming scenarios without consuming material or machine time.

Extended reality technologies form a second major strand of work. Systematic reviews report that XR, including VR and AR, can support complex manual tasks, improve safety, and enhance learner engagement by providing immersive visualization and guided interaction. In CNC machining specifically, VR-based environments have been developed to teach machine setup and error management, showing that trainees can improve error recognition and confidence while retaining conceptual knowledge.

Remote and virtual laboratories represent a third relevant line of work. Virtual laboratories provide simulated experiments via software, while remote laboratories connect students to real equipment through network interfaces and audiovisual channels. In the CNC domain, pandemic-driven experiences with online teaching have demonstrated that streaming from shop-floor machines, combined with interactive discussion, can maintain some level of practical exposure when students cannot attend in person. These initiatives, however, were largely improvised responses to constraints and seldom evolved into systematically designed, permanent remote classrooms with integrated assessment.

Router-based teaching machines form a fourth strand. Designs of three-axis CNC routers for CAM courses show that such systems can deliver valuable hands-on experience at lower cost than full industrial machining centers, and they can be closely integrated with CAD/CAM workflows in educational settings. These implementations generally assume on-site student presence, and while they sometimes include software for toolpath verification, they do not typically address remote video integration or multi-layer automated assessment for distance learners.

The concept presented in this paper builds directly on this body of work. It adopts educational CNC routers as the physical machining platform, uses simulation tools as a preparatory step, and acknowledges XR and digital twins as promising future extensions. Its specific contribution lies in treating a multi-router, camera-instrumented classroom as a *remote physical resource* for external cohorts and in tightly coupling this resource to a web portal that provides automated, data-driven assessment of CNC programs.

3. Course context and educational requirements

The proposed concept is developed for a bachelor-level course titled *Programming of CNC Machine Tools*, delivered primarily to external (part-time) students within a manufacturing technology program. The course aims to equip students with practical skills in programming milling operations, understanding and using machine coordinate systems, and verifying and implementing CNC programs on real equipment. Students enter the course with basic computer literacy and introductory experience in CAD systems, which provides a foundation for linking geometric representation of parts to toolpath generation and G-code.

Key learning outcomes of the course are:

- to create CNC programs for standard milling operations on prismatic parts,
- to correctly apply workpiece and machine coordinate systems,
- to test programs using simulation tools and detect programming and logical errors,
- to implement and execute validated programs on CNC machine tools in a safe and controlled manner.

In the traditional delivery format, the practical part of the course has been realized first with CNC simulators and later with direct programming and operation of CNC machining centers in the university laboratory. External students, however, have limited access to these laboratories due to work commitments and geographical constraints, which restricts their opportunities to observe real machining processes and to link theoretical and simulated programming tasks to the behaviour of physical machine tools. This situation motivates the development of a remote teaching concept that maintains authentic exposure to machining while reducing the number of required on-site sessions.

4. Concept of the remote CNC teaching classroom

To address the constraints of external students while leveraging existing practices in CNC education, a dedicated remote CNC teaching classroom is proposed, built around educational CNC milling routers rather than full-scale production machining centers. Routers are chosen because their control systems are typically PC-based, which simplifies data transfer, screen sharing, and integration with a web portal, and because they are well suited for educational milling tasks on wood, plastics, or, in controlled cases, light metals.

In its initial configuration, the classroom is envisaged to host *four* CNC routers, each equipped with a camera system providing a live view of the machining process. The number four is treated as an illustrative starting point rather than a fixed optimum: it reflects a balance between the desire for multiple parallel demonstrations and the assumption that a single lecturer can feasibly supervise setup, workpiece exchange and monitoring across several routers within a session.

Each router is fitted with at least one camera focused on the cutting zone, with the option of additional views showing the overall machine workspace and the control panel. The camera feeds are connected to the web portal, which distributes synchronized video streams to external students during remote sessions. While detailed bandwidth and latency requirements will depend on chosen resolution and frame rate, the design assumes that the video subsystem must enable sufficiently smooth visualization of tool

motion, chip formation and coolant behaviour on typical home connections of external students; concrete performance targets are to be refined during pilot deployment.

The custom web portal serves as the central interface for external students. Through the portal, students can:

- access live video streams from the routers during scheduled remote practical sessions,
- upload their CNC programs for simulation and potential execution on the routers,
- receive structured feedback and assessment results for their programming tasks,
- complete auto-graded quizzes on theoretical aspects of CNC machining.

The physical handling of workpieces, fixtures, and machine setup remains the responsibility of the lecturer or technical staff on site, ensuring that safety is maintained and that the machines are operated within appropriate limits. External students thus interact primarily through the visual and informational channels provided by the portal and video streams, while still being able to see how their programs affect the real machining process. Viewed against previous three-axis router implementations for CAM courses, the novelty of this concept lies not in the router hardware itself but in its *systematic configuration as a remote resource* (multi-camera instrumentation, streaming integration with a portal) and in the *tight coupling with automated assessment workflows* that are designed from the outset to serve distance and part-time cohorts.

5. Proposed workflow and remote practical exercises

The teaching workflow is organized into several stages that combine simulation, remote observation, and limited on-site practice. This staged approach is designed to support external students, who cannot attend frequent laboratory sessions, while still guiding them from abstract programming concepts to real machining experience.

5.1. Program preparation at home

External students receive the design of a part (drawing or CAD model) and a description of the required milling operations, as part of regular assignments. Based on this, they prepare CNC programs using the designated control formats (e.g., Sinumerik, Heidenhain) and basic process planning guidelines covered in the lectures. The programming tasks are chosen to match the intended learning outcomes, focusing on contour milling, pockets, slots, and simple drilling sequences.

5.2. Program verification in simulation

Students test their programs using CNC simulators that provide graphical toolpath visualization and basic collision detection. The course has previously employed simulators for Sinumerik and Heidenhain controls, which allow students to verify syntax, see graphical representation of toolpaths, and check basic technological parameters. This step enables them to correct errors, adjust toolpaths, and refine process parameters in a risk-free environment, strengthening their understanding of coordinate systems and motion commands.

5.3. Submission and selection for remote execution

Verified programs are uploaded to the web portal, where they may undergo automated checks and preliminary assessment. The lecturer reviews the submissions and selects representative or particularly instructive programs to be executed on the CNC routers during the remote practical session. Criteria for selection may include diversity of approaches, common mistakes, or illustrative examples of good practice. Students whose programs are selected are informed in advance so that they can prepare to discuss their solutions during the session.

5.4. Remote observation of machining on routers

During scheduled online sessions, the lecturer prepares the routers (tool setup, workpiece clamping) and executes the selected student programs while sharing live video and, if needed, the control screen. External students connect through the portal to observe the machining process, listen to explanations, and discuss observed phenomena such as toolpaths, cutting conditions, surface quality, and the impact of programming decisions on process outcomes. The lecturer can pause the execution at key moments to highlight specific aspects (e.g., safe clearance moves, entry into material, transitions between roughing and finishing passes) and relate them back to the G-code segments. Students are encouraged to ask questions via chat or audio, and short formative questions can be posed to check understanding (for example, identifying the coordinate system in use, or explaining why a particular feed rate was chosen).

5.5. Final on-site exercises on production machining centers

To ensure that essential hands-on experience is not lost, the course includes one or two mandatory on-site laboratory sessions at production-grade CNC machining centers (e.g., DMG). In these sessions, students implement and adapt their programming skills in a real industrial environment, under direct supervision, consolidating the competencies acquired through simulation and remote observation. They perform basic setup operations, load and verify programs, and carry out machining tasks on metal workpieces, experiencing the differences in machine behaviour, safety procedures, and process constraints compared to the educational routers. The overall aim of this workflow is to minimize the number of on-site sessions required for external students while still providing a coherent progression from abstract programming concepts through simulation and remote visualization to authentic machining practice.

6. Concept of automated assessment and feedback

The web portal associated with the remote teaching classroom is designed not only as a communication and streaming platform but also as a tool for automated assessment of student CNC programs. The assessment concept builds on multiple layers, inspired by automatic grading approaches in programming courses and by assessment features in modern CNC training software.

6.1. Syntactic and structural validation

Submitted programs are automatically checked for syntactic correctness and for the presence of essential structural elements, such as safety blocks, tool definitions, coordinate system initialization, and proper program termination. This layer is intended to detect basic errors and provide immediate feedback, helping students avoid invalid or potentially dangerous code before simulation or machine execution, in line with experiences from automatic assessment in programming tools courses.

6.2. Simulation-based analysis of toolpaths and parameters

The portal then integrates with CNC simulation tools to evaluate the geometric and technological characteristics of the programmed toolpaths. Metrics may include collision-free motion, total path length and machining time, the distribution of rapid versus feed movements, and the plausibility of feed rates and spindle speeds relative to reference ranges. At the current conceptual stage, these metrics are envisaged to be combined into scoring schemes that reflect both correctness and efficiency of the programming solution; exact weighting and calibration against lecturer grading are reserved for future empirical work.

6.3. Comparison with reference solutions

For typical educational tasks, reference programs representing technically sound solutions can be prepared and maintained. Automated comparison between student and reference toolpaths and

simulated workpiece geometry is expected to allow quantification of deviations in contour accuracy, remaining material, or process time. In combination with logs from router executions and lecturer annotations, this is anticipated to support more consistent and transparent assessment while also generating learning-analytics data on error patterns and progression.

6.4. Integration with remote router execution

Programs that pass the simulated assessment may be marked as candidates for execution on the routers during remote sessions. The portal logs which student program is executed, and the lecturer can add qualitative annotations based on observed machining behaviour, such as surface quality, stability, or practical issues during setup. These annotations complement the automated scores and contribute to a richer feedback profile for each student. Such a combination of automatic scoring and lecturer judgement mirrors approaches used in interactive CNC training and assessment systems, which aim to standardize evaluation while still allowing expert input.

6.5. Automated quizzes and theoretical tests

In addition to program assessment, the portal can host auto-graded quizzes on theoretical aspects of CNC programming and machining (e.g., coordinate systems, G-functions, and safety principles). Immediate feedback on these quizzes supports self-paced learning and helps external students identify gaps in their understanding. Quiz performance can be correlated with practical programming results to gain insight into the relationship between theoretical knowledge and applied skills.

6.6. Anticipated benefits of automated assessment

By combining these layers, the automated assessment concept aims to: - provide external students with rapid, structured feedback on their programming performance, - reduce the manual grading workload of lecturers, - generate data for analysing learning progress and typical error patterns in CNC programming, - support fair and transparent evaluation across large cohorts. At the present design stage, these benefits are understood as anticipated outcomes that require validation in pilot implementations rather than as empirically established effects.

7. Discussion: benefits, limitations, and future developments

The proposed remote CNC teaching classroom and workflow offer several anticipated benefits for manufacturing technology education, especially in the context of external cohorts. For students, the concept is intended to increase accessibility to practically oriented learning activities by enabling them to prepare and test programs at home, observe real machining processes via online video, and receive systematic feedback without frequent travel to campus laboratories. For lecturers, the combination of simulation, remote router execution, and automated assessment is expected to provide a scalable way to manage larger groups, allowing them to concentrate limited on-site time on high-impact sessions at production machining centers rather than on basic programming practice. At the same time, important limitations must be acknowledged. Work on educational routers, even when observed remotely, cannot fully replicate the experience of programming and setting up industrial machining centers, where machine dynamics, safety considerations, and process constraints differ significantly. The concept therefore explicitly retains a minimal number of mandatory on-site laboratory exercises on production machines to ensure that students encounter real industrial conditions. Moreover, the quality of remote observation will depend on camera placement, video resolution, and network stability; these design parameters must be specified and validated during implementation, since inadequate video quality could undermine the pedagogical value of the remote sessions. From a methodological perspective, the current work is limited to concept and design. No empirical data on learning outcomes, student satisfaction, or grading reliability are yet available, and any claimed

benefits must therefore be treated as hypotheses rather than established findings. Planned next steps include pilot deployment with a small cohort of external students, collection of quantitative data (e.g., assessment scores, error rates, time-on-task) and qualitative data (student and lecturer feedback), and comparison with cohorts taught in purely on-site formats. Looking forward, the remote classroom concept can be extended toward more advanced digital manufacturing paradigms. One envisioned direction is the integration of virtual reality representations of the machine tools and the creation of digital twins of the routers and production centers, which would allow students to experience setup and operation in immersive environments while maintaining synchronization with real processes. Beyond machining, similar remote and virtual teaching workplaces could be developed for other manufacturing technologies such as forming, welding, and robotics, supporting a broader modernization of practical education in manufacturing technology programs.

8. Conclusions

This paper has presented a concept for a remote CNC teaching classroom designed to support practically oriented CNC programming education for external bachelor students in manufacturing technology. The concept combines educational CNC milling routers equipped with cameras, a custom web portal, CNC simulation tools, and a structured workflow that leads from program preparation and simulation through remote observation of machining on routers to focused on-site practice at production machining centers. An accompanying automated assessment framework is outlined, which is intended to provide immediate feedback on student programs and to generate data for analysing learning progress and typical error patterns. In contrast to prior work on simulation, XR training and router-based CAM teaching, the proposed design treats a multi-router, camera-instrumented classroom as a remotely accessible physical resource specifically tailored to external cohorts and tightly integrated with an online assessment portal. By reducing the reliance on frequent on-site laboratory sessions while maintaining authentic exposure to machining processes, the concept addresses a concrete challenge in delivering CNC programming courses to part-time and distance learners. At the same time, it is explicitly positioned as a design-stage proposal: empirical validation through pilot implementations and systematic evaluation remains future work. As such, the paper is intended to provide a structured starting point for institutions considering similar investments in remote machining infrastructure for manufacturing education. The authors anticipate that subsequent implementations and studies will refine the design parameters, validate the assessment mechanisms, and explore extensions toward digital twins and VR-based training environments in this domain.

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Welding in the Context of Industrial Development

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Abstract. Welding is a complex process governed by a multitude of factors. Additive manufacturing and Industry 4.0 or 5.0 require the acquisition of specific knowledge related to closed-loop welding processes, supported by highly qualified personnel. The shortage of specialized personnel within the EU is being addressed through the launch of innovative research projects in support of multidisciplinary and multinational consortia. Through consensus, these consortia develop curricula, courses, and evaluation criteria. These materials are disseminated to those interested in specializing as trainers of specialists or process operators, through platforms that are easy to access and use for self-training and for obtaining certificates that confirm the knowledge acquired. These self-training methods are accompanied by the creation of regional centers of excellence and by the launch of innovative ideas for practical implementation, including the creation of start-ups based on the knowledge acquired.

1. Introduction

Welding as a process for making permanent joints was introduced into the repair and manufacturing processes of structures required by the market at the beginning of the 20th century, in Europe and the USA [1,2] due to its net advantages over riveted joints, successfully contributing to the US efforts in World War II by significantly reducing the production cycle of Liberty ships.

During the Cold War, it developed in two directions depending on the interests expressed by the warring camps.

In the USA and its allied countries, welding was oriented towards the creation of structures applicable in the leading industries of aeronautics, energy production and automotive construction through the research and implementation in industrial practice of welding processes with wire and shielding gases of aluminum, stainless steels, super alloys, etc. or/and thin laminates under pressure in points or lines. In the socialist camp states, the development of welding was done for the creation of heavy machinery, which mainly favored the research and implementation in production of mechanized welding under flux layer, or in slag bath.

Current EU directives guide the development of welding through robotic and automated processes specific to production type 4.0 and/or 5.0 [3].

Studies conducted by IIW on the skilled workforce for the fields of the future have highlighted a major shortage of personnel, which has necessitated measures to be taken regarding the education and training of highly qualified personnel with high capabilities in supervising and managing the loop welding processes of structures made of various alloys and/or super alloys. This aim is achieved through the Erasmus Plus program and projects appropriate to the intended purpose of training highly qualified personnel and mentoring them and those involved in welding[4].

2. Knowledge to be acquired

To understand and master the complex processes of making permanent joints through welding, academic-level knowledge in the following disciplines is required:

- welding engineering of circular materials, basics of welding processes, AI-assisted welding;
- circular and advanced materials engineering, including welding filler materials;
- engineering of phenomena adjacent to welding processes;
- robotic welding engineering and/or loop welding in automated cells;
- advanced knowledge of AI algorithms regarding the implementation of innovative ideas in the form of start-ups;
- knowledge of the patenting processes of innovative ideas and their implementation in products created in start-ups;

Acquiring the knowledge necessary to implement industries 4.0 and/or 5.0 can be achieved through individual or team training on

specialized platforms in the field of welding, carried out within the Erasmus Plus program funded by the EU.

3. Structure of the knowledge training process.

The team of authors is involved in two types of projects: one dedicated to innovative learning through digital courses, dual schooling and case studies, structured on learning modules and knowledge test grids [4] with the possibility of mentoring and consulting at regional entities of excellence [4] logically followed by the second type of course with dissemination of knowledge of innovative practices and their implementation in industries 4.0 and 5.0 through the establishment of startups[4].

4. Future prospects

The near future perspective is to promote systemic change and integrate circularity in the development and demonstration of new solutions for digitalized welding manufacturing of socially responsible products that will ensure the transition to a sustainable economy. The future trajectory of advanced welding technologies will be shaped by the continuous integration of intelligent systems for the innovative development of specific welding manufacturing processes for large-scale and/or mass-produced products with high potential for reuse through repeated repairs and long service lives, under harsh operating conditions. In this sense, advanced welding technologies will be shaped by the continuous integration of artificial intelligence (AI) in the innovative development of new advanced processes and materials. AI and machine learning are about to revolutionize the welding industry by enabling predictive maintenance, real-time defect detection and adaptive parameter adjustment. AI algorithms have the ability to analyze vast data sets from past welding processes, allowing them to optimize future operations, predict results, and define benchmarks with remarkable accuracy.

Progress from manual welding, governed by many uncertainties generated by the human presence in the process, to automatic and/or robotic welding involves a set of best practice measures in human-machine collaboration.

The distant future perspective, driven by the need to combine new, diverse, intelligent and sophisticated materials, is a fundamental change in the welding process with self-optimization and reduced dependence on human intervention, leading to greater consistency of welding quality, minimizing material waste and maximizing production.

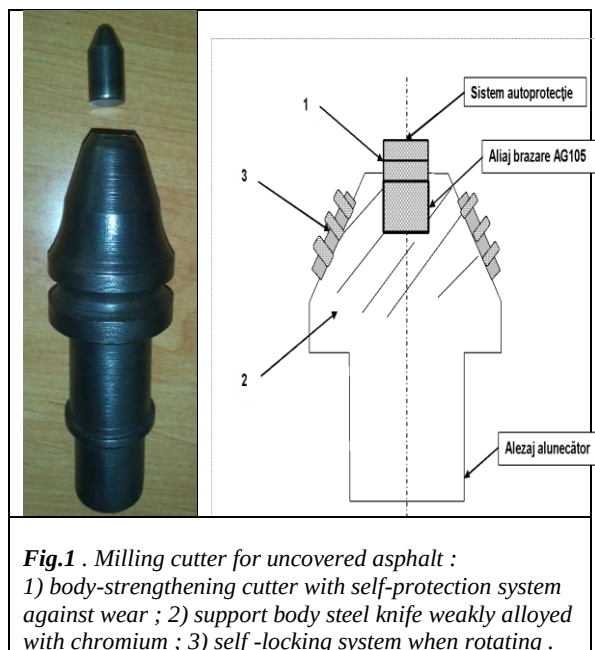
5. Case studies

To illustrate the above, two application examples are presented.

5.1 Asphalt stripping cutter [5]

The milling cutters for asphalt stripping are designed from the design phase for an extended lifespan, generated by a multiple cycle of reuse of the support by equipping it with an intelligent

self-locking protection system when rotating, consisting of a metallic deposit rich in angular tungsten carbides deposited by TIG welding and a tungsten carbide cutting body brazed into the support, figure 1, according to patent RO129863B1, figure 1.



After wear, the knife body is reused by equipping it with the previously mentioned protection systems.

5.2 Pressure and resistance spot welding electrode .

Resistance and pressure welding in spots or lines is a general utility process for thin-thick laminate joints.

The electrode, the active part of the welding process, figure 2, is made of copper alloys whose stock market price has increased dramatically in recent times, which is why the issue of reducing the cost price by assimilating new electrodes or a new process has arisen.



Fig.2 Electrodes for pressure and resistance spot welding

A car manufacturer turned to the robotic laser spot welding process, which required large investments in implementation and long payback periods.

The authors of the paper opted for the creation of a new, cheaper material with possibilities of industrial scaling. To this end, the operating conditions of resistance and spot welding electrodes and the innovative idea of a composite material were presented to AI, which was asked to develop a new material. The solution given by AI was experimented and optimized based on the criteria of the requirements of products with a long service life and reasonable cost.

Conclusions

Welding, a relatively new process, with application in mass production at the beginning of the Great War, has a major importance in the future due to the implementation of loop welding processes, AI-assisted and the requirements for sustainable development.

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Wood defects classification based on hyperspectral imaging

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Abstract: Wood processing requires reliable automated inspection and classification of natural defects. Conventional vision methods are often inadequate for distinguishing visually similar defects, particularly bark and resin pockets. This study compares hyperspectral methods for wood-defect classification. Pine samples were imaged in the VNIR (400–1000 nm) and SWIR (1000–2500 nm) ranges. Images were manually labeled for earlywood, latewood, sound and black knots, resin and bark pockets, and pith. Classification workflows combined raw spectra, PCA, or wavelet-based dimensionality reduction with Pearson correlation, k-NN, and Random Forest classifiers. The results enable comparison of the suitability of individual spectral ranges, feature-reduction methods, and classifiers for automated defect identification.

Keywords: hyperspectral imaging; wood defects; Scots pine; VNIR; SWIR; Random Forest; k-nearest neighbours; PCA; wavelet transform

1. Introduction

Quality control of wooden prefabricated components plays an important role in the furniture industry. It can be performed visually by production workers or automatically using appropriate measurement systems. Rising labor costs are driving the automation of successive production stages, including quality control.

The detection and classification of natural wood defects are essential aspects of quality control. Wood defects include:

- Sound knots – parts of living branches that are permanently fused with the surrounding wood; they are hard and well bonded to the wood structure [1]. They usually do not significantly reduce the material's strength, although they may affect its appearance. In many cases, they are not classified as defects.

- Black knots – remnants of dead branches surrounded by sound wood. They are characterized by dark coloration and weak bonding with the surrounding wood. They are often brittle and may fall out, reducing both the strength and surface appearance of the material.

- Knot holes – voids formed after black knots have fallen out. They constitute a significant defect, reducing the mechanical strength and visual quality of the component.

- Unsound knots – knots that are not fused with the surrounding wood and are loosely embedded. They can easily fall out during processing or use. A characteristic gap is visible around the perimeter of the knot.

- Resin pockets – elongated cavities filled with resin. They may cause resin leakage and problems during machining and surface finishing, for example during painting.

- Bark pockets – cavities filled with bark and surrounded by wood. They appear as irregular, often elongated, darker areas with properties different from those of the surrounding wood. In some cases, they are difficult to distinguish visually from resin pockets.

- Pith – the central part of a tree trunk. It is an elongated, spongy structure with mechanical properties inferior to those of wood. Small areas of pith can easily be mistaken for bark pockets.

- Decay – wood degradation caused by fungi. It manifests itself through a significant loss of strength as well as changes in color and texture.

- Blue stain – discoloration of wood caused by fungi. It does not cause substantial mechanical weakening, but it significantly reduces visual quality.

- Cracks – gaps caused by stress, most commonly during drying or service. They reduce the strength and aesthetic value of wooden components.

- Insect galleries – tunnels bored by insect larvae. They weaken the wood structure and may indicate active or previous pest infestation.

- Tannin stains – brown discolorations on the wood surface [2]. They result from oxidation of tannins contained in the wood following direct exposure to sunlight.

While some of the listed defects are very easy to detect and classify, such as knot holes, the classification of others can be problematic even during manual quality inspection. Resin pockets and bark pockets are examples of defects that may be difficult to distinguish. Therefore, automatic classification of wood defects is a non-trivial task.

Most solutions presented in the literature for wood-defect detection and classification are based on images acquired using RGB cameras and processed with appropriate algorithms.

Xu et al. [3] used a YOLOv5-C3Ghost model with a SimAM module to detect defects such as sound knots, dead knots, cracked knots, cracks, and resin pockets.

Rahiddin et al. [4] presented a method for recognizing wood defects using classical image-processing algorithms. Various variants of Local Binary Pattern (LBP) descriptors were combined with ANN, k-NN, and J48 classifiers. The algorithms were used to investigate defects such as knots, cracks, blue stain, tannin stains, bark pockets, insect galleries, decay, and wane. The highest accuracy, 63.4%, was achieved using the uniform LBP descriptor combined with an ANN classifier.

Chinese patent description CN110310259 [5] presents a knot-detection method based on an improved YOLOv3 algorithm combined with segmentation using the k-means++ algorithm.

The description of Polish patent application P.449481 [6] presents a method for detecting resin-related defects by illuminating a wooden component with a light source of a specified wavelength and measuring the light absorbance of the examined area.

An interesting solution was presented by Sandak et al. [7], who proposed a method for wood-defect recognition using a point spectrometer. Their best results were obtained with a MicroNIR spectrometer operating in the 950–1650 nm range and using classification based on the Support Vector Machine algorithm.

The authors of this work examined wood samples obtained from a furniture manufacturing plant using a hyperspectral camera. The outcome of the study is a set of spectral characteristics of wood defects, which may support the development of automated quality-control systems for wooden components.

2. Materials and methods

Measurements were conducted on a sample of 55 board sections made of Scots pine (*Pinus sylvestris* L.). Each section contained at least one defect. The boards were obtained from a furniture manufacturing plant.

Measurements were performed using a Headwall hyperspectral camera equipped with two sensors: VNIR and SWIR. The VNIR sensor records 370 spectral bands in the 400–1000 nm range and has a spatial resolution of 1600 pixels. The SWIR sensor records 170 spectral bands in the 1000–2500 nm range, with a spatial resolution of 384 pixels. The SWIR sensor requires active cooling provided by a Stirling cooler. During scanning, the samples were illuminated using a halogen light source. Sample movement was

provided by a motorized table integrated with the computer controlling the camera.

Before the measurements, both sensors were calibrated using black and white reference standards: a closed camera cover was used as the black reference, while the white reference material was supplied by the manufacturer. Due to the dimensions of the table, the samples were scanned in two batches, comprising 34 and 21 samples, respectively. During acquisition, the exposure time was 36 ms for the VNIR sensor and 3 ms for the SWIR sensor. The acquired images, visualized using pseudocolors, are presented in Figure 2. Hyperspectral images were saved in HDR format, with each pixel represented by a floating-point value in the range from 0 to 1.

Spatial resolution of VNIR sensor was reduced to adjust it to resolution of SWIR sensor. Hyperspectral cubes obtained from both sensors were merged into one hyperspectral cube.

As part of the study, two data dimensionality-reduction methods were tested. The first was Principal Component Analysis (PCA), with compression to 10 components applied. The second method evaluated was reduction using the wavelet transform. A fourth-order Daubechies wavelet and three decomposition levels were used. The results of both operations were saved as new ENVI images.

Pixel masks were created to assign image regions to individual wood classes or to defect-free wood. The masks were prepared manually in GIMP using pseudocolour representations of the hyperspectral images. Regions whose class assignment was uncertain, as well as markings present on the boards, were intentionally excluded from the labelled dataset. This conservative approach reduced the risk of introducing clearly ambiguous pixels into the reference data, although it did not eliminate all uncertainty associated with manual annotation.

The following classes were distinguished:

spring wood, late wood, sound knots, black knots, resin pockets, bark pockets, piths.

Based on manually labeled masks, reference data for the classification algorithms were prepared. For the Pearson correlation-based classification algorithms and the kNN method, individual reference spectra were created. To this end, the arithmetic mean pixel values were calculated for each spectral band, for each defect, for earlywood and latewood, and for the entire measured wood surface. For the random forest algorithm, reference classes were created as sets of pixel spectra corresponding to the respective masks.

This study investigated the potential of using three classification approaches—Pearson correlation, modified k-nearest neighbors (kNN) algorithm, and Random Forest—to analyze hyperspectral images for the purpose of wood defect detection.

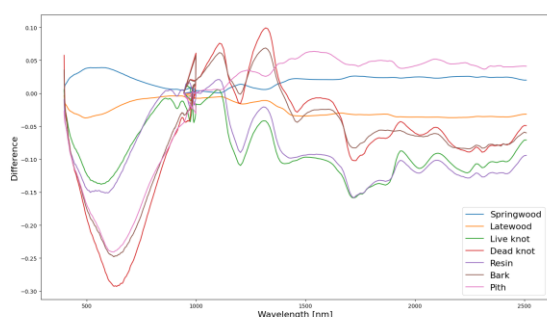


Fig. 1. Differences from the average spectral response

The first approach involved using an algorithm based on the calculation of Pearson correlation. Prior to classification, blank pixel elimination and filtering using a Savitzky–Golay filter were applied to reduce noise. The averaged spectrum of the entire surface of the analyzed wood was subtracted from both the spectra in the image and the reference class spectra. Differences between average classes spectra and average spectrum of wood are shown in Fig. 1. Furthermore, the algorithm was extended with a set of rigid physical rules applied before the classification stage. These rules eliminate classes that do not meet the specified amplitude and structural conditions. The following parameters were considered:

- minimum and maximum number of spectral intersections with the zero axis
- limits of positive signal integral value
- limits of negative signal integral value

Another approach involved the use of a modified kNN algorithm. Instead of the standard Euclidean distance, the similarity metric was defined as an arithmetic combination of the Pearson correlation coefficient and the Manhattan distance between the signals. In this case, Savitzky–Golay filtering and subtraction of the mean wood spectrum were also applied.

The third algorithm employed in this study was the Random Forest classifier, selected because of its ability to model complex and potentially non-linear relationships among hyperspectral features. The model hyperparameters were selected through a systematic tuning procedure using stratified cross-validation. Stratification ensured that the class distribution was preserved across the validation folds, which was particularly important when the numbers of samples representing individual classes were unequal. The optimization process aimed to maximize the macro-average F1 score.

All three classification algorithms were applied to all three prepared datasets (raw data, data reduced with wavelet transform, data reduced with PCA).

3. Results

Fig. 2 presents the classification results obtained using the Pearson correlation-based method, the modified kNN algorithm, and the Random Forest classifier for the raw hyperspectral data and for data reduced using wavelet transformation and PCA.

The obtained classification maps confirm that the selected wood structures and defects exhibit distinguishable spectral characteristics in the combined VNIR–SWIR range. The differences were particularly visible between earlywood, latewood, knots, bark pockets, resin pockets, and pith.

For the Pearson correlation-based method, the raw-data variant produced classification maps in which the main wood structures and larger defects were identified. The application of Savitzky–Golay filtering, subtraction of the mean wood spectrum, and additional physical rules reduced the influence of absolute brightness differences between samples. Nevertheless, the method was sensitive to local spectral variability, especially in areas containing knots, resin pockets, and transitions between earlywood and latewood. The wavelet-reduced data retained the general spatial distribution of the classes, although some local details were lost. In both datasets there was visible problem with false positive detection of big areas of piths. In case of PCA reduced data there were a lot of false recognitions of defects and sound wood.

The modified kNN algorithm provided results similar to Pearson correlation classifier.

The Random Forest classifier in combination with raw data produced the most coherent classification maps among the investigated approaches. Areas of false detected piths were significantly reduced. Instead of that there were small areas of false

detected bark. Classifications provided by Random Forest algorithm in combination with wavelet transform or PCA reductions were false in most of the area.

All examined algorithms have significant problem with distinguishing bark from dead knots and resin from sound knots. In is caused by similarity between spectral characteristics of those defects.

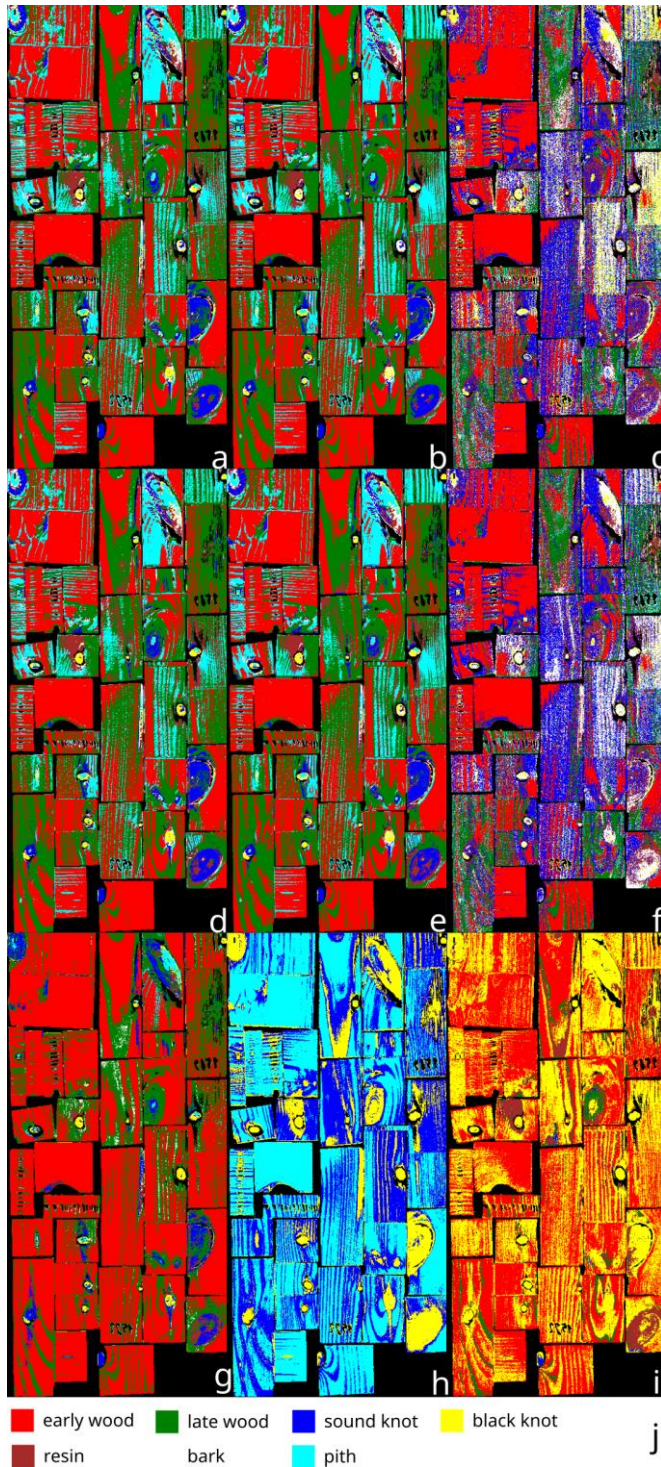


Fig. 2 Results of classification: a) Pearson correlation, raw data, b) Pearson correlation, wavelet transform, c) Pearson correlation, PCA, d) modified kNN, raw data, e) modified kNN, wavelet transform, f) modified kNN, PCA, g) Random Forest, raw data, h) Random Forest, wavelet transform, i) Random Forest, PCA, j) legend

4. Discussion

The results demonstrate that hyperspectral imaging in the combined VNIR–SWIR range can be used to distinguish selected wood structures and natural defects in Scots pine. In particular, the classification maps show that earlywood, latewood, knots, resin pockets, bark pockets, and pith produce spectral responses that are sufficiently different to enable pixel-wise classification. This confirms that hyperspectral data provide more discriminative information than conventional RGB imaging, especially in cases where defects have similar visible colour or texture.

Among the approaches investigated, Random Forest applied to the raw hyperspectral data produced the most coherent classification maps. In comparison with the Pearson correlation and modified kNN methods, it substantially reduced the false-positive assignment of large wood areas to the pith class. This result can be explained by the ability of Random Forest to model nonlinear relationships between spectral bands and to use multiple spectral features simultaneously. In contrast, the Pearson correlation method is based mainly on similarity of spectral shape and therefore may assign spectra with similar relative trends but different physical origin to the same class.

The modified kNN classifier produced results similar to those obtained using Pearson correlation. The combined similarity measure, based on correlation and Manhattan distance, did not provide a clear improvement over the correlation-based approach. This suggests that the remaining classification errors were not caused only by the choice of distance metric, but mainly by overlap between the spectral characteristics of some classes. In particular, bark pockets and black knots, as well as resin pockets and sound knots, were difficult to separate reliably.

The observed confusion between bark and black knots is consistent with their similar material composition and dark appearance. Both classes may contain degraded or bark-related tissue and can exhibit comparable spectral absorption characteristics. Similarly, resin pockets may be confused with sound knots because both are associated with resin-rich regions of wood.

The applied dimensionality-reduction methods had a significant influence on the classification results. Wavelet reduction retained part of the useful spectral information for the Pearson correlation and modified kNN approaches, although some local details were lost. PCA reduction resulted in a larger number of false recognitions for both defects and sound wood. This indicates that the first ten principal components, despite representing a large proportion of the total variance, did not necessarily preserve the spectral features most relevant for defect discrimination. PCA maximizes explained variance rather than class separability; therefore, spectral differences characteristic of small or infrequent defect classes may be suppressed.

A particularly important observation is that Random Forest performed well only when raw spectral data were used. Classification results obtained after wavelet transformation and PCA reduction were incorrect over substantial areas of the boards. This may indicate that the Random Forest model used information distributed across a larger number of spectral bands and was sensitive to the representation of the input data. Therefore, dimensionality reduction should not be assumed to improve classification performance automatically. For practical implementation, it is necessary to select the reduction method and its parameters jointly with the classifier.

The present study has several limitations. The masks were manually prepared and some ambiguous regions were excluded from the reference data. Consequently, the obtained maps should be interpreted as an assessment of the potential of the investigated methods rather than as a final industrial classifier. Moreover, the dataset contained only 55 board sections and the occurrence of individual defect classes was uneven. A limited number of pixels representing bark pockets, resin pockets, or pith may reduce the

ability of the classifiers to learn intra-class variability. Pixel-wise classification can also produce isolated erroneous pixels because it does not explicitly use spatial context.

5. Conclusions

Hyperspectral imaging in the combined 400–2500 nm range provides useful information for automated inspection of Scots pine boards. The investigated wood structures and natural defects exhibited sufficiently distinct spectral responses to support qualitative pixel-wise classification.

The best results in this study were obtained using a Random Forest classifier trained on raw VNIR–SWIR spectra. This approach generated the most coherent classification maps and substantially reduced false-positive pith detections. Pearson-correlation and modified kNN approaches were able to identify major structures and defects but were more sensitive to local spectral variability and produced more extensive false-positive regions.

Dimensionality reduction did not improve performance in the workflows investigated. Wavelet reduction retained part of the relevant information for correlation-based and kNN classification, whereas PCA led to many false classifications. Random Forest performed poorly when applied to the reduced datasets, indicating that raw spectra contained discriminative information that was lost or altered during reduction.

Reliable differentiation between bark pockets and black knots, as well as between resin pockets and sound knots, remains the main challenge. Further work should focus on expanding and balancing the reference dataset, improving ground-truth labelling, and integrating spatial, geometric, and textural information with spectral features. These steps may support the development of robust automated quality-control systems for wood-processing applications.

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Effect of density gradient on energy absorption properties of additively manufactured tpms-d infill pattern via material extrusion method

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Abstract:

As additive manufacturing (AM) technologies continue to gain traction across various industries, researchers have increasingly focused on optimizing the mechanical performance of polymer parts produced through material extrusion (MEX). Among the manufacturing parameters involved, infill pattern and density stand out as key factors that directly determine the mechanical behavior and energy absorption capacity of printed components. This study examines how different infill density gradients influence energy absorption, focusing specifically on samples built from polylactic acid (PLA) filament using a Triply Periodic Minimal Surface (TPMS-D) diamond pattern. TPMS-based infill patterns were selected for this study due to their potential to offer superior mechanical properties and energy absorption capacity compared to conventional two-dimensional infill patterns. The samples were subjected to quasi-static compression tests, from which stress-strain curves were obtained and energy absorption properties were calculated. The findings of this study are expected to inform the optimization of infill pattern and density parameters for energy-absorption applications, while also offering practical guidance for designing lightweight structures capable of withstanding high-energy impacts.

KEYWORDS: ADDITIVE MANUFACTURING VIA MATERIAL EXTRUSION, INFILL PATTERN, INFILL DENSITY GRADIENT, ENERGY ABSORPTION, TPMS PATTERN

1. Introduction

Triply periodic minimal surfaces (TPMS) are mathematically defined structures characterized by zero mean curvature at every point, repeating periodically along three Cartesian axes. Their smooth, non-self-intersecting topology enables systematic control over relative density and enhances load transfer efficiency. Among TPMS architectures, the Diamond (TPMS-D) structure has demonstrated superior load-carrying capacity and specific energy absorption compared to conventional lattice counterparts [1–5].

Material extrusion (MEX), also known as fused filament fabrication (FFF), is widely employed for fabricating TPMS structures due to its capability to produce complex geometries layer-by-layer. Recent studies have shown that TPMS structures fabricated via this method exhibit excellent compression performance and energy absorption capacity [3,4,6,7].

Introducing density gradients into TPMS architecture represents a promising strategy for enhancing mechanical performance. Gradient TPMS structures have been reported to exhibit superior energy absorption compared to homogeneous counterparts, particularly when the gradient direction aligns with the loading direction, as layer-by-layer deformation enhances energy dissipation [3,7].

Despite growing interest, the specific influence of density gradients on the energy absorption properties of TPMS-D structures fabricated via material extrusion remains insufficiently explored. This study aims to address this gap by systematically investigating the effect of density gradient configurations on the compressive and energy absorption behavior of additively manufactured TPMS-D infill patterns.

2. Material and Method

TPMS-Diamond infilled cubic specimens (25×25×25mm) were produced additively using a Creality K1 Max branded 3D printer, which works with the material extrusion method (MeX). Printing parameters were given in Table 1. Each sample coded using the

start and end infill density settings of the slicer gradient along the loading Z-axis (Table 2). Creality Print 7.2 was used for slicing.

Table 1. Printing parameters

Nozzle diameter	Layer height	Nozzle temperature	Bed plate temperature	Print speed
0.4mm	0.20mm	220 C	50 C	90 mm/min

Table 2. Nomenclature of samples

Sample code	Infill gradient	Infill setting (start → end)	Mean weighed mass (g)
1515	Uniform	15 → 15	3.07
2020	Uniform	20 → 20	4.48
2525	Uniform	25 → 25	4.67
3030	Uniform	30 → 30	4.75
4040	Uniform	40 → 40	5.08
8080	Uniform	80 → 80	6.64
9090	Uniform	90 → 90	7.25
2580	Graded	25 → 80	4.75
1590	Graded	15 → 90	4.72

Table 3. Organization of the nine configurations into four comparison sets

Set	Purpose	Configurations compared
I	Effect of gradient severity at constant mass	3030 (uniform), 2580 (graded), 1590 (graded)
II	Graded architecture versus the uniform counterparts of its gradient endpoints	1515, 1590, 9090
III	As Set II, for a milder gradient	2525, 2580, 8080
IV	Uniform architectures bracketing the Set I baseline	2020, 3030, 4040

Two layers of bottom and top layers were printed for each sample to obtain planar loading surfaces. After production, the samples were weighed using a precision balance to be used in calculating the specific mechanical property data. Then, compression tests were carried out on a UVE MNR100 testing machine with a crosshead speed of 20 mm/min. Using the data obtained from compression tests mechanical properties were calculated to evaluate the effect of density parameter. The purpose of the comparison sets with sample codes were given in Table 3.

3. Result and Discussion

3.1 Set I - Effect of gradient severity at constant mass (3030, 2580, 1590)

The aim of the set is to separate the effect of grading from the amount of material. Since the masses are approximately the same, all differences are due solely to the gradient. Obtained compressive response graphs and calculated mechanical properties of the Set are shown in Figure1 and 2.

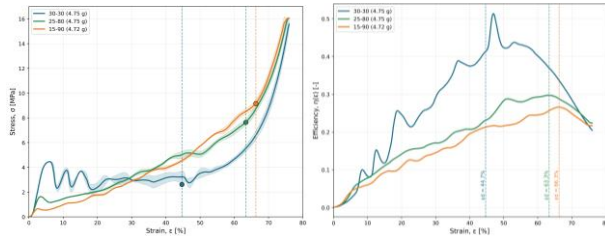


Fig. 1. Compressive response of the equal-mass configurations 30-30, 25-80 and 15-90 (Set I) a) Stress-strain graph, b) Energy absorption efficiency

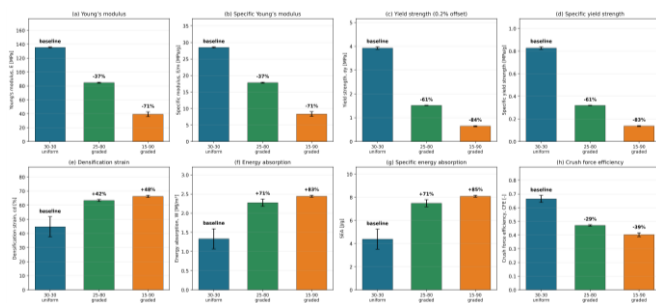


Fig. 2. Mechanical properties of Set I a) Young's modulus, b) Specific Young's modulus, c) Yield strength, d) Specific yield strength, e) Density strain, f) Energy absorption, g) Specific energy absorption, h) Crush force efficiency

Although grading significantly improved energy absorption, it caused deterioration in Young's modulus and yield strength. A moderate gradient reduced the elastic modulus by 37.4% and yield strength by 61.4%, while increasing SEA by 70.8%; a severe gradient reduced the elastic modulus by 71.1% and yield strength by 83.6%, while increasing SEA by 84.6%.

Similar findings regarding the compressive properties of lattice structures were also obtained by Lin et al and Distefano et al [4,8]. Interestingly, in their study, Silva et al. concluded that the use of cell thickness gradients can promote structures with higher rigidity and absorbed energy that can compete with traditional sandwich panel core structures [9].

Increasing the density transition from 2580 to 1590 resulted in a stiffness loss of 33.7 MPa while providing only a 13.8 J/g SEA gain. Therefore, 2580 emerged as a more efficient option in load-bearing applications.

When energy absorption behavior is examined regionally, it is seen that at low strain values, the uniform structure absorbs much more energy per unit mass, but the graded structures take the lead at approximately 50% strain. Therefore, concluding that "grading absorbs better" becomes meaningless without specifying the usable crushing distance.

The SEA scattering decreases from 19.7% to 4.1% and 1.0%. In the uniform structure, the collapse band starts where the worst defect is located, while in the graded structure it always occurs in

the weakest layer. This indicates that the uncertainty in deformation behavior is eliminated with grading.

3.2 Set II - The severe gradient against the uniform counterparts of its endpoints (1515, 1590, 9090)

The aim of the set was to test whether the 1590 gradient provided an intermediate value between uniform 1515 and uniform 9090. Obtained compressive response graphs and calculated mechanical properties of the Set are shown in Figure 3 and 4.

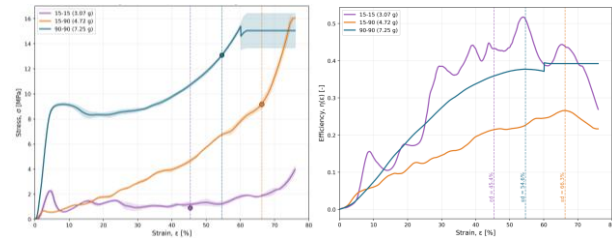


Fig. 3. Compressive response of the graded 15-90 architecture and the uniform architectures of its gradient endpoints, 15-15 and 90-90 (Set II) a) Stress-strain graph, b) Energy absorption efficiency

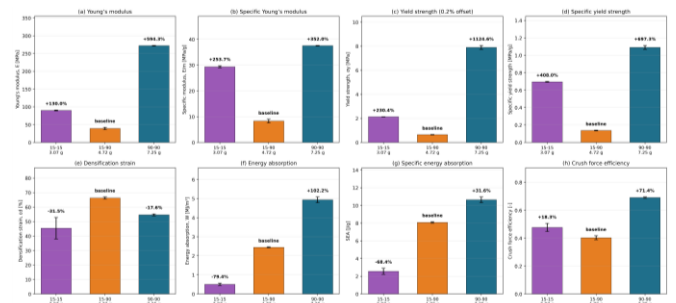


Fig. 4. Mechanical properties of Set II a) Young's modulus, b) Specific Young's modulus, c) Yield strength, d) Specific yield strength, e) Density strain, f) Energy absorption, g) Specific energy absorption, h) Crush force efficiency

The Young's modulus of the 1590 sample was obtained as 39.2 MPa, while the Young's modulus of uniform 1515 was 90.1 MPa. The uniform 1515 sample had 130% higher stiffness and 230% higher yield strength compared to the graded sample, while the graded sample was 54% heavier. This led to even greater increases in specific mechanical properties. However, if the structure in the weakest layers of these two samples is the same, similar behavior, especially in the elastic region, would be expected. This indicates that the mechanical behavior cannot be explained by the logic of series springs. Although analyses to determine the cause of this behavior – such as micro-CT – were not performed within the scope of this study, the possibilities of inadequate interlayer adhesion in the density transition and the rupture of the bond between cells can be evaluated.

Another finding is that the strong gradient eliminates the initial peak. While both uniform structures show distinct initial peaks, this behavior disappears in the graded sample. This indicates that gradient designs can be preferred depending on the need, since the peak force determines the acceleration transmitted to the body.

Unlike other uniform structures, the graded sample exhibited a monotonically increasing stress-strain curve; this resulted in a higher densification point.

If there is no mass constraint, it can be concluded that this behavior improves with increasing mass in terms of energy absorption capacity. This was observed in both energy absorption and specific energy absorption values.

3.3 Set III - The moderate gradient against the uniform counterparts of its endpoints (2525, 2580, 8080)

The configurations examined under Set III were evaluated to validate the severe gradient effects observed in Set II under a more moderate gradient profile. Obtained compressive response graphs and calculated mechanical properties of the Set are shown in Figure 5 and 6.

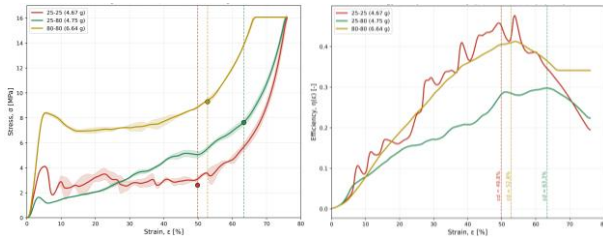


Fig. 5. Compressive response of the graded 25-80 architecture and the uniform architectures of its gradient endpoints, 25-25 and 80-80 (Set III) a) Stress-strain graph, b) Energy absorption efficiency

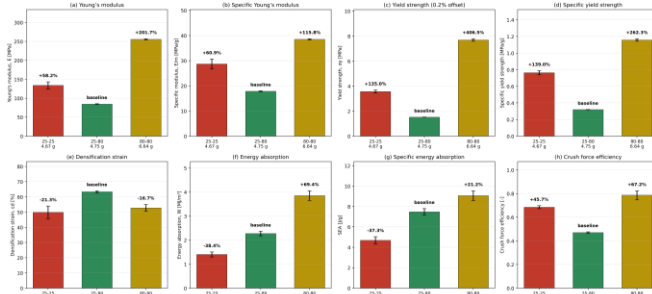


Fig. 6. Mechanical properties of Set III a) Young's modulus, b) Specific Young's modulus, c) Yield strength, d) Specific yield strength, e) Denatification strain, f) Energy absorption, g) Specific energy absorption, h) Crush force efficiency

The tendency for stiffness and strength loss was also maintained in the moderate gradient series. The 8080 configuration exhibited a 201.7% decrease in elastic modulus and a 406.5% decrease in yield strength compared to the 2580 gradient. It was determined that the stiffness difference loss increased as the gradient width increased. This consistent trend obtained in two independent sample sets and at different mass levels indicates that the performance loss is systematic behavior [8].

Unlike the severe gradient structures, it was found that the moderate gradient profile maintained a distinct stress peak. When evaluated together with the Set I results, increasing the gradient amplitude does not eliminate the stress peak alone, but dampens it above a certain critical threshold. This indicates the existence of a threshold value between mechanisms for peak mitigation and complete elimination.

The uniform 8080 configuration yielded the most stable mechanical response throughout the experimental study. The structure exhibited a Crush Efficiency (CFE) of 0.787 and a plateau stress dispersion of only 9.2%. Within the evaluated strain range, it was observed that the thick cell walls deformed via a plastic deformation mechanism rather than brittle buckling.

3.4 Set IV — Uniform architectures around the Set I baseline (2020, 3030, 4040)

Set IV (2020, 3030, and 4040) was investigated to verify the representativeness and boundary conditions of the 3030 cellular configuration, which was considered the basic reference in Set I analyses. Obtained compressive response graphs and calculated mechanical properties of the Set are shown in Figure 7 and 8.

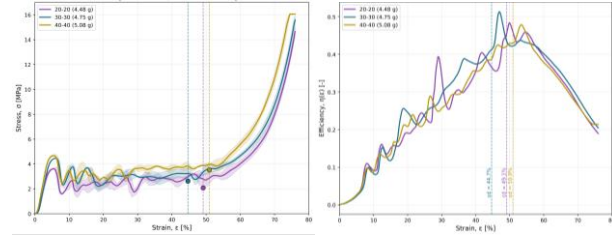


Fig. 7. Compressive response of the uniform architectures 20-20, 30-30 and 40-40 (Set IV) a) Stress-strain graph, b) Energy absorption efficiency

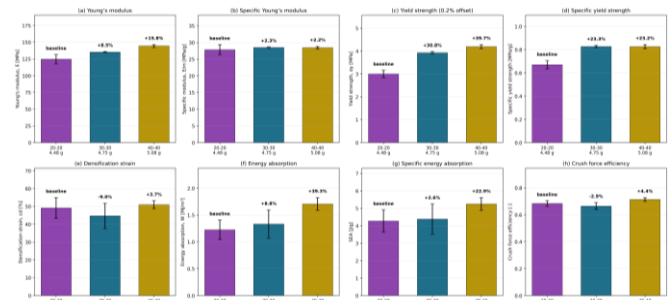


Fig. 8. Mechanical properties of Set IV a) Young's modulus, b) Specific Young's modulus, c) Yield strength, d) Specific yield strength, e) Denatification strain, f) Energy absorption, g) Specific energy absorption, h) Crush force efficiency

It was determined that the 3030 configuration exhibits a monotonic transition between neighboring configurations (2020 and 4040) in terms of elastic modulus, yield strength, and specific modulus values. This confirms that the relationships observed in Set I are not based on a biased reference point. However, the 3030 structure remained somewhat lower than the other samples in terms of densification strain.

In the mass range of 4.48 g to 5.08 g (transition from 2020 to 3030), specific stiffness and specific yield strength showed an increase of 2.3% and 23.3%, respectively. However, in the range of 5.08 g to 7.25 g (from 3030 to 4040), the specific modulus and specific yield strength values remained constant within certain limits, forming a plateau. This indicates saturation in specific mechanical parameters at a certain value and shows that the added material does not contribute to the specific mass efficiency.

It was determined that the configuration with the highest density does not always offer the most efficient mechanical behavior. Plateau uniformity reached the optimum level at 6.64 g mass with 9.2%, then decreased to 13.9% at 7.25 g mass; Simultaneously, the CFE value also decreased from 0.787 to 0.690. This performance loss was accompanied by an increase in the ratio of stress to plateau stress in the densification region from 1.20 to 1.33. It is assessed that the low internal void volume in the high-density configuration compresses the cell wall contacts into a narrower strain range, leading to early densification hardening.

4. Conclusion

In this study, the mechanical response and energy absorption characteristics of additively manufactured PLA components with uniform and functionally graded TPMS-Diamond infill patterns were systematically investigated through quasi-static compression tests. Based on a four-set comparative experimental framework, the principal findings are summarized as follows:

- At constant mass, density grading significantly enhances energy absorption capacity at the expense of elastic modulus and yield strength. A moderate gradient (2580) increased specific energy absorption (SEA) by 70.8%, whereas a severe gradient (1590) yielded an 84.6% increase in SEA compared to uniform counterparts. However, uniform architectures consistently exhibited higher load-bearing capacity and stiffness than graded structures of equivalent average relative density.
- Suppressing the initial stress peak requires exceeding a critical gradient severity threshold. While the moderate gradient (2580) dampened the peak stress by 60.2% (retaining a minor overshoot at $\epsilon \approx 3.4$, the severe gradient (1590) eliminated the stress peak, yielding a monotonic, stable plateau. Furthermore, grading transformed the stochastic collapse typically observed in uniform lattices into a deterministic, layer-by-layer progressive deformation, dramatically reducing the coefficient of variation in SEA from 19.7% to 1.0%.
- The elastic modulus of graded architectures unexpectedly fell below that of the uniform architecture representing their lowest gradient endpoint (by 56.5% for 1590 and 36.8% for 2580). This reproduced phenomenon indicates that global stiffness in graded MEX structures cannot be modeled simply as springs in series, likely due to localized stress concentrations and inter-cell boundary compliance in low-density transition layers.
- Mass efficiency within uniform TPMS-D structures exhibits non-monotonic behavior. Specific stiffness and specific strength saturate in the medium-density range (4.48–5.08 g), whereas maximum structural performance, plateau stability, and crush force efficiency (CFE = 0.787) peak at 6.64 g (80–80 setting). Beyond this point (90–90 setting), early cell-wall contact accelerates densification hardening and degrades CFE to 0.690.

Future work should prioritize examination of the gradient transition zones and the introduction of intermediate gradient severities to resolve the peak-suppression threshold.

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Предложена е методика за проектиране на основните геометрични параметри на дюзи съдържащи отвори с криволинейна спираловидна ос при отчитане на напреженията в заваръчната тел с оглед на избягване на пластично деформиране на телта и прекомерното нарастване на фрикционните сили в отвора на дюзата.

При началните експерименти заготовки с диаметър $D_0=\Phi 50\text{mm}$ и дължина $L=115\text{mm}$ с диаметър на отвора $d_0=\Phi 12,5\text{mm}$ и ексцентрицитет $E=D/4$ и $E=D/8$ след запълване със сол NaNO_3 бяха екструдирани с помощта на инструмент за двустъпална екструзия [2] до крайни продукти с диаметър $D=5,85\text{mm}$ и с отвори с диаметър $d=1,45\text{mm}$ и с ексцентрицитети $e=3,1\text{mm}$ и $1,6\text{mm}$. Прътите бяха насукани за да се получи стъпка на винтовата линия $t=28\text{mm}$, след което от тях бяха изработени дюзи с дължина $l_d=30\text{mm}$. Беше установено, че подходящата тел $d_{\text{тел}}=1,2\text{mm}$ за получените дюзи ($d_0=1,45\text{mm}$) не преминава през техния отвор. Тел с диаметър $\Phi 1,0$ преминава със много голямо съпротивление, като дори получава пластично деформиране. Това я прави също непригодна за заваряване. В получените дюзи тел с диаметър $\Phi 0,8$ (при луфт $\Delta=0,65\text{mm}$) преминава с слабо съпротивление близо до това, с което тел $\Phi 0,8$ преминава през конвенционалната дюза с отвор $\Phi 1,0$ (при луфт $\Delta=0,2\text{mm}$). Опитите с изработените дъзи със тел $\Phi 0,8\text{mm}$ показаха, че заваръчния процес не протича нормално – заваръчната дъга прекъсва, получава се характерно „пукане“ и се генерират много пръски). Описаните резултати, недвусмислено потвърди

изводите от [10], отнасящи се до това, че при дюзите със спираловиден отвор при даден диаметър на тела $d_{\text{тел}}$ параметрите на дюзата (дължина l и характеристиките на отвора: диаметър d , ексцентрицитет e и стъпка на винтовата линия t) влияят съществено върху нейната технологична пригодност и качества. Това обосновава целесъобразността от провеждането на изследвания на влиянието на посочените параметри върху технологичните качествени показатели на дюзата и обоснова целесъобразността от търсенето на подходящи инженерни подходи и набор от параметри за геометричния и силов анализ на двойката дюза-заваръчен тел. Наред с това да се търси възможност за създадена на методика за проектиране на основните геометрични параметри на дюзи съдържащи отвори с криволинейна спираловидна ос, при отчитане напреженията в заваръчната тел с оглед избягване пластичното деформиране на телта и прекомерното нарастване на фрикционните сили в отвора на дюзата.

За целите на анализите са приети означения за параметрите на дюзите и за някои величини характеризиращи двойката тел-отвор, които са посочени в табл.1,

Таблица 1.

Означения	Параметри и величини
$d_{\text{отв}}=2.r_{\text{отв}}$	диаметър на отвора на дюзата
$d_{\text{тел}}=2.r_{\text{тел}}$	диаметър на телта
$\Delta = d_{\text{отв}} - d_{\text{тел}}$	луфт
e	ексцентрицитет на отвора
$l_{\text{дюза}}$	дължина на дюзата
R	радиус на винтова линия
t	стъпка на винтова линия
α	ъгъл на наклона на винтова линия
R_{max}	максимален радиус на винтовата линия на оста на телта – при външен контакт
R_{min}	минимален радиус на винтовата линия на оста на телта – при вътрешен контакт
$d_{\text{св}}=2.r_{\text{св}}$	диаметър на светлото сечение на отвора на дюзата ($d_{\text{св}} < d_{\text{тел}}$)
ρ	радиус на кривина на винтова линия
$\sigma_{\text{ог}}$	огъващо напрежение в телта
E	модул на еластичност на материала на телта

В табл.3.2 са приведени зависимостите, по които се определят основните геометрични и силови параметри на двойката отвор на дюзата-тел (фиг.1 и фиг.2). В същата таблица са дадени и някои пояснения за параметрите, за зависимостите и за величините които влизат в тях. Може би е целесъобразно да бъдат дадени и някои допълнителни пояснения :1. Ако неравенство №1 не е изпълнено светлото сечение на отвора $d_{\text{св}}$ ще бъде по-голямо от диаметъра на телта $d_{\text{тел}}$, следствие на което една тел с праволинейна ос ще преминава с хлабина през отвора и няма да приема формата на винтова линия. 2. В едно напречно сечение оста на телта е на разстояние R ($R_{\text{min}} < R < R_{\text{max}}$)

R_{max}) от оста на дюзата (т.е. на винтовата линия). R_{max} и R_{min} съответстват съответно на максималния и на минималния радиус на разположение на оста на телта. Това дефинира граничните винтови линии на телта. 3. ρ_{min} и ρ_{max} са граничните стойности на радиусите на кривина на винтовата линия на оста на телта. 4.Максималната стойност ρ_{max} дефинира минимално възможна стойност на огъващото напрежение в телта определена с уравнение №11. Това съответства на минимум на еластичния потенциал на деформираната тел т.е. на реалния контакт на телта в отвора на дюзата.

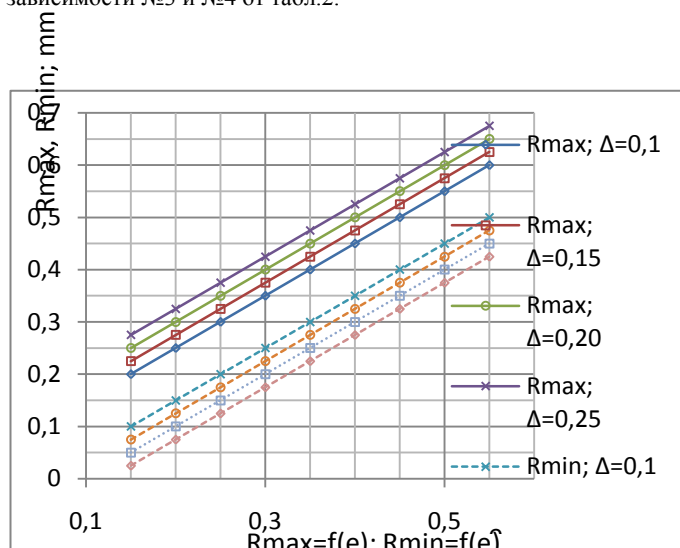
Табл.2

№	Зависимост	Пояснение	Графики
1	$e > \Delta/2$	Условие оста на телта да приема форма на винтова линия.(следва от условието: $r_{\text{св}} = r_{\text{отв}} - e < r_{\text{тел}}$);	
2	R ($R_{\text{min}} < R < R_{\text{max}}$)	Радиус на разположение на оста на телта (отстояние на оста на телта от оста на дюзата)	
3	$R_{\text{max}} = e + \Delta/2$;	Максимален радиус на разположение на оста на телта	Фиг.3-3
4	$R_{\text{min}} = e - \Delta/2$	Минимален радиус на разположение на оста на телта	Фиг.3-3
5	$\text{tg}\alpha = t/2.\pi.R$. ($\alpha_{\text{min}} < \alpha < \alpha_{\text{max}}$)	α - ъгъл на наклон на винтовата линия	
6	$\text{tg}\alpha_{\text{max}} = t/2.\pi.R_{\text{min}}$.	α_{max} - максимално възможен ъгъл на наклон на винтовата линия	
7	$\text{tg}\alpha_{\text{min}} = t/2.\pi.R_{\text{max}}$.	α_{min} - минимално възможен ъгъл на наклон на винтовата линия	
8	$\rho = R.(1 + \text{tg}^2\alpha) = r.[1 +$	Радиус на кривина на винтовата линия	

	$(t/2 \cdot \pi \cdot r)^2]$		
9	$\rho_{\min} = R_{\max} \cdot (1 + \operatorname{tg}^2 \alpha, \min) = R_{\max} \cdot [1 + (t/2 \cdot \pi \cdot R_{\max})^2]$	Минимално възможен радиус на кривина на винтовата линия	
10	$\rho_{\max} = R_{\min} \cdot (1 + \operatorname{tg}^2 \alpha, \max) = R_{\min} \cdot [1 + (t/2 \cdot \pi \cdot R_{\min})^2]$	Максимално възможен радиус на кривина на винтовата линия	
11	$\sigma_{\text{ог}} = E \cdot r_{\text{тел}} / \rho_{\max}$	Огъващо еластично напрежение в телта. Произтича от $\sigma_{\text{ог}} = E \cdot \varepsilon$; $\varepsilon = \Delta l / l_0$; $\Delta l = l - l_0$; $l = 2 \cdot \pi \cdot (\rho + r_{\text{тел}})$; $l_0 = 2 \cdot \pi \cdot \rho$	

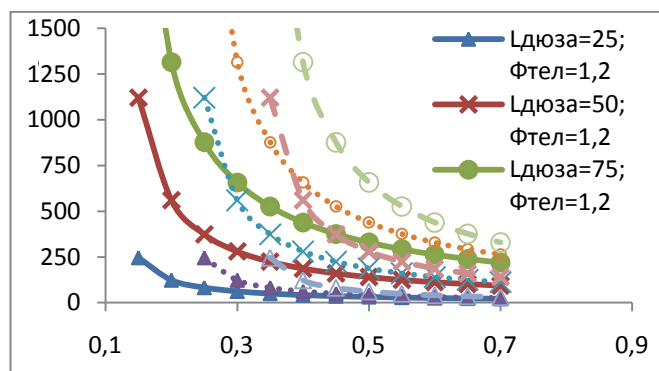
По долу е направна графична интерпретация на зависимостите от табл.2 (фиг.3 до фиг.5), като при пресмятанията е прието: 1)луфт отвор/тел $\Delta=0,2\text{mm}$; 2) $E=2.10^5\text{MPa/m}^2$; 3)телът се деформира еластично; 4) $\rho = \rho_{\max}$ изпълнено е условието на min на потенциалната енергия на еластичната напрегнатост (на еласт. деформация). Обхванати са следните дюзи и телове: $d_{\text{тел}}=0,8; 1,0; 1,2$; $L_{\text{дюза}}=25-75$; $\varepsilon=0,1$ до $0,7$; $D_{\text{дюза}}=5,85$.

На фиг.3 е направена графична интерпретация на зависимости №3 и №4 от табл.2.

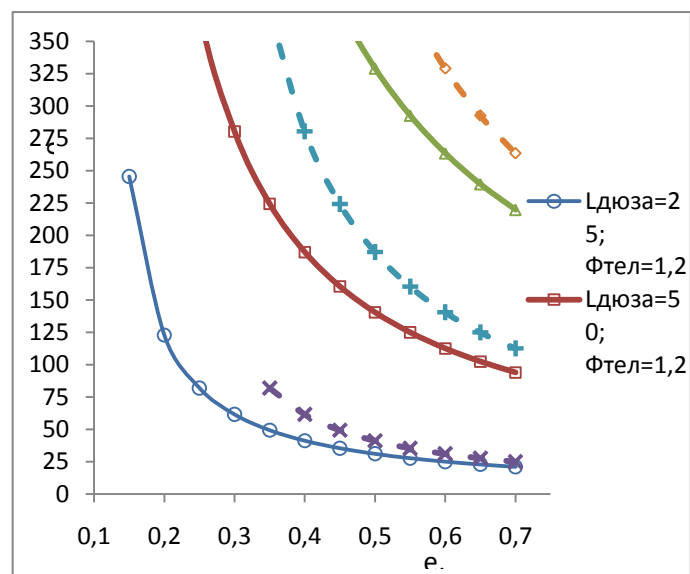


Фиг.3.

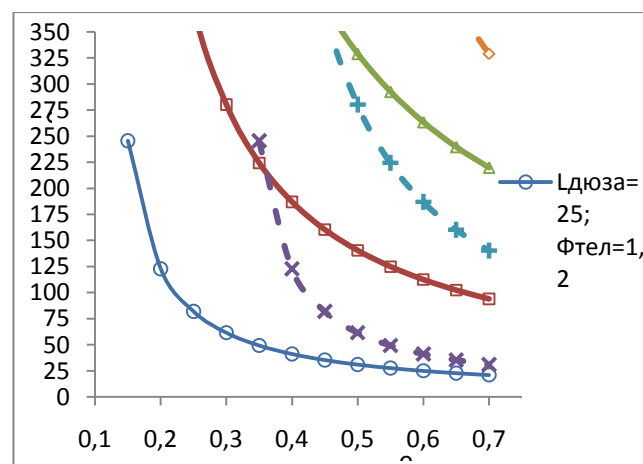
На фиг.4а-г са представени зависимостите на $\rho_{\max}$ (по ординатата $\rho_{\max}$ е означена с ρ) от ексцентрицитете e за различни диаметри на телта и дължина на дюзата, която е равна на стъпката на винтовата линия. Вижда се, че с нарастване на e и с намаляване на $L_{\text{дюза}}$ $\rho_{\max}$ намалява.



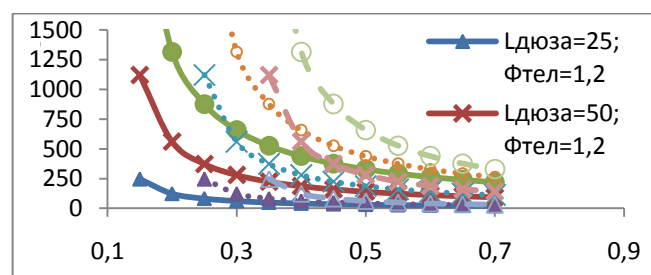
фиг.4а.



фиг.4б.

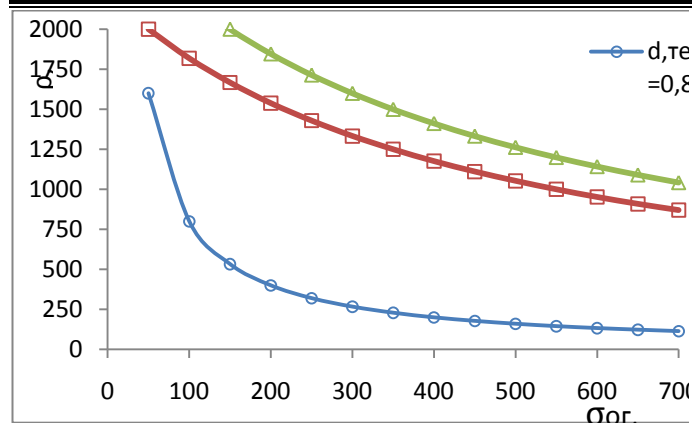


фиг.4в.

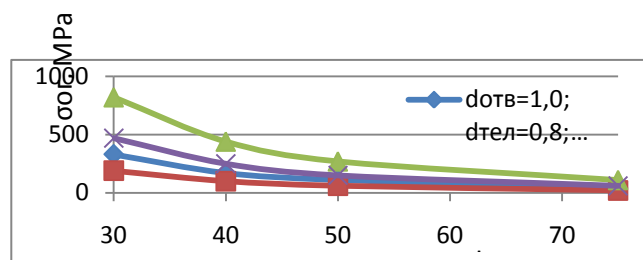


фиг.4г.

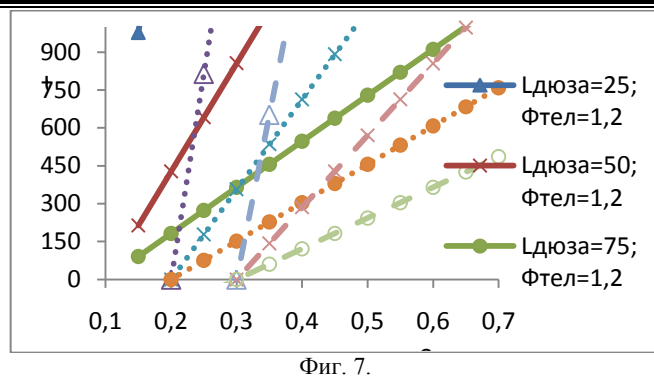
На фиг.5, фиг.6 и фиг.7 са представени съответно зависимостите на $\sigma_{\text{ог}} = f(\rho_{\max})$ /по абсцисата $\rho_{\max}$ е означена с ρ , $\sigma_{\text{ог}} = f(L_{\text{дюза}})$ и $\sigma_{\text{ог}} = f(e)$.



Фиг.5.



Фиг.6.



Фиг. 7.

Зависимостите от табл.2 и графичната им интерпретация от фиг4 до фиг7 дават възможност при зададени изходни параметри $d_{\text{тел}}$, $L_{\text{дюза}}$, $\sigma_{\text{ог}}$ Е да се определи ρ от зависимост №11, респективно фиг.5. След това на базата на зависимости №10 и 4 (фиг4а/г) се подбират основните параметри на дюзата e и t .

Използвайки тази методика за пресмятане на геометричните параметри на дюзите, съобразно допустимите напрежения на огъване, са построени графиките от фиг3-6 до фиг7. На тяхна база са подготвени експериментални дюзи, чиито размери (наред с размерите на заготовките) са дадени в табл.3.

Табл.3 (размери в mm)

№	Dзаг	Lзаг	Dотв.	Eзаг.	Dдюза	Lдюза	dотв.	dтел.	e	t
1	50	115	9	1,50	5,85	30	1,00	0,8	0,18	30
2	50	115	11,2	1,85	5,85	30	1,25	1,0	0,22	30
3	50	115	10,85	2,15	5,85	30	1,20	1,0	0,25	30
4	50	115	10,8	1,15	5,85	30	1,20	1,0	0,13	30

При изпитвания в лабораторни условия на дюзите от табл3 имаше големи отклонения в качеството на заваръчния процес. Това се дължи на голямата чувствителност на този процес към геометричните параметри на дюзите. При преминаване на тела през дюзите с $d_{\text{тел}}=1,0\text{mm}$ в някои от случаите възникваха големи съпротивления и заваръчния процес прекъсваше. При $d_{\text{тел}}=0,8\text{mm}$ преминаването беше с голяма хлабина, което отново не беше благоприятно за заваръчния процес. Опитите със спиралните дюзи показаха, че дължината на дюзата трябва да е по-голяма от наличната и използвана при експеримента апаратура - $L_{\text{дюза}}=30\text{mm}$. С увеличаване на дължината на дюзата ще се получи възможност за увеличаване на радиуса на квинта ρ т.е. до намаляване на огъващите напрежения $\sigma_{\text{ог}}$ на заваръчния тел, при намалена хлабина между отвора и тела. От това ще следва по добро токоподаване и по качествен заваръчен процес.

ЗАКЛЮЧЕНИЕ

Предложена е методика за проектиране на основните геометрични параметри на дюзи съдържащи отвори с криволинейна спираловидна ос при отчитане на напреженията в заваръчната тел с оглед на избягване на пластично деформиране на телта и прекомерното нарастване на трикционните сили в отвора на дюзата.

Многопараметричността на процеса на заваряване изисква извършването на извънредно голяма по обем експериментална работа. Може да се твърди, че постигнатите в настоящата разработка резултати са необходима основа за извършване на бъдещите изследователски дейности по създаване на оптимални конструктивни решения на дюзи със спирална форма на отвора.

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Covered electrode selection for mma, optimal operating parameters and risk of cold cracking (hacc – hydrogen-assisted cold cracking)

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Abstract: This paper examines the critical criteria for selecting covered electrodes in manual metal arc (MMA) welding, focusing on achieving metallurgical compatibility with the base material and preventing welding defects. The performance characteristics, polarity requirements, and specific applications of major coating types—namely rutile, basic/low-hydrogen, and cellulosic electrodes—are evaluated alongside specialized consumables for stainless steels, cast iron, and hardfacing. Special attention is dedicated to the phenomenon of Hydrogen-Assisted Cold Cracking (HACC). Finally, the paper introduces the "Electrode advisor" digital application. By incorporating multi-criteria input parameters—including base material, geometry, thickness, welding position, power source polarity, and HACC risk factors—the software automates the selection of electrode classification, optimal diameter, and operating current. The integration of this application standardizes engineering decisions, streamlines quality assurance documentation, and significantly mitigates the risk of catastrophic weld failure in structural and marine applications.

Keywords: MMA WELDING, COVERED ELECTRODES, ELECTRODE SELECTION, HACC, HYDROGEN-ASSISTED COLD CRACKING, RUTILE, LOW-HYDROGEN, ELECTRODE ADVISOR APPLICATION.

1. Introduction

Covered electrodes are the main consumable for manual metal arc (MMA) welding. The coverings perform numerous technological and metallurgical functions, some of which are: easy striking and stable burning of the arc, creation of reliable slag and gas protection, modification and alloying of the weld metal, deoxidation, control of the cooling rate and many others [13]. The core wire of the electrode must correspond to the metal of the parts being welded and must provide the weld with mechanical and ductile properties that are not worse than those of the base metal.

"Correct selection" means the highest possible match between base material and weld metal (strength, hardness, elongation, impact toughness, corrosion resistance, etc.) and the absence of welding defects (cold cracks, undercuts, notches, bridging effect, ...). This depends on the correctly chosen: welding equipment (AC/DC), welding procedure (diameter and chemical composition of the electrode, welding current, arc length, travel speed, welding position, polarity), and control of diffusible hydrogen (properly selected electrode) [10], [6].

Covered electrodes for carbon (mild) steels are classified according to ISO 2560:2020 (system A/B by strength, impact toughness, type of coating and recovery) [1], [9], and for stainless steels – according to ISO 3581 (chemical composition of the deposited metal and type of coating) [2], [12].

The American AWS specifications use alphanumeric indices (e.g. E7018: E – electrode for manual metal arc welding; "70" – tensile strength 70 kpsi; "1" – all welding positions; "8" – basic/low-hydrogen coating and characteristic current/polarity) [10]. General terms and definitions are codified in AWS A3.0M/A3.0 [13].

2. Types of coatings and performance characteristics

Rutile (R; often E6013) – characterized by easy arc striking, smooth bead surface; suitable for thin sections in all welding positions, including vertical-down [8], [16].

Basic/low-hydrogen (B; E7018/E7016) – provide low diffusible hydrogen and high impact toughness; preferred for thick sections, shipbuilding/structural steels and low service temperatures [7], [12], [6].

Cellulosic (C; E6010/E6011) – deep penetration and high travel speed; typical for pipe root passes and site erection work [16], [10].

Special purpose – for stainless/heat-resisting steels to ISO 3581 [2], [12]; for hardfacing; for cast iron (Ni/NiFe) in cold / semi-hot welding.

3. Polarity and power source

Polarity affects the stability of the arc and the penetration capability of the electrode. Rutile electrodes can be classified as universal from the point of view of power source, working both with alternating and direct current (AC/DC, DC±) [8], [16].

Low-hydrogen (basic) E7018 electrodes are traditionally used on DC+ (DCEP). It should be noted that there are industrial formulations that also allow their use on alternating current (AC) [20], [3].

The choice of current is subject to the ranges given in the manufacturer's data sheet and is optimized for the welding position and diameter [16].

4. Control of diffusible hydrogen and the HxR designation

Cold cracks caused by diffusible hydrogen (HACC) occur after solidification and depend on the source of hydrogen (electrode, contaminated metal, etc.), hardness, microstructure and stresses. In carbon-manganese (C-Mn) steels the cracks often start in the heat-affected zone (HAZ) and may propagate into the weld metal [5]. Prevention includes the use of low-hydrogen electrodes, preheating, dry consumables and clean edges [6].

"H4/H4R" denotes a limited content of diffusible hydrogen (≤ 4 ml/100 g of deposited metal), and the letter "R" indicates additional resistance to moisture pick-up (27 °C / 80 % RH for 9 h) [11], [13].

5. Criteria for electrode selection (algorithm)

1) Material and mechanical requirements.

For S235–S355 (structural steels) and non-critical joints – rutile E6013; for thick sections, dynamic loading, low temperatures – low-hydrogen E7018 [1], [6]; for stainless steels – corresponding 308L/316L/309 class to ISO 3581 [2], [12].

2) Welding position.

All positions / thin material – rutile; vertical-up on thick sections – low-hydrogen; pipe roots – cellulosic [16], [10].

3) Power source and polarity.

Available AC source – rutile or AC-compatible E7018; for DC+ – low-hydrogen and rutile [16], [3].

4) Electrode diameter – selected according to thickness.

Practical guidelines (e.g. $\varnothing 2.5 \sim 2\text{--}5$ mm; $\varnothing 3.2 \sim 4\text{--}8$ mm; $\varnothing 4.0 \geq 8$ mm). For critical joints, the exact values are specified in the data sheet and WPS (Welding Procedure Specification) [16].

5) Hydrogen / cracking limitations.

At high carbon equivalent (CE) or high hardness – H4/H4R low-hydrogen electrodes, preheating and limited heat input in accordance with EN 1011-2 and good practice [6].

As examples from marine practice, the electrodes of manufacturers Unitor and Wilhelmsen can be cited –

LH-314N (LMA, low content of diffusible hydrogen, intended for shipbuilding / structural steels). The data sheet of these electrodes emphasizes their high impact toughness and crack resistance. They are intended for critical joints on "ship quality" steels [7].

GPO-302N (rutile, "general purpose"). Intended for use on low-carbon mild steels in all welding positions, including vertical-down; good bridging effect [8].

These examples illustrate the logic "E6013 for general repairs" versus "E7018 for critical joints / thick sections", in line with ISO/AWS classifications [1], [2], [10].

6. Defects in case of incomplete / wrong selection

- Cold cracks (HACC), often in the HAZ; their symptoms and orientation are described by TWI [5].
- Porosity, slag inclusions – incorrectly selected electrodes (moist electrodes, unsuitable coating for the welding position and/or low heat input – the amount of heat energy introduced per unit length of weld; it affects the HAZ and is usually specified in the WPS) – defined by the welding parameters [6].
- Lack of penetration – wrongly chosen electrode/coating for the specific needs (e.g. rutile instead of cellulosic for a root pass) or too low welding current values [16].

7. Storage, baking and exposure

After baking/drying, low-hydrogen electrodes should be stored in a holding oven at about 120 °C in order to prevent moisture pick-up [3].

Restoring the temperature regime when charging 20–80 kg of cold electrodes is slow (from 45 min to 2.5 h) and has to be taken into account when organizing the work [9].

"R"-classified (H4R) products are designed for increased moisture resistance, but the manufacturer's recommendations and exposure limits still have to be respected [11], [12].

Indicative drying regimes for electrodes:

- E7018 / E7016 (low-hydrogen, H4/H4R) – (260–350) °C for 1–2 h.
- E6013 (rutile) – (100–150) °C for 0.5–1 h (if there is moisture) – high temperatures are not used so as not to damage the coating.
- E6010 / E6011 (cellulosic) – not baked.
- Stainless/nickel electrodes 308L, 316L, Ni/NiFe – (200–300)°C for 1÷2 h.

8. Parameters: current, speed, welding technique

Industrial guidelines (e.g. Lincoln) provide operating ranges by type and diameter, as well as recommendations on polarity and control of arc blow, especially for DC [16].

In practice, the welding current for Ø2.5 / 3.2 / 4.0 mm electrodes usually falls around ~60–90 A / ~90–130 A / ~120–170 A and is fine-tuned according to position, workpiece and coating type, always validated against data sheet / WPS [16].

Figures 1, 2 and 3 show recommended welding current values depending on the thickness of the part being welded, respectively for E6013 (rutile), E7018 (basic) and E6010 (cellulosic) electrodes.

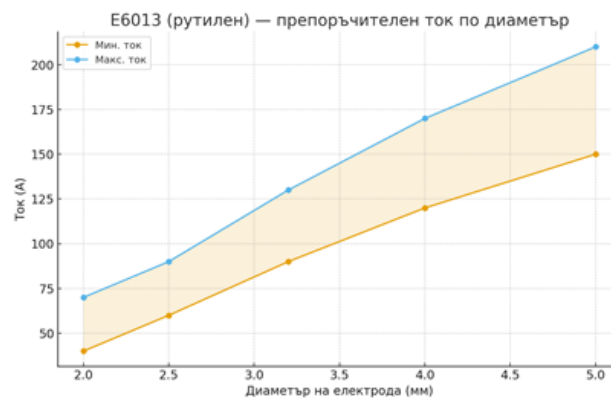


Fig. 1 E6013 (rutile): recommended current by diameter [14], [16]

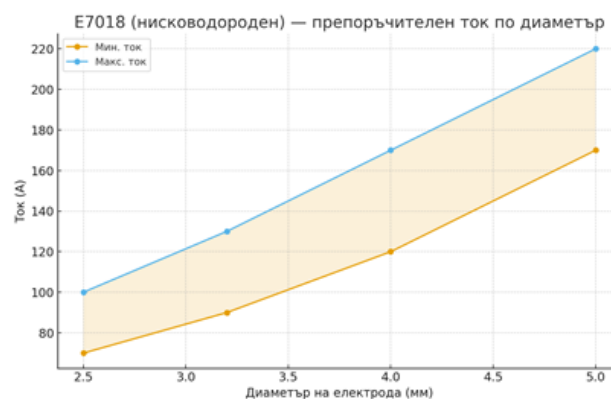


Fig. 2 E7018 (low-hydrogen): recommended current by diameter [14], [16].

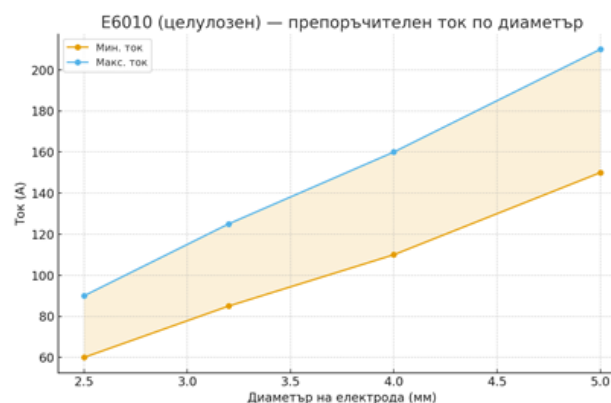


Fig.3 E6010 (cellulosic): recommended current by diameter [14], [16].

9. Cold cracks HACC (hydrogen-assisted cold cracking)

Cold cracks caused by the presence of diffusible hydrogen appear after completion of the welding process at approximately room temperature; most often this occurs in the HAZ or in the weld metal.

The three necessary conditions for this defect are:

- Diffusible hydrogen in the weld/HAZ (caused by moisture, residues of oils, paint, use of rutile or cellulosic electrodes, insufficient baking of electrodes).

- Brittle and hard microstructure (high carbon equivalent CE, large material thickness with martensitic structure and high hardness).

- Presence of tensile stresses (restraint caused by inappropriate welding sequence, rapid cooling).

To control these three components (hydrogen, hard structure, stresses), we propose the following checklist in tabular form for managing the risk of undesired cold cracks.

Mark "Yes" where a risk is present; ≥ 3 "Yes" answers $\Rightarrow$ take measures (E7018 H4/H4R; preheating; dry electrodes; control of interpass temperature; lower HAZ hardness) [5], [6], [11], [12].

Cold cracks (HACC – Hydrogen-Assisted Cold Cracking) – checklist:

Table 1: Risk factors.

Tick	Risk factor	Notes
☐	High carbon equivalent (CE) of the steel or thickness above 15 mm.	
☐	High restraint / fixation of the part (restricted shrinkage).	
☐	Low ambient temperature / no preheating.	
☐	Contaminated edges (moisture, oils, paint, rust).	
☐	Moist electrodes.	

Table 2: Measures when risk is identified

Tick	Measure	Notes
☐	Low-hydrogen electrodes (e.g. E7018 H4/H4R) + correct baking/storage.	
☐	Preheating according to WPS and control of interpass temperature.	
☐	Cleaning of edges to bare, dry metal (no paint/rust/oils).	
☐	Reduction of restraint and optimization of weld sequence.	
☐	Control of heat input and travel speed within the WPS limits.	

10. Electrode selection.

Table 3: Matrix – guideline for electrode selection (carbon steel, pipe root)

Material	Carbon steel S235–S355	Carbon steel (thick ≥ 8 mm)	Pipe root (C-Mn)
Position/ thickness	Flat/fillet, thin ≤ 5 mm	Vertical-up/positional	Vertical-up/-down
Power source	AC or DC $\pm$	DC+	DC+
Recommendation	E6013 (rutile) $\varnothing 2.0$ – 2.5 ; 60–90 A	E7018 (low-hydrogen) $\varnothing 3.2$ – 4.0 ; 110–160 A	E6010 $\varnothing 2.5$ – 3.2 ; 85–125 A
basis/ source	Easy arc striking, smooth bead	Strength/ impact toughness; low H	Deep penetration

Table 4: Matrix – guideline for electrode selection (stainless steel, dissimilar)

Material	Stainless 304 $\leftrightarrow$ 304	Stainless 316 $\leftrightarrow$ 316	Dissimilar 304/316 $\leftrightarrow$ carbon steel
Position/	All positions	All positions	All positions

thickness			
Power source	AC/DC	AC/DC	AC/DC
Recommendation	308L (stainless)	316L (stainless)	309/309 Mo
basis/ source	Matching composition	Mo for corrosion resistance	Buffer composition for dissimilar joints

Table 5: Matrix – guideline for electrode selection (cast iron, low temperature high CE)

Material	Cast iron (repair)	Low temperature / high CE
Position/ thickness	Short runs	Critical joints
Power source	AC/DC	DC+
Recommendation	Ni or NiFe	E7018 H4/H4R
basis/ source	Minimization of cracking; machinability	Control of H; moisture resistance

11. Application for selecting a suitable electrode

On the basis of everything discussed so far, with the aim of fast and correct selection of a suitable electrode, a quick-selection application – "Electrode advisor" – was developed.

Electrode Selection Advisor (SMAW)

Results are indicative. Validate against WPS and manufacturer data sheets (E6013/E7018/E6010; 308L/316L/309Mo; Ni/NiFe).

☒ Expert Mode (positional current correction and recommended setpoint)

1. Material
Dissimilar: Stainless $\leftrightarrow$ Carbon Steel

2. Parameters & Power
Thickness (mm): 6
Position: Horizontal
Power Source: AC
Criticality: General Repairs

3. HACC Risk (Hydrogen-Assisted Cold Cracking)
☐ High CE/Hardness or Thickness ≥ 15 mm ☐ High Restraint / High Stresses ☐ Low Ambient Temperature, No Preheat
☐ Dirty Edges (Moisture/Oil/Paint) ☐ Non-Low-Hydrogen Process/Consumable
Total factors: 0 Risk: LOW

Recommended Selection
CLASS (AWS): 309/309Mo DIAMETER: 3.2 mm CURRENT (GUIDELINE): 86–124 A
Polarity: AC
EXPERT MODE — CURRENT CALCULATION
Base Range (Flat): 90–130 A Position Correction (Horizontal): -5% Recommended Range: 86–124 A
Optimal Setpoint: ≈ 105 A
RATIONALE
• High Cr/Ni acts as a buffer for dissimilar joints.

Fig.4 Screenshot of the quick-selection application "Electrode advisor"

The application uses the following input data, in the form of drop-down menus:

- Base material of the part:
 - Carbon steel (S235–S355);
 - Shipbuilding/structural steel (more critical joints);
 - Stainless 304 / 316;
 - Dissimilar joint (stainless steel – carbon steel);
 - Cast iron (with option "machinability required" – Ni vs NiFe);

- Hardfacing (general guideline).
- Geometry and position
 - Thickness (mm);
 - Welding position: flat / horizontal / vertical-up / vertical-down / overhead;
- Power source / polarity
 - AC, DC+ (DCEP) or DC- (DCEN).
- Criticality of the task
 - General repair vs critical/structural.
- HACC-factors (assessment of hydrogen-assisted cold cracking risk)
 - High carbon equivalent / hardness or thickness ≥ 15 mm;
 - High restraint / restricted deformations;
 - Low ambient temperature without preheating;
 - Contaminated edges (moisture/oils/paint);
 - Use of non-low-hydrogen process/consumable.
- (Cast iron only) Is machinability of the weld required?
 - Determines the choice Ni (better machinability) versus NiFe (higher strength).

The application automatically performs the following operations:

- Calculates HACC risk (low / medium / high) and gives recommendations;
- Selects electrode class (E6013/E7018/E6010; 308L/316L/309Mo; Ni/NiFe);
- Proposes electrode diameter according to part thickness and welding position;
- Indicates approximate current according to diameter and electrode class;
- Adds recommendations on polarity (e.g. "DC+ (DCEP)" for E7018/E6010; "AC/DC±" for E6013).

Conclusions:

The application converts a complex engineering and technological choice into a structured and explained process by:

1. Showing the correct electrode/diameter/polarity for the context.
2. Managing HACC risk with the help of a checklist and giving guidance on its minimization.
3. Generating documents for QA/WPS archiving.
4. Standardizing practice and speeding up the work of the team.

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Innovative approach to determine the type of corrosion damage on hybrid fiber-reinforced industrial concrete floorings in a bulk fertilizer storage warehouse

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Abstract: The industrial concrete floorings of a large warehouse for storing various bulk fertilizer substances in the Varna region are made as hybrid fiber-reinforced one by using a high-quality powdered surface hardener (dry shake topping) to increase their surface hardness and abrasive resistance. The floors are sized and constructed to take on the specific operational loads - a bulk uniform distributed load with a large mass and a mobile front-end loader with a metal rake. Given the expected possible corrosion effect of pre-specified heterogeneous fertilizer substances in the working concrete mix design is used sulfate-resistant cement, which should ensure defect-free operation. Regardless of the measures taken after one year of operation, the presence of surface defects on the floor in strictly limited areas of operation is reported. A reasonable question arises as to the reasons for this. The report discusses an innovative experimental-theoretical approach proposed by the author to identify the causes of this defect with the combined use of a complex method based on the combine use of standardized mechanical and structural direct physical methods of investigation. After specialized sampling and examination of the concrete strength in specific areas, as well as after additional application of X-ray structural and differential thermal analysis, as well as scanning electron microscopy, original scientific results were obtained, which revealed the causes and mechanism of the discussed corrosion damage, and these complex activities definitely have a justified arbitration significance.

Keywords: FERTILIZER SUBSTANCES, INDUSTRIAL CONCRETE FLOORINGS, CONCRETE STRENGTH, X-RAY STRUCTURAL AND DIFFERENTIAL THERMAL ANALYSIS, SCANNING ELECTRON MICROSCOPY

1. Introduction

The industrial concrete floorings of a large warehouse for storing various bulk fertilizer substances in the Varna region are made as hybrid fiber-reinforced one by using a high-quality powdered surface hardener (dry shake topping) to increase their surface hardness and abrasive resistance. The floors are sized and constructed to take on the specific operational loads - a bulk uniform distributed load with a large mass and a mobile front-end loader with a metal rake. Given the expected possible corrosion effect of pre-specified heterogeneous fertilizer substances in the working concrete mix design is used sulfate-resistant cement, which should ensure defect-free operation.

Regardless of the measures taken after one year of operation, the presence of surface defects on the floor in strictly limited areas of operation is reported. A reasonable question arises as to the reasons for this.

It is visible that the defective limited area of the concrete flooring is in front of and in the entrance area of one of the storage cells – numbered as 4 (Fig. 1 and 2).



Fig. 1 and 2 Defective zones of cell 4

Given that the two affected areas were performed in two separate cycles, at different times with an interval of several days between them, it would be difficult to accept the claim that such a single error was made that would lead to this complete identity of the defect. Moreover, if we add the visible corrosion damage to the metal entrance door of this cell, the hypothesis of an unregulated impact from a fertilizer substance remains objectively possible and credible (Fig. 3-6).



Fig. 3 and 4 Defective wall concrete



Fig. 5 and 6 Corrosion in steel entrance doors

It is evident that the loading and unloading activities with bulk fertilizers in the area of the individual storage cells and the maneuvering space in front of them are carried out with a classic front-end loader with a steel shovel on the bucket. A part of the respective fertilizer substance in the process of this activity, in addition to inside the cell, also falls into the space in front of it (Fig. 7 and 8) - i.e. in the presence of the alleged aggressive impact on the concrete, it is clearly manifested both inside the respective cell and in the area immediately in front of it. In addition, the influence of the unregulated mechanical impact from the operation of the front-end loader with a metal shovel on the surface of the flooring in all areas of the warehouse should also be taken into account.



Fig. 7 and 8 Storage sells of bulk fertilizers

In this sense, the hypothesis is that precisely in this area, and for an indefinite period of time, a fertilizer substance with a highly aggressive effect on concrete was stored.

2. Expert and research tests specialized activities

In this direction, the expert and research specialized test activities have been carried out, through which the reasons for the identified defects in the flooring in this area can be objectively and reliably formulate [1,2].

Concrete core sampling (Fig. 9 and 10) and subsequent standard tests were carried out in accordance with the requirements of:

- BDS EN 12504-1:2019 Testing of concrete in structures. Part 1: Cores. Cutting, inspection and compression testing;
- BDS EN 13791:2019 Assessment of the in-situ compressive strength of concrete in structures and precast concrete elements;
- BDS EN 13791:2019/NA:2022 Assessment of the in-situ compressive strength of concrete in structures and precast concrete elements. National Annex (NA);
- BDS EN 12390-7:2019 Testing of hardened concrete. Part 7: Density of hardened concrete.



Fig. 9 and 10 Concrete core sampling

2.1. Concrete compressive strength

The results are also presented in **Table 1**, from which it is evident that the design concrete compressive strength class C35/45 SR has been achieved.

Table 1: Concrete core compressive strength results

N ^o	Density, kg/m ³	Compressive strength, MPa	Correspondance
1	2296	53,10	Accor. BDS EN 13791:2019 for concrete C35/45, For minimum value: Above $0,85(f_{ck, spec} - M) = 0,85(45 - 4) = 34,9 < 53,1$ MPa; For mean value: Above $0,85(f_{ck, spec} + 2) = 0,85(45 + 2) = 40$ MPa $< 57,0$ MPa
2	2294	57,60	
3	2314	60,30	
Mean	2300	57,00	

2.2. Structural tests

The visual inspection of the extracted through-hole cores shows the absence of any defects throughout the entire depth of the core section. Only on the upper surface of **core 1** are these defects observed, reaching a limited depth of about 3-6 mm.

In order to clarify the reasons for this surface defecting of the areas of the flooring in front of and at the beginning of the storage cell, preparation was carried out for conducting specialized structural research, in order to establish the possible presence of structural changes with the depth of the concrete section, due to unregulated corrosion effects from fertilizer substances in a given period of the storage operation.

For this purpose (Fig. 11 and 12) from the uppermost and lowermost part of:

- **core 1** (with the identified surface defects) cylindrical representative sections (discs) with a height of about 10 mm, marked as **1.1.** and **1.2.**;
- **core 3** (without identified defects) cylindrical representative sections (discs) with a height of about 10 mm, marked as **3.1.** and **3.2.**

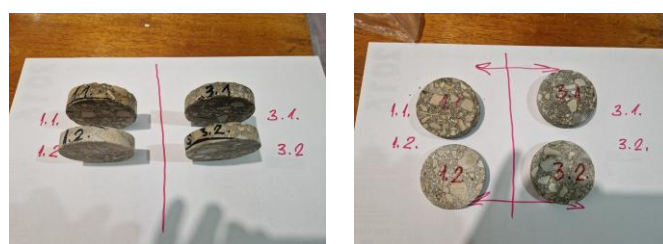


Fig. 11 and 12 Core cut discs for structural tests

The following structural investigations were specified for each of the four individual concrete discs (Fig. 11 and 12) using X-ray diffraction (XRD), differential thermal analysis (DTA) and scanning electron microscopy (SEM). With the help of these direct physical methods, the nature of the internal structure of the concrete can be judged and the extent of the corrosion changes that have occurred in it can be assessed (Fig. 13 - 15).



Fig. 13
XRD apparatus
Bruker D8 Advanced



Fig. 14
Thermograph
SETSYS Evolution



Fig. 15
Electron microscope
INCA Oxford EDS

2.2.1. X-ray phase analysis

X-ray phase analysis is a basic direct physical method for studying the phase composition and phase changes in the crystal lattice of hardened silicate composites (cement concretes and mortars). It is based on the well-known feature of different crystals (even when they are not in the ordered form characteristic of each of them) that their structure repeats the same elements - ions and atoms - phase formations and crystal decomposition as a result of internal and external factors at a certain interval. In essence, the method provides insight into the features of the intra-crystalline structure of silicate composites. Below are presented the obtained X-ray diffraction patterns of the 4 samples studied - sample 1.1., sample 1.2., sample 3.1. and sample 3.2. (Fig. 16-19).

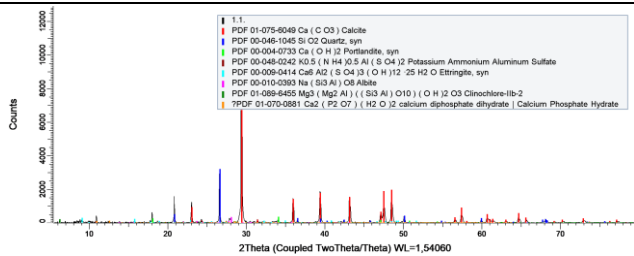


Fig. 16 X-ray diffraction sample 1.1.
(upper surface defected zone cell 4)

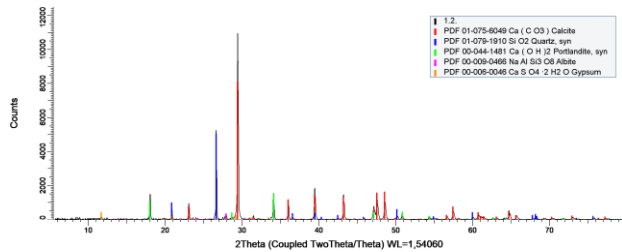


Fig. 17 X-ray diffraction sample 1.2.
(lower surface defected zone cell 4)

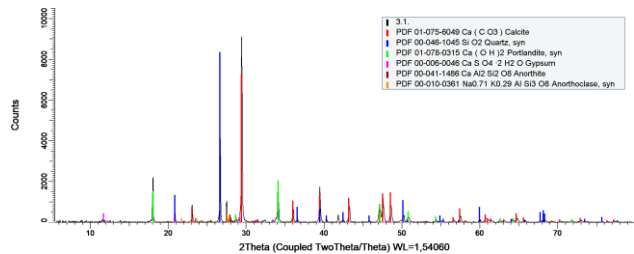


Fig. 18 X-ray diffraction sample 3.1.
(lower surface not defected zone cell 4)

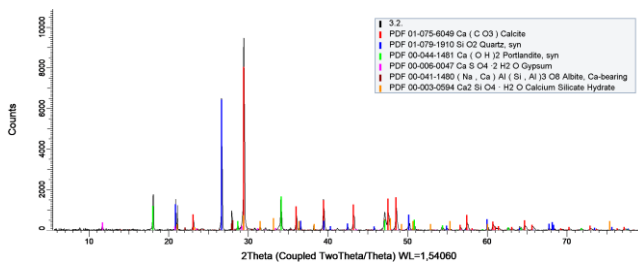


Fig. 19 X-ray diffraction sample 3.2.
(lower surface not defected zone cell 4)

Analysis of the results obtained:

A RADICALLY DIFFERENT REPRESENTATION of the two examined samples from core 1 (from cell 4) is observed:

- In sample 1.1. from the affected upper concrete surface, crystalline compounds typical of sulfate corrosion in concrete are found – potassium-ammonium-sulfate and tricalcium hydro-sulphate-aluminate (ettringite). The latter compound is also known as the so-called "cement bacillus", directly affecting the quality of concrete very negatively;
- In sample 1.2. from the lower surface of the core from the affected area in cell 4, these corrosion products are not found, which means that the corrosion action has occurred only superficially for now.

A PRINCIPALLY SAME REPRESENTATION of the two examined samples from core 3 (from a non-defective area in cell 4) is observed. In samples 3.1. and 3.2. the above-identified products of corrosion on the concrete are absent.

2.2.2. DTA analysis

Differential thermal analysis (DTA) is a method that also belongs to the set of direct physical methods for studying the crystal

structure in silicate composites. It is based on the characteristic feature of hydrate formations in cement stone to dehydrate in a precisely defined temperature range. The corresponding dehydration is accompanied by a characteristic thermal effect that changes the heat balance of the system. Tracking the corresponding endo- and exo-effects allows us to judge the phase transformations identified with the release of chemically bound water. Below are presented the DTA tests of the 4 samples studied – sample 1.1., sample 1.2., sample 3.1. and sample 3.2. (Fig. 20-23).

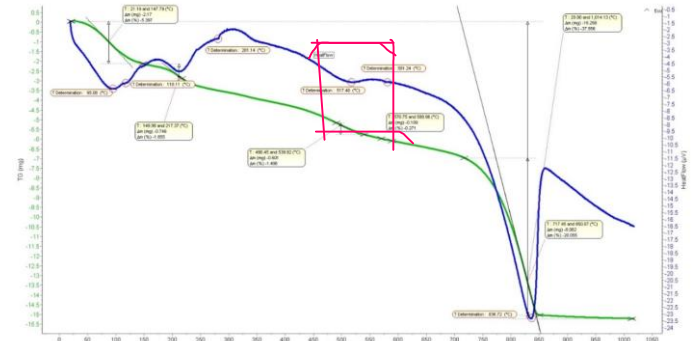


Fig.20 DTA sample 1.1. (upper surface defected zone cell 4)

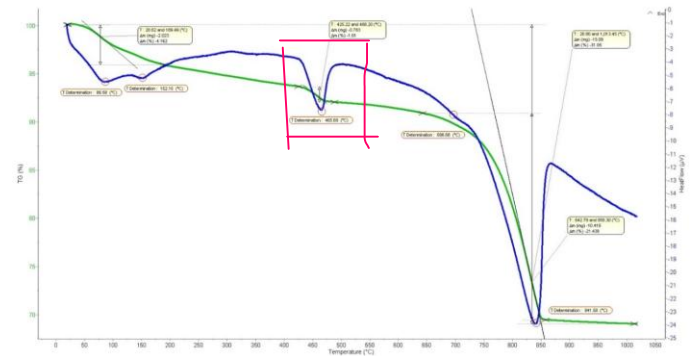


Fig. 21 DTA sample 1.2. (lower surface of defected zone in sell 4)

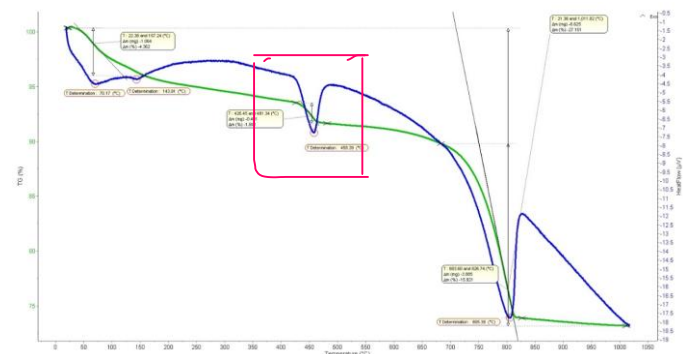


Fig. 22 DTA sample 3.1. (upper surface of non-defected zone in sell 4)

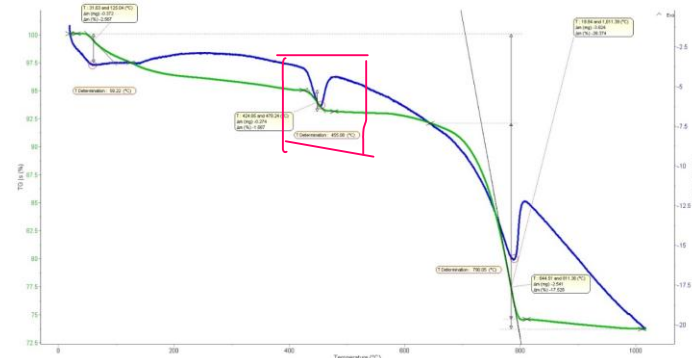


Fig. 23 DTA sample 3.2. (lower surface non-defected zone in sell 4)

Analysis of the results obtained:

A RADICALLY DIFFERENT REPRESENTATION of the two studied samples from core 1 (from cell 4) is observed:

- In **sample 1.1.** from the affected upper concrete surface in the characteristic temperature range 450-5000 C, a COMPLETE LACK OF PORTLANDITE (calcium hydroxide) is observed, which is an environment for the stable existence of the highly basic hydro compounds in the concrete, carriers of its structural and mechanical characteristics. It is the portlandite that is attacked first during the course of sulfate corrosion of the concrete, and its complete absence means that it is completely "consumed" until it is converted into unstable salt products (compounds) with a volume greater than the sum of its constituent products, which in turn creates additional stresses leading to destruction.
- In **sample 1.2.** from the lower surface of the core from the affected area in cell 4 in the discussed temperature range, a characteristic peak (endo-effect of portlandite) is detected, which means that such corrosion has not occurred at the bottom of the concrete section.

A PRINCIPALLY EQUAL REPRESENTATION of the two studied samples from core 3 (from a non-defective area in cell 4) is observed. In **samples 3.1. and 3.2.** the existence of the endo-effect in the same temperature range of 450-500°C is clearly distinguishable, which means a sufficient amount of portlandite is preserved and there is no corrosive effect on the concrete.

2.2.3. Scanning electron microscopy

Scanning electron microscopy (SEM) is performed using an electron microscope with high magnification (up to and over 10 000 times), as a result of which visual data is obtained about the shape and size of individual submicroscopic crystals, the processes of their growth, decomposition and destruction, and on this basis to judge the chemical interactions that have occurred in solution and solid phase.

Below are micrographs at 5000 -10000 times magnification of 2 studied samples – **sample 1.1. and sample 1.2.** (Fig. 24-27).

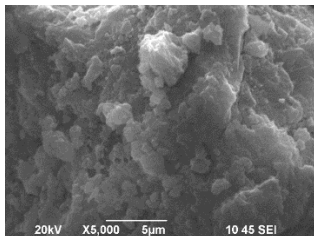


Fig. 24 SEM sample 1.1. x 5 000

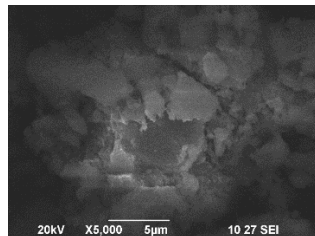


Fig. 25 SEM sample 1.2. x 5 000

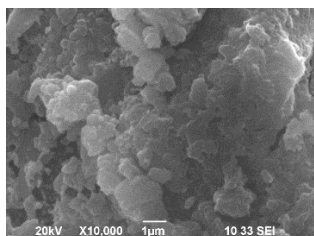


Fig. 26 SEM sample 1.1. x 10 000

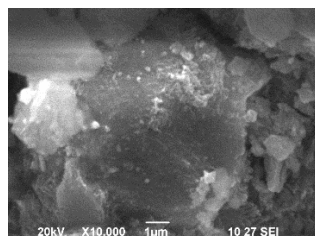


Fig. 27 SEM sample 1.2. x 10 000

Analysis of the obtained results:

A RADICALLY DIFFERENT VISUALIZATION is observed at both magnification steps (5 000 and 10 000 times) of the two examined samples from **core 1 (from cell 4)**:

In **sample 1.1.** from the affected upper concrete surface, the characteristic plate-like (prismatic) crystals of PORTLANDITE (calcium hydroxide) are visually absent, which confirms its absence in comparison with the visible presence in **sample 1.2.** from the bottom of the core in the defective area of cell 4.

3. Briefly about sulphate corrosion of concrete

Sulfates are found in most natural and wastewater, and the presence of sulfate ions SO_4^{2-} is determined by the solubility of sulfate products (Na_2SO_4 , K_2SO_4 , MgSO_4) [3-5].

The interaction between sulfates, on the one hand, and the components of un-hydrated cement or hardening cement paste, on the other, is a complex process that depends on a number of factors. Multiple chemical processes can occur simultaneously or sequentially. Na_2SO_4 and K_2SO_4 , found in water and/or in aqueous solutions of fertilizer substances, contacting cement stone and concrete, are capable, on the one hand, of significantly increasing the solubility of the components of cement stone and thus accelerating the development of type I corrosion (dissolution and leaching). On the other hand, exchange reactions can be initiated with the replacement of the K^+ , Na^+ , Mg^{2+} ions in the sulfates with the Ca^{2+} ion from the cement stone - i.e. the development of type II corrosion. Under certain conditions, the action of sulfates can also cause the development of type III corrosion. In this case, an important circumstance is that as a result of the exchange reactions, the water in contact with the cement stone and concrete gradually becomes saturated with CaSO_4 . The precipitation and growth of gypsum crystals ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) are possible when reaching a concentration of (2020 – 2100) mg/l. This value can change in the presence of dissolved substances in the water - CaCl_2 , Na_2SO_4 , etc.

Factors influencing the course of corrosion processes include composition of the cement or hardened cement paste, form of the sulfates involved in the process, type of interaction – whether sulfates are part of the composition of the cement or concrete mixture, which hydrate (internal sulfate attack) or enter the hardened paste/mortar/concrete from the environment (external sulfate attack) and the temperature at which the interaction occurs []

The final result of the reactions taking place is expressed in an increase in the volume of the cement paste, chemical decomposition of the hydrate products, associated with a deterioration in the strength characteristics of the hardened cement paste and concrete and formation of cracks in the structure of the hardened material, surface collapse, chipping or delamination of the cement paste/mortar/concrete.

A distinctive feature of sulfate aggression in cement-based materials is that the final result of the action of sulfates does not depend on the type of their source (external or internal), but is determined by the conditions of the interaction.

The aggressive effect of sulfates on cement stone was known as early as the end of the 19-th century - it was identified by a number of famous researchers in the field of silicates, such as Vika, Le Chatelier and Michaelis. The two most popular forms of sulfate attack are the so-called gypsum (with the formation of dihydrate gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and sulphate-aluminate corrosion.

It has been established that of all the complex salts formed in concrete under the action of aggressive sulfate waters, calcium hydro-sulphate-aluminate (ettringite) has the greatest importance for the resistance of cement stone. Depending on the conditions of formation, it has two modifications – tri-sulfate hydro-sulphate-aluminate ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) and mono sulfate hydro-sulphate-aluminate ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 12-13\text{H}_2\text{O}$).

The tri-sulfate form, which contains a large amount of water of crystallization, has a particularly great influence on the development of corrosion processes in concrete. The growth of its crystals in hardened concrete causes significant destruction in the cement stone and concrete, due to the appearance of internal stresses due to an increase in the volume of the solid phase.

4. Concluding remarks

4.1. General part

Transferring the above considerations to the specific case, it should be categorically summarized that the experimentally established sulfate corrosion was caused by an external source - given the use of sulfate-resistant concrete for the industrial concrete pavements of the site.

How this effect was achieved cannot be established categorically, given the storage of different fertilizer substances in different periods of operation of the warehouse.

In the assignment for the design of the industrial concrete pavements, a group of fertilizer substances subject to processing and storage at the site was announced, namely ammonium sulfate, NDK 10-26-26, di-ammonium sulfate, MA, UREA, GENEZIS CAN 27%, NPS 16:20 +12 (S) and NPS 16:20 +12 (S).

There are also data on the values of the angle of natural slope during bulk storage in a loose bulk state from 29 to 41°. This indicator is important for determining the dominant load on the pavements as a bulk load.

Based on the presented relevant MSDS, a number of chemical agents with a potential corrosive effect on the alkalinity of ordinary concrete are specified in their composition - ammonium, nitrate and sulfur oxides, phosphates, chlorides, amines, etc., in various combinations and percentages. For most of these fertilizer mixtures, the limits of the pH-indicator (at 5% aqueous solution) are specified within 4,0 to 9,5, as well as their limits of solubility in water - usually within 100 g/l.

In addition, data are also provided on their degree of hygroscopicity, as a function of the relative humidity of the outside air and temperatures. According to their degree of solubility in the presence of moisture of different origin, aqueous solutions can have different levels of corrosive effect on concrete, as a function of its degree of permeability, porous structure and method of reinforcement.

Based on the above, given the many possible combinations of different fertilizer substances during storage, it is not possible to determine with absolute accuracy the degree of their corrosive effect on concrete flooring.

It is for this reason that sulfate-resistant concrete was used - a logically justified measure, given the proven technological impossibility of applying secondary surface protection to the concrete by applying a polymer coating, since it would be immediately compromised by the mechanical impact during the operation of the front loader.

4.2. Reasoned conclusion

The design strength characteristics of the concrete for industrial concrete flooring have been achieved.

The partial surface defecting of the industrial concrete flooring IN FRONT and IN THE FRONT PART OF THE INTERIOR of storage cell 4 of the site is due to the occurrence of highly aggressive sulfate corrosion of the concrete, caused by the manipulation of an unidentified single batch of fertilizer substance, which, due to the presence of external influences (temperature difference, change in the humidity of the environment and air) has released a limiting concentration of sulfate ions in an aqueous environment, which have entered into an exchange reaction with the hydration products of the sulfate-resistant concrete.

A clear sign of the objectivity of such a statement is the categorically established experimentally presence of zero portlandite content, accompanied by the formation of tricalcium hydro-sulphate-aluminate (ettringite) only and exclusively in the upper surface of the defective zone of the space in front of and at the beginning of the flooring in the commented cell 4.

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Application of Machine Learning Algorithms for Predicting the Hydrodynamic Characteristics of High-Voltage Electric Discharge Processing of Al–Ti Powder Mixtures with Different Mass Compositions by Spark Discharge in Ethanol

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Abstract: This study investigates the hydrodynamic and thermal characteristics of spark discharge during electric discharge treatment of Al–Ti powder mixtures in ethanol. Machine learning methods were applied to identify relationships between mixture composition, interelectrode gap, and discharge parameters. Among the tested algorithms, the Gradient Boosting model demonstrated the highest predictive accuracy for describing nonlinear plasma-dynamic processes.

The analysis revealed a linear increase in plasma channel pressure from approximately 270 to 450 MPa as the interelectrode gap increased from 5 to 35 mm, regardless of the Al–Ti ratio. In contrast, the pressure acting on the discharge chamber walls was strongly dependent on powder composition. Titanium-rich mixtures exhibited a decrease in wall pressure with increasing gap, whereas mixtures containing 80 wt.% Al showed the opposite trend.

The developed Gradient Boosting model demonstrated that thermal characteristics of the discharge are governed primarily by pulse and liquid parameters, while peripheral hydrodynamic effects are controlled by the composition of the powder mixture. The obtained results provide new insights into the optimization of electric discharge processing of metal powder systems.

Keywords: Electric Discharge Treatment, Aluminum–Titanium Powders, Spark Discharge, Machine Learning, Gradient Boosting, Plasma Channel Pressure, Plasma Temperature.

1. Introduction

Materials of the Al–Ti–C system, including Ti_2AlC and Ti_3AlC_2 MAX phases, as well as TiAl-based alloys, combine the properties of metals and ceramics, such as high thermal stability, specific stiffness, and electrical conductivity, which makes them attractive for aerospace, transportation, and energy applications [1, 2]. Conventional synthesis methods for these materials, including hot isostatic pressing (HIP) and reactive sintering, require extensive preparation of the starting materials and do not always ensure the desired phase composition [3, 4].

At the same time, high-voltage electric discharge (HVED) treatment of powder mixtures in hydrocarbon liquids combines particle fragmentation, surface activation, and in-situ synthesis of carbon-containing phases through the decomposition of the working liquid within the discharge channel [5, 6]. The use of ethanol as a working liquid is particularly promising due to its stable chemical composition and the high reproducibility of the synthesis process [7].

Therefore, HVED treatment of Al–Ti powder mixtures in ethanol offers a potential route for producing Al–Ti–C system materials with a controlled phase composition and morphology while minimizing the number of technological processing steps.

Despite significant progress in the study of HVED processing of powder materials, the hydrodynamic and thermal characteristics of the process in Al–Ti mixtures with different mass compositions remain insufficiently investigated. Existing analytical and numerical models of HVED generally do not account for the influence of the dispersed system composition on discharge characteristics [8, 9]. This considerably complicates the targeted selection of processing parameters for obtaining Al–Ti–C materials with a desired structure and phase composition. This challenge can be addressed through the application of machine learning algorithms, which enable the identification of complex relationships between process parameters and discharge characteristics [10].

The goal of this study was to determine the patterns governing changes in the hydrodynamic characteristics of the discharge during high-voltage electric discharge treatment of Al–Ti powder mixtures with different mass compositions using spark discharge in ethanol, based on machine learning algorithms.

2. Methodology

Elemental titanium powder PT-6 (TU U 14-10-026-98, mean particle size, $d_m = 60 \mu\text{m}$) and aluminum powder PA1 (GOST 6058-73, mean particle size, $d_m = 20 \mu\text{m}$) were used as precursor materials. Experiments were conducted on Al–Ti powder mixtures containing 15, 25, 50, and 75 wt.% Al. High-voltage electric discharge processing was performed in ethanol (DSTU 4221:2003) using a spark discharge (SD) regime.

High-voltage electric discharge treatment was performed at a single-pulse energy of $W_1 = 1 \text{ kJ}$ and a specific processing energy (W_{sp}) of 10–40 MJ/kg. The interelectrode gap served as the main variable parameter. The discharge channel pressure (P_{ch}), chamber wall pressure (P_w), and plasma temperature in the discharge channel (T) were selected as response variables and were determined using the methodology described in [11].

Five machine learning algorithms were considered for prediction: Gradient Boosting, Random Forest, Linear Regression, artificial neural networks (ANN), and support vector regression (SVR) [12–16]. The selection of the most accurate model was based on the normalized root mean square error (NRMSE) metric [17].

All computations were implemented in the Python programming language.

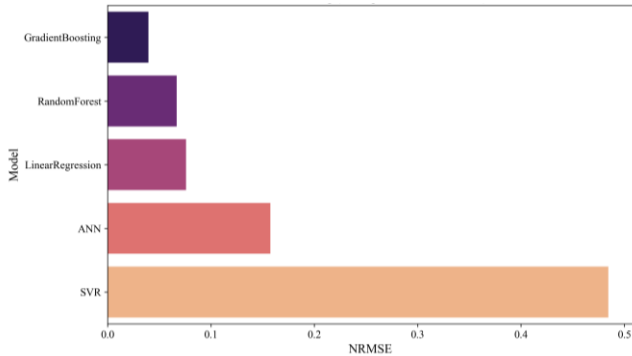
3. Results and discussion

To establish complex nonlinear relationships between the conditions of high-voltage electric discharge processing (aluminum content, interelectrode gap, and liquid properties) and the target process characteristics (discharge channel pressure, chamber wall pressure, and plasma temperature), a machine learning-based approach was employed. Modeling was performed using five conceptually different regression algorithms: Gradient Boosting, Random Forest, Linear Regression, artificial neural networks (ANN), and support vector regression (SVR).

The initial experimental dataset was randomly divided into training (80%) and testing (20%) subsets, with cross-validation applied to prevent overfitting. Considering the different physical units of the input parameters, data standardization was performed prior to model training to ensure zero mean and unit variance for each variable.

The error analysis (see Figure 1) clearly demonstrates the predictive performance of each model. The baseline models (Linear Regression and SVR) exhibited the highest prediction errors, confirming the strongly nonlinear nature of the investigated plasmadynamic processes. In contrast, the tree-based ensemble methods (Random Forest and Gradient Boosting) achieved the lowest NRMSE values and the highest prediction stability. Their superior performance can be attributed to their ability to automatically capture complex interactions among the process parameters without requiring a predefined mathematical relationship.

Figure 1: Comparative analysis of prediction accuracy for the



target parameters using different machine learning algorithms

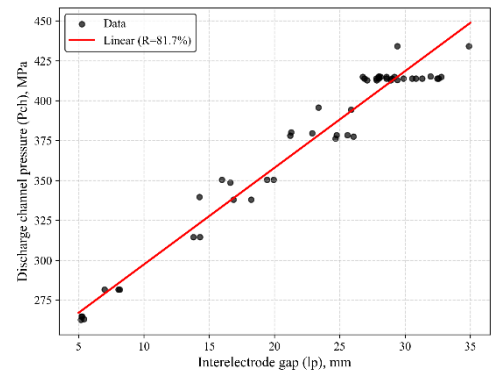
Based on the prediction results for all target variables, Gradient Boosting was identified as the best-performing algorithm, achieving the lowest prediction error. The resulting optimized model was then saved and used to generate all subsequent results reported in this study.

It should be noted that the original experimental dataset, obtained for mixtures containing 15, 25, 50, and 75 wt.% Al, was used exclusively for model training and cross-validation. After the high generalization capability of the Gradient Boosting model had been confirmed, its predictive capability was employed to generate the target parameters on a standardized composition grid with uniform increments (20, 40, 60, and 80 wt.% Al). This approach enabled both interpolation and extrapolation of the underlying physical relationships across the investigated composition range. Furthermore, it demonstrated the model's ability to predict hydrodynamic and thermal characteristics for arbitrary intermediate compositions without the need for additional resource-intensive experiments.

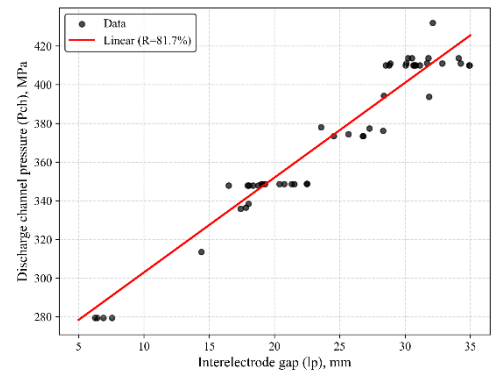
The predicted discharge channel pressure (see Figure 2) exhibits a clear increasing trend with increasing interelectrode gap during HVED treatment of the mixtures. The relationship is distinctly linear and is accurately described by the approximation equations (1) – (4) obtained for each investigated composition.

For the 20 wt.% Al + 80 wt.% Ti mixture, the discharge channel pressure increased markedly from approximately 265 MPa at an interelectrode gap of 5 mm to approximately 450 MPa at 35 mm. For the 40 wt.% Al + 60 wt.% Ti mixture, the values predicted by Equation (2) exhibited a similar linear trend but reached a slightly lower maximum pressure. The predicted discharge channel pressure ranged from approximately 278 to 425 MPa over the investigated interelectrode gap.

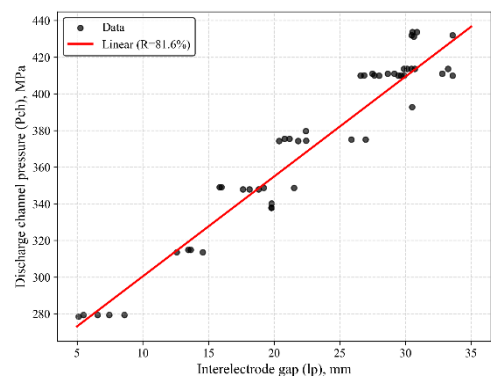
For the 60 wt.% Al + 40 wt.% Ti mixture, the discharge channel pressure increased from approximately 273 to 437 MPa with increasing interelectrode gap. For the 80 wt.% Al + 20 wt.% Ti mixture, in which the aluminum matrix predominated, the linear relationship described by Equation (4) showed the highest goodness of fit ($R^2 = 82.9\%$). The corresponding discharge channel pressure ranged from approximately 270 to 435 MPa.



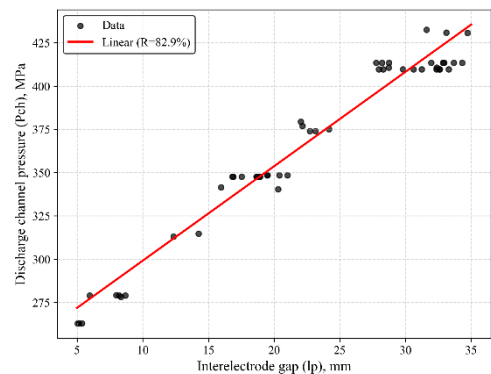
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(a) 20 wt.% Al + 80 wt.% Ti; (b) 40 wt.% Al + 60 wt.% Ti; (c) 60 wt.% Al + 40 wt.% Ti; (d) 80 wt.% Al + 20 wt.% Ti

Figure 2: Dependence of the discharge channel pressure on the interelectrode gap during HVED treatment of Al–Ti mixtures with different compositions in ethanol

Overall, the results indicate that the composition of the Al–Ti system does not fundamentally affect the relationship between discharge channel pressure and the interelectrode gap during HVED treatment in ethanol. A stable positive linear relationship between the interelectrode gap and the discharge channel pressure was observed for all investigated compositions. This behavior can be attributed to the increase in plasma channel volume and the corresponding increase in the energy released within the discharge channel as the interelectrode gap increases.

$$P_{ch}(Al20) = 6 \cdot l_p + 237, \quad (1)$$

$$P_{ch}(Al40) = 5 \cdot l_p + 254, \quad (2)$$

$$P_{ch}(Al60) = 5,5 \cdot l_p + 246, \quad (3)$$

$$P_{ch}(Al80) = 55 \cdot l_p + 245, \quad (4)$$

where $P_{ch}(Al(x))$ – the discharge channel pressure for Al–Ti mixtures containing different amounts of aluminum at a discharge energy of $W_1 = 1$ kJ, MPa;

l_p – the interelectrode gap, mm.

The simulation results presented in Figure 3 reveal the dependence of the pressure acting on the discharge chamber walls (P_w) on the interelectrode gap (l_p) for Al–Ti powder mixtures with different compositions. Unlike the discharge channel pressure, the pressure on the chamber walls exhibits a pronounced dependence on the chemical and phase composition of the dispersed system. This is reflected in both the absolute pressure values and the direction of the linear trends described by Equations (5)–(8).

$$P_w(Al20) = -0,4 \cdot l_p + 70, \quad (5)$$

$$P_w(Al40) = -0,3 \cdot l_p + 72, \quad (6)$$

$$P_w(Al60) = -0,04 \cdot l_p + 67, \quad (7)$$

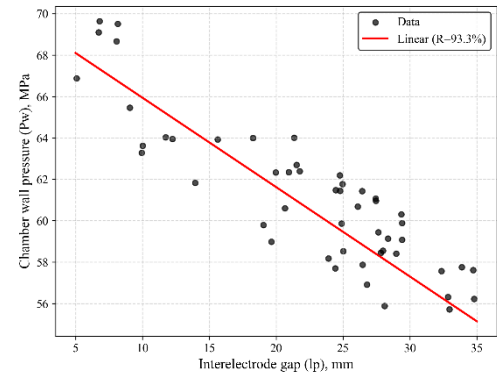
$$P_w(Al80) = 0,09 \cdot l_p + 62, \quad (8)$$

where $P_w(Al(x))$ – pressure acting on the discharge chamber walls for Al–Ti mixtures containing different amounts of aluminum, MPa;

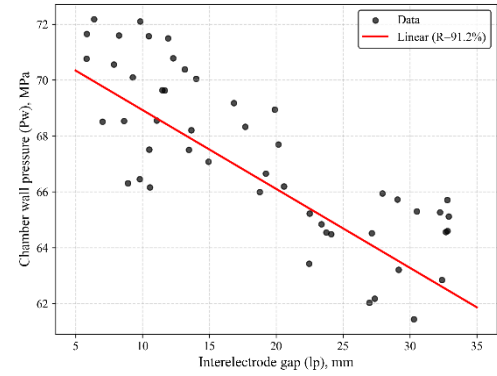
l_p – the interelectrode gap, mm.

Overall, the results indicate that the composition of the Al–Ti powder mixture is the key factor governing the hydrodynamic pressure field at the periphery of the discharge chamber. When the titanium content predominates (20–40 wt.% Al), increasing the interelectrode gap promotes more effective attenuation of shock waves in the heterogeneous medium due to the high inertia and acoustic stiffness of the titanium particles. As a result, the pressure acting on the chamber walls decreases with increasing discharge channel length.

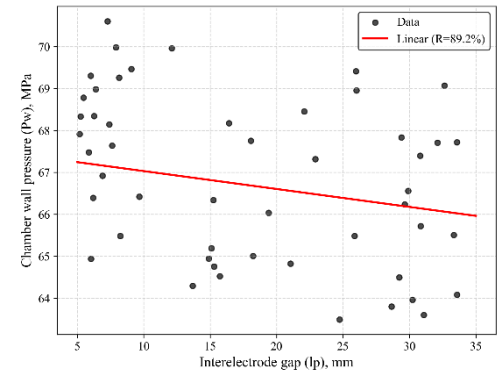
In contrast, increasing the aluminum content to 80 wt.% changes the hydrodynamic response of the system. The lower density and higher ductility of aluminum reduce the initial hydrodynamic resistance at short interelectrode gaps. However, as the discharge channel becomes longer, more aluminum particles are involved in the discharge zone, while intensified ethanol decomposition generates an additional gaseous phase. These effects provide a physical explanation for the reversal of the pressure gradient and the emergence of an increasing pressure trend.



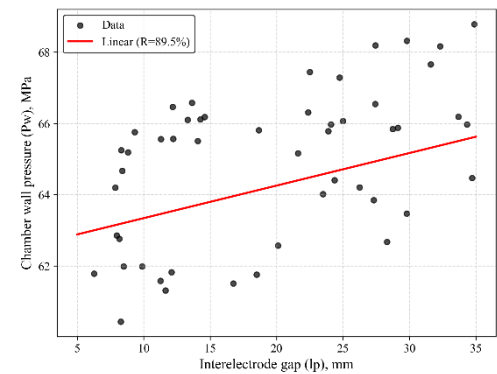
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(a) 20 wt.% Al + 80 wt.% Ti; (b) 40 wt.% Al + 60 wt.% Ti; (c) 60 wt.% Al + 40 wt.% Ti; (d) 80 wt.% Al + 20 wt.% Ti

Figure 3: Dependence of the pressure acting on the discharge chamber walls on the interelectrode gap during HVED treatment of Al–Ti mixtures with different compositions in ethanol

The simulation results presented in Figure 4 reveal the dependence of the plasma temperature in the discharge channel (T) on the interelectrode gap (l_p) for Al–Ti powder mixtures with different compositions during HVED treatment in ethanol. Unlike the pressure behavior, the plasma temperature exhibits a consistent decreasing trend with increasing interelectrode gap for all investigated compositions. This relationship is well described by the linear approximation equations (9)–(12).

$$T(\text{Al}20) = -101 \cdot l_p + 33509, \quad (9)$$

$$T(\text{Al}40) = -103.6 \cdot l_p + 33491, \quad (10)$$

$$T(\text{Al}60) = -84,3 \cdot l_p + 33266, \quad (11)$$

$$T(\text{Al}80) = -100 \cdot l_p + 33494, \quad (12)$$

where $T(\text{Al}(x))$ – plasma temperature into the discharge channel for Al–Ti mixtures containing different amounts of aluminum, K;

l_p – the interelectrode gap, mm.

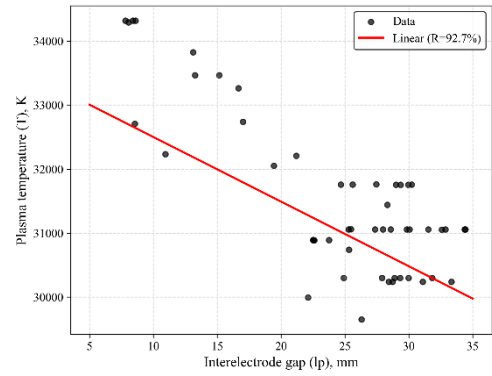
For the 20 wt.% Al + 80 wt.% Ti composition, the temperature dependence is described by the linear relationship given by Equation (9), with a correlation coefficient of $R^2=92.7$. For the minimal interelectrode gap ($l_p=5$ mm), the plasma temperature reaches approximately 33,000 K and then decreases monotonically to approximately 30,000 K as the interelectrode gap increases to the maximum investigated value of 35 mm.

For the 40 wt.% Al + 60 wt.% Ti composition, the plasma temperature follows the linear relationship described by Equation (10). At an interelectrode gap of 5 mm, the plasma temperature is approximately 33,000 K. As the interelectrode gap increases to 35 mm, the plasma temperature gradually decreases to approximately 29,900 K.

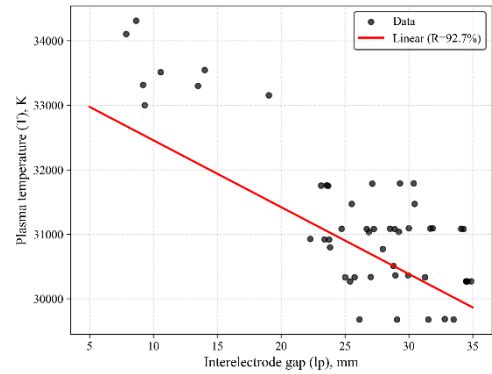
For the 60 wt.% Al + 40 wt.% Ti composition, the values predicted by Equation (11) exhibit a similar decreasing trend. The linear approximation provides the best fit to the data, with a correlation coefficient of $R^2=93.2$. The plasma temperature decreases gradually from approximately 32,850 K at the minimum interelectrode gap to approximately 30,300 K at an interelectrode gap of 35 mm.

For the Al-rich composition containing 80 wt.% Al + 20 wt.% Ti, the mathematical model represented by Equation (12) confirms the general trend. The predicted plasma temperature in the discharge channel decreases linearly from approximately 33,000 K at an interelectrode gap of 5 mm to approximately 30,000 K as the interelectrode gap increases to 35 mm.

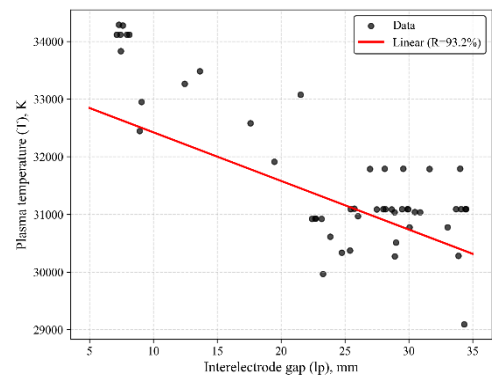
Overall, the results indicate that the composition of the Al–Ti powder system does not significantly affect either the nature of the relationship or the temperature gradient with respect to the interelectrode gap during HVED treatment by spark discharge in ethanol. All investigated compositions exhibit an almost identical linear decreasing trend with high statistical significance. From a physical standpoint, the monotonic decrease in plasma temperature with increasing interelectrode gap can be attributed to the proportional increase in the volume and lateral surface area of the plasma channel. The small differences in the absolute temperature values (within 1–2%) among the investigated compositions indicate that the thermodynamic characteristics of the spark discharge in ethanol are governed primarily by the energy characteristics of the discharge pulse and the intense decomposition of the working liquid. In contrast, the solid dispersed phase acts mainly as a stabilizing and heat-absorbing component.



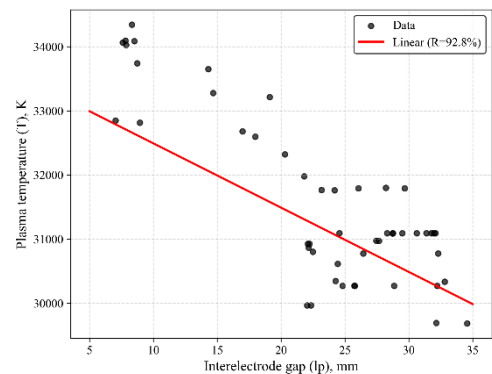
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(a) 20 wt.% Al + 80 wt.% Ti; (b) 40 wt.% Al + 60 wt.% Ti; (c) 60 wt.% Al + 40 wt.% Ti; (d) 80 wt.% Al + 20 wt.% Ti

Figure 4: Dependence of the the plasma temperature in the discharge channel on the interelectrode gap during HVED treatment of Al–Ti mixtures with different compositions in ethanol

4. Conclusions

1. The relationships governing the hydrodynamic characteristics of spark discharge in ethanol during HVED treatment of Al–Ti mixtures were established using machine learning algorithms. It was demonstrated that, for the analysis of highly nonlinear plasmadynamic processes, tree-based ensemble methods, particularly Gradient Boosting, provide the highest predictive accuracy by effectively capturing the complex interactions among mixture composition, interelectrode gap, and discharge energy parameters.

2. The discharge channel pressure was found to increase linearly from approximately 270 to 450 MPa as the interelectrode gap increased from 5 to 35 mm. This relationship was independent of the Al–Ti mixture composition and can be attributed to the proportional increase in plasma channel volume and the corresponding increase in energy released within the discharge channel.

3. The pressure acting on the discharge chamber walls was found to be highly sensitive to the powder composition. For Ti-rich mixtures, increasing the interelectrode gap reduced the chamber wall pressure because of enhanced attenuation of shock waves by the acoustically stiff titanium particles. In contrast, Al-rich mixtures (80 wt.% Al) exhibited the opposite trend, with increasing chamber wall pressure resulting from intensified gaseous phase generation.

4. A common linear decreasing trend in plasma temperature was identified for all investigated compositions. The plasma temperature decreased from approximately 33,000 to 30,000 K as the interelectrode gap increased from 5 to 35 mm. This behavior can be attributed to the increase in the lateral surface area of the plasma channel, which enhances plasma cooling by the surrounding heterogeneous medium.

5. The optimized Gradient Boosting predictive model demonstrated that the thermal characteristics of the process are governed primarily by the discharge pulse and the properties of the working liquid, whereas the peripheral hydrodynamic behavior is controlled mainly by the powder composition.

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5. References

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Complex-composition ceramics based on Al_2O_3 and basalt glass

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Abstract: To reduce the sintering temperature in the microstructural design of $\alpha\text{-Al}_2\text{O}_3$ -based ceramics, a mixture of basalt glass and yttria-stabilized zirconia (YSZ) was used. The effect of isothermal holding time during liquid-phase sintering of a composite containing 85 wt.% $\alpha\text{-Al}_2\text{O}_3$ and 15 wt.% (basalt glass + YSZ) on its mechanical properties was investigated. The samples were sintered at temperatures of 1450 and 1500 °C for 2–6 hours. It was established that sintering occurs in the presence of a liquid phase that persists throughout the entire holding period, and the densification of the material is determined by the rearrangement and Ostwald ripening processes of Al_2O_3 grains. The maximum hardness reached 7.73 GPa, while the fracture toughness reached 7.95 $\text{MPa}\cdot\text{m}^{1/2}$. The effect of microstructural evolution on the hardness and fracture toughness of the composite was demonstrated.

KEYWORDS: ALUMINA, BASALT GLASS, ZIRCONIA, LIQUID PHASE SINTERING, HARDNESS, FRACTURE TOUGHNESS.

1. Introduction

Al_2O_3 is characterized by one of the best cost-to-properties ratios among modern ceramic materials [1]. The widespread application of ceramic materials based on $\alpha\text{-Al}_2\text{O}_3$ in various industrial sectors is attributed to the combination of their chemical, mechanical, thermal, electrical, and optical properties. However, despite these advantages, the using of Al_2O_3 as a structural material is limited by its low fracture toughness [2]. Microstructural design of advanced structural materials based on $\alpha\text{-Al}_2\text{O}_3$ is aimed at reducing the sintering temperature and overcoming the brittleness problem [3], [4].

2. Prerequisites and means for solving the problem

The reduction of the sintering temperature of Al_2O_3 based ceramics below 1600–1700 °C is promoted by additives that form a liquid phase during heating. The liquid phase appears either the melting temperature of the additives is lower than the sintering temperature or new phases with melting temperatures below the sintering temperature are formed as a result of interactions between the components [4]. Various additives are used in the sintering of $\alpha\text{-Al}_2\text{O}_3$ based ceramics, among which multicomponent additives have attracted considerable attention [5] – [7]. This promotes to lowering the sintering temperature, increasing the density, and improving the mechanical properties of the ceramics.

To improve fracture toughness of Al_2O_3 -based ceramics, zirconia-toughened alumina (ZTA) have been developed. In these ceramics, particles of unstabilized ZrO_2 or a tetragonal solid solution based on ZrO_2 stabilized with Y_2O_3 are dispersed within an $\alpha\text{-Al}_2\text{O}_3$ matrix. The improvement in fracture toughness of ZTA composites is achieved through the combined action of transformation toughening caused by the stress-induced martensitic phase transformation of tetragonal ZrO_2 -based solid-solution particles into the monoclinic phase ($\text{T-ZrO}_2 \rightarrow \text{M-ZrO}_2$), together with microcracking, crack deflection, and the formation of compressive surface stresses. ZTA composites are typically sintered at 1600 °C [8] – [10].

3. Solution of the examined problem

Various types of natural rocks can be utilized in the development of materials with varied microstructures and physic mechanical properties due to their unique multicomponent mineralogical and chemical compositions [11]. Igneous basalt-like rocks, which undergo a special technological treatment involving melting, are finding increasingly widespread applications in the production of various materials and products [12]. Since rocks are products of the solidification of natural silicate melts, their melting begins with softening at 1050–1070 °C and progresses to intensive melting at approximately 1450 °C. Melts of igneous rocks differ from glass melts in that they are produced at higher temperatures and do not involve a silicate-forming stage. Unlike glasses obtained by melting a charge (a blend of raw materials in the required proportions), the components involved in the melting of rocks are not individual oxides but rather mineral compounds, including

pyroxene, augite, olivine, diopside, anorthite, and others. Furthermore, the structure characteristic of the parent rock, together with its specific mineralogical composition, is retained in the melt [13]. The use of basalt glass to reduce the sintering temperature of $\alpha\text{-Al}_2\text{O}_3$ -based ceramics is consistent with the current trend in oxide ceramic development, which is based on the principles of designing medium-entropy complex-composition oxide materials [14].

To the best of the authors' knowledge, studies on Al_2O_3 /basalt glass composites have not yet been reported in the literature.

In the present study, a blend of basalt glass (BG) and a powder of a tetragonal ZrO_2 -based solid solution stabilized with Y_2O_3 (YSZ) was used. ZrO_2 affects the physicochemical characteristics of the basalt glass melts; however, no chemical interaction between them occurs [15]. It is known that increasing the Al_2O_3 content raises the glass-transition and crystallization temperatures, increases the viscosity of rock-derived melts, and improves the mechanical properties of fibers [16]. However, data regarding the chemical interaction between Al_2O_3 and basalt glass melts are not available in the literature.

The aim of this study was to investigate the effect of the isothermal holding time during liquid-phase sintering on the mechanical properties of a composite with the composition 85 wt.% $\alpha\text{-Al}_2\text{O}_3$ –15 wt.% (BG + YSZ), where the liquid phase persists throughout the entire isothermal holding period.

Alumina powder of $\Gamma\text{-OO}$ grade and basalt glass powder were selected for the study; the latter was obtained by melting a rock of transitional composition between basic (basalts) and intermediate (andesites), followed by grinding in a ball mill. The powder of a ZrO_2 -based solid solution stabilized with 3 mol.% Y_2O_3 (YSZ) was synthesized by the hydrothermal method in an alkaline medium [17], [18].

A powder blend with the composition 85 wt.% $\alpha\text{-Al}_2\text{O}_3$ – 15 wt.% (BG + YSZ) was mixed in a ball mill in an isopropyl alcohol medium for 2 h and subsequently dried in air at 80 °C for 24 h. Green bodies were formed by cold uniaxial double-sided pressing in a steel die under a pressure of 50 MPa. A 5 wt.% aqueous solution of polyvinyl alcohol in distilled water was used as a binder during powder preparation.

The formed specimens (three samples for each sintering holding time) were dried in air at 80 °C for 24 h. The green compacts, with a diameter of 20 mm and a height of 6 mm, exhibited a relative density of 0.40. The samples were sintered in a Nabertherm LTH08/17 laboratory electric furnace at 1450 °C and 1500 °C for 2, 4, and 6 h, followed by cooling in furnace. Fracture surfaces and polished longitudinal cross-sections of the sintered specimens had been prepared for investigations.

The properties of the samples were investigated by X-ray diffraction (XRD) using a DRON-1.5 diffractometer equipped with a digital data acquisition system ($\text{CuK}\alpha$ radiation, scanning rate 1–4° min^{-1} , $2\theta = 15\text{--}90^\circ$) and by scanning electron microscopy (SEM) using an Axia ChemiSEM microscope (Thermo Fisher Scientific, USA). Mechanical properties were evaluated using a Falkon 509 hardness tester (Netherlands) with an indentation load of 300 N (30 kgf). The fracture toughness factor (K_{IC}) of the composites was calculated according to [19].

4. Results and discussion

The specific surface area of the initial powder mixture with the composition (wt.%) 85 α -Al₂O₃ – 15 (BG + YSZ) was 53.14 m²/g. The mixture contained (wt.%): 84.852 Al₂O₃, 6.763 SiO₂, 1.907 Fe₂O₃, 0.527 MgO, 3.190 ZrO₂, 0.262 Y₂O₃, 0.290 Gd₂O₃, 0.413 K₂O, and 0.215 TiO₂. The theoretical density, calculated using the rule of mixtures, was 3.67×10^3 kg/m³. According to the X-ray diffraction (XRD) analysis, reflections characteristic of α -Al₂O₃ and a low-angle amorphous halo associated with the presence of basalt glass in the mixture were identified.

It is well known that liquid-phase sintering is conventionally divided into three stages according to the dominant densification mechanisms: (1) viscous flow of the liquid phase accompanied by rearrangement of solid particles; (2) dissolution–precipitation, or recrystallization through the liquid phase; and (3) formation of a rigid skeleton followed by sintering controlled by solid-state diffusion mechanisms [20]. The temperatures of 1450 °C and 1500 °C correspond to the melting range of basalt glass. Regardless of the holding time, sintering of the composite with the composition 85 wt.% α -Al₂O₃ – 15 wt.% (BG + YSZ) were proceeded in the presence of a liquid phase that persists throughout the entire isothermal holding period. Therefore, the solid-state sintering stage was absent under the present experimental conditions.

The relative density of the sintered samples was determined by hydrostatic weighing (Archimedes method). After sintering at 1450 °C, the relative density increased to 0.89, whereas after sintering at 1500 °C it reached 0.96. The volumetric shrinkage of the specimens was 57–58%. Such high shrinkage is a characteristic feature of liquid-phase sintering and depends, to a certain extent, on the wettability of the solid phase by the liquid. During volumetric densification, the principal driving forces are capillary forces generated by the liquid phase [20].

It can be assumed that the decisive factor in the high shrinkage of the samples during sintering is the nanocrystallinity of the initial α -Al₂O₃ powder and the wettability of Al₂O₃ by the basalt glass melt. The main mechanisms responsible for shrinkage during liquid-phase sintering of the composite are particle rearrangement and Ostwald ripening of α -Al₂O₃ grains [21].

After 2 h of sintering at both temperatures, Al₂O₃ grains of rounded and platelet-like morphology with various sizes were observed in the samples (Fig. 1a, d). While nanosized rounded Al₂O₃ grains remained after sintering at 1450 °C, their size increased to 1–2 μ m after sintering at 1500 °C. The lateral dimension of the platelet-like grains varied from 2–5 μ m up to 10 μ m. It is evident that their size was considerably larger after sintering at 1500 °C. The diameter of rounded pores ranged from 1–2 μ m to 5–7 μ m. Elongated pores reached lengths of up to 10 μ m after sintering at 1450 °C, whereas their length decreased by approximately two times after sintering at 1500 °C (Fig. 1a, d).

When the holding time is increased to 4 hours during sintering, the basalt glass melt fills the large pores and pore channels, leaving exclusively isolated pores. After sintering at 1450 °C, the Al₂O₃ grain size was approximately 2–3 μ m. At 1500 °C, the size of Al₂O₃ grains of various morphologies increased by a factor of 1.5–2 (Fig. 1b, e).

The grains size of Al₂O₃ practically does not increase after sintering at 1450 °C for 6 hours (Fig. 1c). After sintering at 1500 °C, rounded Al₂O₃ grains up to 5 μ m in diameter and plate-like grains measuring up to up to 5–7 μ m remained. The number of Al₂O₃ grains in the form of plates up to 10 μ m in length increased (Fig. 1f). Isolated rounded pores were preserved in the samples.

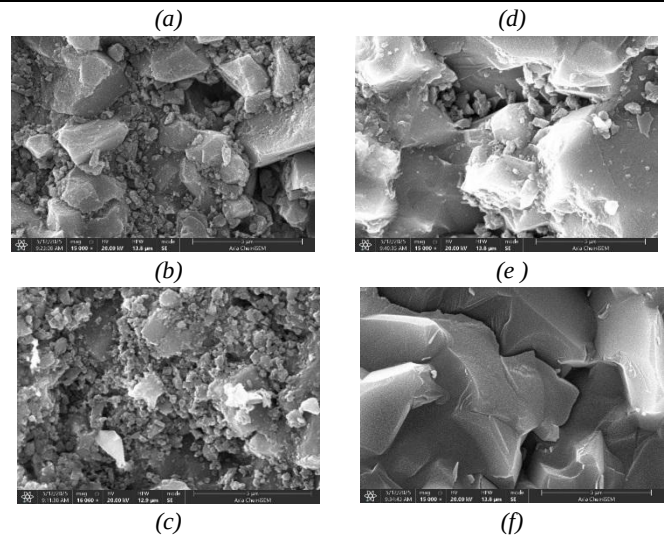
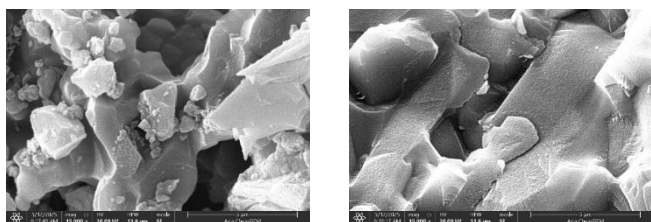


Fig. 1. SEM images of fracture surfaces of samples sintered for 2 h (a), 4 h (b), and 6 h (c) at 1450 °C, and for 2 h (d), 4 h (e), and 6 h (f) at 1500 °C

At both sintering temperatures, increasing the holding time resulted in the formation of agglomerates of irregularly shaped YSZ grains. This is a characteristic feature of ZrO₂ behavior in basalt glass melts [18]. It may therefore be assumed that, in the present case, the evolution of microstructural constituents has a greater influence on the mechanical properties of the composite with the composition 85 wt.% α -Al₂O₃ – 15 wt.% (BG + YSZ) than the presence of ZrO₂ itself.

Self-organization of the composite with the composition 85 wt.% α -Al₂O₃ – 15 wt.% (BG + YSZ) during sintering at 1450 °C leads to the formation of a two-scale structure. Within a matrix of smaller α -Al₂O₃ grain conglomerates, "densified" zones are formed, containing platelet-shaped α -Al₂O₃ grains up to 3 μ m in length within the solidified basalt glass (Fig. 2).

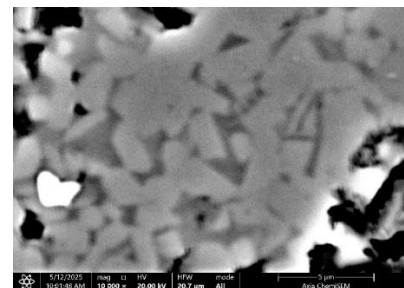


Fig. 2. Microstructure of the "densified" zones formed during sintering at 1450 °C. The bright phase corresponds to YSZ particles

The microstructural evolution during sintering at 1500 °C differs in that no "densified" zones are formed after 2 and 4 h of sintering. Under these conditions, isolated rounded and elongated pores are observed, with sizes decreasing to 1–2 μ m and approximately 10 μ m, respectively. Increasing the holding time to 6 h results in the formation of a two-scale microstructure within the composite. Isolated regions are formed in which platelet-like and polygonal Al₂O₃ grains are embedded in the solidified basalt glass melt.

At 1450 °C, the hardness of the ceramic continuously increases from 6.56 GPa to 7.56 GPa as the holding time is extended from 2 to 6 h (Fig. 3a). At 1500 °C, the hardness increases from 6.65 GPa to 7.71 GPa when the holding time is increased from 2 to 4 h and remains essentially unchanged (7.73 GPa) after 6 h of sintering (Fig. 3a).

The fracture toughness (K_{IC}) of the ceramic increases from 5.55 MPa·m^{1/2} to 5.95 MPa·m^{1/2} after sintering at 1450 °C. In contrast, after sintering at 1500 °C, the fracture toughness decreases from

7.95 MPa·m^{1/2} to 6.14 MPa·m^{1/2} when the holding time is increased from 4 to 6 h (Fig. 3b).

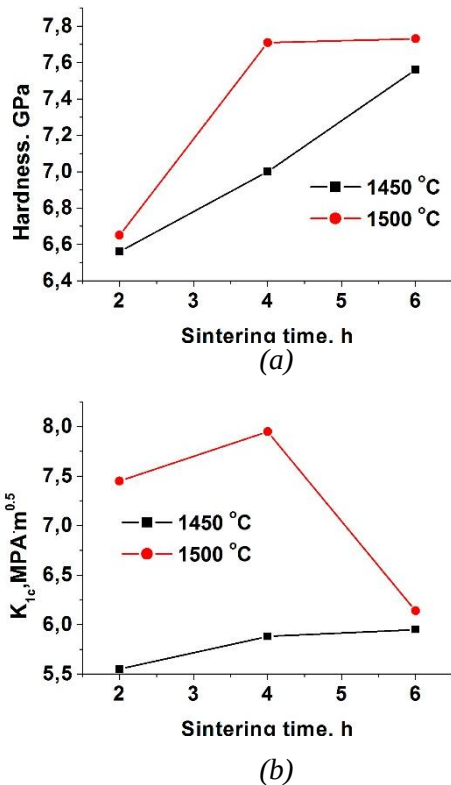


Fig. 3. Dependence of hardness (a) and fracture toughness, K_{IC} (b), of the sintered samples on the sintering holding time

The evolution of the composite microstructure results in an increase in hardness to 7.56 GPa and fracture toughness (K_{IC}) to 5.95 MPa·m^{1/2} in the presence of densified regions formed during sintering at 1450 °C. The absence of these regions and the higher density achieved after sintering at 1500 °C lead to increases in hardness and fracture toughness (K_{IC}) to 7.71 GPa and 7.95 MPa·m^{1/2}, respectively. However, the subsequent formation of densified regions during prolonged sintering at 1500 °C reduces the mechanical properties of the ceramic to values comparable to those obtained after sintering at 1450 °C.

5. Conclusions

Microstructural designing of ceramic composites is based on understanding the mechanisms of formation of the structure elements under various thermodynamic conditions, as well as the nature of their influence on the properties of ceramics.

Sintering of the composite with a composition of 85 wt.% α -Al₂O₃ – 15 wt.% (BG + YSZ) at 1450–1500 °C occurs in the presence of a liquid phase that persists throughout the entire isothermal holding period. The sintering behavior of the composite is predominantly determined by the rearrangement and Ostwald ripening of Al₂O₃ grains.

The features of structure formation during sintering are determined by the synergistic effect of α -Al₂O₃, YSZ, and basalt glass, due to the absence of interaction between them, as well as the evolution of the structural components. A characteristic feature is the formation of a two-scale microstructure. "Densified" zones are formed in the samples, containing α -Al₂O₃ platelets in the solidified basalt glass.

Upon the formation of a two-scale microstructure after sintering the composite at 1450 °C, its hardness increases to 7.56 GPa and the fracture toughness (K_{IC}) reaches 5.95 MPa·m^{1/2}. Raising the sintering temperature to 1500 °C and extending the holding time from 2 to 6 hours leads to an increase in the composite's hardness from 6.65 GPa to 7.73 GPa. The formation of the two-scale microstructure contributes to a decrease in fracture toughness from 7.95 MPa·m^{1/2} to 6.14 MPa·m^{1/2} as the holding time is increased from 4 to 6 hours.

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Precision determination of metal content in neutron super-mirrors by X-ray fluorescence method

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Abstract: Neutron polarizing super-mirrors are aperiodic multilayer structures made of alternating magnetic CoFe and non-magnetic TiZr layers deposited on glass or silicon substrates using the magnetron sputtering method. The polarizing ability of such super-mirrors and the efficiency of their use depend on the fine ratio of the thickness of the layers and the relative metal content in the layers [1, 2]. X-ray fluorescence analysis (XRF) is highly sensitive, but it has a rather large relative error, usually not less than 5%. An original X-ray spectrometer with a radioisotope source based on ¹⁰⁹Cd was used to determine the content of Fe, Co, Ti, and Zr in super-mirror samples with sufficient accuracy to control their manufacturing technology. The XRF analysis was based on the direct external standard method taking into account absorption corrections. As a result, the relative metal content in the super-mirror layers was determined with a relative error of less than 1%.

KEYWORDS: NEUTRON SUPER-MIRRORS, X-RAY FLUORESCENCE ANALYSIS, X-RAY SPECTRA OF Ti, Mn, Fe, Co, Sr, Zr

1. Introduction

Neutron super-mirrors are the basic components of neutron-optical devices such as neutron guides, neutron polarizers, and neutron beam polarization analyzers. They are used in neutron studies of the structure of new materials, biological objects, quantum liquids, superconductors, etc. Neutron super-mirror polarizing elements are aperiodic multilayer structures made of alternating magnetic, based on Fe and Co, and non-magnetic, based on Ti and Zr, layers deposited by magnetron sputtering on glass or silicon substrates [1,2]. The polarizing and reflecting ability of such super-mirrors depends on the fine ratio of the thickness of the layers and the relative metal content in the layer.

The X-ray fluorescence (XRF) analysis has a number of significant advantages over other methods for determining the elemental composition of a substance. The first and probably the most significant advantage is the possibility of conducting an elemental analysis without taking into account the aggregate state and type of chemical compound of the sample. The second is the relative simplicity and unambiguity of the X-ray spectrum, which facilitate the interpretation of the results. The third feature of the X-ray spectral method is its non-destructive nature, i.e. the sample is not destroyed during the examination. The non-destructive nature of the analytical signal excitation makes it possible to analyze unique samples and samples, and, if necessary, repeat measurements under different conditions, but with the same sample.

A significant limitation of the use of XRF methods is the presence of strong matrix effects, i.e. the influence of the bulk of the sample on the signal extraction from small components of its complex composition. In this case, the matrix is a substrate material that differs significantly in its elemental composition (silicon) from the analyzed metal layers. Modern XRF techniques are based on well-developed methods and techniques for performing individual operations. A special place is occupied by the processing of measurement results. Here, the development of computing capabilities opens up new prospects for significantly reducing the detection limits of small impurities in samples.

The main objective of this study was to develop a methodology for determining the relative content of Fe, Co, and Ti, Zr in samples of super-mirrors using the X-ray fluorescence method with an errors of less than 1%, sufficient for confident control of their manufacturing technology.

2. X-ray fluorescence spectrometer with a radioisotope source

Our XRF-spectrometer uses a ring radioisotope source based on ¹⁰⁹Cd [3]. Its activity at the time of manufacture was 0.36 GBq, which ensures a photon flux into a solid angle of 10⁻⁴ sr of the order of 10³ s⁻¹. It is a source of X-ray radiation with an average energy of about 22 keV and gamma radiation with an energy of 88 keV (the latter cannot be considered as an intense source of gamma radiation, but requires special protection measures).

A fundamental feature of the design of the irradiation chamber in the spectrometer [4] is that the primary radiation from the source falls on the sample, but due to the collimator it does not enter the detector. The fluorescent radiation from the sample freely enters the detector. The variable height of the irradiation chamber ensures optimal conditions for irradiation and registration of secondary radiation, for which an HPGe-based detector is used. The detector's working area is 30 mm², and the energy resolution is 200 eV for the K α line of iron with an energy of 6.4 keV. The detector operates at a "nitrogen" temperature, so the volume of the irradiation chamber is separated by a beryllium window from the detector area. The latter is surrounded by a multilayer (aluminum-cadmium-lead) passive detector protection from the external radiation.

In order to determine the contribution of the source's primary radiation to the background, two series of measurements were carried out. In the first one, the spectrometer worked without a sample and measured the background directly. In the second series, the primary radiation from the source was partially directed into the detector. The recording time of the pure background was 2 hours and the radiation from the source was 1 hour. The corresponding X-ray spectra, reduced to one second, are shown in Fig. 1.

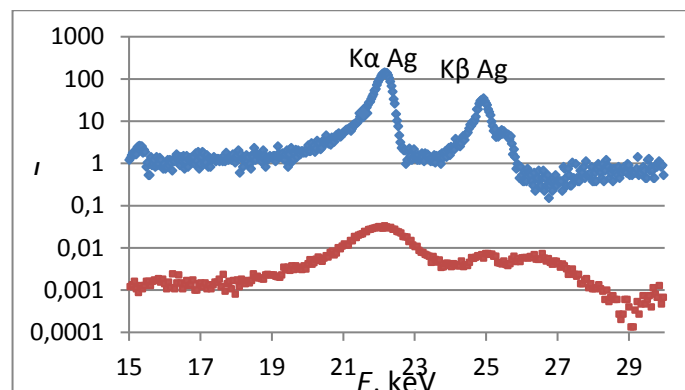


Fig. 1. Radiation spectra of the source (upper curve, blue dots) and background of the spectrometer (lower curve, red dots)

3. High-precision technique of XRF analysis

All XRF methods for determining the content of elements are based on comparing the line intensities of the characteristic X-ray spectra of the analyzed samples and reference samples. The so-called standard samples are often used as a reference sample. These are solids or liquids with precisely known elemental composition and concentrations, which are indicated in the sample passport. The error with which the content of the element in the reference sample is determined defines the systematic error of the method. It is generally assumed that this error should be no more than 1/3 of the error characterizing the reproducibility of the analysis method or its accuracy [5].

For the quantitative XRF analysis is most often used the direct method of the external standard [5,6]. The intensity of the analytical line is proportional to the concentration c_A^x of the detected element A in the sample:

$$I_x = \frac{K \cdot c_A^x}{(\mu_{m1}^x / \sin \varphi + \mu_{mi}^x / \sin \psi)} \quad (1)$$

For a reference sample –

$$I_0 = \frac{K \cdot c_A^0}{(\mu_{m1}^0 / \sin \varphi + \mu_{mi}^0 / \sin \psi)} \quad (2)$$

where

$$\mu_{m1} = c_A \mu_{m1}^A + (1 - c_A) \mu_{m1}^H \quad (3a)$$

$$\mu_{mi} = c_A \mu_{mi}^A + (1 - c_A) \mu_{mi}^H \quad (3b)$$

μ_{m1}^A, μ_{mi}^A – are the mass absorption coefficients of the atoms of the element being determined, respectively, of the primary radiation and the analytical line; μ_{m1}^H, μ_{mi}^H are the mass absorption coefficients of the packing atoms of the primary radiation and the analytical line. The angles φ and ψ are the angles of incidence of the primary radiation on the sample and the departure of the fluorescent radiation, respectively.

When determining the desired concentration using the direct method of the external standard, the intensity ratio is applied

$$\frac{I_x}{I_0} = \frac{c_A^x}{c_A^0} \cdot \frac{\mu_{m1}^0 / \sin \varphi + \mu_{mi}^0 / \sin \psi}{\mu_{m1}^x / \sin \varphi + \mu_{mi}^x / \sin \psi} \quad (4)$$

If the groups of the samples and the reference sample have the same chemical composition, and the concentration of the element to be determined in the samples varies little, then with a small assumption it can be assumed that the values of μ_{m1}^x and μ_{mi}^x are constant for all samples. With a reasonable choice of the concentration of element A in the comparison sample in relation to the concentrations of this element in the analyzed samples, the second multiplier on the right side of expression (4) is approximately equal to one, and the ratio is valid

$$c_A^x = \left(\frac{I_x}{I_0} \right) c_A^0 \quad (5)$$

Experimentally, the intensity is defined as the area of the analytical peak in the spectrum. The peak shape was approximated by the Gaussian function. The parameters of the analytical peaks, amplitude and width, were calculated using the method of the first and second moments. When processing the experimental spectra, the background was previously subtracted.

The errors are calculated using standard formulas. Statistical errors in analytical measurements are related to the discrete nature of secondary radiation detection, and the number of pulses in a given analyzer channel is a useful signal. The expected standard deviation (variance) of the total count is equal to the square root of the number of pulses N . When calculating the error of the intensity of the analytical line, which is equal to the area under the line in the spectrum, one speaks of the "full" standard deviation σ_t :

$$\sigma_t^2 = \sigma_N^2 + \sigma_1^2 + \sigma_2^2 + \sigma_3^2 + \dots \quad (6)$$

where σ_N is a statistical error, and σ_i are errors related to instrument error or calibration, operator actions, sample preparation, etc. These errors are often attributed to the background.

A fundamental feature of X-ray diffraction is the possibility of reducing the relative error and detection limits with increasing exposure time, i.e. increasing the statistics of X-ray spectra. Usually, the relative error for standard samples is much less than the error of the test sample. In this case, the latter is determined by the statistical error in determining the intensity of the analytical line.

4. Results

To determine the elemental composition of the neutron super-mirror, the state standard sample KS-1, salts of Ti and Sr, and metallic Zr in the form of powders were used as reference samples. The weight of the super-mirror sample was 19.2(1) mg. The time to set the spectra of the test sample was 10000 seconds. XRF spectra of the test and reference samples are shown in Fig. 2 and Fig. 3. The spectra are reduced to a registration time of 1 second. The results of the determination of the content of Mn, Fe, Co, Ti, Zr and Sr along the $K\alpha_1$ line in a sample of a neutron super-mirror are presented in Table 1. The relative content of the "working" metals Fe, Co, Ti,

and Zr (% by weight) in the neutron super-mirror, which showed the best properties, is shown in Table 2.

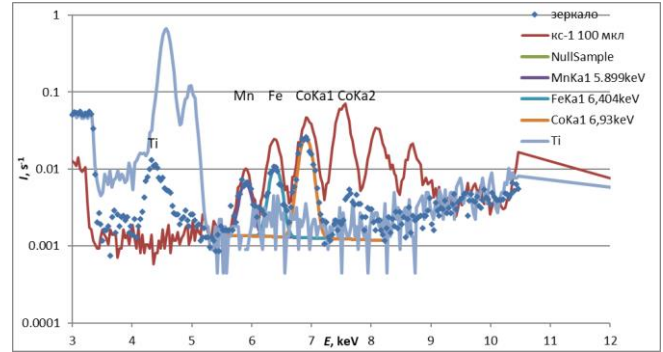


Fig. 2. Fluorescence X-ray spectra of a super-mirror sample and reference samples of Ti, Mn, Fe, Co ("soft" part).

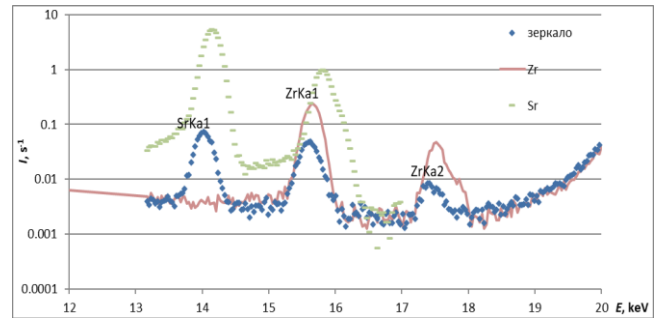


Fig. 3. Fluorescence X-ray spectra of a super-mirror sample and reference samples of Sr and Zr (the "hard" part)

Table 1. Metal content in the neutron super-mirror.

Element	Mn	Fe	Co	Ti	Zr	Sr
Concentration	0.16(5)	0.21(3)	0.54(3)	0.72(3)	0.114(3)	0.83(5)
C, % by weight	0.031(9)	0.040(6)	0.103(9)	0.14(2)	0.022(2)	0.16(1)

Table 2. The relative content of Fe, Co, Ti, and Zr in the super-mirror

Element	Fe	Co	Ti	Zr
Relative content, % by weight	13.25(3)	34.04(7)	45.5(1)	7.24(1)

5. Conclusions

The paper describes a precision technique for X-ray fluorescence determination of the content of Fe, Co, Ti, and Zr in neutron super-mirrors. The main part of this technique is the use of a spectrometer with a radioisotope source. The main factors influencing the result are the activity of the radioisotope source and the energy range of its radiation, the preparation of reference samples with the appropriate elemental composition, and the processing of secondary radiation spectra.

As a result, the following metal content was determined in a sample of a super-mirror with the best polarizing properties: Fe - 0.21(1)%; Co - 0.54(3)%; Ti - 0.72(3)%; Zr - 0.114(3) %. These results allowed us to determine the relative metal content in the super-mirror layers: Fe/Co - 28/72 and Ti/Zr - 86/14, with an error of less than 1%.

The principal conclusion of the work is that the proposed precision XRF technique makes it possible to control the process of magnetron sputtering of metals onto a substrate for the production of neutron polarizing super-mirrors with specified properties.

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Transformation of waste polystyrene into high surface area activated carbon: structural and surface insights

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Abstract: Activated carbon was prepared from waste polystyrene through acid treatment and pyrolysis to obtain a porous adsorbent with controlled structural features. Nitrogen adsorption reveals a type IV isotherm with H4 hysteresis and a high specific surface area of 1382 m²/g, indicating combined micro- and mesoporosity. SEM imaging shows irregular carbon agglomerates with a highly textured surface, while elemental and FTIR analyses confirm an aromatic carbon matrix with limited heteroatom functionality. These structural and chemical features demonstrate that the synthesized carbon material is well-developed and suitable for further application-oriented studies, including detailed adsorption performance evaluation.

Keywords: ACTIVATED CARBON, POLYSTYRENE WASTE, PYROLYSIS, SURFACE CHARACTERISTICS

1. Introduction

Plastic waste has become one of the most persistent environmental challenges of modern society [1]. The continuous growth in global plastic production, combined with limited recycling capacity, results in the accumulation of large quantities of polymeric materials in landfills and natural ecosystems. Polystyrene, widely used in packaging and consumer products, is particularly problematic due to its high consumption rate, low biodegradability, and the scarcity of efficient recycling routes. Once released into the environment, polystyrene fragments slowly degrade into microplastics, which disperse through soil and water systems and may enter biological food chains. Their presence has raised concerns about potential ecological toxicity and long-term impacts on human health [2].

Beyond environmental persistence, the production and disposal of plastics contribute significantly to greenhouse gas emissions, reinforcing the need for sustainable waste-management strategies. In recent years, the concept of converting polymeric waste into value-added carbon materials has gained attention as a promising approach to both waste reduction and resource recovery. Polystyrene, with its aromatic backbone and high carbon content, is a suitable precursor for producing activated carbon through controlled thermochemical treatment [3, 4].

Transforming waste polystyrene into activated carbon not only mitigates environmental pollution but also provides access to porous materials with desirable structural and surface characteristics. Such carbons are relevant for adsorption, catalysis, energy storage, and other technological applications [4].

The present study focuses on the synthesis and characterization of activated carbon obtained from waste polystyrene through acid pretreatment and pyrolysis. The aim is to examine the development of porosity, morphology, and surface chemistry in the resulting material and to establish its potential for further application-oriented research.

2. Materials and Methods

2.1. Preparation of the Carbon Material

The activated carbon was synthesized through a two-stage thermochemical conversion process involving acid-induced oxidation followed by high-temperature pyrolysis. In the first stage, the waste polystyrene was melted and subjected to oxidation with concentrated sulfuric acid at a 1:1 weight ratio. The mixture was maintained at 350 °C under continuous stirring until a solid carbonaceous product was formed. This treatment promoted extensive structural rearrangement and partial aromatization of the polymer matrix, preparing the material for subsequent carbonization.

In the second stage, the obtained solid was pyrolyzed at 900 °C in a carbon dioxide atmosphere. The temperature was increased at a controlled heating rate of 10 °C/min, and the sample was held at the

final temperature to ensure complete thermal decomposition and pore development. After cooling to room temperature under CO₂ flow, the resulting carbon material was collected, and stored for further characterization. This combined oxidation-pyrolysis route enabled the formation of a porous, predominantly aromatic carbon structure suitable for detailed textural and surface analysis.

2.2. BET Surface Area and Porosity Characterization

Nitrogen adsorption-desorption isotherms at 77 K were recorded to evaluate the specific surface area and textural properties of the carbon material. Prior to analysis, the samples were degassed under vacuum at elevated temperature to remove adsorbed moisture and volatile species. The amount of nitrogen adsorbed on the carbon surface was measured over a range of relative pressures, and the specific surface area was calculated according to the Brunauer-Emmett-Teller (BET) method. Pore size distribution and total pore volume were obtained from the desorption branch using the Barrett-Joyner-Halenda (BJH) model. Measurements were performed using a Quantachrome gas sorption analyzer.

2.3. SEM/EDS analysis

Scanning electron microscopy (SEM) was used to examine the morphology of the carbon material, including particle structure, surface texture and pore development. The samples were mounted on conductive stubs and sputter-coated to minimize charging during imaging. Elemental composition was determined by energy-dispersive X-ray spectroscopy (EDS) integrated into the SEM system, providing qualitative and quantitative information on the main elements present in the material.

2.4. FTIR analysis

Fourier-transform infrared (FTIR) spectroscopy was used to identify the surface functional groups present in the carbon material. Spectra were recorded in the mid-infrared region, and characteristic absorption bands were analyzed to evaluate the presence of aromatic structures, oxygen-containing functionalities and other groups formed or modified during the oxidation and pyrolysis steps.

2.5. Raman analysis

Raman spectroscopy was employed to assess the structural ordering of the carbon material. The spectra were used to evaluate the relative intensity of the D and G bands, providing insight into the degree of graphitization, defect density and the overall structural evolution resulting from the thermochemical conversion of polystyrene.

3. Results and Discussion

3.1. Textural Properties

N₂ adsorption at 77 K reveals a type IV isotherm with an H4 hysteresis loop, characteristic of materials combining microporosity with slit-like mesopores (Fig.1) [5]. The steep increase in adsorbed volume at low relative pressures ($p/p_0 < 0.05$) indicates well-

developed microporosity, while the presence of hysteresis confirms the contribution of mesopores to the overall pore structure.

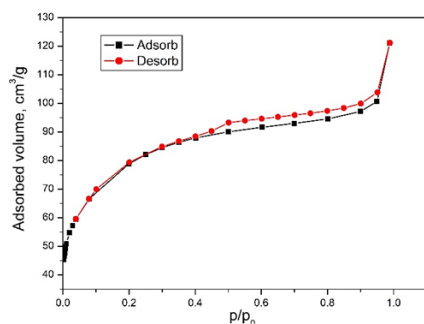


Fig. 1 N_2 adsorption-desorption isotherm at 77.4 K for the activated carbon

The carbon material exhibits a highly developed and hierarchically organized pore structure (Table 1). The exceptionally high specific surface area (1382 m²/g) confirms the dominant contribution of micropores, which account for approximately 41% of the total pore volume. Mesopores and macropores form comparable fractions of the structure, supporting efficient diffusion and enhanced mass transport. Overall, the combination of micro-, meso- and macropores provides both strong adsorption potential and favorable transport properties, characteristic of carbon materials produced through acid oxidation followed by high-temperature CO₂ activation.

Table 1: Specific surface area and textural parameters of the activated carbon

Textural parameter	Measured value
Total pore volume, cm ³ /g	1.025
Micropore volume, cm ³ /g	0.424
Mesopore volume, cm ³ /g	0.284
Macropore volume, cm ³ /g	0.317
BET specific surface area, m ² /g	1382

3.2. Morphology and Elemental Composition

The morphology of the activated carbon was examined by scanning electron microscopy (Figure 2). The SEM images reveal irregular, fragmented particles with a rough and highly textured surface. Numerous surface irregularities, fissures and pore openings are visible, consistent with the hierarchical pore structure identified through nitrogen adsorption. This morphology is typical for activated carbons obtained through thermochemical conversion of polymeric precursors.

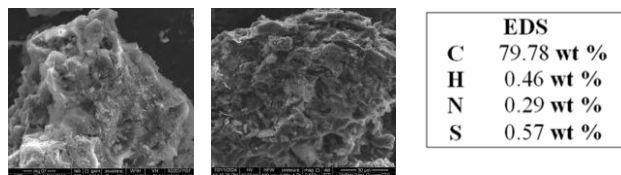


Fig. 2 SEM morphology and EDS elemental composition of the activated carbon

The elemental composition (Fig. 2) is dominated by carbon (79.78%), confirming efficient carbonization of the polystyrene precursor. The very low hydrogen (0.46%) and nitrogen (0.29%) contents are characteristic of highly aromatized carbon structures formed during high-temperature pyrolysis. A minor sulfur content (0.57%) reflects the acid treatment step and suggests the presence of sulfur-containing surface functionalities. Overall, the elemental profile aligns with the observed morphology and the textural parameters derived from nitrogen adsorption, supporting the

conclusion that the material is extensively carbonized and possesses a well-developed porous structure.

3.3. FTIR spectrum of the activated carbon

The FTIR spectrum of the activated carbon (Fig. 3) displays weak and broad absorption bands, typical of highly carbonized materials with a limited number of surface functional groups. The broad band at 3435 cm⁻¹ corresponds to O–H stretching vibrations, most likely associated with adsorbed moisture. The weak peaks in the 2969 – 2852 cm⁻¹ region arise from residual aliphatic C–H groups. The band at 1631 cm⁻¹ is attributed to aromatic C=C vibrations and contributions from adsorbed water.

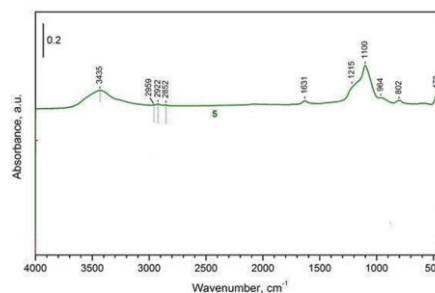


Fig. 3 FTIR spectrum of the activated carbon

Several bands in the 1215 – 1100 cm⁻¹ region indicate C–O stretching in oxygen-containing functionalities and may also reflect S–O/S=O vibrations introduced during H₂SO₄ treatment, consistent with the elemental analysis. The peaks at 964 and 802 cm⁻¹ correspond to out-of-plane deformations of aromatic C–H bonds, while the low-frequency band at 473 cm⁻¹ is associated with skeletal vibrations of the carbon matrix [6].

Overall, the FTIR spectrum confirms the presence of an aromatized carbon structure containing small amounts of oxygen- and sulfur-bearing groups, in agreement with the high degree of carbonization indicated by the textural, morphological and elemental analyses.

3.4. Raman analysis of the activated carbon

The Raman spectrum of the activated carbon (Fig. 4) is characterized by two prominent bands: the D band at ~1350 cm⁻¹ and the G band at ~1580 – 1600 cm⁻¹, accompanied by a weaker and broader 2D band near 2700 cm⁻¹. The relatively high intensity ratio I_D/I_G indicates a substantial degree of structural disorder and the presence of small graphitic domains. This spectral profile is typical of turbostratic carbon, where graphene layers are misaligned and only weakly ordered.

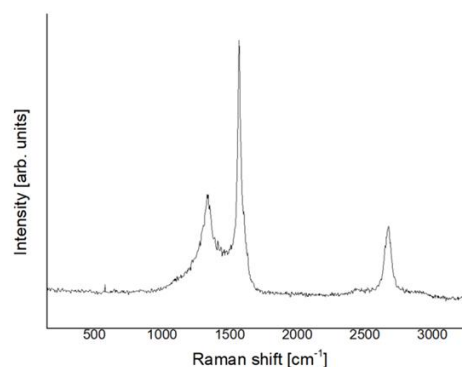


Fig. 4 Raman spectrum of the activated carbon

The Raman results are consistent with the textural data, which reveal a high specific surface area and a well-developed porous structure. Overall, the spectrum confirms that the material is a defect-rich, turbostratic carbon with low crystallinity and an extended network of sp²-hybridized domains, supporting its potential for adsorption and catalytic applications [7, 8].

4. Conclusion

The comprehensive characterization of the activated carbon demonstrates that the applied synthesis route – acid treatment of the polystyrene precursor followed by high-temperature CO₂ activation – successfully produces a highly porous, defect-rich carbon material with well-developed surface functionality. Nitrogen adsorption measurements reveal a hierarchical pore structure dominated by micropores and complemented by meso- and macropores, resulting in a high specific surface area and favorable mass-transport properties. SEM imaging confirms the presence of irregular, fragmented particles with a rough and textured surface, while EDS analysis verifies efficient carbonization and the incorporation of oxygen- and sulfur-containing groups originating from the oxidation step.

FTIR spectroscopy further supports the formation of an aromatized carbon matrix enriched with limited but identifiable oxygen- and sulfur-bearing functionalities. Raman analysis indicates a turbostratic, defect-rich structure with a high I_D/I_G ratio, consistent with small graphitic domains and low crystallinity. Together, these findings confirm that the obtained material is a highly disordered, microporous activated carbon with an extended network of sp²-hybridized domains.

Overall, the structural, morphological and spectroscopic results demonstrate that the synthesized activated carbon possesses properties favorable for adsorption and catalytic applications, including high surface area, hierarchical porosity, abundant defect sites and tailored surface chemistry. These characteristics highlight its potential for use in processes requiring efficient mass transfer, strong adsorptive interactions and tunable surface reactivity.

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Utilization of polystyrene waste for the development of an efficient adsorbent for water purification

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Abstract: Activated carbon derived from polystyrene waste was evaluated as an adsorbent for the removal of the reactive dye Drimaren Red K-7B from aqueous solutions at different temperatures. The adsorption equilibrium was analyzed using Langmuir, Freundlich and Dubinin-Radushkevich models, with the latter providing the best fit ($R^2=0.97$). Kinetic data followed the pseudo-second-order model at all temperatures ($R^2=0.99$). The maximum adsorption capacity reached 35 mg/g at 20 °C. Thermodynamic analysis revealed negative Gibbs free energy values, confirming the spontaneous nature of the process, while positive enthalpy and entropy indicated an endothermic mechanism accompanied by increased interfacial disorder. These results demonstrate the potential of polystyrene-derived activated carbon as an efficient adsorbent for water treatment applications.

Keywords: ACTIVATED CARBON, POLYSTYRENE WASTE, ADSORPTION, ADSORPTION MODELING, WATER PURIFICATION

1. Introduction

Polystyrene waste represents a significant fraction of global plastic pollution due to its extremely low biodegradability and widespread use in packaging, construction, electronics and medical applications. Conventional waste-management strategies such as mechanical recycling, landfilling and incineration often show limited efficiency and may lead to downcycling. In this context, the concept of upcycling has gained increasing attention, aiming to convert polystyrene waste into value-added materials with improved functional properties. Thermochemical conversion routes are particularly promising, as they enable the production of carbonaceous materials with developed porosity and potential for further functionalization [1].

Recent studies indicate that polystyrene-derived materials can exhibit notable adsorption performance toward organic dyes, highlighting their potential as low-cost sorbents for water treatment [2, 3]. Reactive dyes, widely used in the textile industry, are resistant to biodegradation and can cause severe coloration, reduced light penetration and ecological toxicity even at low concentrations. Therefore, the development of efficient and sustainable adsorbents for their removal remains an important technological challenge [4, 5].

This study focuses on the upcycling of polystyrene packaging waste into activated carbon and on the evaluation of its adsorption performance toward a model reactive dye. The adsorption behavior was examined through equilibrium, kinetic and thermodynamic analyses to assess the applicability of the material for water purification.

2. Materials and methods

2.1. Preparation of the carbon material

The activated carbon used in this study was synthesized from waste polystyrene through high temperature CO₂ activation. The polystyrene precursor was carbonized and subsequently activated under a CO₂ flow at elevated temperature, yielding a porous carbon material. The structural, textural and surface chemical properties of the obtained adsorbent were characterized in a separate study, confirming its suitability for the removal of organic contaminants.

2.2. Adsorption experiments

The adsorption experiments were performed in batch mode using a fixed volume of dye solution and continuous magnetic stirring at 300 rpm to ensure homogeneous suspension and effective contact between the adsorbent and the dye molecules. A mass of 0.025 g of the activated carbon was added to 12.5 mL of aqueous Drimaren Red K-7B solutions. To evaluate the effect of the initial dye concentration, solutions in the range of 10–100 mg/L were prepared. The influence of temperature on the adsorption process

was examined at 20 °C, 40 °C and 60 °C under identical experimental conditions.

The change in dye concentration during the adsorption process was monitored using a UV-Vis spectrophotometer (ONDA UV-31 SCAN), recording absorbance at $\lambda = 548$ nm in the range 190–600 nm. The adsorption capacity was calculated according to:

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (1)$$

where q_e is the adsorption capacity (mg/g), C_0 and C_e are the initial and equilibrium dye concentrations (mg/L), V is the solution volume (L), and m is the mass of adsorbent (g). The percentage of dye removal was determined using:

$$D(\%) = \frac{C_0 - C_e}{C_0} 100 \quad (2)$$

Isotherm experiments were conducted at 20 °C using initial dye concentrations of 10–100 mg/L, a contact time of 60 min and an adsorbent dose of 0.025 g. Kinetic experiments were performed at initial dye concentration of 100 mg/L, contact times between 5 and 60 min, and temperatures of 20 °C, 40 °C and 60 °C.

Equilibrium, kinetic and thermodynamic analyses were performed using commonly applied adsorption models, and the corresponding parameters were obtained from experimental data collected at different initial dye concentrations, contact times and temperatures.

3. Results and discussion

3.1. Effect of initial dye concentration

The effect of the initial dye concentration on the adsorption performance of the polystyrene-derived activated carbon is presented in Fig.1. The adsorption capacity increased steadily from 3 mg/g at 10 mg/L to 35 mg/g at 100 mg/L, indicating that higher dye concentrations provide a stronger driving force for mass transfer toward the adsorbent surface.

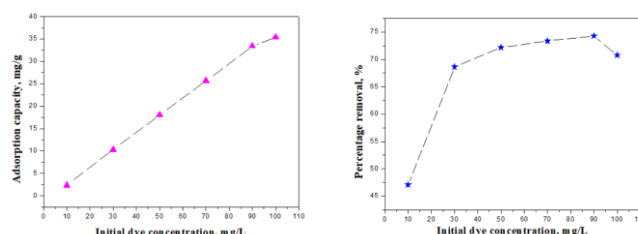


Fig.1 Effect of initial dye concentration on adsorption capacity and removal efficiency

In contrast, the percentage removal exhibited a different trend: it increased from 45% to approximately 75% in the range 10-90 mg/L, followed by a slight decrease at 100 mg/L.

This behavior reflects the interplay between the number of dye molecules in solution and the availability of active sites on the carbon surface. At low concentrations, the limited number of dye molecules results in lower removal efficiency, despite the presence of numerous unoccupied adsorption sites. As the concentration increases, more dye molecules interact with the surface, leading to higher adsorption capacity and improved removal efficiency. At the highest concentration, partial saturation of the active sites likely occurs, which reduces the percentage removal even though the adsorption capacity continues to rise.

Overall, the results confirm that the adsorption process is strongly influenced by the initial dye concentration, with higher concentrations enhancing the driving force for adsorption but also approaching the limits of surface saturation.

3.2. Effect of contact time and temperature on dye removal efficiency

The effect of contact time and temperature on the adsorption performance of the polystyrene-derived activated carbon is illustrated in Fig. 2. The percentage removal increased continuously with time for all temperatures, indicating progressive occupation of the available adsorption sites. Higher temperatures resulted in markedly higher removal efficiencies: at 60 °C the system reached above 90% removal within 60 min, whereas at 20 °C the maximum removal remained below 80%. This clear separation between the kinetic curves confirms that the adsorption process is temperature-dependent and exhibits endothermic behavior.

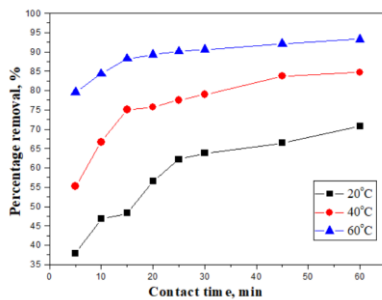


Fig.2 Effect of contact time and temperature on dye removal efficiency

The enhanced adsorption at elevated temperatures can be attributed to several factors. First, increasing temperature accelerates the diffusion of dye molecules through the boundary layer surrounding the adsorbent particles, reducing external mass-transfer resistance. Second, higher thermal energy facilitates intraparticle diffusion within the porous structure of the carbon, allowing dye molecules to access deeper adsorption sites. Third, temperature may promote partial activation or increased mobility of surface functional groups, effectively increasing the number of energetically favorable adsorption sites. As a result, both the rate of adsorption and the final removal efficiency improve with temperature.

In summary, the kinetic profiles demonstrate that the adsorption of Drimaren Red K-7B onto the polystyrene-derived activated carbon is strongly influenced by temperature, with higher temperatures accelerating mass transfer and enabling more efficient utilization of the adsorbent's porous structure. These observations are consistent with the thermodynamic parameters discussed later, which further confirm the endothermic nature of the process.

3.3. Isotherm analysis

Adsorption isotherms describe the relationship between the equilibrium dye concentration and the amount adsorbed onto the activated carbon, providing insight into the surface properties and adsorption mechanism. The equilibrium adsorption data were evaluated using the Langmuir (Eq. 3), Freundlich (Eq. 4) and Dubinin-Radushkevich (Eq. 5, 6, 7) isotherm models. The corresponding linearized equations are:

$$\frac{C_e}{q_e} = \frac{1}{K_L \cdot q_{\max}} + \frac{C_e}{q_{\max}} \quad (3)$$

where $q_{\max}$ (mg/g) is the monolayer capacity and K_L (L/mg) is the Langmuir constant.

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

Where K_F ((mg/g)) is the Freundlich constant and n indicates adsorption intensity.

$$\ln q_e = \ln q_{\max} - K_{DR} \varepsilon^2 \quad (5)$$

$$\varepsilon = RT \ln \left(1 + \frac{1}{C_e} \right) \quad (6)$$

The mean adsorption energy is:

$$E = \frac{1}{\sqrt{2K_{DR}}} \quad (7)$$

where K_{DR} is Dubinin-Radushkevich constant related to adsorption energy (mol^2/kJ^2); ε is Polanyi potential, describing the work required to transfer a molecule from solution to the adsorbent surface; E is mean adsorption energy (kJ/mol), used to distinguish physical adsorption ($E < 8$ kJ/mol) from stronger interactions.

The equilibrium data were fitted to the Langmuir, Freundlich and Dubinin-Radushkevich isotherm models, and the obtained parameters are summarized in Table 1. The Langmuir model yielded a relatively low correlation coefficient ($R^2=0.44$), indicating that monolayer adsorption on a homogeneous surface does not adequately describe the system. In contrast, the Freundlich model showed a much better fit ($R^2=0.94$), with $n=0.63$, suggesting adsorption on a heterogeneous surface and the presence of sites with different affinities. The Dubinin-Radushkevich model provided the highest correlation ($R^2=0.97$), and the calculated mean adsorption energy $E=5.77$ kJ/mol indicates that the process proceeds predominantly through physical adsorption [6].

Table 1: Fitted Parameters of Langmuir, Freundlich and Dubinin-Radushkevich isotherms.

Model	Key parameters	R^2
Langmuir	$q_{\max} = 22.22$ mg/g, $K_L = 0.045$ L/mg	0.44
Freundlich	$K_F = 0.2248$ mg/g, $n = 0.63$	0.94
Dubinin-Radushkevich	$q_{\max} = 31.44$ mg/g, $K_{DR} = 14.99 \cdot 10^{-3} \text{ mol}^2/\text{kJ}^2$, $E = 5.77$ kJ/mol	0.97

3.4. Adsorption kinetic models

The adsorption kinetics were evaluated using the pseudo-first-order (PFO), (Eq. 8), pseudo-second-order (PSO), (Eq. 9) and intraparticle diffusion models (Weber-Morris), (Eq. 10). These kinetic models were applied to describe the rate of dye uptake and to identify the controlling steps in the adsorption process. The linearized forms of the equations were used to determine the kinetic constants and to assess the suitability of each model for describing the experimental data.

$$\lg(q_e - q_t) = \lg q_e - \left(\frac{k_1}{2.303} \right) t \quad (8)$$

$$\frac{t}{q_t} \left(\frac{1}{k_2 q_e^2} \right) + \left(\frac{t}{q_e} \right) \quad (9)$$

$$q_t = k_{id} t^{1/2} + C \quad (10),$$

where q_t is adsorption capacity at time t (mg/g); q_e – equilibrium adsorption capacity (mg/g); k_1 – PFO rate constant (1/min); k_2 – PSO rate constant (g/mg.min); k_{id} – intraparticle diffusion rate constant; C – boundary layer thickness parameter.

Based on the experimentally obtained kinetic data (Table 2) both PFO and PSO equations provided good fits to the experimental data; however, the PSO model showed consistently higher correlation coefficients ($R^2 > 0.99$), indicating that the adsorption

process is better described by a second order kinetic mechanism. This suggests that the rate of dye uptake is primarily governed by the availability of active sites on the adsorbent surface [7].

The Weber-Morris plots were used to assess the contribution of intraparticle diffusion to the overall adsorption rate. The increasing values of the boundary layer thickness parameter C with temperature indicate enhanced external mass transfer resistance [8]. The intraparticle diffusion rate constant k_{id} increased from 20°C to 40°C and then decreased at 60°C, reflecting temperature dependent changes in dye mobility within the pores. Although diffusion contributes to the overall adsorption process, the relatively low correlation coefficients ($R^2=0.84-0.96$) demonstrate that intraparticle diffusion cannot be considered the sole rate controlling step.

Taken together, the kinetic analysis confirms that the adsorption of Drimaren Red K-7B proceeds through a multi step mechanism, where surface interactions dominate, while diffusion plays a secondary but non negligible role.

Table 2: Kinetic model parameters at different temperatures

T (°C)	Model	R ²	k ₁ /k ₂ /k _{id} (1/min or g/mg.min)	q _e mg/g	C
20	PFO	0.96	36*10 ⁻³	20.56	-
20	PSO	0.99	3.7*10 ⁻³	39.04	-
20	Diffusion	0.96	2.27	-	18.1
40	PFO	0.95	78*10 ⁻³	21.11	-
40	PSO	0.99	65*10 ⁻³	44.66	-
40	Diffusion	0.84	2.45	-	25.52
60	PFO	0.97	59*10 ⁻³	7.56	-
60	PSO	0.99	17*10-3	47.41	-
60	Diffusion	0.85	1.14	-	38.7

3.5. Thermodynamic parameters

To further elucidate the nature of the adsorption process, the thermodynamic parameters ΔG° , ΔH° and ΔS° were calculated based on the temperature-dependent equilibrium data (Table 3). The thermodynamic parameters (ΔG° , ΔH° , ΔS°) were determined from the equilibrium constants using the Van't Hoff approach [9]. These parameters provide insight into the spontaneity, energetic changes and molecular ordering associated with the adsorption of Drimaren Red K-7B onto the activated carbon. The values of the equilibrium constant at different temperatures were used to construct the Van't Hoff plot, from which the enthalpy and entropy changes were determined, while the Gibbs free energy was subsequently calculated to assess the feasibility of the adsorption process.

Table 3: Thermodynamic parameters (ΔG° , ΔH° , ΔS°) for the adsorption of Drimaren Red K-7B

T (°C)	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/mol.K)
20	-0.46	35	120
40	-2.65	35	120
60	-5.37	35	120

The thermodynamic parameters indicate that the adsorption of Drimaren Red K-7B onto the activated carbon is spontaneous at all studied temperatures, as evidenced by the negative values of ΔG° . The positive enthalpy change ($\Delta H^\circ \approx +35$ kJ/mol) confirms that the process is endothermic, while the positive entropy change ($\Delta S^\circ \approx +120$ J/mol.K) suggests increased randomness at the solid-liquid interface during adsorption. The more negative ΔG° values at higher temperatures further demonstrate that the adsorption becomes thermodynamically more favorable with increasing temperature.

3.6. Mechanism of adsorption

The combined kinetic and thermodynamic results indicate that the adsorption of Drimaren Red K-7B onto the activated carbon proceeds through a multi-step mechanism. The excellent fit of the pseudo-second-order model suggests that surface interactions and the availability of active sites play a dominant role in the adsorption process. The Weber-Morris analysis shows that intraparticle diffusion contributes to the overall rate but is not the sole rate-controlling step. The positive enthalpy change ($\Delta H^\circ \approx +35$ kJ/mol) confirms that the adsorption is endothermic, while the positive entropy change ($\Delta S^\circ \approx +120$ J/mol.K) reflects increased disorder at the solid-liquid interface. These findings collectively indicate that adsorption is governed primarily by surface binding accompanied by temperature-enhanced diffusion into the porous structure of the adsorbent.

3. Conclusion

Activated carbon produced from polystyrene waste demonstrated efficient adsorption performance toward Drimaren Red K-7B, with increasing capacity at higher initial dye concentrations and temperatures. The equilibrium data were best described by the Dubinin-Radushkevich model, indicating heterogeneous surface characteristics and predominantly physical adsorption. Kinetic analysis confirmed that the pseudo-second-order model governs the adsorption rate, while intraparticle diffusion plays a secondary role. The thermodynamic parameters showed that the process is spontaneous and endothermic, becoming more favorable with temperature. Overall, the results highlight polystyrene-derived activated carbon as a promising upcycled material for the removal of reactive dyes from aqueous solutions.

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Influence of thermodynamic properties of metallic materials on the efficiency of hydro-vacuum atomization and hydriding processes

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Abstract: *In this study, we present the key results of computer modeling and experimental series of a combined technological process that integrates hydro-vacuum atomization and hydrogenation of metallic melts. The simulations considered several representative cases in which lead, aluminum, magnesium, Armco iron, and magnesium–aluminum alloys were used as initial materials. A comparative analysis of the obtained data revealed that, during hydro-vacuum atomization, the cooling, solidification, and hydrogenation kinetics of atomized droplets are strongly governed by the specific thermodynamic characteristics of the metals, such as the solidification interval, latent heat of fusion/solidification, and specific heat capacity. It was established that the relationship between the melt overheating temperature and the width of its solidification interval constitutes a key controlling factor in the combined processes of atomization and hydrogenation. This factor is critical because it determines both the duration and the nature of the residence of atomized metallic droplets in the transitional liquid–crystalline state, thereby defining the temporal window and the depth of highly effective physico-technological influences that promote hydrogen uptake. These influences arise during the entrainment and breakup of the melt jet in the water stream and include hydromechanical activation, structural metastabilization, and interstitial hydrogen incorporation released from collapsing cavitation cavities in water due to the thermobaric energy generated during their directed cumulative implosion.*

KEYWORDS: HYDRO-VACUUM ATOMIZATION, CRYSTALLIZATION KINETICS, HYDROGENATION, STRUCTURAL METASTABILIZATION, OVERHEATING EFFECT.

1. Introduction

The use of metallic powders as solid-state hydrogen carriers opens new prospects for the development of safe, energy-efficient, and compact systems for the storage and transportation of renewable energy.

In contrast to conventional methods of hydrogen storage in gaseous or cryogenically liquefied form, metallic powder materials enable hydrogen retention in a chemically bound state (as metal hydrides formed through chemisorption mechanisms) or physically, through adsorption on the developed surface of the particles. This approach provides a substantially higher level of operational safety and allows long- term hydrogen storage under ambient conditions without the need to maintain extremely high pressures or ultralow temperatures. Moreover, the use of powder-based metal hydride materials completely eliminates the risks and operational costs associated with high- pressure composite vessels (600–700 bar) or cryogenic cooling systems operating at 20–70 K, both of which require complex infrastructure and expensive reinforced containment shells [1]. Unlike conventional hydrogen storage technologies, the retention of hydrogen in a chemically bound metal- hydride state, followed by its controlled thermal release upon heating the metal to approximately 100–300 °C, is characterized by a high degree of controllability and operational reliability. The desorption process proceeds smoothly, and its kinetics can be effectively regulated by adjusting the temperature of the hydrogenated material. This enables precise modulation of the hydrogen release rate in accordance with the specific demands of the system [2].

An equally important operational criterion is the high reversibility and stability of the hydrogenation–dehydrogenation cycles. The ability of a material to withstand numerous working cycles without significant structural degradation or loss of reversible hydrogen capacity determines the long- term durability of powder- based metal hydride storage systems and their technological applicability in the practical energy sector.

High- cycle metal hydride systems are particularly in demand in hydrogen- powered transportation, from unmanned aerial vehicles to heavy commercial machinery, as well as in autonomous and backup energy complexes for remote facilities, in decentralized

storage systems for renewable energy, and in stationary hydrogen reservoirs used in “green” metallurgy and the chemical industry [3, 4].

The successful integration of metal hydride systems into the aforementioned high- technology sectors is largely determined by the morphological and thermodynamic characteristics of the hydrogen- storage materials. A highly developed specific surface area in powder- based materials provides a dense population of active sorption sites, significantly accelerating the kinetics of diffusive or chemisorptive hydrogen uptake and saturation. However, morphological optimization alone is insufficient to overcome the fundamental limitations that hinder large- scale commercial deployment of such storage media. The primary barrier remains the stringent thermodynamic constraints associated with the high thermal stability of certain hydride phases, which result in sluggish desorption kinetics and necessitate elevated temperatures for reversible hydrogen release [5]. Moreover, these kinetic limitations become progressively aggravated over time due to the advancing structural degradation of the material. During high- temperature operation, cyclic phase hardening induces fragmentation (cracking) of the particles, followed by thermocyclic sintering and passivation [6, 7]. These processes lead to an irreversible reduction in sorption capacity and effectively negate the initial advantages associated with a highly developed specific surface area. Consequently, this indicates that the operational efficiency of reversible metal hydride systems is governed by a fundamental compromise between the thermodynamic stability of the hydride phases formed and the kinetic parameters of the sorption–desorption processes. For example, on the one hand, hydrides of light metals and their complex compounds (MgH₂, LiBH₄, Mg(BH₄)₂, AlH₃, TiH₂) exhibit exceptionally high theoretical hydrogen capacities, ranging from 4 to 18.5 wt.% H₂. However, such high storage capacities are achieved at the expense of thermodynamically robust metal–hydrogen bonds, which require substantial energy input for subsequent dissociation and shift the desorption temperature range into the domain of elevated temperatures (>250–500 °C). This is undesirable both from the standpoint of process efficiency and due to the reduction in the operational lifetime of the storage system. On the other hand, intermetallic compounds, such as Laves phases based on AB₂- and

AB₅- type alloys (LaNi₅, TiFe, ZrMn₂), enable operation under moderate, near- ambient thermal conditions (≈ 50 – 100 °C) [8, 9]. Nevertheless, their hydrogen storage capacity is significantly lower than that of light- metal hydrides and typically does not exceed 1.4–2 wt.% H₂. This limitation restricts their applicability in technical systems where high specific energy density is a critically important requirement.

A compromise-oriented attempt to resolve the thermodynamic–kinetic contradiction involves transitioning to lighter binary systems, among which intermetallic alloys of the Mg–Al system are of particular interest. The Mg₁₇Al₁₂ and Mg₂Al₃ phases occupy an intermediate position between high- capacity yet thermodynamically stable light- metal hydrides and low- temperature but low- capacity intermetallic compounds. The incorporation of aluminum into the magnesium matrix partially reduces its thermodynamic stability while maintaining a relatively high specific hydrogen capacity. For the Mg₁₇Al₁₂ phase, the theoretical hydrogen storage capacity is approximately 4.0–4.4 wt.% H₂, and due to the destabilizing influence of aluminum, the desorption enthalpy decreases to 65–70 kJ/mol H₂ (compared to ~ 75 kJ/mol for the pure Mg–H system). This results in a shift of the hydrogen release temperature threshold into the range of 150–280 °C and a noticeable improvement in desorption kinetics [10, 11].

Nevertheless, even the use of such metastabilized alloys does not fully eliminate the fundamental thermodynamic–kinetic dualism and ultimately imposes strict limitations on the choice of metallic materials and the methods by which they can be hydrogenated.

2. Modern approaches to solving the existing problem and substantiating the effectiveness of the hydro-vacuum method

Based on current scientific knowledge and accumulated practical experience, a wide range of methods has been developed to address the aforementioned problem. For example, modern catalytic approaches [12] demonstrate high efficiency in improving hydrogenation kinetics; however, the use of noble metals (Pt, Pd) is limited by their high cost and susceptibility to deactivation due to oxidation and impurity poisoning, which reduces the cyclic durability of such metal-based systems.

Mechanical alloying [13] provides high dispersion (< 50 μm) and stable formation of defect-rich states, but requires prolonged processing and is accompanied by powder contamination from wear products of the milling media.

Plasma- arc synthesis [14], in turn, is applicable only to a narrow class of hydride systems, primarily to compounds such as Alane (AlH₃), and is characterized by extreme thermal gradients that complicate control over the phase composition. Other high- energy hydride- forming techniques, such as electro- pulse treatment of solid- phase suspensions in liquid dielectrics or acoustic- cavitation sonoprocessing [15–17], face challenges associated with low process stability and high energy consumption per unit of product, which significantly limits their productivity and scalability.

The combination of the aforementioned limitations and the need to resolve the existing thermodynamic–kinetic contradiction has led to a clear demand for a new- generation technology capable of integrating the stage of metallic powder structure formation with the operation of its hydrogenation. Such a technology is the innovative hydrovacuum atomization (HVA) process developed by the authors of this study [18–20].

The combined atomization–hydrogenation process implemented in the HVA method is based on a recently identified hydrophysical mechanism of condensational sonomorphosis, which is induced and sustained by the thermobaric shock- wave effects of cavitation fluctuations [21]. This process is accompanied by the in-situ incorporation of hydrogen atoms released from molecules within a supercavitating, cumulatively collapsing, and sonopulsating vapor–water mixture stream, into both the crystalline lattices of the atomized metal and energetically favorable activated

intercrystalline regions containing free electrons, resulting in the formation of interstitial covalent bonds, see Fig. 1.

Against the background of existing technological approaches for producing hydrogenated metallic powders, it can be emphasized that the synergistic combination of atomization and hydrogenation offered by the HVA technology represents a unique engineering solution that integrates the advantages of mechanochemical activation and ultrasonic (sonochemical) treatment while mitigating their key drawbacks. The principal synergistic benefit of this method lies in the coupling of deep powder dispersion with in situ hydrogen saturation within a locally vacuumized and non- stationarily cavitating hydrodynamic medium, wherein the energy released during cavitation bubble collapse and the accompanying cumulative microjets not only contribute to material fragmentation or hydrogen liberation from collapsing cavitation cavities, but also induce a directed sonomorphosis of metastable substructures. These substructures are specifically adapted for prolonged reversible cycling, mutually reversible absorption and release of atomic hydrogen, without significant deterioration of the initial hydrogen accommodation capacity.

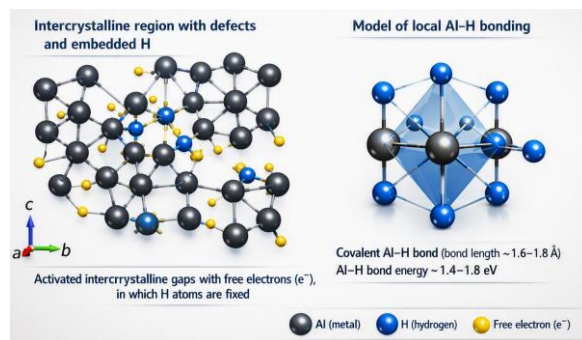


Fig. 1. Physical model of intercrystalline region and local Al–H bonding

The high density of generated crystal structure defects and the continuous exposure of pristine juvenile surfaces free from passivating oxide films ensure unobstructed diffusion and capture of hydrogen deep within the nonequilibrium metallic matrix, which acts as a structure containing numerous hydrogen traps. It

should also be noted that an accompanying hydrodynamic Leidenfrost effect [22], arising at the “hot particle–liquid” interface, plays an important role in stabilizing such energetically activated structural states. The quasi- stable vapor envelope temporarily reduces the heat- transfer rate; however, its subsequent explosive collapse generates extreme quenching velocities and continuing intense acoustic- cavitation micro- impacts. These processes inhibit defect relaxation and rigidly fix the metastable, Kirkendall- effect- induced submicron- foamed structure of the powder particles formed during processing (Fig. 2).

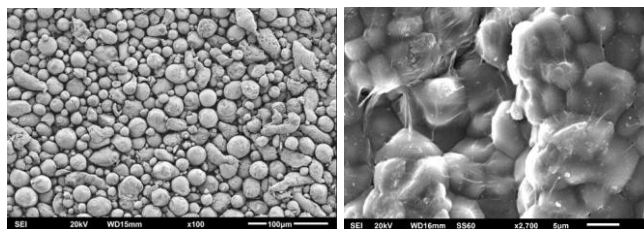


Fig. 2. SEI images of aluminum powder and surface morphology of its particles obtained by hydrovacuum atomization and hydrogenation (Bulk density not exceeding 0.5 g/cm³)

Undoubtedly, a deep understanding of the physical nature of the formation of the described structures and their subsequent controlled engineering, first requires an analysis of the fundamental thermophysical parameters of the hydrovacuum atomization process, with identification of those most critical in terms of their influence on structure formation. This emphasizes the importance

not only of selecting appropriate technological regimes for the target hydrovacuum dispersion process, but also of establishing rational conditions for the preliminary thermophysical preparation prior to feeding the liquid metallic material into the rarefied core of the counter-gravity suction and atomization chamber Fig. 3, position 1.

Consequently, understanding the mutual influence of these factors on the final (operational) characteristics of the powders represents a fundamental basis for improving the conditions of their structural formation and for more effective adaptation to the saturation and solid-state binding of atomic hydrogen.

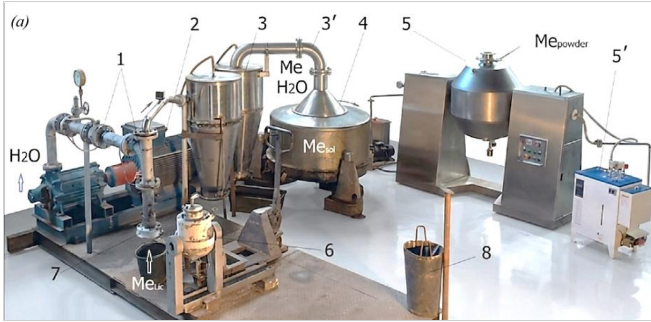


Fig. 3. General view of the hydro-vacuum atomization and hydrogenation installation for metallic melts:

- 1 — Hydro-vacuum suction, dispersion, and melt-cooling chamber (Me_{lic})
- 2 — High-pressure pump for high-velocity water injection into the chamber (water delivery pressure 12–20 bar, flow rate 20–22 L/s)
- 3 — Pair of scrubbers for primary gravitational separation of coarse powder fractions (1–2.5 mm) from the forming slurry (Me)
- 3' — Pipeline for slurry transfer from the scrubbers (3) to the dewatering centrifuge (4)
- 5 — Vacuum drying chamber for dehydrated powder
- 5' — Thermovacuum station of the drying chamber (5)
- 6 — Unit for metered feeding of the melt to the disperser-cooler (1)
- 7 — Vertical melt-suction head
- 8 — Ladle supplying the melt to the feeding unit (6)

Addressing the stated problem requires a predictive assessment of the thermophysical regimes governing the structure formation of the atomized liquid metallic material. Achieving such predictive accuracy becomes feasible through computer simulation of the cooling and quenching processes experienced by the fragmented particles formed during hydro-vacuum atomization.

The application of numerical computer modeling makes it possible, in an inverse formulation, to determine the ranges of initial (input) parameters for melt preparation that are most closely aligned with the optimal conditions required for the target hydro-vacuum treatment. In this way, modeling functions as a tool that provides a closed optimization loop: from forecasting the structural states of the atomized particles to adjusting the initial technological conditions that ensure the desired properties of the final material.

3. Factors and assumptions taken into account in the simulation

1) The interaction of a high-energy water jet (velocity 200 m/s) and the melt occurs by dispersing the latter into spherical droplets with a diameter of 10–1000 μm , the cooling of which is carried out in the presence of a gasified film formed as a result of the Leidenfrost effect; 2) Under the influence of the impact force developed during the spraying of water in the disperser, the vapor film is expected to degrade and be completely removed from the surface; 3) The temperature field in the atomized metal is distributed homogeneously $T_{liq}(t)=\text{const.}$; 4) The heat exchange process between the metal and water includes both convective and radiative (thermal radiation) components; 5) The melt solidifies at a fixed temperature $T_m=T_{lic}=T_{sol}$.

Modeling and its results: For a spherical particle with an average radius r , the volume (V), mass (m) and surface area (A) are defined as follows:

$$V = (4/3) \pi r^3, m = \rho V, A = 4\pi r^2.$$

However, considering that the metal melt is dispersed into N equal number of droplet fragments so that the total mass remains unchanged, the effective surface area increases approximately as follows: $A_{eff} = AN^{1/3}$. Then, the heat radiation intensity (q_{dot} , W) from the metal fragments with a temperature T_{liq} and a velocity v will be:

$$q_{dot} = A_{eff} [h(T_{f,v})(T_{liq} - T_w) + \varepsilon \sigma ((T_{liq} + 273.15)^4 - (T_w + 273.15)^4)] \quad (1)$$

Where, $h(T_{f,v})$ -convective heat transfer coefficient of the boundary layer, depending on the effective temperature of the cooling medium (water/steam) ($T_{film}=0.5(T_{liq}+T_w)$, $W/(m^2 \cdot K)$; T_{liq} - temperature of the metal entering the dispersion chamber, $^{\circ}\text{C}$; T_w - temperature of the dispersing and cooling water, $^{\circ}\text{C}$; ε - blackness of the metal surface (emissivity); σ - Stefan-Boltzmann constant, $5.670374419 \times 10^{-8} W/(m^2 \cdot K^4)$; v - water jet velocity, m/s.

To account for the specific heat capacity (c_v , J/kg·K) of the boundary layer, the computer script uses the following simple approximation:

$$h(T_{f,v}) = \min(h_{base} + c_v \sqrt{v}, h_{max}) \quad (2)$$

Where, h_{base} is the base heat transfer coefficient under stationary conditions, and h_{max} is the maximum allowable limit (upper limit) of the coefficient. For the case of Leidenfrost stability, we obtain the following:

$$h(T_{f,v}) = 8000 + 600 \sqrt{200} \approx 16485 \quad (3)$$

When the vapor barrier degrades (breaks), the coefficient takes on the following value:

$$h(T_{f,v}) = 25000 + 600 \sqrt{200} \approx 33485 \quad (4)$$

In addition, the calculation model for the cooling rate of the metal is based on enthalpy balance and, taking into account its specific heat capacity $c_{p,liq}$ and the condition $T_{liq} > T_{sol}$, takes the following form:

$$m c_{p,liq} dT/dt = -q_{dot} \Rightarrow dT/dt = -q_{dot} / (m c_{p,liq}) \quad (5)$$

In expanded form, the heat transfer equation is formed as the following mathematical representation:

$$dT/dt = -A_{eff} [h(T_{f,v})(T_{liq} - T_w) + \varepsilon \sigma ((T_{liq} + 273.15)^4 - (T_w + 273.15)^4)] / (m c_{p,liq}) \quad (6)$$

We assume that the actual temperature of the liquid metal T_{liq} remains unchanged until the latent heat of crystallization L_f (J/kg) is completely released from the cooling fragments. That is, the metal completely solidifies when the current latent heat supply becomes zero ($L(t) = 0$):

$$T = T_{sol}, T_{liq, Initial} = m L_f, dL/dt = -q_{dot}, dT/dt = 0 \quad (7)$$

After the latent heat of crystallization is completely released $L(t) = 0$ and the prerequisite for further cooling of the solid phase $T < T_{sol}$ is created, the subsequent rate of temperature change is described by the following differential equation:

$$m c_{p,sol} dT/dt = -q_{dot}, \text{ from where: } dT/dt = -q_{dot} / (m c_{p,sol}) \quad (8)$$

Where, $c_{p,sol}$ is the specific heat capacity of the metal in the solid state. And dT/dt is calculated in a similar way to the expanded heat transfer equation (6), by replacing the heat capacity of the liquid phase in it with the solid.

4. Results and discussion

The obtained modeling results, specifically t_{sol} (cooling time to the solidus temperature T_{sol}) and $t_{100^\circ C}$ (cooling time to the boiling point of water, $100^\circ C$) — were obtained for atomized particles of lead, aluminum, magnesium, Armco- iron, and Al–Mg alloys. The simulations were performed using the initial thermophysical and thermodynamic parameters (ρ , T_{lic} , $c_{p,liq}$, $c_{p,sol}$, L_f , and T_{liq}) listed in Table 1.

The resulting cooling- kinetics contour maps, shown in Figure 4, illustrate the temperature evolution of particles of different diameters during hydrovacuum atomization. These cooling kinetics maps (isotherms) reveal distinct cooling behaviors governed by the intrinsic thermal properties of each material. In particular:

Lead (Pb) demonstrates the slowest cooling due to its high density and relatively low thermal diffusivity.

Aluminum (Al) cools significantly faster, with steep temperature gradients even for larger particle diameters.

Magnesium (Mg) exhibits the most rapid cooling, driven by its low density and high thermal conductivity.

Armco- iron (Fe) shows prolonged high- temperature retention, reflecting its high melting point and substantial latent heat of fusion.

Al–Mg alloys display intermediate cooling kinetics, with the 30%Al – 70%Mg and 70%Al – 30%Mg compositions forming distinct thermal- response profiles.

Table 1

Met.	ρ , kg/m ³	T_{lic} , °C	$c_{p,liq}$, J/kg·K	$c_{p,sol}$, J/kg·K	L_f , x10 ³ , J/kg	T_{liq} , °C
Pb	10660	327.5	160	128	23	370
Al	2375	660.3	1170	900	397	700
Mg	1570	650.0	1370	1020	370	700
Fe	6980	1538	820	450	247	1600

These cooling- time fields directly determine the structurogenesis pathways of atomized droplets. The combination of particle diameter and cooling rate defines whether the droplets undergo: full crystallization, partial solidification with retained liquid cores, formation of fine- grained or amorphous structures, development of dendritic, cellular, or equiaxed morphologies, suppression or activation of diffusion- controlled transformations, hydrogen uptake and defect stabilization during hydrovacuum processing.

Comparative analysis of the modeled thermal processes shows that, following the primary influence of particle dispersity (their diameter, d) on the cooling rate, the next most significant factors are the thermophysical properties of the material and the initial temperature of the melt supplied to the hydrovacuum disperser.

Analysis of the time required for the particles to reach the boiling point of water ($100^\circ C$) reveals several key regularities:

- Effect of particle size:

For all investigated materials, the fine fraction with a diameter of $d = 0.1$ mm ($100\ \mu m$) cools to $100^\circ C$ approximately ten times faster than larger particles with $d = 1.0$ mm ($1000\ \mu m$). This behavior is governed by the increase in specific surface area as particle size decreases.

- Armco- Fe exhibits the highest cooling rate among all materials considered. Due to its high initial temperature ($1530^\circ C$) and the intense radiative-convective heat transfer, a particle with a diameter of 0.1 mm cools to $100^\circ C$ in only 0.006 s (average cooling rate $2.38 \times 10^5\ ^\circ C/s$), whereas a particle of 1.0 mm reaches $100^\circ C$ in 0.062 s (average cooling rate $2.3 \times 10^4\ ^\circ C/s$).

- Magnesium (Mg) and aluminum (Al), despite having nearly identical initial temperatures ($720^\circ C$), exhibit different cooling characteristics. Specifically, magnesium particles cool faster (for $d = 0.1$ mm: $t \approx 0.007$ s, $v \approx 8.86 \times 10^4\ ^\circ C/s$) than aluminum particles ($t \approx 0.009$ s, $v \approx 6.89 \times 10^4\ ^\circ C/s$). This difference is attributed to the lower density and caloric properties of magnesium.

- In Al–Mg alloy systems, an increase in the magnesium content leads to an intensification of the cooling process.

- For the alloy containing 30% Al – 70% Mg, the cooling time of a 0.1 mm particle down to $100^\circ C$ is $t \approx 0.008$ s ($v \approx 7.75 \times 10^4\ ^\circ C/s$), whereas for the alloy with 30% Mg – 70% Al this time increases to $t \approx 0.010$ s ($v \approx 6.20 \times 10^4\ ^\circ C/s$).

- For lead (Pb), due to its comparatively low initial temperature ($370^\circ C$), the average cooling rate varies from $5.4 \times 10^4\ ^\circ C/s$ (for $d = 0.1$ mm, $t \approx 0.005$ s) to $5.19 \times 10^3\ ^\circ C/s$ (for $d = 1.0$ mm, $t \approx 0.052$ s).

Analysis of the obtained results shows that the solidification rate (the $Me_{liq} \rightarrow Me_{sol}$ phase transition) is primarily governed by the latent heat of crystallization, the specific heat capacity, and the initial thermal interval — i.e., the degree of superheating ($T_{liq} - T_{lic}$). For example, although the crystallization temperature of Armco iron is nearly twice that of aluminum, and although aluminum is less superheated during dispersion compared to Armco iron, its solidification duration is nevertheless longer (0.03312 s vs. 0.02736 s). This behavior is explained by the fact that the latent heat of aluminum ($397\ 000$ J/kg) is significantly higher than that of iron ($247\ 000$ J/kg), and its specific heat capacity in the solid state ($c_{p,sol}$) is approximately twice that of iron.

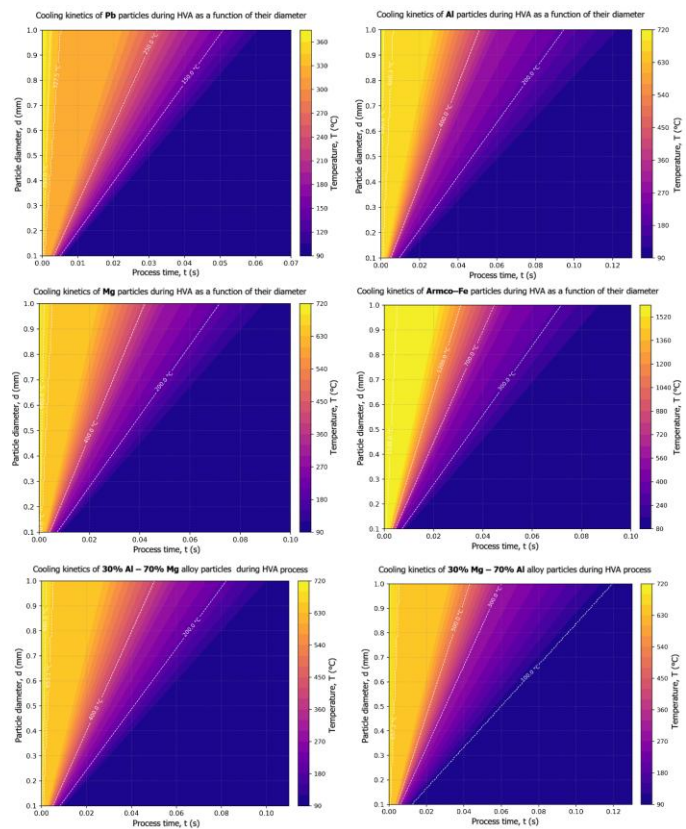


Fig. 4. Modeling results: Dependence of the cooling rate of hydrovacuum- atomized particles of lead, aluminum, magnesium, Armco-Fe, and Al–Mg alloys on dispersion duration and particle size - fraction 100–1000 μm .

In turn, analysis of the diagrams shown in Fig. 5 indicates that, due to the reduction of powder particle size to 0.01 mm ($10\ \mu m$) — and, consequently, the increase in the surface- area- to- mass ratio (A/m) — the cooling rates in this range rise sharply and reach values on the order of $10^5\ ^\circ C/s$.

In particular, analysis of the time required for particles to reach $100^\circ C$ for the extreme boundaries of the fraction ($d = 0.01$ mm ($10\ \mu m$) and $d = 0.10$ mm ($100\ \mu m$)) leads to the following findings:

For aluminum particles with a size of 0.01 mm ($10\ \mu m$), the cooling time from the initial melt temperature ($\approx 720^\circ C$) down to $100^\circ C$ is less than 0.002 s (approximately 1.8 – 2.0 ms). The

corresponding average cooling rate exceeds $6.5 \times 10^5 - 7.7 \times 10^5$ °C/s, which is characteristic of ultra- rapid solidification conditions.

For aluminum particles with a size of 0.10 mm (100 μ m), the time required to reach 100 °C is approximately 0.0125 s, and the average cooling rate is about 5.3×10^4 °C/s.

The 30% Mg – 70% Al alloy exhibits cooling dynamics similar to those of pure aluminum: a 0.10 mm particle cools to 100 °C in 0.012 s, with an average cooling rate of $\approx 5.5 \times 10^4$ °C/s.

In the 50% Al – 50% Mg alloy, due to reduced density and modified heat capacity, the cooling time of a 0.10 mm particle decreases to 0.0105 s (cooling rate $\approx 5.9 \times 10^4$ °C/s).

In turn, the 70% Mg – 30% Al alloy demonstrates the highest cooling rate within the investigated particle- size fraction: a 0.10 mm particle reaches 100 °C in 0.0115 s, with an average cooling rate of $\approx 6.4 \times 10^4$ °C/s, which is attributed to the predominance of the magnesium matrix with lower specific density.

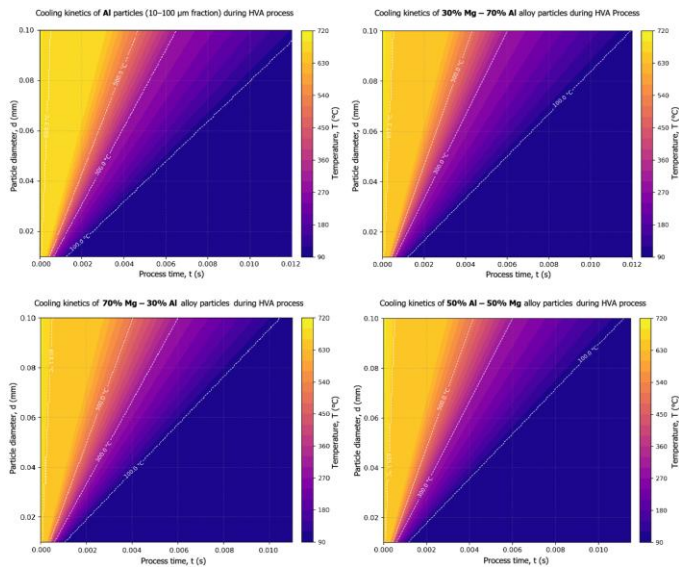


Fig. 5. Modeling results: Dependence of the cooling rate of hydrovacuum-atomized particles of aluminum and Al-Mg alloys on dispersion duration and particle size - fraction 10–100 μ m.

In all cases, a reduction in particle diameter within the investigated range from 100 μ m to 10 μ m leads to an increase in cooling rate by approximately a factor of 10–12.

This allows us to conclude that using the 10–100 μ m fraction during hydrovacuum atomization of Al-Mg alloys enables the stable attainment of ultra- high cooling rates on the order of $10^4 - 10^5$ °C/s, thereby creating favorable conditions for the formation of fine- crystalline, highly heterogeneous, metastabilized substructures in the resulting powders.

This trend is confirmed by the experimental data presented in Fig. 6.

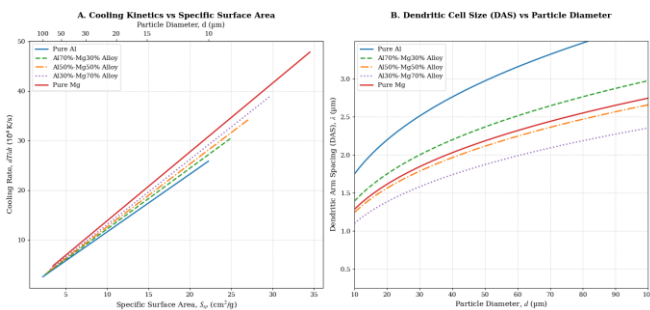


Fig. 6. Influence of the specific surface area of powder particles and their diameter on the cooling kinetics and the size of the resulting dendritic cells.

As seen in Fig. 6, a reduction in particle size from 100 μ m to 10 μ m and an increase in specific surface area from 2–3 to 25–35 cm²/g lead to a pronounced rise in cooling rate (diagram A), which in turn results in a refinement of the dendritic cell size from 2.5–3.0 μ m down to 1.2–1.6 μ m (diagram B).

In binary Al–Mg alloys (particularly in compositions Al 50% – Mg 50% and Al 30% – Mg 70%), the combined effects of constitutional supercooling and segregation processes produce a more dispersed dendritic structure compared to the pure components.

For fine powder fractions ($d < 50$ μ m), the formation of an ultra- fine microstructure (< 2.5 μ m) is reliably ensured, which predetermines a high degree of thermodynamic nonequilibrium and metastable structural state in the resulting powders.

Quenching from the liquid state stabilizes metastable phases and supersaturated solid solutions characterized by a high level of excess internal energy (Gibbs energy).

This substantially reduces both the thermodynamic and kinetic barriers for hydrogen–material interaction, lowering the activation energy of chemisorption.

The extreme cooling kinetics generate a high- density defect structure (high concentrations of vacancies, dislocations, and microstrains).

Local lattice distortions facilitate the incorporation of hydrogen atoms into tetrahedral and octahedral interstitial sites, while microscale segregation and phase heterogeneity create localized regions with increased chemical affinity toward hydrogen.

As a result, hydrogen absorption proceeds with high kinetics at reduced temperatures and pressures. Moreover, in the case of repeated hydrogenation of powders previously dehydrogenated by conventional methods, no prolonged high- temperature, mechanical, or catalytic activation is required, since the metastable structural state remains preserved.

A comprehensive, integrated analysis of the obtained data shows that the key factor governing the kinetics of the phase transition and the resulting metastabilized morphogenesis of powder particles is the coupled influence of the latent heat of crystallization (L_f), which depends on density (ρ), and the melt superheating ($\Delta T = T_{liq} - T_{lic/sol}$), which in turn is determined by the actual melt preparation temperature (T_{liq}) prior to the onset of HVA.

For example, according to numerical thermodynamic modeling, the anomalously high latent heat of crystallization of aluminum ($L_f = 397\,000$ J/kg), combined with the elevated specific heat capacity of its solid phase ($c_{p,sol} = 900$ J/(kg·K)), substantially prolongs the overall solidification duration of a droplet compared with other metallic systems.

In particular, when compared with a magnesium matrix, which possesses a similar latent heat ($L_f = 370\,000$ J/kg) and even higher specific heat capacity ($c_{p,sol} = 1020$ J/(kg·K)), the calculated time required for complete cooling of aluminum particles down to 100 °C is significantly longer ($t_{100^\circ C} = 0.12345$ s vs. 0.08182 s for magnesium, Fig. 4).

This behavior is explained by the substantial difference in density between the compared molten metals (2375 kg/m³ vs. 1570 kg/m³). The higher density of aluminum results in a proportionally greater droplet mass at an equal diameter d (mm), which leads to a significantly larger volumetric heat- storage capacity ($\rho \cdot L_f$ and $\rho \cdot c_p$).

As a consequence, the specific surface area available for heat removal per unit mass ($A/m = 4\pi r^2 / \rho V$) is lower for aluminum, which ultimately prolongs both the phase- transition processes and subsequent cooling. This trend is reflected in Fig. 6A (blue curve).

Additionally, the melt- superheating factor (ΔT) makes a substantial contribution.

A high melt- preparation temperature T_{liq} generates a significant excess of thermal energy in the liquid phase ($Q_{overheat} = m \cdot c_{p,liq} \cdot \Delta T$), which must be dissipated before the onset of solidification.

In combination with the high volumetric heat capacity of aluminum ($\rho \cdot c_p$) and its latent heat (L_f), this leads to an overall

increase in the thermal inertia of the droplet and explains the pronounced prolongation of the time required for complete cooling ($t_{100^\circ\text{C}}$). Consequently, aluminum exhibits a comparatively coarse dendritic structure (cell sizes $> 3 \mu\text{m}$, Fig. 6B, blue curve).

On the other hand, the large magnitude of the phase enthalpy reflected in the latent heat of crystallization (L_f), when combined with extreme convective–radiative heat extraction, creates unique conditions for the quasi- instantaneous fixation of strongly nonequilibrium substructures.

The latent heat released during aluminum crystallization briefly maintains high matrix plasticity within the temperature interval of the phase transition. Against the background of continuous ultrasonic shock- wave loading from the HVA medium, this promotes the intensive development of shear deformations, the generation of dislocation ensembles, and the stabilization of a pronounced “spongy” porosity throughout the volume of the forming powder particle (Fig. 7).

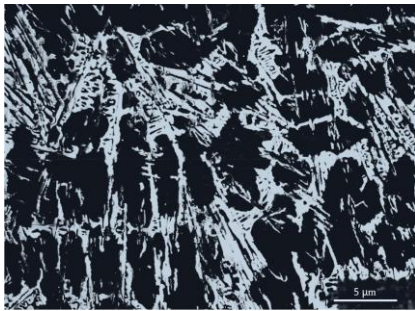


Fig. 7. Microsection of a particle produced by hydrovacuum atomization from the Al–Mg alloy (Al 70% – Mg 30%), particle from the 100 μm fraction.

Since, in the HVA process, the primary adjustable parameter governing the cooling (and, accordingly, supercooling) regime is the degree of initial superheating of the melt supplied for atomization, it can be stated that targeted control of this parameter enables regulation of the actual residence time of the dispersed fragment in both the liquid and the transitional liquid–crystalline states. This residence time is critically important for structural formation, activation, and the degree of hydrogenation.

It is noteworthy that, in the case of Al–Mg and other binary alloys possessing a defined solidification interval, an equally important control criterion becomes the establishment of an optimal balance between the melt- superheating level and the temperature range (interval) of crystallization (as determined from phase diagrams).

As demonstrated by the experimental results, this factor acts as the key technological lever in the combined processes of structure formation and hydrogenation (Figs. 8 and 9).

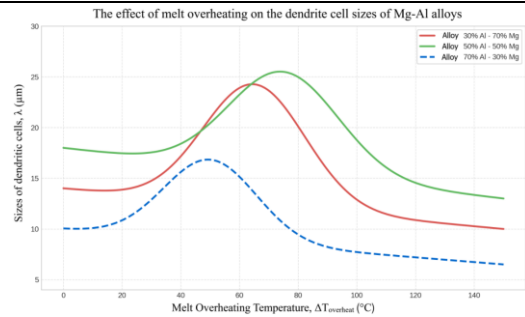
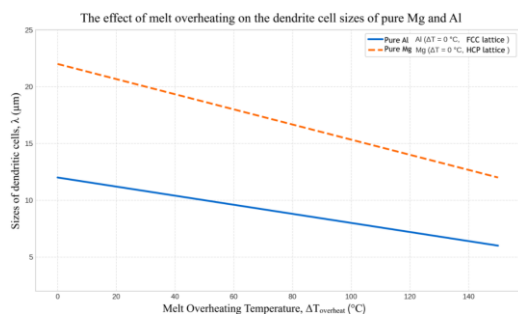


Fig. 8. Influence of melt superheating on the size of dendritic cells.

High melt superheating reduces melt viscosity and surface tension, which, under hydrovacuum atomization, ensures finer droplet fragmentation and an increased cooling rate.

Since pure metals do not exhibit complex eutectic transformations or a dendrite- forming interval ($T_{lic} \approx T_{sol}$), a simple direct inverse relationship is observed: - the higher the superheating, the finer the resulting structure.

Pure magnesium, due to the characteristics of its hexagonal close- packed lattice and its stronger tendency for directional crystal growth, initially exhibits a coarser grain size than pure aluminum with its face- centered cubic lattice; however, the overall trend of the curves for both metals remains descending.

In contrast to pure metals, binary Al–Mg alloys possess a wide solidification interval ($\Delta T > 0$), which is determined from the phase diagram.

Within the range of moderate superheating (typically around 50–80 $^\circ\text{C}$), the curve shows a pronounced rise, - an increase in the average grain size (the peak region).

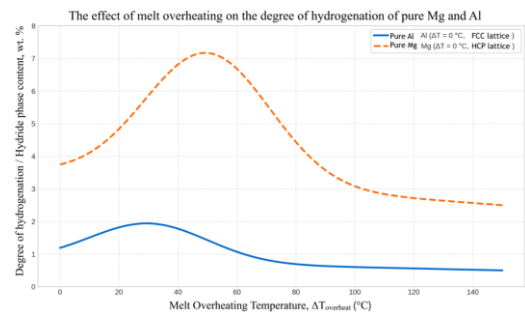
This behavior is associated with the persistence of nucleating microclusters in the melt and enhanced liquation (component segregation), both of which promote the coarsening of dendritic structures during solidification.

Compositions enriched in magnesium or located near the eutectic exhibit a wider solidification interval, which makes the grain- coarsening peak more pronounced.

The aluminum- rich alloy (70% Al, 30% Mg — blue dashed line) has a narrower solidification interval and correspondingly smaller grain- size values.

Notably, when the melt- superheating temperature exceeds a critical threshold (100–120 $^\circ\text{C}$), all inherited micro- inhomogeneities and clusters are completely destroyed.

The melt becomes fully homogeneous, which under subsequent rapid cooling allows the solidification interval to be traversed with suppressed grain growth, ensuring the formation of minimal grain sizes in the final powder.



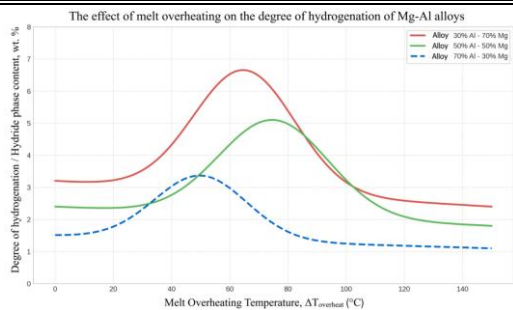


Fig. 9. Influence of melt superheating on the degree of hydrogenation.

Alloys with high magnesium content (70%) exhibit the strongest tendency to interact with hydrogen and to form metastable MgH_2 phases, showing the highest hydrogenation degrees. Alloys with 50% Mg contain a well- developed eutectic component and a highly defective microstructure at moderate superheating, forming a pronounced peak. Aluminum- rich alloys (30% Mg), due to the partial protective influence of finely dispersed nanoporous aluminum- oxide films, demonstrate the lowest hydrogenation values.

However, aluminum is not a passive component; on the contrary, it actively participates in hydrogen chemisorption, forming surface Al–H complexes and promoting hydrogen redistribution within the solid phase.

The thermodynamic stability of aluminum and its higher Al–H bond energy partially “counterbalance” the excessive reactivity of magnesium, weakening Mg–H interactions and facilitating reversible low- temperature dehydrogenation, - a particularly important factor for cyclic processes.

This behavior is confirmed by the X- ray diffraction data illustrated in Fig. 10.

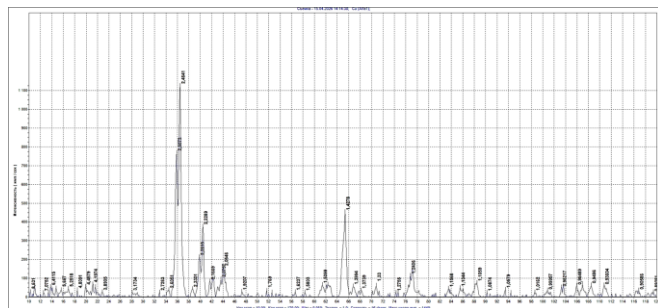


Fig. 10. X- ray diffraction pattern of the atomized Al–Mg alloy powder (70% Mg – 30% Al).

The presented X- ray diffraction pattern shows that the investigated sample exhibits well- defined, intense diffraction peaks corresponding to the coexistence of crystallographic phases of the initial metallic components (magnesium and aluminum), as well as the products of their interaction with hydrogen.

The presence of distinct reflections confirms the formation of the magnesium hydride phase MgH_2 , along with stable intermediate structures and surface Al–H complexes, which directly correlate with hydrogen redistribution processes within the solid phase.

The presence of characteristic peaks with well- defined interplanar spacings reflects the high degree of dispersity and defectiveness of the crystalline structure, which is governed by the developed eutectic component and the influence of protective nanoscale oxide films.

These structural features subsequently reduce the overall energy required for reversible activation and enable low- temperature reversible dehydrogenation of the alloy.

In particular, magnesium constitutes the primary component of the matrix structure. The most intense analytical peak is recorded at $2\theta = 36.43^\circ$ ($d = 2.4641 \text{ \AA}$, relative intensity 100%, contributing ~21.78% to the volumetric phase content). High- angle magnesium reflections are observed at $2\theta = 106.95^\circ$ ($d = 0.9585 \text{ \AA}$), 108.41° ($d = 0.9496 \text{ \AA}$), and 110.75° ($d = 0.9360 \text{ \AA}$).

Aluminum, in turn, participates in forming the structural framework of the alloy. A characteristic aluminum peak is detected at $2\theta = 65.29^\circ$ ($d = 1.4278 \text{ \AA}$, intensity 288 imp/s, mass fraction ~12.85%), as well as at $2\theta = 38.61^\circ$ ($d = 2.3301 \text{ \AA}$).

The intermetallic compounds $\text{Al}_{12}\text{Mg}_{17}$ and AlMg_3 represent products of alloying and eutectic solidification. The $\text{Al}_{12}\text{Mg}_{17}$ phase is identified at $2\theta = 65.29^\circ / 88.29^\circ$ ($d = 1.1059 \text{ \AA}$), while AlMg_3 appears at $2\theta = 21.15^\circ$ ($d = 4.1974 \text{ \AA}$).

The presence of these phases confirms the well- developed eutectic structure of the alloy.

Diffraction peaks corresponding to AlH_3 are registered in the medium- and high- angle regions at $2\theta = 41.64^\circ$ ($d = 2.1669 \text{ \AA}$), 52.55° ($d = 1.7400 \text{ \AA}$), 61.35° ($d = 1.5099 \text{ \AA}$), and within the $83\text{--}106^\circ$ range ($2\theta = 83.49^\circ, 85.54^\circ, 93.45^\circ, 105.93^\circ$).

This confirms that aluminum is not inert but actively participates in hydrogen chemisorption, forming surface Al–H complexes.

Characteristic reflections of orthorhombic MgH_2 (typically strong peaks at $2\theta \approx 27.9^\circ, 35.7^\circ, 38.2^\circ$, and 39.9°) appear near the magnesium peaks; however, in the experimental diffractogram they merge into broadened composite peaks due to the nanoscale grain state, — high defect density and small crystallite thickness ($17\text{--}19 \text{ nm}$).

The recorded significant peak half- width ($0.4639^\circ \approx 0.0081 \text{ rad}$) combined with high intensity ($I = 734 \text{ imp/s}$) indicates not amorphization but pronounced subgrain fragmentation and a high dislocation density, both induced by the extreme thermal gradients inherent to hydro-vacuum atomization.

Hydroxide–hydride complexes ($\text{AlH}_3 \cdot \text{O}_3$) are also present in the diffractogram, appearing in the low- angle region at $2\theta = 13.80^\circ$ and 14.73° , reflecting the specific surface interactions of moisture/oxygen with the forming hydride structures.

Notably, the intermetallic $\text{Al}_{12}\text{Mg}_{17}$ reflection at $2\theta = 65.29^\circ$ ($d = 1.4278 \text{ \AA}$) exhibits a peak half- width of 0.6971° , corresponding to an ultrafine crystallite size of approximately $14\text{--}15 \text{ nm}$.

For the oxide phase periclase (MgO), the half- width at $2\theta = 35.78^\circ$ is 0.4407° , while for Al_2O_3 at $2\theta = 39.83^\circ$ it is 0.4272° .

These small coherent- scattering domain sizes confirm the formation of protective nanoporous oxide frameworks.

The maximum peak broadening is observed for reflections at $2\theta = 16.87^\circ$ (0.8080° for $\text{Al}_{12}\text{Mg}_{17}$), $2\theta = 62.35^\circ$ (0.9210° for Mg), and $2\theta = 77.07^\circ$ (0.7190°).

Such pronounced broadening at low and medium intensities indicates a high level of type- II microstrains within the crystal lattice, caused by local lattice distortions due to incorporated hydrogen atoms and vacancies.

Thus, the dependencies of λ and the hydrogenation degree on $\Delta T_{\text{overheat}}$, presented in Figs. 8 and 9, consistently demonstrate that the thermodynamically governed evolution of the dendritic- structure scale is the key factor determining the hydrogen- saturation capacity and functional properties of powders produced by hydrovacuum atomization.

5. Generalized semi- empirical model for the formation of crystalline- structure parameters.

Based on the theoretical and experimental data obtained in this study, a semi- empirical generalized expression was developed to mathematically describe the combined influence of melt- superheating temperature and the solidification interval on the resulting structural scale (dendritic- cell or grain size, λ).

The model incorporates three principal factors: 1) Baseline structural refinement driven by increasing superheating ($\Delta T_{\text{overheat}}$); 2) Structural coarsening in the moderate- superheating region, associated with the persistence of micro- inhomogeneities and the

width of the solidification interval ($\Delta T_{\text{interval}}$); 3) An individual component- affinity coefficient, reflecting the tendency of alloy constituents to form clusters or protective films, - in this case, governed by the Al/Mg ratio.

$$\lambda = \lambda_0 \cdot \exp(-\Delta T_{\text{overheat}}/k_1) + A_1 \cdot (\Delta T_{\text{interval}}/T_{\text{ref}}) \cdot \exp(-(\Delta T_{\text{overheat}} - T_{\text{crit}})^2 / 2 \sigma^2) + C \quad (9)$$

Where: λ - resulting dendritic- cell or grain size (μm); λ_0 - initial structural scale of the base pure metal; $\Delta T_{\text{overheat}}$ - melt- superheating temperature above the liquidus ($^{\circ}\text{C}$); $\Delta T_{\text{interval}}$ - alloy solidification interval ($T_{\text{liquidus}} - T_{\text{solidus}}$), defining the width of the phase- transition zone ($^{\circ}\text{C}$); k_1 - thermal- damping coefficient (responsible for monotonic cell narrowing under deep superheating); A_1 - coefficient describing the influence of liquation and inherited clusters (directly proportional to the fraction of components with a wide solidification interval, e.g., 30% Al–70% Mg or 50% Al–50% Mg compositions); T_{crit} - critical moderate- superheating temperature (typically 50–80 $^{\circ}\text{C}$) at which the structural peak (melt heterogeneity) is most pronounced; T_{ref} - reference normalization temperature (100 $^{\circ}\text{C}$); σ - width of the melt- instability peak; C - technological limit of structural refinement under extremely high cooling rates during hydrovacuum dispersion.

The proposed generalized relationship makes it possible to quantitatively describe how the thermodynamic properties of metallic materials influence structure formation during hydrovacuum atomization.

Specifically: – the first exponential term reflects the dominance of thermodynamic factors; as the melt- superheating temperature increases ($\Delta T_{\text{overheat}} \gg k_1$), its viscosity and surface tension decrease exponentially, leading to a monotonic reduction in the dendritic- cell scale (λ).

– the second term, a Gaussian bell- shaped function, describes the “anomalous” structural peak that arises within the moderate- superheating range (T_{crit}), where liquation- induced heterogeneities and cluster groupings persist.

The amplitude and width of this peak are governed by the solidification interval ($\Delta T_{\text{interval}}$): it vanishes for pure metals ($\Delta T_{\text{interval}} \rightarrow 0$), while in Al–Mg alloys it produces a pronounced maximum.

Equation (9), together with the experimental trends shown in Figs. 8 and 9, demonstrates a direct correlation between the dendritic- structure scale λ and the hydrogen- saturation capacity of the particles. For pure metals (Al, Mg), the monotonic decrease in λ with increasing $\Delta T_{\text{overheat}}$ (Fig. 8) leads to a higher fraction of defective boundaries and active surface sites, which manifests as an increased degree of hydrogenation and a higher content of hydride phases (Fig. 9). This effect is especially pronounced for magnesium and Mg- rich systems.

In Al–Mg alloys, the nonlinear, hump- shaped dependence $\lambda(\Delta T_{\text{overheat}})$, governed by the Gaussian term in Equation (9), leads to the fact that within the intermediate- superheating region ($T_{\text{crit}} \approx 50\text{--}80\text{ }^{\circ}\text{C}$), dendritic- cell coarsening is accompanied by a reduction in the effective area of active boundaries and a decrease in the hydride- phase fraction.

Upon transitioning to deep superheating ($>120\text{ }^{\circ}\text{C}$) followed by rapid cooling, λ decreases again, and the structure becomes fine- dispersed and defect- enriched.

This promotes enhanced hydrogen diffusion and uptake, resulting in higher hydrogenation degrees, - particularly in Mg- rich compositions.

According to the derived equation, the resulting dendritic- cell or grain size, being itself dependent on the solidification duration of the particles, directly influences the depth and character of hydrogen uptake. Consequently, the longer a particle remains within the high- temperature transitional solidification interval, the more time is available for sonovolumetric action and condensational sonomorphosis, both of which arise under hydrovacuum

atomization conditions. These effects enhance hydrogen diffusion and capture, stabilize defect- rich states, and generate activated structures capable of sustaining multiple hydrogenation–dehydrogenation cycles.

Taken together, these findings lead to the key conclusion that the thermodynamics of phase transformations, alloy composition, and solidification kinetics jointly determine the efficiency of structure formation and its targeted adaptation to subsequent hydrogenation–dehydrogenation cycles.

This establishes a fundamental basis for developing methods of purposeful design of highly stable functional powders for hydrogen- energy applications.

6. Conclusion

The analysis of the thermodynamic properties of metallic materials, combined with thermal- regime modeling of particle cooling during hydrovacuum atomization, demonstrates that differences in heat capacity, thermal conductivity, and latent heat of fusion generate unique temperature–time cooling trajectories for the produced particles, thereby defining their structural state.

The modeling results, together with data from parallel experimental investigations, establish a closed optimization loop for the technological process: thermodynamic properties $\rightarrow$ thermal history of particles $\rightarrow$ structure formation $\rightarrow$ hydride formation $\rightarrow$ adjustment of melt parameters $\rightarrow$ increased efficiency of atomization and hydrogenation.

This approach enables predictive control over the structural evolution and reactive capability of atomized metallic systems, providing a scientifically grounded platform for designing highly efficient regimes of hydrovacuum processing and hydrogenation.

The constructed thermal maps (isotherms) of particle cooling demonstrate that the parameters t_{sol} and $t_{100^{\circ}\text{C}}$ serve as reliable indicators of solidification and cooling kinetics and can be used to predict the critical intervals of dendritic, highly activated, and therefore hydrogen- intensive structure formation.

It was found that rapidly cooling systems (Mg, Al–Mg alloys) tend to form fine- dispersed, highly defective, and metastable structures, whereas materials with high thermal inertia (Pb, Armco- Fe) retain coarse structural elements and prolonged high- temperature states. This explains their lack of competitiveness compared to the lightweight metals investigated in this study.

It is shown that the reactive (chemisorptive) hydrogen- uptake capability is determined not only by composition but also by the combined thermodynamic properties of the alloy components.

Mg- rich systems (50–70% Mg) indeed exhibit a strong tendency toward hydride formation (MgH_2). However, aluminum, when present alongside magnesium, does not act as a passivating component; rather, it actively participates in hydrogen chemisorption, forming surface Al–H complexes and facilitating hydrogen redistribution within the solid phase.

The thermodynamic stability of aluminum and its higher Al–H bond energy partially offset the excessive reactivity of magnesium, attenuating Mg–H interactions and enabling reversible low- temperature dehydrogenation, a feature of particular importance for cyclic hydrogenation–dehydrogenation processes.

Two scientifically substantiated pathways for the purposeful production of HVA- powders for different applied tasks have been identified.

For obtaining dense, homogeneous particles of globular morphology, eutectic and near- eutectic compositions are optimal, as they ensure the condition $\Delta T_{\text{interval}} \rightarrow 0$, maximize the solidification rate, and minimize the free energy of the system.

For generating metastable, activated structures adapted to repeated hydrogenation–dehydrogenation cycles, alloys with a wide solidification interval ($\Delta T_{\text{interval}} \neq 0$) are preferable, provided that the melt- superheating level $\Delta T_{\text{overheat}}$ is strictly controlled and satisfies the condition $\Delta T_{\text{overheat}} \geq \Delta T_{\text{interval}}$ during the preparation stage prior to hydrovacuum atomization.

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Study of the influence of the cross-linker on the modification of cotton fabric with chitosan for controlled release of diclofenac and preparation of antimicrobial textiles

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Abstract: Composite materials with potential application in medical textiles were developed by modifying cotton fabric with chitosan containing diclofenac. The influence of the concentration of citric acid as a crosslinking agent on the properties of the materials was studied. The resulting composite materials were characterized by gravimetric analysis, optical microscopy, contact angle measurement and thermogravimetric analysis. The antimicrobial activity of the cotton samples was evaluated against Gram-positive and Gram-negative model bacterial strains. A synergistic antimicrobial effect of chitosan and diclofenac and a uniform release of diclofenac *in vitro* under physiological conditions were established.

Keywords: MEDICAL TEXTILES, COTTON FABRIC, CHITOSAN, CITRIC ACID, CROSSLINKING, DICLOFENAC, ANTIBACTERIAL ACTIVITY

1. Introduction

Medical textiles are one of the most dynamically developing areas of technical textiles, widely used in modern healthcare. The development of new materials and technologies has significantly expanded the possibilities for creating functional textile products intended for wound treatment, infection control, local delivery of medicinal substances and other medical applications [1, 2]. Among them, wound dressings occupy a special place, which, in addition to a protective function, must provide an optimal environment for tissue regeneration, limit the development of microorganisms and, if necessary, carry out a controlled release of biologically active substances [3].

Cotton fabrics are widely used as a base for medical textiles due to their biocompatibility, good absorbency, air permeability and mechanical strength. Despite these advantages, the lack of intrinsic antimicrobial activity necessitates their surface modification with biologically active polymers [4].

Among natural polymers, chitosan has attracted considerable scientific interest due to its biocompatibility, biodegradability, non-toxicity, film-forming ability and pronounced antimicrobial activity. Due to its reactive amino and hydroxyl groups, it can be chemically crosslinked and used as a carrier for medicinal substances, which allows the development of multifunctional textile materials for wound healing [5]. One of the most promising crosslinking agents is citric acid, which provides stable crosslinked of chitosan on cellulose fibers and can influence the structure, surface properties and functional characteristics of the resulting materials [6].

Despite a significant number of publications, the influence of citric acid concentration on the properties of cotton fabric modified with chitosan containing diclofenac is still insufficiently studied. The present work aims to evaluate the influence of the concentration of the crosslinking agent on the physicochemical characteristics of the developed composite materials and to analyze their suitability for application as functional medical textiles.

2. Materials and methods

Cotton fabric (100% cellulose) was used as the textile substrate. Chitosan, diclofenac sodium as an anti-inflammatory biologically active substance, and citric acid as a crosslinking agent were used to modify it.

2.1. Preparation of composite materials

The experimental procedure was developed based on our previous studies, with the present work introducing a change only in the concentration of the crosslinking agent used [4, 7].

The cotton fabrics were pre-treated by impregnation with aqueous solutions of citric acid (CA) with concentrations of 1 %, 5 % and 10 % w/w chitosan, then dried at room temperature. In parallel, a chitosan solution (2.7 % w/v) was prepared, to which diclofenac sodium was added under continuous mechanical stirring

until a homogeneous mixture was obtained. After homogenization, the solution was left for 2 h, which allowed the separation of air bubbles and ensured uniform formation of the coating. The pretreated textile samples were impregnated with the resulting chitosan solution containing diclofenac and subsequently subjected to a heat treatment at 80 °C, which resulted in crosslinking of chitosan with citric acid and formation of a stable polymer film on the surface of the cellulose fibers. The resulting materials were designated according to the concentration of the crosslinking agent as follows: CHTD1, CHTD5 and CHTD10.

To assess the influence of the degree of crosslinking, all other technological parameters – chitosan and diclofenac concentration, processing time and thermal fixation conditions – were kept constant.

2.2. Methods for the characterization of composite materials

The change in fabric weight following processing was calculated using Equation (1):

$$(1) \text{Weight gain (\%)} = \frac{w - w_0}{w_0} \times 100$$

where w_0 and w represent the weight of the tissue before treatment and after modification and immersion in distilled water and subsequent drying, respectively.

Microscopic images of the textile samples were obtained using a B- 290TB optical microscope (Optika®, Pontenica, Italy), equipped with a high-quality illumination system and the ability to operate at different magnifications. Observations were performed at 4× and 10× magnifications, which allowed visualization of both the general surface structure and finer morphological details.

The thermal behavior of the studied samples was evaluated by thermogravimetric and differential thermal analysis using a STA PT1600 TG-DTG/DSC (LINSEIS Messgeräte GmbH, Selb, Germany). The measurements were performed at a heating rate of 10 °C min⁻¹ in the temperature range from room temperature (20 °C) to 600 °C, in air.

The contact angle of the studied textile samples with distilled water was measured using the sitting drop method, implemented with an optical tensiometer THETA Flow Auto 1, equipped with a Basler acA2500- 60 µm digital camera. The method is based on recording the profile of a water drop placed on the surface of the textile material and subsequent geometric evaluation of the angle formed between the liquid and the solid surface.

The pH value of the prepared solutions was determined with a Hanna Instruments microprocessor pH meter.

In vitro release of the biologically active substance

The release of diclofenac sodium from the three composite materials obtained using 1%, 5% and 10% citric acid as a crosslinking agent was studied *in vitro* in phosphate buffer with a

concentration of 0.01 M and pH = 7.4 at a temperature of 37 ± 0.5 °C. The amount of diclofenac released was determined spectrophotometrically by measuring the absorption at a wavelength of 277 nm with a UV-Vis spectrophotometer ONDA UV-31SCAN in the range of 190–1100 nm. Each sample was immersed in 20.0 mL of the buffer solution, and at predetermined time intervals 5.0 mL of the medium were taken and replaced with an equal volume of fresh buffer, tempered to 37 °C. The concentration of the released active substance was determined by a previously established calibration curve.

Antimicrobial activity

The antimicrobial properties of the three composite materials were evaluated against representative Gram-positive and Gram-negative bacterial strains – *Bacillus cereus* and *Pseudomonas aeruginosa*. The study was conducted by contacting textile samples with a size of 10×10 mm with bacterial suspensions in sterile meat-peptone broth. Untreated cotton fabric and bacterial medium without textile material were used as control samples.

After incubation of the samples at 28 °C for 18 h with stirring at a speed of 240 rpm, bacterial growth was assessed spectrophotometrically by measuring the optical density of the medium at 600 nm (OD_{600}). The antimicrobial effectiveness of the materials was determined by comparative assessment of the bacterial cell density compared to the control samples. The tests were conducted in three independent replicates, and the influence of the concentration of citric acid on the antimicrobial activity of the resulting composite materials was monitored.

3. Results and Discussion

Gravimetric analysis revealed a gradual increase in the mass of the modified materials with increasing citric acid concentration – from 21.42% for CHTD1 to 22.33% for CHTD10. The observed trend can be associated with an increase in the amount of fixed chitosan layer on the cellulose fibers due to more intensive crosslinking at higher citric acid concentrations.

Figure 1 shows optical micrographs of untreated cotton fabric (CO) and the composite material CHTD10, obtained at $4\times$ and $10\times$ magnifications. In the untreated textile sample, the characteristic configuration of the fibers for the woven structure is observed, forming free spaces in the interweaving zones. The individual cellulose fibers are clearly distinguishable, with characteristic fibrillar structures standing out on their surface. In the modified sample CHTD10, significant morphological changes are observed. At $4\times$ magnification, a formed chitosan coating is observed on the surface of the fibers, while at $10\times$ a dense polymer layer is visualized, which covers the fibers and partially fills the spaces between the individual fibers [8].

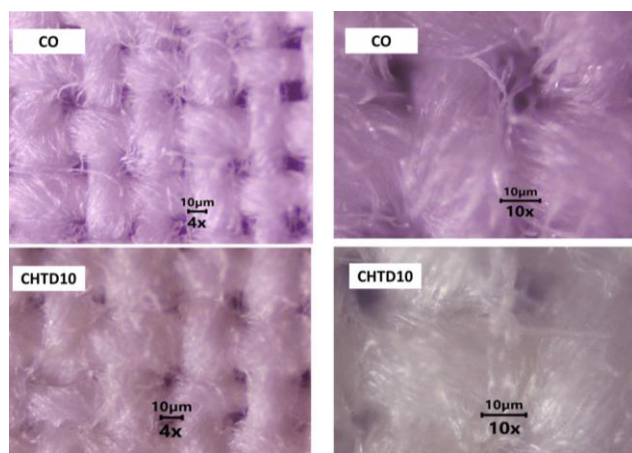


Fig. 1 Optical microscope micrographs of cotton fabric (CO) and composite material CHTD10 (magnification of the photos is $4\times$ and $10\times$).

The thermogravimetric analysis (TGA) and its first derivative – differential thermogravimetric (DTG) curve of the starting cotton fabric and the composite materials CHTD1, CHTD5, and CHTD10 are presented in Fig. 2A and Fig. 2B, respectively. It can be seen that the thermal degradation of all samples proceeds in three stages. After the initial small loss in the weight of the materials at 119 °C, which is due to the release of moisture from the materials (4 % for the untreated cotton fabric, 5.0 % for CHTD1, 5.5 % for CHTD5 and 8.1 % for CHTD10). The second stage is with the most significant thermal degradation, as for the starting cotton fabric it starts at 271 °C (CO - 5.85 %), and for the composite materials at a lower temperature of 234 °C, due to the presence of acids causing degradation of chitosan and cellulose [9]. Respectively for the materials 6.6 % for CHTD1, 8.9 % for CHTD5 and 10.8 % for CHTD10. The mass loss at 370 °C is 73.9 % for CO, 66.7 % for CHTD1, 68.2 % for CHTD5 and 69.3 % for CHTD10. The first derivative also shows that the inflection points for the starting material CO are at 335 °C, for CHTD1 it is at 336 °C, for CHTD5 it is at 337 °C and for CHTD10 it is at 335 °C (Fig. 2B). At 600 °C without leaving dry residues, while the cotton fabric burns at 100 %, and for samples CHTD1, CHTD5 and CHTD10 the dry residues are 15.1 %, 12.4 % and 7.2 %, respectively. With increasing citric acid concentration, a gradual decrease in the residual mass after thermal degradation is observed – from 15.1% for CHTD1 to 7.2% for CHTD10, which indicates a dependence of the thermal behavior on the concentration of the crosslinking agent.

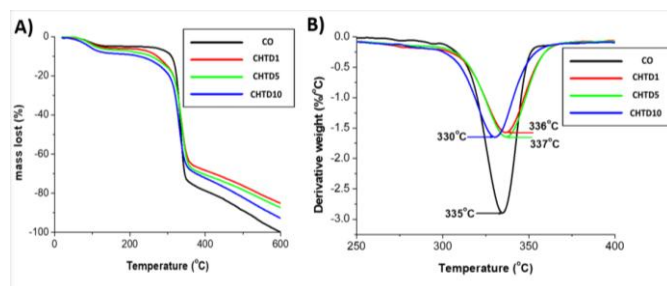


Fig. 2 (A) TG and (B) DTG curves of untreated cotton fabric (CO) and composite materials (CHTD1, CHTD5, and CHTD10).

Figure 3 shows the contact angle values of the composite materials CHTD1, CHTD5 and CHTD10. The original cotton fabric is not included in the figure, since due to its pronounced hydrophilicity, the water droplet is absorbed directly by the surface and a value of 0° is recorded [10]. For the modified materials, values of 91.57°, 92.28° and 94.22° were measured, respectively, and a gradual increase in the contact angle with increasing citric acid concentration was found. The results obtained show a change in the surface wettability of the cotton fabric as a result of the modification, with all the studied composite materials exhibiting a hydrophobic character.

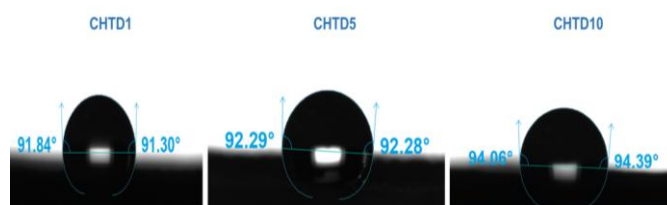


Fig. 3 Evaluation of hydrophilic-hydrophobic properties by measuring the contact angle of wetting with water on composite materials (CHTD1, CHTD5, and CHTD10).

Figure 4 shows the cumulative release profile of diclofenac sodium from samples CHTD1, CHTD5 and CHTD10 in phosphate buffer with pH = 7.4 at 37 °C for 26 hours. During the first 6 hours, a more intensive release of the drug substance from the studied composite materials was established, with the amounts reached being $93.28 \mu\text{g cm}^{-2}$ for CHTD1, $107.82 \mu\text{g cm}^{-2}$ for CHTD5 and 112.98

$\mu\text{g cm}^{-2}$ for CHTD10, respectively. This initial faster stage is probably associated with the desorption of diclofenac located near or on the surface of the materials. After the initial phase of more intensive release, a gradual and more uniform release of diclofenac from the composite matrices was observed until the end of the study period. After 26 hours, the cumulative released amount reached $109.95 \mu\text{g cm}^{-2}$ for CHTD1, $127.52 \mu\text{g cm}^{-2}$ for CHTD5 and $136.07 \mu\text{g cm}^{-2}$ for CHTD10. The observed differences between the individual samples can be related to the peculiarities of the structure and degree of crosslinking of the hydrogel matrix, the way of distribution and retention of the drug substance in it, as well as the processing conditions of the textile materials. These factors determine the accessibility of diclofenac to the aqueous environment and, accordingly, influence the rate, amount and duration of its release.

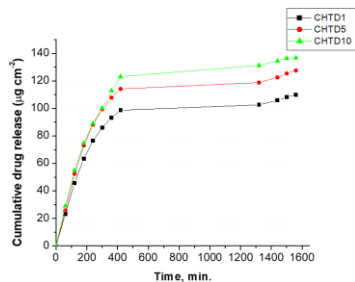


Fig. 4 Release of diclofenac in phosphate buffer (pH=7.4) at 37 °C from composite materials (CHTD1, CHTD5, and CHTD10).

Figure 5 shows the results of the study of the antimicrobial activity of the composite materials CHTD1, CHTD5 and CHTD10 against A) *P. aeruginosa* and B) *B. cereus*, with the untreated cotton fabric (CO) included for comparison. The control sample showed 100% bacterial contamination, with no antimicrobial activity noted. For *P. aeruginosa*, the bacterial reduction for CHTD1, CHTD5 and CHTD10 was 98.6%, 98.4% and 98.0%, respectively, and for *B. cereus* – 98.0%, 97.5% and 98.2%. The high antimicrobial activity is probably due to the combined action of chitosan and diclofenac, as well as to the changes in the surface characteristics of the modified textile materials. The obtained bacterial reduction values are higher than those found for the materials previously studied by us, containing indomethacin and diclofenac. This highlights the potential of the developed composites for application as antimicrobial materials for medical textiles [4,7, 11].

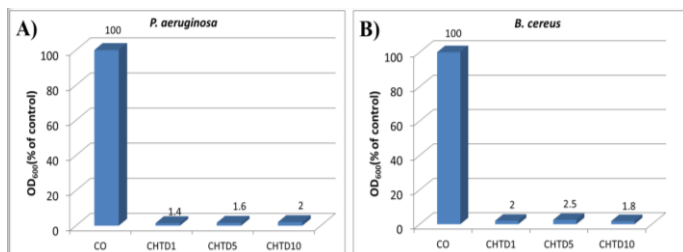


Fig. 5 Antimicrobial activity of cotton fabric (control) and composite materials: CHTD1, CHTD5, and CHTD10 on the growth of A) *P. aeruginosa* and B) *B. cereus*.

4. Conclusion

New composite textile materials were obtained by modifying cotton fabric with chitosan, diclofenac and different concentrations of citric acid. It was found that the concentration of the crosslinking agent affects the mass growth, surface properties and thermal behavior of the materials. All three composites showed high antimicrobial activity against Gram-positive and Gram-negative bacteria, with a bacterial reduction of over 97%. The obtained results define the developed materials as a promising basis for functional medical textiles with potential application in wound dressings and controlled release of diclofenac.

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Integrated comparative assessment of material, energy and construction-time efficiency of prefabricated and conventional reinforced concrete systems

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Abstract: The selection of an appropriate structural and construction system significantly influences material consumption, energy demand, construction duration and overall project efficiency. This study presents an integrated parametric methodology for comparing prefabricated and conventional cast-in-situ reinforced concrete systems under equivalent functional and structural requirements. A representative five-storey building with a gross floor area of approximately 5,000 m² is analysed using two construction scenarios: CIS (conventional cast-in-situ reinforced concrete) and PRE (prefabricated reinforced concrete). The assessment incorporates concrete and reinforcement consumption, material losses, structural construction duration, embodied energy of principal materials, additional production energy, transportation and site energy. The analysed performance dimensions are integrated into a dimensionless Construction Efficiency Index (CEI), while sensitivity analysis evaluates the influence of decision priorities. At a reference factory-to-site distance of 50 km, the parametric scenario indicates a reduction of approximately 7.8% in total structural-material input, 25% in structural construction duration and 6.2% in total energy demand for PRE relative to CIS. The baseline CEI increases from 1.000 for CIS to 1.153 for PRE. The study additionally introduces a project-specific break-even transportation-distance model. Under the adopted assumptions, the energy advantage of prefabrication disappears at approximately 185 km, whereas the integrated CEI advantage persists to a substantially larger theoretical distance because material and time benefits remain. The proposed framework demonstrates that prefabrication should not be evaluated through a single indicator. Its relative advantage depends on the combined effects of materials, energy, time and logistics. The methodology is intended as an early-stage engineering decision-support approach and requires project-specific inventory data for application to real construction projects.

KEYWORDS: PREFABRICATED CONSTRUCTION; REINFORCED CONCRETE; MATERIAL EFFICIENCY; EMBODIED ENERGY; CONSTRUCTION TIME; TRANSPORT; CONSTRUCTION TECHNOLOGY; CONSTRUCTION EFFICIENCY INDEX.

1. Introduction

The construction sector is increasingly required to improve resource efficiency, reduce environmental impacts and shorten project delivery periods while maintaining structural performance, quality and durability. In this context, industrialised and prefabricated construction methods have gained significant attention as alternatives to conventional cast-in-situ construction. Their principal distinction lies in transferring a considerable part of the production process from the construction site to a controlled factory environment, followed by transportation and assembly of prefabricated components on site. This change in production logic affects material consumption, labour organisation, construction time, energy demand, transportation requirements and the generation of construction waste [3].

Reinforced concrete remains one of the dominant structural materials in multi-storey buildings, and both conventional cast-in-situ and prefabricated concrete systems rely heavily on concrete and reinforcing steel. However, the distribution of material and energy inputs differs considerably between the two construction approaches. Prefabrication can provide improved production control and reduce certain on-site activities, but it may simultaneously require additional energy for factory processing, lifting and transportation of large structural elements. Recent life-cycle research therefore supports stage-by-stage comparison rather than simplified assumptions about overall environmental superiority [1].

Material efficiency represents one of the principal criteria for comparing alternative construction systems. Factory-based production can potentially improve dimensional control, standardisation and material utilisation, while reduced dependence on temporary formwork and repeated on-site operations may decrease certain categories of construction waste. Nevertheless, prefabrication does not automatically result in lower consumption of all structural materials. Comparative life-cycle studies have shown that specific prefabricated projects may consume more concrete or reinforcing steel than comparable cast-in-situ solutions, depending on structural configuration, prefabrication rate and component design [2]. Consequently, material efficiency should be evaluated quantitatively for the specific structural system rather than assumed as an inherent advantage of prefabrication.

Construction duration is another important factor. The separation of factory production from on-site preparation enables certain activities to be performed in parallel and can reduce the duration of on-site structural works. Previous life-cycle assessments of prefabricated buildings have identified shorter construction periods among the potential advantages of industrialised construction, although the realised benefit depends on project organisation, production capacity, logistics and assembly efficiency [3]. Therefore, construction-time comparison should consider the complete delivery process rather than only the final on-site erection phase.

The environmental and energy implications of prefabrication are more complex. Embodied energy associated with construction is distributed among material production, component fabrication, transportation, site activities and equipment operation. Abey and Anand compared the embodied energy of prefabricated and conventional residential construction and showed that energy-intensive materials dominate both systems, while transportation may become a significant component when prefabricated elements are produced far from the site [4]. Their case study illustrates that prefabrication should not automatically be assumed to provide an energy advantage under all project conditions.

Conversely, other comparative studies have reported lower environmental impacts for prefabricated construction. Mao et al. found lower construction-stage greenhouse-gas emissions for a semi-prefabricated residential alternative, although transportation of prefabricated components partly offset the achieved reduction [5]. Wang et al. also reported favourable life-cycle energy and cost performance for the prefabricated building examined in their case study [3]. These contrasting findings indicate that relative performance is highly dependent on system boundaries, material quantities, component production methods, transportation distances and project-specific assumptions.

Transportation is therefore a particularly important parameter in the assessment of prefabricated reinforced-concrete systems. Unlike conventional construction, in which raw materials or ready-mixed concrete may often be supplied from relatively local sources, prefabricated construction requires the transport of completed structural elements whose mass and dimensions may result in considerable logistics-related energy consumption. Recent carbon-footprint research similarly

identifies material production and transportation as important contributors to the life-cycle impacts of prefabricated buildings [6].

This relationship suggests that transportation distance may represent a decisive variable when selecting between construction systems. For a sufficiently short distance between the prefabrication plant and the construction site, reductions in site energy, waste and construction duration may outweigh the additional transport requirement. As transport distance increases, however, the energy associated with moving prefabricated components also increases and may progressively reduce or eliminate the overall energy advantage.

The present study therefore introduces the concept of a project-specific break-even transportation distance, representing the transport distance at which the total energy performance of the prefabricated alternative becomes equivalent to that of the corresponding conventional system. The break-even value is not treated as a universal constant because it depends on component mass, vehicle characteristics, transport-energy intensity and the initial energy difference between the compared systems.

Another methodological challenge is that material efficiency, energy consumption and construction duration are expressed in different physical units and may lead to conflicting conclusions. A prefabricated system may provide a substantial reduction in construction time while exhibiting only a marginal improvement, or even a disadvantage, in embodied energy. Accordingly, the objective of this study is to develop an integrated comparative methodology for evaluating the material, energy and construction-time efficiency of prefabricated and conventional

reinforced-concrete systems. The principal scientific contribution is twofold: development of a dimensionless Construction Efficiency Index (CEI), and formulation of a project-specific break-even transportation-distance model for prefabricated reinforced-concrete construction.

2. Methodology

2.1. Research Framework

The methodology was developed to provide an integrated comparison between conventional cast-in-situ reinforced-concrete construction (CIS) and prefabricated reinforced-concrete construction (PRE) under equivalent functional and structural requirements. The analysis considers material efficiency, energy efficiency and construction-time efficiency. The resulting indicators are integrated into a dimensionless Construction Efficiency Index, while a separate transport-distance sensitivity model evaluates the logistical conditions under which prefabrication retains an energy advantage.

2.2. Definition of the Reference Building

A representative five-storey reinforced-concrete building, denoted G+4, was adopted as the reference case. The assumed gross floor area is 5,000 m², or approximately 1,000 m² per storey. The same architectural function and equivalent load-bearing requirements are assumed for both construction alternatives. The comparison focuses on the load-bearing superstructure; components assumed to be identical in both alternatives, such as finishes and building services, are excluded from the comparative calculation.

Table 1. Principal characteristics of the reference building

Parameter	Adopted value
Number of storeys	G+4
Gross floor area	5,000 m ²
Approximate floor area per level	1,000 m ²
Primary structural material	Reinforced concrete
Scenario 1	CIS – Cast-in-situ RC
Scenario 2	PRE – Prefabricated RC

2.3. Material Consumption Assessment

The principal permanent structural materials considered in the analysis are concrete volume V_c and reinforcement mass M_s . Material losses generated during production, transport and construction are considered separately so that the total structural-material resource input includes both installed material and expected losses.

$$M_{str,j} = \rho_c V_{c,j} + M_{s,j} \quad (1)$$

$$M_{res,j} = M_{str,j} + M_{w,j} \quad (2)$$

$$L_{m,j} = (M_{w,j} / M_{res,j}) \times 100 \quad (3)$$

The material-efficiency indicator is defined relative to the conventional CIS system. Values above 1.00 for PRE indicate a lower total structural-material resource input than the reference conventional alternative.

$$I_{M,j} = M_{res,CIS} / M_{res,j} \quad (4)$$

2.4. Construction-Time Assessment

Construction-time analysis is limited to the structural superstructure phase. The relative time reduction and corresponding time-efficiency indicator are defined as follows:

$$R_T = (1 - T_{PRE} / T_{CIS}) \times 100 \quad (5)$$

$$I_{T,j} = T_{CIS} / T_j \quad (6)$$

2.5. Embodied-Energy Assessment

The total energy demand associated with each construction scenario is divided into the embodied energy of structural materials, additional component-production energy, transportation energy and construction-site energy. This formulation is intended to represent a redistribution of energy demand rather than assume that prefabrication necessarily reduces every energy component.

$$E_{tot,j} = E_{mat,j} + E_{prod,j} + E_{tr,j} + E_{site,j} \quad (7)$$

$$E_{mat,j} = M_{c,j} e_c + M_{s,j} e_s \quad (8)$$

$$E_{tr,j} = \sum (M_{k,j} d_{k,j} e_{tr,k} \lambda_k) \quad (9)$$

$$E_{tr,PRE} = M_{PRE} d_{PRE} e_{tr} \lambda \quad (10)$$

$$E_{site} = E_{crane} + E_{pump} + E_{equipment} + E_{temporary} \quad (11)$$

$$I_{E,j} = E_{tot,CIS} / E_{tot,j} \quad (12)$$

2.6. Construction Efficiency Index – CEI

The three efficiency indicators are integrated into the proposed Construction Efficiency Index. The CIS system is used as the reference and therefore has CEI = 1.00. A PRE value above 1.00 indicates an integrated advantage under the adopted weighting system.

$$CEI_j = w_M I_{M,j} + w_E I_{E,j} + w_T I_{T,j} \quad (13)$$

$$w_M + w_E + w_T = 1 \quad (14)$$

$$CEI_j = 0.35 I_{M,j} + 0.35 I_{E,j} + 0.30 I_{T,j} \quad (15)$$

Table 2. CEI weighting scenarios

Scenario	w_M	w_E	w_T	Main priority
B	0.35	0.35	0.30	Balanced baseline
M	0.50	0.25	0.25	Material efficiency
E	0.25	0.50	0.25	Energy efficiency
T	0.25	0.25	0.50	Construction time

$$\Delta CEI = CEI_{\max} - CEI_{\min} \quad (16)$$

2.7. Break-Even Transportation Distance

The energy demand of the prefabricated alternative is expressed as a function of factory-to-site distance. The break-even distance occurs when the total energy demand of PRE becomes equal to that of CIS. The resulting value is project-specific and depends on the adopted system boundaries, component mass, transport-energy intensity and logistical correction factor.

$$E_{PRE}(d) = E_{PRE,0} + M_{PRE} e_{tr} \lambda d \quad (17)$$

$$E_{PRE}(d_{BE}) = E_{CIS} \quad (18)$$

$$d_{BE} = (E_{CIS} - E_{PRE,0}) / (M_{PRE} e_{tr} \lambda) \quad (19)$$

Transport-distance sensitivity is evaluated for 25, 50, 100, 150, 200 and 300 km, with recalculation of transportation

energy, total PRE energy, energy-efficiency indicator and CEI. This enables separate identification of the distance at which the pure energy advantage disappears and the distance at which the overall integrated CEI advantage disappears.

3. Results and Discussion

3.1. Definition of the Parametric Scenario

To evaluate the proposed methodology, a representative numerical scenario was defined for both construction systems. The selected values provide an internally consistent basis for comparative analysis. They represent modelling assumptions and not measured data from an executed building.

Table 3. Parametric input data for the comparative analysis

Parameter	CIS	PRE
Installed concrete volume (m ³)	1,850	1,750
Installed reinforcement mass (t)	215	225
Concrete material-loss rate (%)	5.0	2.0
Reinforcement material-loss rate (%)	3.0	1.0
Structural construction duration (days)	220	165
Additional production energy (GJ)	100	450
Fixed transportation energy (GJ)	450	150
Site energy (GJ)	850	300

The adopted material and transport assumptions were: concrete density $\rho_c = 2.4 \text{ t/m}^3$, unit energy factor for concrete $e_c = 0.95 \text{ MJ/kg}$, unit energy factor for reinforcing steel $e_s = 15.0 \text{ MJ/kg}$, transport-energy intensity $e_{tr} = 0.80 \text{ MJ/(t·km)}$, and logistics factor $\lambda = 1.20$. These values are used solely as parametric inputs and should be replaced or verified using a consistent project-specific life-cycle inventory dataset in a real application.

3.2. Material Consumption and Material Losses

Accounting for the assumed loss rates, the total structural-material resource input was calculated as approximately 4,895.3 t for CIS and 4,513.0 t for PRE. The prefabricated alternative therefore required approximately 7.8% less total structural-material input in the adopted scenario. The reduction was primarily associated with lower concrete volume and lower production losses, while PRE required a slightly higher reinforcement mass.

Table 4. Comparative material-efficiency results

Indicator	CIS	PRE
Gross concrete input (t)	4,673.7	4,285.7
Gross reinforcement input (t)	221.6	227.3
Total structural-material input (t)	4,895.3	4,513.0
Estimated material losses (t)	240.3	88.0
Overall loss ratio (%)	4.91	1.95
Material-efficiency indicator I_M	1.000	1.085

The calculated material-efficiency indicator for PRE was $I_M = 1.085$. This illustrates that improved material efficiency does not require every individual material quantity to decrease; rather, the final result depends on the combined balance of concrete, reinforcement and losses.

3.3. Construction-Time Efficiency

The structural construction duration was assumed to be 220 days for CIS and 165 days for PRE. This corresponds to a 25.0% reduction and a time-efficiency indicator of $I_T = 1.333$. The assumed time advantage reflects the possibility of overlapping factory production with site preparation and reducing repeated on-site formwork, reinforcement, casting and curing cycles. The result nevertheless depends on uninterrupted production, transport and assembly logistics.

3.4. Energy Comparison at the Reference Transport Distance

A factory-to-site transport distance of 50 km was adopted as the reference logistics scenario. At this distance, the calculated material-related energy was 7,764.7 GJ for CIS and 7,480.5 GJ for PRE. For a transported prefabricated-element mass of approximately 4,425 t, the variable transportation energy was 212.4 GJ. The resulting total energy demand was 9,164.7 GJ for CIS and 8,592.9 GJ for PRE.

Table 5. Energy-demand comparison at a 50 km transport distance

Energy component	CIS (GJ)	PRE (GJ)
Structural materials	7,764.7	7,480.5
Additional production	100.0	450.0
Transportation	450.0	362.4
Site activities	850.0	300.0
Total	9,164.7	8,592.9

At the reference distance, the PRE system exhibited approximately 6.2% lower total energy demand, corresponding to $I_E = 1.067$. The energy benefit results from the combined balance of lower material-related energy and substantially lower site energy, partly offset by higher factory-production energy.

3.5. Influence of Transportation Distance

The transport distance of prefabricated elements was varied between 25 and 300 km. The results show a continuous reduction in the isolated energy advantage of PRE as transportation distance increases. At 25 km the energy-efficiency indicator was 1.080, whereas at 300 km it decreased to 0.949, indicating that the prefabricated alternative became less favourable than CIS from the energy perspective alone.

Table 6. Influence of transport distance on PRE energy performance and CEI

Distance (km)	Variable transport energy (GJ)	Total PRE energy (GJ)	I_E	CEI_B
25	106.2	8,486.7	1.080	1.158
50	212.4	8,592.9	1.067	1.153
100	424.8	8,805.3	1.041	1.144
150	637.2	9,017.7	1.016	1.135
200	849.6	9,230.1	0.993	1.127
300	1,274.4	9,654.9	0.949	1.112

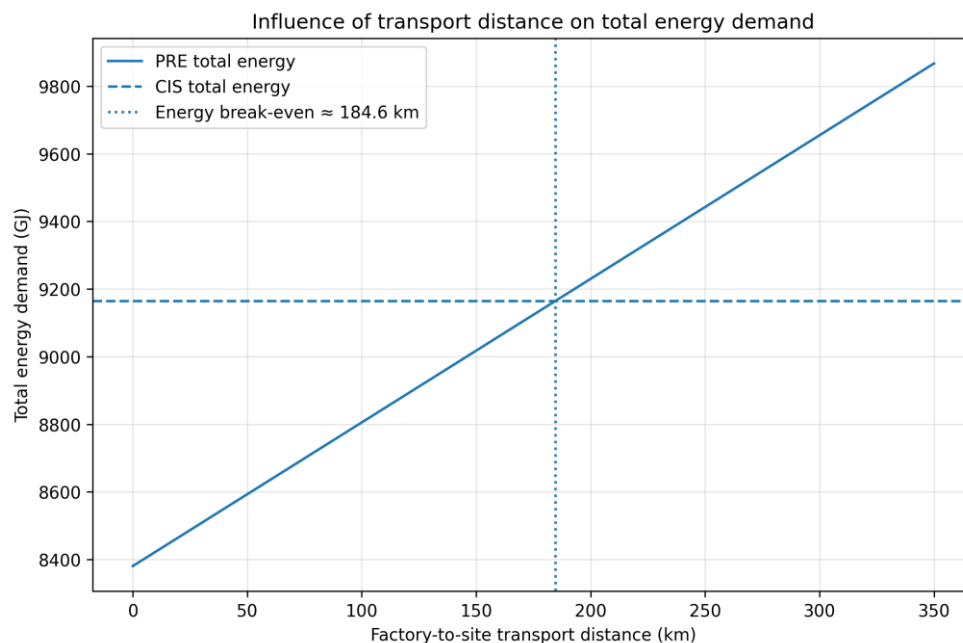


Figure 1. Influence of transport distance on total energy demand and the energy break-even point.

3.6. Break-Even Transportation Distance

The prefabricated-system energy demand excluding variable factory-to-site transport was $E_{PRE,0} = 8,380.5$ GJ. The energy increase per kilometre was 4.248 GJ/km. The resulting project-specific energy break-even transportation distance was approximately 184.6 km. Below this distance, PRE exhibits lower total energy demand than CIS under the adopted assumptions. Beyond this point, the additional transport burden causes total PRE energy demand to exceed that of the conventional alternative.

$$d_{energy, BE} \approx 184.6 \text{ km} \quad (20)$$

The calculated value is not a universal threshold for prefabricated construction. It is directly dependent on structural

configuration, transported mass, production system, vehicle performance, energy intensity and system boundaries. Its significance lies in demonstrating how logistics can be explicitly incorporated into technology selection.

3.7. Construction Efficiency Index at the Reference Distance

At 50 km, the three calculated efficiency indicators for PRE were $I_M = 1.085$, $I_E = 1.067$ and $I_T = 1.333$. Under the baseline weighting model, the resulting Construction Efficiency Index was $CEI_{PRE} = 1.153$, compared with $CEI_{CIS} = 1.000$. Thus, the prefabricated alternative exhibited an integrated efficiency advantage of approximately 15.3% in index terms. This difference should not be interpreted directly as an economic saving or life-cycle environmental reduction.

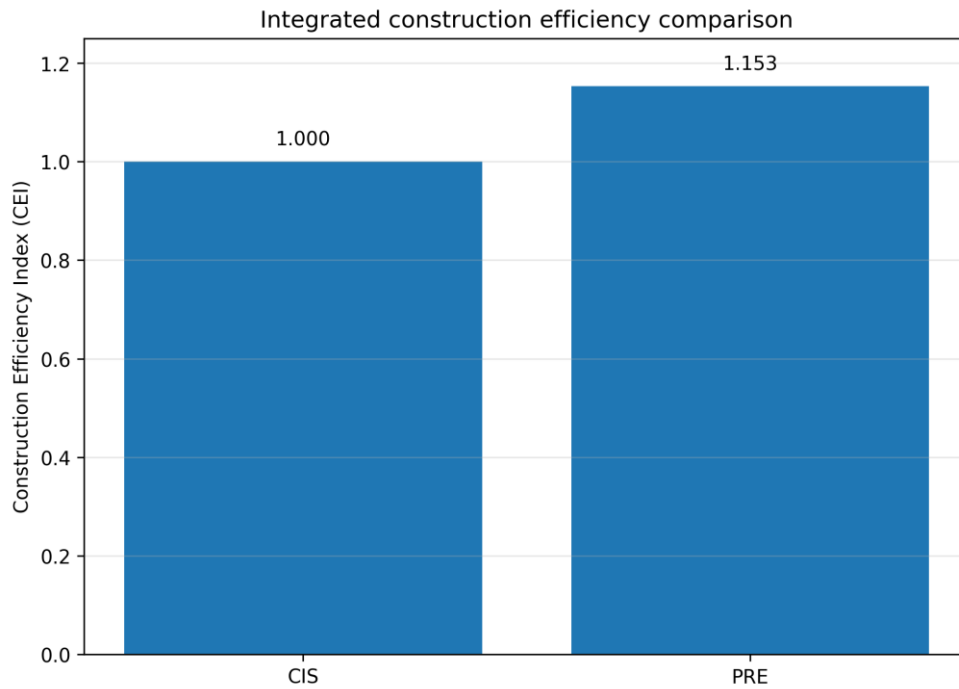


Figure 2. Integrated construction-efficiency comparison between CIS and PRE systems at the 50 km reference distance.

3.8. Sensitivity to Decision Priorities

Table 7. CEI sensitivity at the reference distance of 50 km

Weighting scenario	CEI – CIS	CEI – PRE
Balanced baseline – B	1.000	1.153
Material-oriented – M	1.000	1.142
Energy-oriented – E	1.000	1.138
Time-oriented – T	1.000	1.204

The prefabricated alternative remained preferable under all four weighting scenarios. The highest CEI was obtained for the time-oriented model, reflecting the strong relative advantage associated with construction duration. The variation between maximum and minimum PRE CEI values was $\Delta CEI = 0.066$, indicating that the system ranking remained relatively stable under the analysed decision priorities.

3.9. Energy Break-Even Versus Overall CEI Break-Even

An important result is that the energy break-even distance and the overall CEI break-even distance are not identical. The pure energy advantage disappears at approximately 185 km,

but PRE continues to achieve $CEI > 1.00$ because material and construction-time advantages remain. Under the baseline weighting model, the theoretical distance at which $CEI_{PRE} = CEI_{CIS}$ was approximately 1,454 km.

$$d_{energy, BE} \approx 185 \text{ km} < d_{CEI, BE} \approx 1454 \text{ km} \quad (21)$$

The very large theoretical CEI break-even value should be interpreted cautiously. Its significance is conceptual rather than prescriptive: a construction system can cease to be preferable according to one isolated criterion while still remaining favourable according to an integrated multi-criteria assessment.

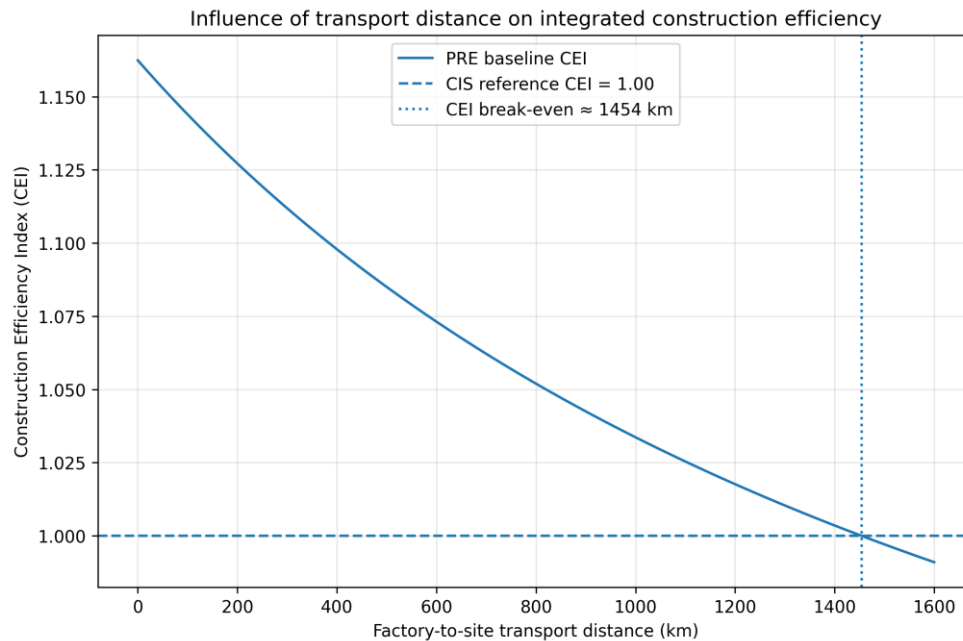


Figure 3. Influence of factory-to-site transport distance on the integrated Construction Efficiency Index.

3.10. Comparative Performance Summary

Table 8. Summary of comparative performance at a 50 km transport distance

Performance indicator	CIS	PRE	Relative PRE effect
Total material input (t)	4,895.3	4,513.0	−7.8%
Material losses (t)	240.3	88.0	Substantial reduction
Construction time (days)	220	165	−25.0%
Total energy demand (GJ)	9,164.7	8,592.9	−6.2%
I_M	1.000	1.085	PRE advantage
I_E	1.000	1.067	PRE advantage
I_T	1.000	1.333	PRE advantage
Baseline CEI	1.000	1.153	PRE advantage

Within the adopted scenario, construction-time efficiency represented the strongest relative advantage of prefabrication, followed by material efficiency and then the energy advantage. Transportation emerged as the principal variable capable of reversing the isolated energy ranking. The results therefore demonstrate that prefabricated construction should not be classified as universally superior or inferior to conventional cast-in-situ construction; its performance depends on the combined effects of structural design, material consumption, production efficiency, site productivity and logistics.

4. Conclusions

This study developed an integrated comparative methodology for evaluating the material, energy and construction-time efficiency of conventional cast-in-situ and prefabricated reinforced-concrete systems. A representative five-storey building with a gross floor area of approximately 5,000 m² was analysed through a clearly defined parametric numerical scenario.

The prefabricated system demonstrated higher material efficiency within the adopted assumptions. Total structural-material input decreased from approximately 4,895.3 t for CIS to 4,513.0 t for PRE, corresponding to a reduction of approximately 7.8%, while estimated material losses were substantially lower in the prefabricated scenario.

Construction-time efficiency represented the strongest comparative advantage of prefabrication. The structural construction duration decreased from 220 to 165 days,

corresponding to a 25% reduction and a time-efficiency indicator of $I_T = 1.333$.

At the reference transport distance of 50 km, the calculated total energy demand was approximately 9,164.7 GJ for CIS and 8,592.9 GJ for PRE. The energy advantage of approximately 6.2% was smaller than the time advantage and was strongly dependent on transportation distance.

The project-specific energy break-even transport distance was approximately 185 km. Below this distance, the prefabricated alternative exhibited lower total energy demand under the adopted assumptions; beyond this distance, the additional transport burden reversed the isolated energy ranking.

The proposed Construction Efficiency Index integrated material, energy and time performance. At the reference distance, $CEI_{CIS} = 1.000$ and $CEI_{PRE} = 1.153$. The prefabricated alternative remained preferable under balanced, material-oriented, energy-oriented and time-oriented weighting scenarios.

The energy break-even distance and integrated CEI break-even distance were substantially different. Although the isolated energy advantage disappeared at approximately 185 km, the overall CEI advantage persisted to a much larger theoretical distance because material and time benefits remained. This demonstrates the importance of multi-criteria assessment when individual indicators produce different conclusions.

The principal scientific contribution of the study is the combination of two decision-support elements: the dimensionless Construction Efficiency Index, $CEI = w_M I_M + w_E I_E + w_T I_T$, and the project-specific break-even transportation-

distance model, $d_{BE} = (E_{CIS} - E_{PRE,0}) / (M_{PRE} e_{tr} \lambda)$. Together they provide a transparent framework for analysing trade-offs between construction technologies.

A major limitation is that the numerical results are based on a parametrically defined reference building rather than measured data from a completed project. The adopted material quantities, loss rates, energy intensities, site-energy requirements and transport factors are modelling assumptions and should not be interpreted as universal values.

Future research should apply the methodology to completed reinforced-concrete projects using verified bills of quantities, construction schedules, factory-energy records, transport logs and project-specific life-cycle inventory data. The CEI framework may also be extended to incorporate cost, greenhouse-gas emissions, labour productivity, durability and end-of-life scenarios. The overall conclusion is that optimal construction-system selection is a function of materials, energy, time and logistics rather than a single universal claim of technological superiority.

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Техническа и софтуерна архитектура на работна станция за анализ на киберинциденти в облачно-ориентирана среда за съвместна работа

Technical and Software Architecture of a Cyber Incident Analysis Workstation in a Cloud-Oriented Collaborative Environment for Defence and Security

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Abstract: This paper presents a methodology for the architectural configuration of information infrastructure components of a cybersecurity system, in which the NATO Common Operating Environment (NCOE) model is used as an architectural basis for ensuring interoperability. The methodology systematizes the functional, technical, and software aspects of configuration and considers the possibilities for integrating allied and national information systems.

Based on the proposed methodology, a technical and software configuration of a workstation for cyber incident analysis is developed and considered as a component of a virtualized and cloud-oriented collaborative environment. The architecture integrates virtualization, Docker and Kubernetes, REST API and gRPC, OpenID Connect, as well as cybersecurity monitoring and analysis tools, including Security Onion, Elasticsearch, Snort, Suricata, Zeek, and Wazuh.

The proposed model provides a basis for integrating workstations, virtual servers, applications, and information resources into a common information and communication environment for collaborative work and shared use of resources. Thus, the development contributes to the implementation of cloud-based and virtualized solutions, enhanced interoperability, and support for the management process in the field of defence and security.

Keywords: ARCHITECTURAL CONFIGURATION, CLOUD TECHNOLOGIES, VIRTUALIZATION, CLOUD ARCHITECTURE, CYBERSECURITY, CYBER INCIDENT ANALYSIS, COLLABORATION

1. Въведение

Развитието на облачните технологии, виртуализацията и разпределените информационни системи създава нови възможности за изграждане на високоскоростни, мащабируеми и сигурни информационно-комуникационни среди за нуждите на отбраната и сигурността. Особено значение придобиват архитектурите, които осигуряват споделено използване на информационни ресурси, сигурен обмен на данни, оперативна съвместимост и ефективна съвместна работа на потребители и специализирани приложения.

Цифровата трансформация на процесите в областта на отбраната и сигурността води до непрекъснато увеличаване на обема, разнообразието и динамиката на обработваната информация. Управленските структури и специализираните звена трябва да осигуряват своевременен достъп до информационни ресурси, обмен на данни между различни системи и ефективна съвместна работа между потребители, които могат да бъдат физически и организационно разпределени.

Тези изисквания поставят нови задачи пред информационно-комуникационната инфраструктура. Тя трябва да бъде не само надеждна и производителна, но и гъвкава, мащабируема и способна да интегрира хетерогенни информационни системи и приложения. Допълнително изискване при системите за отбраната и сигурността е осигуряването на високо ниво на защита на информационните ресурси, контрол на достъпа, проследимост на действията и устойчивост при възникване на инциденти.

Облачните технологии и виртуализацията предоставят подходяща технологична основа за реализиране на подобни среди. Чрез виртуализацията физическите изчислителни ресурси могат да бъдат абстрахирани и предоставяни на различни приложения и потребители в зависимост от текущите потребности. Облачният подход допълнително позволява централизирано управление на ресурсите, динамично разпределение на изчислителна мощност, съхранение на данни и предоставяне на приложения като услуги.

Прилагането на облачни технологии в областта на отбраната и сигурността обаче не следва да се разглежда единствено като технологична миграция от локална към

отдалечена инфраструктура [8]. Необходимо е да се разработи цялостна архитектурна концепция, която да отчита взаимодействието между инфраструктурните ресурси, виртуалните машини, приложенията, потребителските работни места, комуникационните интерфейси и средствата за защита.

В този контекст работната станция на анализатора по киберсигурност може да бъде разглеждана като функционален компонент на по-широка информационна инфраструктура. Тя предоставя достъп до специализирани инструменти за мониторинг и анализ, но същевременно взаимодейства с виртуални сървъри, бази данни, мрежови услуги и приложения за съвместна работа.

В предоставената архитектура **техническата** конфигурация е определена като съвкупност от компоненти и стандарти, които взаимодействат с други технически конфигурации посредством профили за оперативна съвместимост. Този подход позволява работната станция да бъде разглеждана не като изолирано устройство, а като част от интегрирана архитектура.

Съществено значение за подобна интеграция има използването на стандартизирани комуникационни протоколи и програмни интерфейси. В разработения модел са предвидени REST API за обмен на данни между работната станция и виртуалния сървър, gRPC за комуникация между микросервиси, HTTPS за сигурен достъп до приложения и OpenID Connect за удостоверяване на потребителите.

Допълнителна възможност за повишаване на гъвкавостта на архитектурата предоставят контейнеризацията и оркестрацията. Docker позволява приложенията и техните зависимости да бъдат пакетирани в преносими среди, докато Kubernetes осигурява механизми за управление и оркестрация на контейнеризирани приложения.

Целта на настоящата разработка е да се предложи техническа и софтуерна архитектура на работна станция за анализ на киберинциденти, която да може да бъде интегрирана в облачно-ориентирана, виртуализирана информационно-комуникационна среда за съвместна работа и споделено използване на информационни ресурси в областта на отбраната и сигурността.

За постигане на тази цел са поставени следните основни задачи:

1. Да се определят техническите и софтуерните компоненти на работната среда.
2. Да се анализират възможностите за виртуализация и контейнеризация на приложенията.
3. Да се определят комуникационните интерфейси и механизмите за оперативна съвместимост.
4. Да се интегрират средства за мониторинг, откриване и анализ на киберинциденти.
5. Да се определят технологии за сигурен достъп и обмен на информация.
6. Да се оцени приложимостта на архитектурния модел като компонент на среда за съвместна работа и споделяне използване на информационни ресурси.

За целта се предлага използване на методика за архитектурно конфигуриране на компонентите на информационната инфраструктура на система за киберсигурност, функционираща в обща оперативна среда на НАТО (NATO Common Operating Environment – NCOE). Методиката доразвива архитектурния подход чрез систематизиране на функционалните, техническите и софтуерните аспекти на конфигурирането и отчитане на изискванията за оперативна съвместимост. Тя е приложима както при интеграция в съюзни инфраструктури, така и при взаимодействие с национални и хетерогенни информационни среди [1, 3, 4].

Моделът NCOE осигурява връзката между профилите на общите стандарти на НАТО (NCSP/NISP – NATO Common/Interoperability Standards Profile) и практическото изграждане на оперативна съвместими автоматизирани информационни системи. Той определя основни услуги, стандарти, интерфейси и правила за внедряване, необходими за осигуряване на оперативна съвместимост на стратегическо, оперативное и тактическо ниво.

В рамките на методиката архитектурното конфигуриране се разглежда като последователен преход от функционалните изисквания към техническите средства и софтуерните компоненти. Съответно се разграничават три основни нива: функционална конфигурация (Functional Configuration – FC), определяща необходимите приложения и основни услуги; техническа конфигурация (Technical Configuration – TC), определяща компонентите и стандартите за тяхното взаимодействие; и софтуерна конфигурация (Software Configuration – SC), определяща логическите съвкупности от софтуерни продукти и интерфейси.

Прилагането на този подход позволява проследимо свързване на функционалните изисквания с техническите и софтуерните решения и създава основа за изграждане на оперативна съвместими системи за киберсигурност. Въз основа на методиката е разработен вариант на техническа и софтуерна на конфигурация на работна станция, предназначена за анализ на киберинциденти и подпомагане дейността на специалисти по киберсигурност.

2. Архитектурен подход и архитектурно конфигуриране на компонент на информационната инфраструктура

Предлаганата архитектура се основава на разделяне на информационната среда на функционални и технологични слоеве. Подобен подход позволява ясно разграничаване на мрежовите услуги, инфраструктурните услуги, общите приложения и приложенията със специфично предназначение.

Основните компоненти на модела NCOE включват:

- Приложения за обща поддръжка;
- Инфраструктурни услуги;
- Услуги на ядрата (на операционните системи);
- Интерфейси на приложните програми;
- Компонент на данни;
- Услуги поддръжка и част от мрежовите услуги.

В архитектурата се разграничават:

- слой на мрежовите услуги;
- слой на услугите на ядрото;
- слой на инфраструктурните услуги;
- слой на приложенията за обща поддръжка;
- слой на приложенията за специфична задача.

Това разделяне позволява различните компоненти да бъдат управлявани независимо и същевременно да бъдат интегрирани посредством стандартизирани интерфейси. В техническата конфигурация са разгледани IPv4 и IPv6, VLAN, SSH, виртуални мрежови интерфейси и различни механизми за достъп до виртуалната машина.

Архитектурният подход позволява работната станция да изпълнява ролята на потребителски и аналитичен компонент, докато изчислителните, комуникационните и информационните ресурси могат да бъдат предоставяни от виртуализирана инфраструктура.

Това е особено подходящо за облачно-ориентирана среда, при която ресурсите не са задължително физически свързани с конкретна работна станция, а могат да бъдат предоставяни динамично като услуги.

За архитектурното конфигуриране на компонент на информационната инфраструктура – автоматизирано работно място (APM) на анализатор по киберсигурност (машина Cyberops), е необходимо да се разработят функционална, техническа и софтуерна конфигурация - NATO Functional Configurations (FC), Technical Configurations (TC), Software Configurations (SC), представени съответно с **графично** изображение, съгласно модела NCOE и шаблон в **табличен** вид –NATO System View (NSV), NATO Technical View (NTV) (FC- NSV-12, TC- NTV-3, SC – NTV-4), за всеки от компонентите от информационната инфраструктура. В примера са разработени техническа и софтуерна конфигурации

3. Техническа конфигурация

3.1. Общи характеристики

Техническата конфигурация определя физическите и виртуалните компоненти, посредством които се реализира работната среда на анализатора по киберсигурност. Техническите конфигурации (TC) са сбор от компоненти и стандарти, които взаимодействат с други технически конфигурации посредством Профилите за Оперативна съвместимост..

В първоначалния архитектурен модел са разгледани базова машина, виртуална среда и специализирани виртуални машини. За виртуализация е използвана средата Oracle VM VirtualBox, а като специализирана среда за анализ е разгледана CyberOps Security Onion.

Виртуализацията позволява разделяне на различни функционални среди и намалява зависимостта между приложението и конкретната физическа инфраструктура. Това създава предпоставки за по-лесно преместване, възстановяване и мащабиране на приложенията при промяна на инфраструктурните изисквания.

3.2. Мрежова свързаност

Мрежовата комуникация е реализирана посредством стандартни протоколи и технологии за сигурен достъп. Предвидени са IPv4 и IPv6, VLAN, SSH и механизми за виртуален мрежов достъп.

Особено значение има разделянето на различните мрежови интерфейси. В софтуерната конфигурация са разгледани вътрешни мрежови интерфейси, DMZ и NAT, което позволява логическо разделяне на комуникационните потоци.

Този модел съответно може да бъде разширен в облачна инфраструктура чрез използване на виртуални мрежи, сегментация и политики за контрол на комуникациите между различните услуги.

3.3. Профили за вътрешна оперативна съвместимост

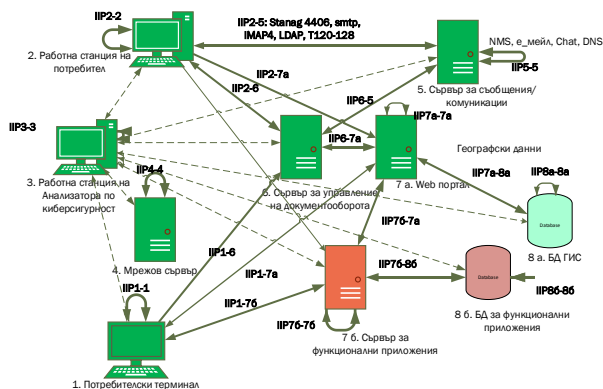
За осигуряване на взаимодействието между работната станция и виртуализираната инфраструктура са дефинирани профили за вътрешна оперативна съвместимост IIP (Internal Interoperability Profiles).

Те включват:

- обмен на данни между работната станция и виртуалния сървър;
- обмен на данни между работната станция и WEB приложения;
- RSS функционалност;
- интеграция с централизирана антивирусна система;
- комплексна подсистема за сигурност на информацията.

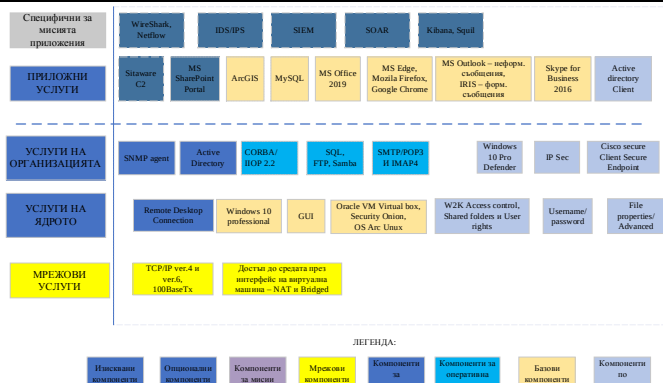
Тези профили представляват важна предпоставка за интегриране на работната станция в обща информационно-комуникационна среда.

Вариант на техническата конфигурация на работната станция на „Анализатор по киберсигурност“, включваща неговото разположение и свързаност посредством профилите за вътрешна оперативна съвместимост (IIP) в общата информационна среда е представен на фиг. 1.



Фиг. 1. Място на техническата конфигурация на Анализатора по киберсигурност и профилите за вътрешна оперативна съвместимост в общата информационна среда

Вариант на техническа конфигурация според модела NCOE на потребителска работна станция на „Анализатор по киберсигурност“ е представена на фиг. 2.



Фиг. 2. Техническа конфигурация на работно място на Анализатора по киберсигурност съгласно модела NCOE.

За техническа конфигурация от архитектурната рамка на NATO - Technical Configurations се из ползва табличен шаблон (NTV-3) описан в Implementation handbook [2, 5, 6, 7],

Вариант на техническа конфигурация на потребителска работна станция на „Анализатор по киберсигурност“ според модела NTRM (NATO Technical Reference Model), попълнена в шаблон NTV-3 са представена в таблица 1 и 2.

4. Софтуерна конфигурация

Софтуерната конфигурация включва операционните системи, системните услуги, комуникационните компоненти, приложенията за обща поддръжка и специализираните средства за анализ на киберсигурността.

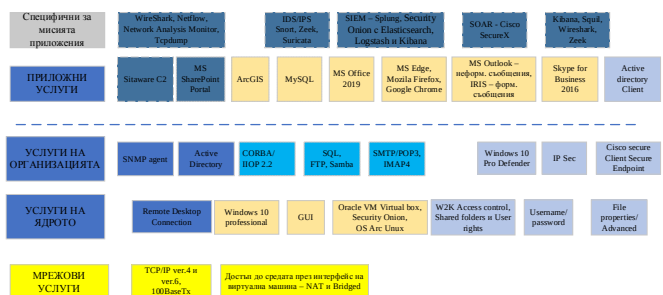
Софтуерните конфигурации представляват сбор от софтуерни продукти и сегменти, които осигуряват интерфейсите с други софтуерни конфигурации. В архитектурната рамка те са свързани с техническите конфигурации и стандартите, които трябва да бъдат поддържани.

Софтуерните конфигурации (SC) са физически сбор на софтуерни продукти и сегменти, които осигуряват Софтуерните интерфейси с други софтуерни конфигурации (Фиг.3)

В Implementation handbook [2]. е представен шаблон За софтуерна конфигурация от архитектурната рамка на NATO т.е. SOFTWARE CONFIGURATIONS (NTV-4). Вариант на софтуерна конфигурация NTV-4 е представен в таблица 3.

Шаблонът на технически изглед (NTV-4) дефинира софтуерните конфигурации на системите (таблица 3). Софтуерните конфигурации са основни шаблони, които са резултат от проектите технически конфигурации. Те съдържат задължителните софтуерни продукти, които са необходими за прилагане на техническите стандарти.

Вариант на софтуерна конфигурация според модела NCOE на потребителска работна станция на „Анализатор по киберсигурност“ е представена на фиг.3.



Фиг. 3. Софтуерна конфигурация на работно място на „Анализатора по киберсигурност“

4.1. Операционни системи и виртуализация

В архитектурата са разгледани Windows и Linux базирани среди, виртуализация посредством Oracle VM VirtualBox и специализирани виртуални машини.

Linux базираната среда позволява използване на инструменти за мрежов анализ, защита и управление на системните ресурси. Разгледани са Ubuntu, UFW, ClamAV и различни клиентски приложения.

4.2. Приложения за съвместна работа

В средата са включени услуги за документооборот, електронна поща, обмен на файлове и видеоконферентна връзка. В първоначалния модел са разгледани Microsoft SharePoint, Microsoft Exchange, Outlook и Skype for Business.

От гледна точка на Задача 1.2.7 тези компоненти имат значение не само като стандартни потребителски приложения, а като основни елементи на среда за съвместна работа, в която потребителите могат да обменят документи, съобщения, файлове и информация в реално време.

4.3. Специализирани приложения за киберсигурност

Ключов компонент на архитектурата е средата за анализ на киберинциденти Security Onion.

В нея са интегрирани средства за пълно улавяне и анализ на мрежови пакети, SIEM функционалност, системи за откриване на прониквания и инструменти за анализ на мрежовата активност. Разгледани са приложения като Wireshark, Tcpdump, Netflow, Elasticsearch, Logstash, Kibana, Sguil, Snort, Suricata, Zeek и Wazuh.

Интегрирането на тези инструменти в обща среда позволява събиране на информация от различни източници и подпомага анализа на събитията по киберсигурност.

4.4. Контейнеризация и оркестрация

Една от основните насоки за развитие на архитектурата е използването на контейнеризация и оркестрация.

Docker позволява приложенията да бъдат реализирани като самостоятелни контейнеризирани компоненти с ясно дефинирани зависимости. Това улеснява разгръщането на приложенията в различни инфраструктурни среди.

Kubernetes предоставя възможност за управление на контейнерите, автоматизирано разполагане, мащабиране и управление на жизнения цикъл на приложенията. В разработения модел Docker и Kubernetes са определени като технологични компоненти на слоя за проектиране и интеграция на софтуера.

Прилагането на контейнеризация е особено подходящо за архитектури, изградени от микросервиси. Всеки функционален компонент може да бъде реализиран като отделна услуга, която взаимодейства с останалите чрез стандартизирани интерфейси.

Този подход повишава гъвкавостта на архитектурата и създава възможности за постепенно реинженеринг на съществуващи приложения при миграцията им към облачни услуги.

4.5. Комуникационни интерфейси и интеграция на приложенията

За осигуряване на взаимодействието между отделните компоненти на системата се използват стандартизирани програмни и комуникационни интерфейси.

REST API се използва за обмен на данни между работната станция и виртуалния сървър. gRPC е предвиден за комуникация между микросервисите. HTTPS осигурява защитен достъп до уеб приложенията, а OpenID Connect се използва за удостоверяване на потребителите.

Този модел позволява отделните компоненти да бъдат разработвани и актуализирани независимо, като същевременно запазват възможността за интеграция.

От гледна точка на облачната архитектура това е важно условие за изграждане на модулна и мащабируема информационна среда.

4.6. Информационна сигурност

Системите, предназначени за нуждите на отбраната и сигурността, трябва да осигуряват защита на информацията на всички архитектурни нива.

В разработения модел са предвидени IPSec, защитна стена, антивирусна защита, TLS/SSL и механизми за удостоверяване. В софтуерната конфигурация са разгледани Windows Defender, ClamAV и IPSec настройки.

За защита на комуникациите са разгледани HTTPS и TLS, като в архитектурния модел са посочени TLS 1.2/1.3. Като перспективна архитектурна насока е включен и подходът Zero Trust Architecture, който предполага непрекъсната проверка на потребители, устройства и комуникационни взаимодействия.

Сигурността следва да бъде разглеждана като хоризонтален архитектурен слой, а не като отделна функция. Това означава, че контролът на достъпа, криптирането, мониторингът и откриването на аномалии трябва да бъдат интегрирани във всички етапи от обработката и обмена на информация.

4.7. Управление и мониторинг на облачно-ориентираната среда

Ефективното управление на виртуализирана и облачно-ориентирана инфраструктура изисква непрекъснато наблюдение на състоянието на системата.

В предложената архитектура Prometheus е определен като средство за мониторинг, а Grafana – за визуализация на метриките.

Чрез мониторинга могат да се наблюдават натоварването на ресурсите, състоянието на приложенията, комуникационните параметри и други показатели, свързани с надеждността и производителността.

За среда, предназначена за отбраната и сигурността, мониторингът има и допълнителна функция – подпомага откриването на отклонения от нормалното поведение и идентифицирането на потенциални инциденти.

Управление на данните и споделяне използване на информационни ресурси

Ефективното споделяне на информационни ресурси е един от основните елементи на средата за съвместна работа. [4, 9]

В архитектурата са разгледани PostgreSQL, Elasticsearch и ZFS като компоненти за управление и съхранение на данни. Като перспективни решения са посочени Distributed SQL бази данни и облачно базирани решения за съхранение.

Разделянето между приложенията и слоя за управление на данните позволява да се реализира архитектура, при която различни приложения могат да използват общи информационни ресурси посредством стандартизирани интерфейси.

Това е особено важно при анализ на киберинциденти, тъй като информацията може да постъпва от множество източници – мрежови устройства, крайни системи, приложения, системи за сигурност и потребителски работни станции [10].

Таблица 1. Примерна техническа конфигурация на потребителска работна станция на анализатора по киберсигурност

NTV-3	ТЕХНИЧЕСКА КОНФИГУРАЦИЯ (ТК) (да се попълни във фазата на доставките за целевата архитектура)				
ТК компоненти (продукти) и/или стандарти. (пояснение – продуктите се задават с наклонен шрифт, с стандартите с нормален)	Слой на мрежови услуги	Слой на услугите на ядрото	Слой на инфраструктурните услуги	Слой на приложенията за обща поддръжка	Слой на приложенията за специфична задача
	- LAN за работа с IPv4 и IPv6, Netware на Novell; - IPv4 - IETF Standard 5/RFC 791/RFC 950/RFC 919/RFC 922/RFC 792/RFC 1112; Internet Protocol, September 1981; - IPv6 - IETF Standard RFC 8200; - IPX/SPX Compatible Protocol; - Поддръжка на VLANs – IEEE 802.1Q; - Telnet - Telnet (IETF STD 8:1983, RFC 0854:1983, 0855:1983); - SSH - RFC 4250, The Secure Shell (SSH) Protocol Assigned Numbers Достъп до средата през интерфейса на виртуалната машина – NAT и Bridged	Операционна система: - на базовата машина Windows 10 Pro; - среда за виртуализация Oracle VM VirtualBox; - Виртуална машина CyberOps Security Onion; - ОС на виртуалната машина Arg Linux; - Система 4096 MB;	IPSec - IETF Standard RFC 1108; Windows 10 Defender Firewall; Antivirus Software – Cisco secure Client Secure Endpoint, Domain Name Services - IETF Standard 13/RFC 1034/RFC 1035, Domain Name System, November 1987. Active Directory services - LDAP v3 (RFC 2251:1997); Услуги за Документооборот – MS SharePoint Portal Server 2012; Уеб Услуги (некриптирани - HTTP v1.1 (RFC 2616:1999 updated by 2817:2000), URL (RFC 1738:1994 updated by 1808:1995, 2368:1998, 2396:1998, 2732:1999), URI (RFC 2396:1998 updated by 2732:1999) и криптирани RFC 2818: HTTP Over TLS) Електронна поща за обмен на неформализовани документи - SMTP (RFCs 1870:1995, 2821:2001); POP3 (RFC 1939:1996), IMAP4 (RFC 3501:2003) Обмен на файлове - FTP (IETF STD 9, RFC 959:1985 updated by 2228:1997, 2640:1999, 2773:2000) FIPS Pub 158 (X-Window System) Видеотелеконференция – Skype for Business 2016 Управление на обекти при разпределено изчисление - CORBA/IIOP 2.2	C2 софтуер Systematic - C2 System „Sitaware“: - HQ (щаб на бригада и нагоре) Dismounted soldier – спешен войник или C2 system на ПИКИС или C2PC на BAE System MS Office 2010; Microsoft SharePoint; Microsoft Edge; Acrobat Reader, MySQL Клиент Email клиент MS Outlook; Географска ИС – ArcGIS; Персонална база данни MySQL	- интегрирано в пакета Security Onion, директно преминаване от Sguil към други инструменти, пълни заснети пакети, подходящи за анализ; - NIDS, изпраща прикачени файлове за анализ на злонамерен софтуер, локален мониторинг, , изобразени с инструментите tcpflow, HIDS; - поддръжка за многопоточност на графичния процесор (GPU);
Профили за вътрешна оперативна съвместимост (IP) между АРМ и виртуалния сървър	Профил IP1 - Обмен на данни между АРМ и виртуалния сървър Профил IP2 - Обмен на данни между АРМ и WEB приложения Профил IP3 - RSS функционалност на работното място – работна среда, база данни, storage пространство Профил IP4 - Антивирусен клиент – елемент на централизираната система за антивирусна поддръжка Профил IP5 - Комплексна подсистема за сигурност на информацията				

Таблица 2. Техническа Конфигурация на работна станция на анализатора по киберсигурност в табличен вид.

Обхват на услугата	Услуга	Стандарти	Нови стандарти
Операционни системи	- Управление на ресурсите - Поддръжка на мрежови услуги - Защита и сигурност	- Ubuntu 20.04 LTS (64-bit) - TCP/IP стек - IPSec - UFW (Uncomplicated Firewall)	- Поддръжка на многопоточност на графичния процесор (GPU) - Виртуализация чрез Oracle VM VirtualBox
Проектиране на софтуер	- Разработка на софтуер - Интеграция на различни системи	- Agile методологии - DevOps практики	- Контейнеризация чрез Docker - Kubernetes за оркестрация
Потребителски интерфейси	- Удобство за потребителя - Интуитивност - Поддръжка на множество платформи	- HTML - CSS - JavaScript - REST API	- Progressive Web Apps (PWA) - WebAssembly (WASM)
Управление на данните	- Съхранение на данни - Архивиране - Възстановяване при инциденти	- PostgreSQL - ElasticSearch - ZFS файлови системи	- Distributed SQL databases (e.g., CockroachDB) - Облак-базирани решения за съхранение на данни
Обмяна на данни	- Сигурен трансфер на данни - Съвместимост между различни системи	- HTTPS (TLS 1.2/1.3) - SMTP, POP3, IMAP4 - FTP - SSH	- HTTP/3 - QUIC протокол - Zero Trust Architecture (ZTA)
Стандарти за пренос на информацията	TCP	IETF Standard 7/RFC 793, Transmission Control Protocol, September 1981.	
	IP	IETF Standard 5/RFC 791/RFC 950/RFC 919/RFC 922/RFC 792/RFC 1112, Internet Protocol, September 1981.	
	OSI/IP	IETF Standard 35/RFC 1006, ISO Transport Service on top of the TCP, May 1987	
	Access to LAN	IEEE 802.3u:1995, Supplement to ISO/IEC 8802-3:1993, Local and Metropolitan Area Networks: Media Access Control (MAC) Parameters, Physical Layer, Medium Attachment Units, and Repeater for 100 Mbps Operation, Type 100 BASE-T.	
	Networks Management	IETF RFC 2011, SNMPv2 Management Information Base for the Internet Protocol, November 1996.	
	Internet/Intranet Protocols	IETF RFC 2401, Security Architecture For The Internet Protocol, November 1998.	

Таблица 3. Вариант на софтуерна конфигурация на потребителска работна станция на анализатора по киберсигурност

SC Продукти в:	Слой на мрежовите услуги	Слой на услугите на ядрото	Слой на инфраструктурните услуги	Слой на приложенията за обща поддръжка	Слой на приложенията за специфична задача
Бележка: Показва номера на софтуерната версия и стандарт(и), които са покрити от софтуерния пакет	TCP/IP стек; - Поддържащ IPv4 и IPv6, Remote accesss (Telnet, SHH - HyperTerminal, PuTTY) Отдалечен достъп до мрежата; - Адаптер 1: Intel PRO/1000 MT Desktop (Вътрешна мрежа, 'дмз') Адаптер 2: Intel PRO/1000 MT Desktop (Вътрешна мрежа, 'дмз') Адаптер 3: Intel PRO/1000 MT Desktop (NAT)		IPSec настройки на Windows 2010 Windows Defender Услуги за Документооборот на сървер с MS SharePoint Portal Server, достъпвани през браузър – Microsoft Edge Електронна поща за обмен на неформализовани документи на Windows 2013 Enterprise Server със MS Exchange Server (SMTP, POP3, IMAP4) – MS Outlook клиент Видеотелеконференция – Skype for Business 2016 Достъп до база данни Oracle – SQL client - IPSec настройки на Ubuntu 20.04; - Софтуерен продукт UFW (Uncomplicated Firewall); - Antivirus Software ClamAV; - Достъпвани през браузър – Firefox, Chrome; - Thunderbird клиент; - PostgreSQL клиент за достъп до бази данни;	C2 софтуер Systematic - C2 System „Sitaware“; - HQ (щаб на бригада и нагоре) Dismounted soldier – спешен войник или C2 system на ПИКИС или C2PC на BAE System; - Mozilla Firefox; - Google Chrome;	Security Onion (интегрирано решение за мрежова сигурност): - пълно улавяне на пакети - Wireshark , Tcpdump , Netflow , Network Analysis Monitor на системата Cisco Prime Infrastructure; - SIEM система - Splunk , Security Onion с ELK , което се състои от интегрираните приложения Elasticsearch, Logstash и Kibana За анализ на пакети и предупреждения - Sguil , Kibana ; Intrusion Detection System – Snort , Suricata , Wazuh ; Инструмент за наблюдение на мрежовата сигурност и Network Intrusion Detection System - Zeek (Bro) -; Host-based Intrusion Detection System - OSSEC ; - CapME; - Пакети данни: PCAPS, Content Data, Transaction Data, Session Data, Host Logs, Alert Data, Syslog Data, Metadata; Elasticsearch за управление на данни;
Сегменти: Бележка: Опишете продуктите които съставляват различните сегменти	Сегмент 1 Вътрешен интерфейс: - HTTP интерфейс - порт 80, 8080; - HTTPS интерфейс - порт 443; - SMTP интерфейс за обмен на съобщения - порт 25; - SSL интерфейс за проверка за вируси – порт 443 - FTP интерфейс за трансфер на файлове – порт 21; - SSH интерфейс за отдалечен достъп – порт 22;	Сегмент 2 Вътрешен интерфейс: - REST API за обмяна на данни между APM и виртуалния сървър; - gRPC за комуникация между микросервизите; - Docker за контейнеризация; - Kubernetes за оркестрация на контейнерите;	Сегмент 3 Външен интерфейс: - HTTPS за сигурен достъп до уеб приложения; - OpenID Connect за удостоверяване на потребителите;	Сегмент 4 Външен интерфейс: - Prometheus за мониторинг на системата; - Grafana за визуализация на метриците; - SSL/TLS за криптиране на комуникациите;	Сегмент 5
Интерфейс	Вътрешен	Вътрешен	Външен	Външен	Външен

и Бележка: Опишете интерфейсите по отношение на APIs (Приложно- програмни интерфейси) вградени в продукти или като отделни продукти. Споменете поддържаните стандарти и възможните настройки на параметрите.					
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	Networks Management	IETF RFC 2011, SNMPv2 Management Information Base for the Internet Protocol, November 1996.	
	Internet/Intranet Protocols	IETF RFC 2401, Security Architecture For The Internet Protocol, November 1998.	

Обработката и корелацията на тази информация създават възможност за формиране на единна информационна картина, която може да подпомага вземането на управленски решения.

4.8. Архитектурен модел на среда за съвместна работа

На базата на разгледаните компоненти може да бъде формулиран архитектурен модел на среда за съвместна работа, включващ:

1. Потребителски слой – работни станции и потребителски интерфейси.

2. Слой за приложения – специализирани приложения за анализ, документооборот, комуникация и управление.

3. Слой за микросъзвеси – контейнеризирани функционални компоненти.

4. Слой за интеграция – REST API, gRPC и други интерфейси за обмен на данни.

5. Слой за данни – бази данни, Elasticsearch и файлово/обектно съхранение.

6. Инфраструктурен слой – виртуални машини, виртуални мрежи, изчислителни и дискови ресурси.

7. Слой за сигурност – удостоверяване, контрол на достъпа, криптиране, защитни механизми и мониторинг.

8. Слой за управление и наблюдение – инструменти за мониторинг, визуализация и управление на инфраструктурата.

Тази многослойна архитектура позволява отделните компоненти да бъдат развивани независимо и постепенно да бъдат интегрирани в по-голяма облачна инфраструктура.

В този смисъл работната станция на анализатора по киберсигурност представлява не само крайно потребителско устройство, а функционален възел в система за споделено използване на информационни ресурси.

Приложимост на архитектурния модел в областта на отбраната и сигурността

Предложеният архитектурен модел може да намери приложение в среди, в които е необходимо взаимодействие между различни потребители, информационни системи и специализирани звена.

Сред основните направления на приложение и свързаните с тях услуги са:

- анализ и мониторинг на киберинциденти;
- наблюдение на комуникационната инфраструктура;
- обмен на информация между аналитични звена;
- централизирано или разпределено управление на информационни ресурси;
- споделено използване на бази данни и приложения;
- дистанционен достъп до специализирани информационни ресурси;
- подпомагане на процесите по вземане на управленски решения;
- интегриране на съществуващи приложения в облачно-ориентирана инфраструктура.

Особено значение има възможността архитектурата да бъде разширявана чрез добавяне на нови виртуални машини, контейнеризирани приложения и микросервиси, без да се налага цялостно реструктуриране на съществуващата среда.

Това създава предпоставки за поэтапна миграция на съществуващи приложения към облачни услуги и за реинженеринг на приложенията с цел повишаване на тяхната оперативна съвместимост и мащабируемост.

Предложеният модел съответства на основните направления на Задача 1.2.7 „Облачни технологии и системи за съвместна работа с приложения в отбраната на държавата и сигурността на обществото“.

Първото направление е свързано с анализа на облачните архитектури и съвременните центрове за данни. В разработката са разгледани виртуализирани среди, виртуални сървъри, контейнеризация, оркестрация и различни подходи за управление на изчислителни и информационни ресурси.

Второто направление е изграждането на информационно-комуникационна среда за съвместна работа. Тази възможност се реализира чрез интегриране на комуникационни услуги, услуги за документооборот, електронна поща, обмен на файлове, специализирани приложения и стандартизирани програмни интерфейси.

Третото направление е разработването на прототип на архитектура за споделено използване на информационни ресурси и подпомагане на управленския процес. Представената архитектура на работната станция и виртуализираната среда може да бъде разглеждана като функционален компонент на такъв прототип.

В този смисъл разработката не се ограничава до техническо описание на отделна работна станция, а представя принципи за нейното интегриране в по-широка облачно-ориентирана архитектура.

Заключение

В настоящия доклад е представен разработен вариант на техническа и софтуерна архитектура на работна станция за анализ на киберинциденти, разглеждана като компонент на облачно-ориентирана среда за съвместна работа и споделено използване на информационни ресурси.

Анализът показва, че комбинацията от виртуализация, контейнеризация, стандартизирани комуникационни интерфейси, средства за управление на данни и интегрирани инструменти за киберсигурност създава подходяща технологична основа за изграждане на гъвкави и разширяеми информационно-комуникационни среди.

В архитектурата са определени техническите и софтуерните конфигурации, комуникационните интерфейси и механизмите за вътрешна оперативна съвместимост. Разгледани са възможностите за интегриране на виртуални машини, контейнеризирани приложения и микросервиси посредством REST API и gRPC. Предвидени са технологии за удостоверяване, криптиране и мониторинг, които допринасят за повишаване на сигурността и надеждността на средата.

Интегрирането на Security Onion и специализираните средства за анализ – Elasticsearch, Snort, Suricata, Zeek, Wazuh и други – позволява формиране на среда за събиране и анализ на информация за събитията по киберсигурността.

Съществен резултат от разработката е прилагането на архитектурен подход, при който работната станция не се разглежда като изолирана крайна система, а като компонент на виртуализирана и облачно-ориентирана информационно-комуникационна среда. Това позволява споделяне на информационни ресурси, интегриране на специализирани приложения и реализиране на съвместна работа между различни потребители и функционални звена.

Предложените техническа и софтуерна архитектурни конфигурации създава основа за последващо прототипиране на система за споделено използване на информационни ресурси и подпомагане на управленския процес в областта на отбраната и сигурността. Тя позволява поэтапно интегриране на съществуващи приложения, включително чрез тяхното реинженериране и адаптиране към облачно-ориентиран модел.

В резултат разработката допринася към основните резултати на Задача 1.2.7 чрез:

- анализ и структуриране на архитектурни решения за виртуализирана и облачно-ориентирана среда;
- определяне на компоненти на информационно-комуникационна среда за съвместна работа;
- разработване на архитектурен модел за споделяно използване на информационни ресурси;
- определяне на механизми за оперативна съвместимост между компонентите;
- интегриране на средства за киберсигурност, мониторинг и анализ;
- създаване на архитектурна основа за последващо разработване на прототип на система за подпомагане на управленския процес.

По този начин предложената архитектура представлява технологична основа за развитие на сигурни, мащабируеми и интегрирани информационно-комуникационни среди, приложими към съвременните изисквания на отбраната и сигурността.

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Maturity assessment and transformation roadmap for smart manufacturing in micro and small enterprises

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Abstract—A practical maturity assessment is required before resource-constrained enterprises invest in smart and intelligent manufacturing. This paper proposes and demonstrates a compact method for micro and small enterprises in furniture and wood processing. Thirty questions assess fifteen capability areas grouped into People, Processes and Technologies on a 0–4 scale. Area, dimension and overall scores are complemented by a target gap and a priority index that combines business relevance and implementation complexity. A Google Sheets prototype implements transparent calculations, rule-based recommendations and an optional generative-AI layer that converts validated scores into structured initiatives. In an illustrative application to an 8–10-person furniture enterprise, the overall maturity index was 1.2/4; People scored 1.7, Processes 1.1 and Technologies 0.8. The resulting roadmap prioritizes downtime coding, production-order traceability, machine-data collection and MES-light functions before advanced analytics. The contribution is an evidence-based, dependency-constrained mechanism that converts maturity scores into prioritized and measurable transformation initiatives.

Keywords—MSE; Maturity Assessment; Industry 4.0; Smart Manufacturing; Intelligent Manufacturing; Furniture Production; Roadmap; Generative AI.

1. Introduction

Micro, small and medium-sized enterprises (MSMEs) rarely begin digital transformation with integrated information systems and dedicated teams. Furniture and wood-processing enterprises commonly combine partially automated machines, operator-dependent knowledge and fragmented production records. A maturity assessment is therefore needed to identify prerequisites, capability gaps and feasible investment sequences [1]–[4].

Readiness indicates whether transformation can start, whereas maturity describes the achieved capability level and the next development step [1], [2]. Existing models provide valuable strategic and technological perspectives [2]–[7], but their direct application may be demanding for micro enterprises. This study adapts their logic into a short, evidence-oriented instrument and asks: (1) how can maturity be measured transparently with limited effort; (2) how can weak capabilities be converted into prioritized initiatives; and (3) how can generative AI improve explanations without replacing deterministic scoring and rules?

The instrument is optimized for micro and small enterprises, where organizational roles are compact and assessment resources are limited. It may also serve as a preliminary diagnostic instrument in medium-sized enterprises, but transfer to more complex organizational structures requires additional validation and, where necessary, extension of the capability set.

2. Methodology and Model Design

The method was developed by comparative synthesis of representative readiness and maturity models and by translating recurring requirements into a sector-specific design: low complexity, actionability, people–process–technology balance, roadmap support and furniture-sector fit. The result comprises 15 capability areas and 30 questions. Each assessment answer must be supported, where possible, by observable evidence such as order records, downtime logs, machine signals, maintenance schedules, work instructions or dashboards. The sector-specific capability selection and enterprise constraints were adapted from the broader methodological and experimental work reported in [9].

2.1. Design requirements derived from existing models

The reviewed approaches contribute complementary elements. IMPULS provides an accessible readiness profile [2]; the PwC framework connects transformation with the digital-enterprise strategy [3]; the Acatech index describes progression from computerization and connectivity toward visibility, predictive capability and adaptability [4]; Ganzarain and Errasti and the VDMA guideline emphasize staged implementation in SMEs [5],

[7]; and Schumacher et al. and Akdil et al. demonstrate multidimensional and strategy-oriented assessment [6], [10]. However, their direct application in micro-enterprises is constrained by extensive questionnaires, reliance on expert-based weighting, assumptions regarding integrated information systems, and limited guidance for translating assessment results into affordable improvement initiatives. Smart-manufacturing architectures additionally clarify the role of connected assets and data-driven services [8], while recent furniture-sector research demonstrates the value of real-time Industry 4.0 implementation for operational and energy performance [12].

Five requirements were consequently adopted. First, the assessment must be completed during a short facilitated workshop. Second, every score must be traceable to observable evidence. Third, technology must be evaluated together with organizational and process prerequisites. Fourth, weak areas must be linked to concrete initiatives, dependencies and measurable effects. Fifth, the resulting roadmap must respect the investment capacity and operational specifics of furniture production, including customized orders, variable batch sizes, machine retrofits, dust extraction and electrical power constraints. The resulting dimensions, capability areas and assessment purposes are summarized in Table 1.

TABLE 1. Structure of the proposed assessment model

Dimension	Capability areas	Assessment purpose
People	Leadership and strategy; operator skills; digital skills; improvement culture; maintenance roles	Management commitment, competence and organizational capacity
Processes	Standard operations; production orders; downtime management; quality control; planned maintenance	Standardization, traceability and process control
Technologies	Machine automation; data collection; connectivity; MES/operations systems; analytics and prediction	Automation, data infrastructure and decision support

Each capability area j is assessed by two questions on a five-point scale: 0—absent; 1—ad hoc; 2—basic/local; 3—systematic/standardized; and 4—optimized/data-driven. The area score C_j is the arithmetic mean of its two question scores. Dimension scores and the overall index M are arithmetic means of the relevant area scores. Equal weighting is used in the basic version to retain interpretability; optional weights may be introduced only after stakeholder agreement. The ordinal scales

used to determine business relevance and implementation complexity are presented in Table 2.

TABLE 2. Ordinal scales used for prioritization

Value	Business relevance B_j	Implementation complexity K_j
1	Limited effect	Low: local change
2	Moderate performance effect	Medium: training or limited integration
3	High safety, delivery, quality or cost effect	High: major investment or organizational change

For a 12-month target T_j , the gap is $G_j = \max(0, T_j - C_j)$, and the priority index is $P_j = G_j \times B_j / K_j$. A large gap with high relevance and low or moderate complexity therefore receives early attention. The 12-month targets are agreed in the third workshop using strategic need, available budget, technical dependencies and feasible implementation effort; they are planning values rather than universal benchmarks.

The seven maturity bands—traditional (0–0.5), digitized (>0.5–1.0), connected (>1.0–1.5), visible (>1.5–2.0), managed (>2.0–2.5), optimized (>2.5–3.25) and smart (>3.25–4.0)—provide a pragmatic interpretation derived from the staged transformation logic adapted from the reviewed maturity models [2], [4], [5], [7]. The unequal upper intervals reflect the broader organizational change required for optimization and smart operation. These thresholds are not statistically calibrated and will be tested in future multi-company validation.

2.2. Assessment procedure

The procedure is organized in three working sessions. The first session characterizes the enterprise: product families, production routes, critical machines, recurring disturbances, available records and strategic objectives. The second session scores the 30 questions. The owner or manager, production supervisor and a representative operator discuss each answer and attach evidence. When evidence is unavailable, the score is treated as provisional

and the missing record becomes an improvement need. The third session validates target levels, business relevance and complexity, reviews the automatically calculated priorities and assigns owners to accepted initiatives.

This separation reduces two common sources of bias. It prevents a modern isolated machine from raising the perceived maturity of the entire production system, and it prevents a low global score from concealing useful local capabilities. It also makes disagreement explicit: if assessors provide different ratings, they must compare evidence and record the agreed score. Future validation will quantify inter-rater agreement, but the facilitated evidence rule already improves auditability in the pilot version. Two representative downtime-management questions and their evidence criteria are provided in Table 3, while the complete assessment-to-roadmap workflow is summarized in Fig. 1.

TABLE 3. Example questions and evidence for downtime management

Question	Scoring evidence
Are downtime events recorded using standardized reason codes?	0: no record; 1: informal notes; 2: local spreadsheet/form; 3: systematic digital log; 4: integrated event capture and analysis
Are downtime causes reviewed and converted into actions?	0: never; 1: reactive discussion; 2: occasional review; 3: regular Pareto/KPI review; 4: closed-loop improvement with verified effects

3. Transparent Digital Implementation

The prototype is implemented in Google Sheets using nine linked worksheets: Input Data, Questionnaire, Maturity Assessment, Recommendation Rules, AI Prompt, AI Recommendations, Roadmap, Dashboard and Lists. Spreadsheet formulas calculate C_j , M , G_j and P_j , while conditional formatting visualizes capability gaps. This design allows users to inspect the calculation path and adapt target levels without a proprietary platform.

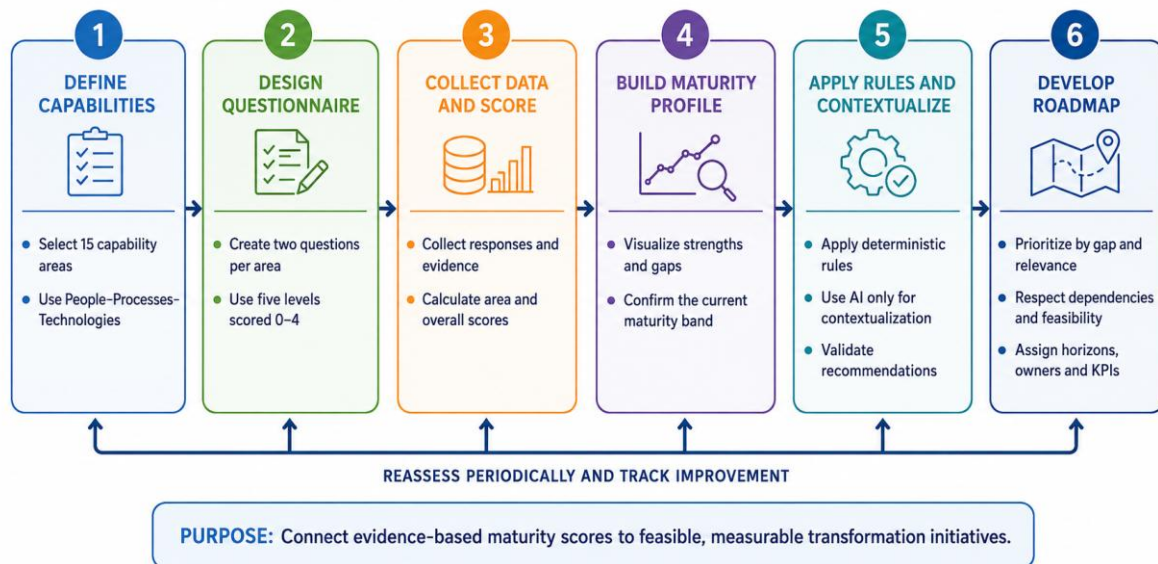


Fig. 1. Workflow of the assessment and roadmap method.

In this study, MES-light denotes a deliberately limited set of manufacturing operations functions suitable for a small enterprise: order and operation tracking, machine-status and downtime registration, basic quality and maintenance records, and KPI dashboards. It does not imply a full-scale MES with comprehensive ERP, warehouse, genealogy and enterprise-wide integration.

The rule layer provides the safety and consistency boundary. For example, a downtime-management score below 2 triggers downtime codes and weekly Pareto review; a data-collection score below 2 triggers machine-state, runtime, alarm and selected electrical-variable capture; low connectivity triggers a gateway and

a common communication interface; and missing MES capability triggers order and operation tracking through MES-light functionality. If analytics is absent, rules first require reliable data archiving and KPI dashboards instead of directly recommending advanced AI. Representative conditions, rule-based initiatives and verification indicators are listed in Table 4.

Generative AI is an optional interpretation layer, not a scoring engine. The structured prompt contains capability ID, dimension, current and target scores, gap, business relevance, complexity, enterprise context and constraints. The permitted output fields are initiative, horizon, KPI, resources, dependencies, expected effect

and status. Rule-selected actions constrain the request, and a manager or engineer validates every output against equipment, budget, safety and workforce constraints before roadmap inclusion. No confidential operational data should be transferred to an external model without organizational authorization and appropriate data-protection controls.

TABLE 4. Examples of deterministic recommendation rules

Condition	Rule-based initiative	Expected evidence/KPI
Production orders < 2	Introduce order and part identifiers; digital production cards	Traceability coverage
Downtime management < 2	Define downtime codes and weekly top-cause review	Unplanned downtime –15%
Data collection < 2	Capture runtime, status, alarms and selected machine variables	Data from all identified critical machines
Connectivity < 2	Install edge/gateway connectivity using a common interface	Connected critical assets
Analytics = 0	Build data history and basic OEE/downtime/quality dashboards	Regular KPI review

The hybrid workflow preserves a clear division of responsibility. Spreadsheet formulas produce the numerical profile; deterministic rules select a bounded set of admissible actions; the generative model reformulates these actions for the enterprise context; and a human decision-maker approves, edits or rejects the output. For downtime management, for example, the prompt may contain $C_j = 0.5$, $T_j = 2.0$, high business relevance, low complexity, a wide-belt sander and pellet press, no MES and a six-month horizon. A valid output proposes downtime categories, operator entry on an HMI or simple form, a weekly top-three-cause review and a target such as a 15% reduction in unplanned downtime.

The requested AI response is structured rather than free-form. Mandatory fields are initiative, rationale, prerequisite, owner, resources, time horizon, KPI, expected effect and verification evidence. The model is instructed not to modify scores, invent available equipment or bypass dependency rules. Outputs that recommend prediction without data history, autonomous control without safety analysis, or investments outside the declared budget are rejected. This arrangement uses language generation for contextualization while keeping prioritization reproducible and controllable. Recent research in the furniture sector confirms the growing relevance of AI while also emphasizing the need for appropriate data and organizational readiness [11].

4. Illustrative Application and Results

The model was applied to an illustrative case representing a small furniture/wood-processing enterprise with approximately 8–10 employees, a wide-belt sanding machine and a pellet press. Automation is partial, ERP support is absent or fragmented, no MES is installed, and the principal problems are downtime, weak order traceability and lack of centralized production data. The application demonstrates calculation and prioritization; it is not presented as statistical validation.

The overall index was 1.2/4, corresponding to the connected range. Dimension averages were 1.7 for People, 1.1 for Processes and 0.8 for Technologies. Operator skills (3.0) and machine automation (2.5) provide a useful foundation. MES/operations systems and analytics/prediction scored 0, while production orders, downtime management and connectivity scored 0.5. The profile therefore indicates that the principal constraint is not only equipment age but the absence of systematic event registration, production traceability and data exchange. The complete current-to-target capability profile is visualized in Fig. 2, and the aggregated assessment results are summarized in Table 5.

TABLE 5. Summary of the illustrative assessment

Indicator	Score	Interpretation
Overall maturity	1.2/4	Connected: first links among processes, roles and production data
People	1.7/4	Strongest dimension; skills available, improvement culture weak
Processes	1.1/4	Orders, downtime and maintenance insufficiently traceable
Technologies	0.8/4	Data, connectivity, MES and analytics are major gaps

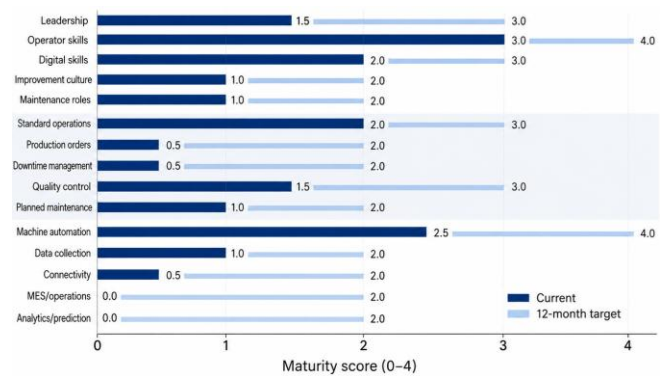


Fig. 2. Current and 12-month target scores for all 15 capability areas.

The 15-area profile reveals an uneven transformation baseline. Within People, operator skills are already systematic, digital skills are at a basic level, and leadership, improvement culture and maintenance responsibilities require formalization. Within Processes, standard operations provide a partial foundation, but order identifiers, downtime causes and planned maintenance records are incomplete. Within Technologies, local automation exists, yet the machines are not sufficiently connected and no operational information layer consolidates their states, alarms, orders and performance indicators. The current and 12-month target scores for all capability areas are detailed in Table 6.

TABLE 6. Capability scores and 12-month targets

ID	Capability	Current	Target
P1	Leadership and strategy	1.5	3.0
P2	Operator skills	3.0	4.0
P3	Digital skills	2.0	3.0
P4	Improvement culture	1.0	2.0
P5	Maintenance roles	1.0	2.0
PR1	Standard operations	2.0	3.0
PR2	Production orders	0.5	2.0
PR3	Downtime management	0.5	2.0
PR4	Quality control	1.5	3.0
PR5	Planned maintenance	1.0	2.0
T1	Machine automation	2.5	4.0
T2	Data collection	1.0	2.0
T3	Connectivity	0.5	2.0
T4	MES/operations systems	0.0	2.0
T5	Analytics and prediction	0.0	2.0

The targets deliberately do not require maximum maturity in every area. The first 12-month cycle aims at repeatable records, visible production status and basic operational coordination. Analytics and prediction have a nominal target of 2 only after the prerequisite data-collection and connectivity actions are completed. This dependency-aware interpretation is more informative than ranking areas exclusively by score. Representative calculations showing how the gap, business relevance and complexity determine the priority index are presented in Table 7. A target score of 2 for analytics denotes a basic local capability—data history, KPI calculation and a bounded pilot—not validated predictive operation. The 12-month target vector therefore defines the first reassessment cycle, whereas the roadmap in Table 8 extends the transformation sequence to 36 months.

The values in Table 7 illustrate the rule application used in the pilot. They are workshop-assigned planning estimates, not universal constants. Dependencies are applied after numerical ranking; therefore, MES-light, analytics or optimization cannot precede the required data, connectivity and process foundations even when their numerical priority is high. Formally, let x_A , x_D and $x_C \in \{0,1\}$ denote selection of analytics, data-collection and connectivity initiatives, respectively. The precedence constraints are $x_A \leq x_D$ and $x_A \leq x_C$. The final implementation sequence is obtained by applying these constraints to the numerical ranking.

TABLE 7. Example priority calculations for leading gaps

Capability	G_j	B_j	K_j	P_j
Downtime management	1.5	3	1	4.50
Improvement culture	1.0	2	1	2.00
Maintenance roles	1.0	2	1	2.00
MES/operations systems	2.0	3	3	2.00
Production orders	1.5	2	2	1.50
Data collection	1.0	3	2	1.50
Connectivity	1.5	3	3	1.50

5. Roadmap and Discussion

The priority index placed downtime management first because its gap and business relevance are high while initial implementation is inexpensive. After precedence constraints were applied to the numerical ranking, the dependency-aware sequence continued with improvement culture, maintenance responsibilities, production-order tracking, data collection, connectivity and MES-light. This ordering avoids premature investment in predictive or optimization functions before reliable operational data exists. The corresponding staged initiatives, implementation horizons and measurable targets are presented in Table 8.

TABLE 8. Roadmap derived from the illustrative assessment

Horizon	Selected initiatives	Measurable targets
0–6 months	Downtime codes; order/part cards; maintenance roles and schedule; machine-data capture & archiving; gateway connectivity; initial MES-light tracking; basic dashboards	Unplanned downtime –15%; traceability >50%; data from all identified critical machines
6–18 months	12-month digital plan; HMI/data training; standardized operations; structured quality-event log; systematic KPI reviews	Stable weekly reviews; improved quality and schedule adherence
18–36 months	Competence matrix; retrofit of critical machines with PLC/ HMI/ VFD and protection; validated analytics and optimization	Expanded connectivity; predictive and optimization pilots

The application shows why an overall score alone is insufficient. A company can possess skilled operators and locally automated machines yet be unable to coordinate orders or leverage production data. Separate capability scores expose these asymmetries and connect them to proportional investments. Reassessment after each improvement cycle establishes a baseline-to-target comparison and turns the roadmap into a living management instrument.

The roadmap links maturity development with operational evidence. Downtime codes and weekly analysis should reduce the frequency and duration of recurrent stoppages. Production cards and consistent identifiers should increase the percentage of orders and parts that can be traced through processing. Machine connectivity should increase the proportion of critical assets that report status and alarms. MES-light functions should improve order-progress visibility and schedule adherence. Only after these indicators stabilize should the enterprise pilot forecasting, predictive maintenance or optimization-based scheduling. The percentages and coverage levels in Table 8 are planning targets; they have not yet been verified through a longitudinal post-implementation assessment.

Repeated assessment is essential. After each six- or twelve-month cycle, the same evidence criteria and formulas are applied, current values are compared with the baseline, and targets are revised. Improvement is accepted only when a higher maturity score is accompanied by implemented practices and observable evidence. This prevents maturity inflation caused by planned but incomplete investments.

The method has limitations. The example is illustrative; business relevance and complexity are partly expert-assigned, and the questionnaire has not yet been statistically validated. Inter-rater agreement, sensitivity to weights, repeatability and correlation with operational indicators—lead time, utilization, setup loss, downtime, energy use and rework—must be tested across additional enterprises. Generative AI outputs also require controlled prompts, structured outputs and human approval.

Further case studies should include enterprises of different sizes and equipment mixes. Content validity can be assessed by panels of automation, production management and furniture-sector experts; reliability can be investigated through repeated scoring and inter-rater statistics; and criterion validity can be explored by relating maturity changes to OEE, lead time, quality loss and specific energy consumption. These steps are required before the model can be used for external benchmarking.

6. Conclusion

A compact maturity assessment can provide a credible entry point to smart and intelligent manufacturing for resource-constrained furniture MSEs. The proposed method makes its structure and calculations explicit: 30 questions assess 15 capabilities on a 0–4 scale; current-to-target gaps are combined with business relevance and complexity; deterministic rules select feasible actions; and generative AI is limited to contextualized, human-validated explanations. The illustrative application produced an overall score of 1.2/4 and a prioritization consistent with the predefined rules: traceability, downtime management, data collection, connectivity and MES-light foundations precede advanced analytics. Future work will validate the instrument in multiple enterprises and link maturity improvement to operational and energy performance indicators.

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Ontology engineering for iso 50001-based industrial energy management systems

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Abstract—This paper presents an implemented prototype of the ontoEnMS ontology, which formalizes selected ISO 50001 energy-management knowledge and connects it with manufacturing and measurement contexts. A hybrid METHONTOLOGY/NeOn workflow is used to develop an OWL 2 DL TBox and an enterprise ABox. The model represents energy reviews, significant energy uses (SEU), energy baselines (EnB), energy performance indicators (EnPI), operational control and improvement actions. Alignment hooks connect ontoEnMS with an author-developed IEC 62264-based OntoMES model and SOSA/SSN without asserting unjustified class equivalence. The prototype is checked in Protégé 5.6.4 through Hermit/Pellet consistency reasoning and four competency questions implemented as SPARQL query patterns. An industrial mining-and-processing ABox links production units, equipment, electricity meters, observations and area-level EnPI values. For area K2-B, 18.40 kWh/t is compared with a 17.92 kWh/t baseline, identifying a 0.48 kWh/t (2.68%) adverse deviation. SHACL completeness constraints are specified as the next closed-world validation layer rather than reported as executed tests.

KEYWORDS: ISO 50001, ONTOLOGY ENGINEERING, ENMS, OWL 2 DL, TBOX, ABOX, KNOWLEDGE GRAPH, SOSA/SSN, IEC 62264

1. Introduction and Related Work

Industrial Energy Management Systems (EnMS) integrate measurements from meters and power-quality analysers with PLC/SCADA, MES, ERP and IIoT data. ISO 50001:2018 defines the management framework for continual improvement, while ISO 50006 guides EnPI and EnB selection [1], [2]. Neither standard supplies a machine-interpretable domain model. Consequently, identical terms, units, periods and asset references are often represented differently across systems, limiting traceability and automated analysis.

Ontologies address this gap by defining explicit classes, relations and constraints. Previous work has demonstrated ontology-driven energy management in discrete manufacturing [6], while SOSA/SSN provides reusable semantics for sensors and observations [5]. The contributions are: (1) an ISO 50001-traceable OWL 2 DL TBox; (2) conservative alignment with an author-developed IEC 62264-based OntoMES model and SOSA/SSN; and (3) an industrial ABox evaluated through competency questions and an EnPI/EnB comparison. The approach also separates OWL consistency reasoning from closed-world data-quality validation.

Existing energy ontologies frequently focus on buildings, device descriptions or isolated consumption values. Manufacturing EnMS requires a broader context: the same measured value must be related to the organizational scope, production hierarchy, process segment, operating state, production quantity, relevant variable and reference period. The ontology-driven manufacturing approach in [6] confirms the value of semantic integration, but it does not provide a direct ISO 50001 traceability model for energy review, SEU, EnB, EnPI, targets and corrective actions. This gap motivates a dedicated

management ontology aligned with, rather than merged into, manufacturing and observation models.

Recent work further confirms the need for reusable industrial semantics. A systematic review found that manufacturing knowledge graphs are used mainly for knowledge fusion, while empirical industrial validation remains limited [12]. Energy-efficiency ontology research reports fragmented vocabularies and a need for reusable cross-domain models [13], and SemIoE demonstrates a lightweight reuse-based ontology for an industrial Internet-of-Everything knowledge graph [14]. Unlike these broader models, ontoEnMS focuses on traceability from ISO 50001 management concepts to EnPI/EnB, production hierarchy and measurement evidence.

The research questions are therefore: RQ1—how can the main ISO 50001 concepts and dependencies be represented in OWL 2 DL; RQ2—how can energy-management entities be connected to IEC 62264 equipment and SOSA/SSN observations without unsafe equivalence; and RQ3—whether a plant-specific ABox can answer operational competency questions and expose deviations from an energy baseline. These questions determine the ontology modules, alignment strategy and evaluation criteria used below.

2. Ontology Engineering Method

The method combines METHONTOLOGY specification, conceptualization, formalization and evaluation [7] with the reuse and alignment principles of NeOn [8]. It comprises six stages: domain analysis; TBox/ABox conceptualization; OWL 2 DL formalization; semantic alignment; validation; and reuse in knowledge-graph analytics. Figure 1 summarizes the workflow

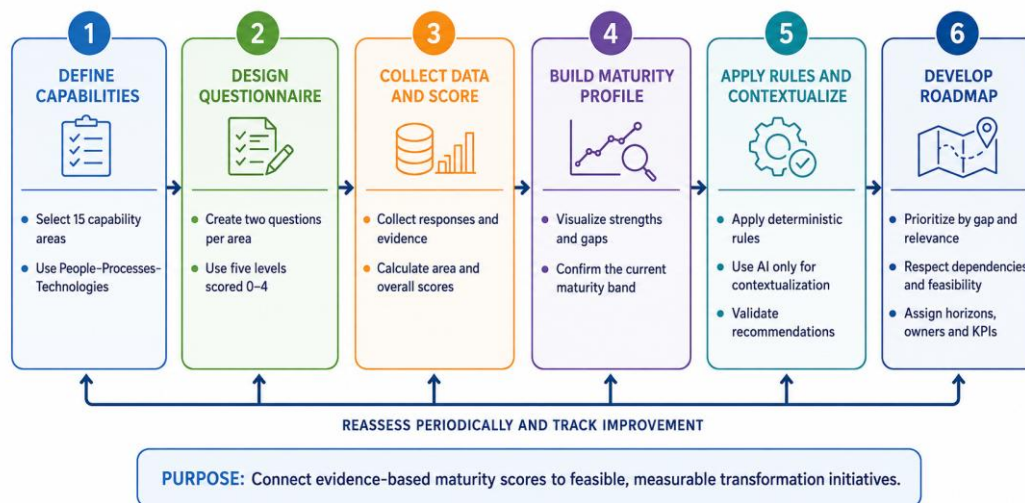


Fig. 1. Ontology engineering workflow for ontoEnMS

Domain terms were derived from ISO 50001, ISO 50006 and ISO 50015 [1]–[3]. The reusable TBox contains classes, object properties, datatype properties and axioms; the ABox stores plant-specific assets, observations, baselines and indicators. This separation allows one conceptual model to be reused by multiple plants without mixing schema and operational facts.

During specification, the scope was limited to knowledge needed for planning, operational control, monitoring, performance evaluation and improvement. Competency questions were defined before formalization, so that every major relation was justified by a need for retrieval or validation. Conceptualization produced class hierarchies and relation diagrams; formalization translated them into OWL 2 DL; alignment introduced explicit bridge properties; and validation combined logical, data-quality and query-based checks. This sequence reduces the risk of developing an expressive ontology that cannot answer the energy manager's practical questions.

3. TBox Formalization and Standards Traceability

Fig.2 presents a simplified ontology class diagram of the core TBox. The central class, EnergyManagementSystem, is connected to EnergyPolicy, EnergyReview, MonitoringMeasurementPlan, and EnergyObjective through the object properties hasPolicy, hasReview, hasPlan, and hasObjective, respectively. EnergyReview identifies EnergyUse and defines EnergyBaseline and EnergyPerformanceIndicator. SignificantEnergyUse is modeled as a subclass of EnergyUse and is linked to OperationalControl through controlledBy, while EnergyPerformanceIndicator is related to MeteringPoint through supportedBy. EnergyObjective is connected to EnergyTarget through hasTarget, and EnergyTarget is linked to EnergyActionPlan through hasPlan. Finally, CorrectiveAction is connected to ContinualImprovement through leadsTo. The diagram therefore summarizes the principal ontology classes and semantic relations supporting energy policy, review, performance monitoring, objective setting, operational control, and continual improvement.

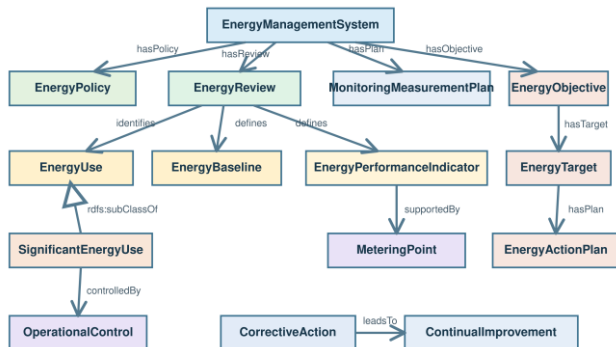


Fig. 2. Simplified ontology class diagram of the core TBox

Table 1 makes the standards-to-model traceability explicit. Object properties define relations such as hasEnergyPolicy, identifiesSEU, definesBaseline and comparedWithBaseline. Datatype properties represent baseline and EnPI values, units and periods using typed literals. Representative axioms are given in Listing 1.

TABLE 1. Traceability from standards to ontology elements

Standard concept	ontoEnMS representation	Validation/operation
Energy review	EnergyReview; identifiesEnergyUse; definesEnPI	OWL restriction; CQ
SEU	SignificantEnergyUse $\sqsubseteq$ EnergyUse; appliesToEquipment	Reasoner classification
EnB	EnergyBaseline; value, unit, reference period	SHACL completeness
EnPI	EnergyPerformanceIndicator; value, unit, reporting period	SPARQL comparison
Monitoring	Plan, metering point, SOSA observation	Trace to meter/asset

The existential restrictions support classification and consistency testing; they do not prove that an ABox record is complete under OWL's open-world assumption. Missing value, unit

or period assertions are therefore checked with SHACL shapes [9] or SPARQL validation queries. This distinction prevents absence of a statement from being interpreted as a logical contradiction.

The object-property model is organized into functional groups. Structural properties link EnergyManagementSystem to Organization, EnergyPolicy, EnergyManagementTeam, ManagementReview, EnergyReview and MonitoringMeasurementPlan. Planning properties connect EnergyReview with EnergyUse, SEU, EnB, EnPI, RelevantVariable, StaticFactor and ImprovementOpportunity. Evaluation properties connect EnergyPerformanceEvaluation with the evaluated EnPI, a generated result and BaselineComparison. Finally, improvement properties link Nonconformity, CorrectiveAction and ContinualImprovement. Explicit domain and range axioms are used only when they express stable semantics; integration hooks such as appliesToArea, appliesToEquipment and derivedFromObservation remain weakly constrained until the external ontology is imported.

```

SignificantEnergyUse  $\sqsubseteq$  EnergyUse
EnergyReview  $\sqsubseteq$ 
   $\exists$  identifiesEnergyUse.EnergyUse
EnergyReview  $\sqsubseteq$ 
   $\exists$  definesEnPI.EnergyPerformanceIndicator
ControlledSEU  $\equiv$  SignificantEnergyUse
 $\sqcap \exists$  hasOperationalControl.OperationalControl
EnergyUse  $\sqcap$  MeteringPoint  $\sqsubseteq \perp$ 
  
```

Listing 1. Representative OWL 2 DL axioms

The datatype profile distinguishes numerical values from their context. hasBaselineValue, hasEnPIValue, hasEnergyConsumptionValue, hasTargetValue, hasActualValue, hasDeviationValue and hasImprovementPercent use xsd:decimal. hasStartDate and hasEndDate use xsd:dateTime. Units and periods are currently represented by hasUnit, hasReportingPeriod and hasReferencePeriod; controlled QUDT unit resources are planned [10]. QuantifiedEnergyBaseline and QuantifiedEnPI are defined by intersections requiring a value, unit and period, while proposed SHACL shapes express the corresponding closed-world cardinality and datatype checks.

Cardinality constraints are applied selectively. An EnergyManagementSystem is expected to have an energy policy and at least one energy review; an EnergyReview identifies energy uses and defines at least one EnPI; and a complete performance evaluation refers to an EnPI, produces a result and includes a baseline comparison. Excessively restrictive global cardinalities were avoided because industrial data are often populated incrementally. Required ABox fields are placed in SHACL shapes, where violation severity and actionable messages can be reported without making the ontology logically inconsistent during ingestion.

Disjointness axioms separate management activities, physical devices, information objects and energy-use concepts. For example, EnergyUse is disjoint with MeteringPoint, while EnergyBaseline and EnergyPerformanceIndicator remain distinct even when they share a numerical value and unit. This prevents a reasoner from collapsing conceptually different entities and enables provenance to be attached separately to the baseline definition, current calculation and comparison result.

4. Alignment and Enterprise ABox

The integration model keeps three independent TBoxes—iec62264_tbox.owl, iso50001_tbox.owl and sosa_ssn.owl—and connects them through nfme_energy_abox.owl. OntoMES is an author-developed OWL prototype that formalizes selected IEC 62264 production-hierarchy concepts—Enterprise, Site, Area, ProductionUnit and Equipment [4], [11]. SOSA/SSN represents Sensor, Observation, ObservableProperty and Result [5]. Alignment uses subclass and linking properties where concepts overlap only partially; owl:equivalentClass is reserved for demonstrably identical semantics.

TABLE 2. Semantic alignment patterns

ontoEnMS entity	Related external entity	Relation/purpose
SEU	Equipment / ProcessSegment	appliesToEquipment / appliesToProcessSegment
MeteringPoint	sosa:Sensor	hasMeter; point is not the device
EnergyMeasurement	sosa:Observation	rdfs:subClassOf sosa:Observation
EnPI	Observation/result set	derivedFromObservation; calculated indicator

The enterprise ABox represents NFME, sites K1 and K2, areas K2-A/K2-B/K2-C, production units MFC1/MFC2, mills M1–M5, flotation rows, blowers, meters PF-P301÷PF-P317 and transformers Tr_1÷Tr_36. PF_P301 is the physical ElectricityMeter and is typed as sosa:Sensor; the separate individual MP_P301 represents the logical MeteringPoint and is linked to the device by hasMeter. PF_P301 measures mill M1 and produces timestamped sosa:Observation individuals. Thus, an observation can be traversed through its sensor and metering point to equipment, production unit, area, SEU and energy review without conflating a measurement location with a device.

ISO 50001 individuals are added per area. The numerical values are anonymized representative operational values derived from the dissertation's industrial case study; they support ontology population and query evaluation and are not presented as a complete audited dataset. Baseline_K2_B_2026 has value 17.92 kWh/t and applies to K2-B; EnPI_K2_B_2026_04_16 has value 18.40 kWh/t. BaselineComparison links both values. The adverse deviation is $18.40 - 17.92 = 0.48$ kWh/t, or 2.68% relative to the baseline. K2-B milling and flotation are instantiated as an SEU that includes MFC1 and MFC2. The result is an auditable path from the indicator to production assets and observations rather than an isolated numerical record.

TABLE 3. Area-level energy baselines in the industrial ABox

Area	Process	EnB	Unit
K2-B	Ore processing	17.92	kWh/t
K2-A	Ore crushing	2.59	kWh/t
K2-C	Tailings/water system	1.77	kWh/t
K1-A	Mining	1.38	kWh/m ³
K1-B	Primary crushing	0.74	plant-defined unit
K1-C	Ore transport	0.30	plant-defined unit

Table 3 summarizes the baselines instantiated from the enterprise energy review. Area identifiers retain the Latin suffixes B and C used in the source case study. Each EnPI is calculated as the electricity consumed by an area divided by the corresponding production quantity: $EnPI = E/G$. Separate indicators are required because the production denominators differ. K2-A, K2-B and K2-C use tonnes of processed ore, while K1-A uses cubic metres of mined mass. Before automated cross-area comparison, the units for K1-B and K1-C must be confirmed in the source EnMS; the ontology intentionally preserves these as data-quality issues rather than silently assigning a unit.

At the equipment level, the shared ABox represents the chain NFME → K2 → K2_B → MFC1 → M1. The meter PF_P301 observes ElectricityConsumption_M1, and an observation stores 1250.4 as xsd:decimal with result time 2026-04-16T08:00:00+03:00. Electricity-supply relations add transformer and 6-kV switchgear context. This permits queries by production hierarchy, measurement chain or electrical feeder and avoids duplicating equipment definitions inside ontoEnMS.

The production hierarchy also preserves different aggregation levels. NFME is typed as Enterprise; K1 and K2 as Site; K2-A, K2-B and K2-C as Area; and MFC1/MFC2 as ProductionUnit. MFC1 contains mills M1–M5, flotation rows FR1, FR2, FR4 and FR7, and blowers VD1, VD2 and VD4. Equipment-to-meter and suppliedBy relations enable an EnPI deviation to be investigated first by production unit and subsequently by individual asset or electrical supply section. This graph structure is particularly useful when one

meter supplies several assets or when an area indicator aggregates multiple observations.

The ABox population pattern is deliberately modular. The TBoxes contain no enterprise individuals, whereas nfme_energy_abox.owl imports the three domain models and holds only individuals, assertions, observations, baselines and indicators. New plants can create their own ABox without copying the class definitions. Likewise, changes in a sensor identifier or production hierarchy do not require modification of the ISO 50001 TBox. This division supports version management and preserves provenance across model updates.

5. Validation and Industrial Evaluation

The prototype was evaluated across four categories—syntactic, logical, semantic, and query-based—while SHACL closed-world validation was specified but not executed. The RDF/OWL model and namespace declarations were inspected in Protégé 5.6.4. HermiT and Pellet were used locally for consistency, class-satisfiability, disjointness and inferred-classification checks; the ontology remained consistent and the tested named classes were satisfiable. Semantic evaluation examined terminology correspondence, class separation, relation direction, domain/range compatibility and traceability to the selected ISO 50001 concepts. Four SPARQL query patterns were executed against the populated NFME ABox: CQ1 identifies an SEU; CQ2 retrieves an EnPI linked to equipment; CQ3 identifies metering devices and observations for a production area; and CQ4 retrieves EnPI values exceeding their EnB. Cross-domain traversal forms part of the query-based interoperability check. The paper reports the retrieved individuals, but exact ontology metrics, reasoner logs and a public repository remain future reproducibility deliverables.

```
# enms: denotes the local prototype namespace
SELECT ?enpi ?value ?baseline ?baseValue ?area
?unit
WHERE {
  ?enpi a enms:EnergyPerformanceIndicator ;
    enms:hasEnPIValue ?value ;
    enms:appliesToArea ?area ; enms:hasUnit ?unit ;
    enms:comparedWithBaseline ?baseline .
  ?baseline enms:hasBaselineValue ?baseValue ;
    enms:appliesToArea ?area ; enms:hasUnit ?unit .
  FILTER(?value > ?baseValue)
}
```

Listing 2. Simplified CQ4 query pattern for EnPI-over-baseline detection

CQ4 returns the K2-B indicator because 18.40 kWh/t exceeds 17.92 kWh/t for the same area and unit. It demonstrates a direct, non-normalized comparison; production deployment additionally requires temporal applicability and relevant-variable checks. The displayed query is abbreviated for presentation: the prefix declaration is omitted because the current prototype uses a local namespace that must be replaced by a persistent IRI before publication. A production implementation should also verify compatible reporting/reference contexts and use QUDT unit identifiers rather than free-text unit strings [10]. Proposed SHACL shapes require every EnergyBaseline to have a numeric value, unit and reference period, and every observation to have a sensor, observed property, result and timestamp. These constraints complement, rather than replace, OWL reasoning.

TABLE 4. Validation results and planned checks

Check	Mechanism	Result / status
Syntax	Protégé 5.6.4	Valid RDF/OWL and namespaces - passed
Logic	HermiT; Pellet	Consistent; tested named classes satisfiable — passed
CQ1	SPARQL pattern	Expected SEU individual retrieved
CQ2	SPARQL pattern	Expected EnPI/equipment context retrieved
CQ3	Graph/SPARQL	MP_P301, PF_P301 and observation retrieved
CQ4	SPARQL pattern	Expected K2-B comparison returned
Data quality	SHACL shapes	Specified; execution report is future work

Table 4 distinguishes executed checks from planned validation. Hermit and Pellet can infer that every SignificantEnergyUse is an EnergyUse and detect an individual asserted simultaneously as disjoint EnergyUse and MeteringPoint. They cannot, by the absence of a triple alone, conclude that an EnergyReview record is incomplete. Conversely, SHACL can report a missing unit or timestamp after the shapes are executed, but it does not perform OWL class classification. Competency questions assess whether the combined schema and ABox answer domain needs, while graph traversal demonstrates cross-model traceability.

Execution of the four query patterns against the populated NFME ABox produced the expected individuals. CQ1 returned SEU_K2_B_MillingFlotation and its associated area and production units. CQ2 followed the SEU/equipment relations to retrieve the K2-B EnPI and MFC1/MFC2 context. CQ3 traversed from K2-B and M1 through MP_P301 to the physical meter PF_P301 and its SOSA observation. CQ4 returned EnPI_K2_B_2026_04_16 because 18.40 kWh/t is higher than the 17.92 kWh/t baseline. These locally obtained results support management-level and equipment-level navigation; independent repetition requires publication of the ontology, ABox and complete query files.

For a robust CQ4 implementation, value comparison is performed only after verifying unit compatibility and matching the indicator scope with the baseline scope. The reporting period and reference period are not assumed to be identical: the former identifies the current assessment interval, while the latter records the interval from which the baseline was established. Relevant variables and static factors can be attached to both the baseline and evaluation context, preparing the model for normalized EnPI calculations required when production load or operating conditions change materially.

Data-quality validation targets the ingestion boundary. A baseline shape requires exactly one decimal value, at least one unit and one reference period. An EnPI shape requires a decimal value, unit, reporting period and link to its applicable area. An observation shape requires madeBySensor, observedProperty, hasSimpleResult and resultTime. A comparison shape verifies links to both EnPI and EnB and stores the calculated deviation. Violations are reported as incomplete operational records rather than logical contradictions, so acquisition can continue while corrective data-governance actions are initiated.

The evaluation demonstrates functional coverage and traceability, but it is not yet a scalability benchmark. The current ABox is a representative industrial model; reasoning time, streaming update latency and large-volume observation storage remain future evaluation tasks. High-frequency observations should remain in a time-series store, while the knowledge graph maintains selected observations, context and provenance.

The present evaluation also has three boundary conditions. First, ontoEnMS formalizes selected ISO concepts and relations; it does not certify organizational conformity with ISO 50001. Second, the case values illustrate ontology population and query behaviour and are not a complete energy audit. Third, persistent ontology IRIs, a public repository, published model metrics and an executable SHACL report are required for independent reproduction. These limitations define concrete deliverables for the next implementation cycle.

Versioning is particularly important for EnB and EnPI. A baseline is not only a number; it has an approval state, reference period, applicability scope, unit, relevant variables and possible adjustment history. When equipment is replaced or the production structure changes, a new baseline individual should be created instead of overwriting the previous one. The same principle applies to indicator definitions. Linking every calculated EnPI to the version of its definition and baseline makes historical comparisons reproducible and supports an auditor in establishing why a result was accepted at a specific time.

Provenance should distinguish measured, calculated, asserted and inferred statements. Sensor observations originate from a

metering device; production quantities may originate from MES or laboratory records; EnPI values are calculated from identified inputs; and reasoner classifications follow from ontology axioms. Source system, import time and transformation rule can therefore be retained as provenance metadata when the ABox is updated.

The conservative alignment strategy improves maintainability. Declaring MeteringPoint equivalent to `sosa:Sensor` would incorrectly identify a logical measurement location with a physical or virtual sensing device. Similarly, an EnPI is normally a calculated indicator and not simply an `ObservableProperty`, while an SEU may correspond to equipment, a process, an installation or a combination of assets. Bridge properties preserve these distinctions and still permit integrated queries. Strong equivalence can be introduced later for particular subclasses only when necessary and sufficient semantic correspondence has been established.

Taken together, the case study answers the three research questions. RQ1 is addressed by the OWL 2 DL TBox and the standards-traceability mapping. RQ2 is addressed by modular imports and bridge properties connecting IEC 62264 and SOSA/SSN contexts without broad equivalence assertions. RQ3 is addressed by the enterprise ABox, competency queries and the K2-B baseline comparison. The presented results provide preliminary evidence of semantic feasibility and functional usefulness, while independent reproduction, quantitative scalability and live-system integration remain subjects for subsequent work.

6. Conclusion

The ontoEnMS prototype transforms selected ISO 50001 concepts into a reusable OWL 2 DL schema and connects them to enterprise facts through a shared ABox. Its scientific contribution is the explicit standards traceability, conservative cross-ontology alignment and separation of open-world reasoning from closed-world data validation. The NFME case shows how EnPI/EnB comparison can be traced to production areas, equipment, meters and observations. Future work will publish the ontology and SHACL profile with persistent IRIs, report exact class/property/axiom/ABox metrics, execute and publish the SHACL validation report, evaluate query and reasoning performance, replace textual units with QUDT resources, and connect the model to live OPC UA data and hybrid AI modules.

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Operational methods for assessing the mechanical and filtering properties of permeable materials based on metal powders

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Abstract: This article presents information about methods for evaluating the permeable materials based on metal powders mechanical and filtration properties. It describes methods for determining the permeability coefficient and local permeability coefficient, filtration fineness, and shear strength of powder materials, assessing the sintering quality of composite permeable materials containing fibers, and testing for strength under operating pressure differentials (up to 1 atm). The role of 3D-modeling in improving the efficiency of research is highlighted, enabling the rapid development and manufacture of fixtures for hermetically sealed specimens as their shape and size change.

Keywords: RESEARCH, PERMEABLE MATERIALS, METAL POWDERS, PERMEABILITY COEFFICIENTS, LOCAL PERMEABILITY COEFFICIENT

1. Introduction

Permeable materials based on metal powders have found widespread use in various fields of technology to solve a variety of problems, including the purification of liquids and gases from impurities, flame suppression, noise reduction, dispersion of liquid and gas flows, capillary transport in heat exchange devices, implantology, intensification of chemical processes, and other purposes [1-3]. The most frequently used materials in practice are those classified [4] as filtering materials. The operation of this group is characterized by the presence of excess pressure applied to the gas or liquid, as a result of which the latter, passing through pore channels, are purified, homogenized, separated, mixed, and distributed over the filtration surface. The creation and development of powder filter materials (PFM), like any permeable materials, is impossible without studying their properties [5]. There are a fairly large number of parameters, the combination of which provides a complete understanding of the properties of a porous material [6-8]. However, this is a rather labor-intensive and time-consuming process. At the same time, a preliminary, rapid, and high-quality results assessment of a given operation allows us to reduce research time, clarify its direction, and determine the feasibility of further investigation. This paper presents information on the authors' operational methods for assessing the mechanical and filtration properties of permeable materials based on metal powders.

2. Materials and research methods

The key characteristics of PFMs, reflecting their effectiveness, include permeability, pore size, filtration fineness, and uniformity of property distribution across the working surface (filtration area). Furthermore, since PFM products are subject to certain mechanical loads during operation, they must possess sufficient mechanical strength. When a structure is subjected to loads, its strength is assessed based on the ultimate limit state of the materials within the structural elements; analyzing the strength of a structure requires the use of strength criteria to assess the materials under load resistance [9]. The authors chose the shear strength limit (σ_{middle}) as such a strength criterion, which has become widely used in the practice of powder metallurgy [2, 10 – 13] and can be determined directly on the same samples that were used to study other properties.

Permeability – the property of a porous material to allow liquid or gas to pass through itself under excess pressure. It is characterized by a permeability coefficient, which for laminar flow, in accordance with Darcy's law, has the form (1) [1.4]:

$$k = \frac{Qh\mu}{S\Delta p} \quad (1)$$

where Q is the air flow rate per unit time through the sample, m³/s; h is the thickness of the sample in the direction of air

flow, m; μ – is the coefficient of dynamic viscosity of air, Pa · s; S – filtration area, m²; Δp – pressure differential on the sample, Pa.

The schematic setup for determining permeability is shown in Figure 1.

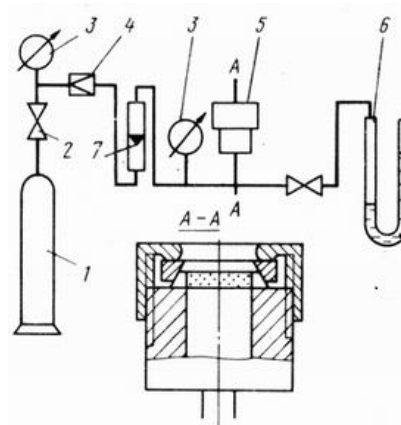


Fig. 1. Schematic diagram of the permeability determination setup: excess pressure source (1); tap (2); pressure gauge (3); reducer (4); cartridge with sample (5); U-shaped pressure gauge (6); flow meter (7).

The design of the installation is simple and easy to maintain, and the measurement method is simple and efficient. The laminar flow regime of the filtered medium was chosen for several reasons: it minimizes pressure losses during the movement of the liquid or gas flow [14], prevents the possibility of detachment of retained particles (during direct filtration) by vortex forces [15], and facilitates the filtration process parameters calculation [16].

As a rule, earlier studies were carried out on samples in the form of disks with a diameter of 30 and a thickness of 3–5 mm; in recent years, in connection with the commissioning of the POROLUXTM 500 porometer (manufacturer IB-FT GmbH, Berlin, Germany), a sample with a diameter of 25 mm has been adopted as the second typical sample. The POROLUXTM 500 porometer (Figure 2) operates on the principle of gas-liquid phase displacement and is designed for rapid measurement of through pores in materials such as textiles, paper, hollow fiber membranes, non-woven materials, etc., while it also allows for rapid measurement of the gas permeability coefficient in the range from 0.01 to 50 Darcy (from 1×10^{-14} to 50×10^{-12} m²) [17]. As follows from the above, samples in the form of disks with a diameter of 25 and a thickness of 3–5 mm are used for measurements in the device. The advantage of the device is its automation, that is, there is no need for direct human participation in the measurement process.

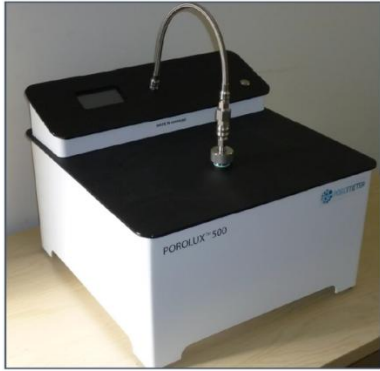


Fig.2. Porometer POROLUX™ 500.

Pore sizes. Pore sizes, along with permeability, are among the most important characteristics of filter materials. Maximum pore sizes determine the maximum size of pollutant particles that can pass through the material, i.e. they are used to evaluate the filtration fineness, while average pore sizes are used to compare different permeable materials, conduct hydrodynamic calculations, and also to evaluate the filtration fineness [2,4,18]. The simplest and most common method for quickly determining pore sizes is the liquid displacement method by gas [19]; the setup and equipment shown in Figure 1 are used for measurements. The method is based on measuring the pressure required for gas bubbles to pass through the pores of a porous sample filled with liquid with a known surface tension. When modeling a porous medium using cylindrical capillaries and in the case of complete wetting, the capillary (pore) diameter is determined from expression (2):

$$d = \frac{4\sigma}{\Delta P}, \quad (2)$$

where σ – surface tension of the wetting liquid, N/m; ΔP – pressure drop across the sample, Pa.

The appearance of the first three chains of gas bubbles above the sample surface corresponds to the opening of maximum-sized pores, while intense gas bubble release across the entire sample surface corresponds to the opening of medium-sized pores. Clearly, in the latter case, the accuracy of the measurement is affected by the skill of the researcher. Another method disadvantage is the limited size of the pores being measured, with a measurement range of 1–250 μm . The lower limit is limited by the required gas pressure (3–4 MPa), while the upper limit is limited by the inability to fill the pores with liquid (capillary forces no longer balance the gravity of the liquid) [4].

For more accurate data, the POROLUX™ 500 porometer is used. The device has an operating pressure in the range from 0 to 34.5 bar (3.447 MPa) and allows measuring pores with sizes from 0.013 to 500 μm [17].

Filtration fineness is the primary filter characteristic, regulating the efficiency of separating pollutant particles of a given size from the purified gas or liquid stream. According to [20], a distinction is made between absolute filtration fineness (corresponding to the maximum size of spherical artificial pollutant particles detected in the filtrate) and nominal filtration fineness (corresponding to the minimum size of pollutant particles detected in the filtered liquid for which the obtained filtration coefficient is not lower than the specified value). With a certain degree of error, the filtration fineness of a PFM can be estimated based on pore size measurements. Thus, in [4, 21] the pore sizes values and the corresponding nominal fineness of PFM purification are presented with data on the characteristics of the original powders and the process material production parameters, which is the basis for predicting the efficiency of the latter. For a more accurate operational assessment of the filtration fineness of PFM, a suspension with a reference

pollutant is passed through the sample under study and the sizes of solid particles in the original suspension and filtrate are analyzed. The obtained results allow us to evaluate the main filtration characteristics, including the "beta-ratio" coefficient (β_x), which is widespread abroad, which is defined as the ratio of the number of particles of a certain size present in the unfiltered (original) liquid to the number of particles of the same size in the filtered liquid (filtrate) [22]. The particle size distribution curve of the pollutant for both suspensions was obtained using a Mastersizer 2000 laser diffraction analyzer of particle size distribution with a HydroS automatic dispersion and sample feed module (Malvern Instruments Ltd., UK). An example of a device for testing filtration fineness in laboratory conditions, assembled in-house, is shown in Figure 3, and a photo of the Mastersizer 2000 device is – in Figure 4.



Fig. 3. Device for determining the filtration fineness of liquid during the filtration process.



Fig. 4. Mastersizer 2000 laser diffraction particle size analyzer with HydroS automatic sample dispersion and feed module (Malvern Instruments Ltd., UK).

It should be noted that in order to obtain the most reliable test results, the test conditions should be as close as possible to the operating conditions (operating viscosity, temperature and pressure of the area, flow conditions, filter material thickness).

Uniform properties distribution over the working surface (filtration area). The simplest and most efficient methods for assessing this characteristic include the maximum pore size ratio to the average pore size, used in [1, 6, 23, 24], which is designated as the homogeneity index in [24]. The closer the value of this index to 1, the better the uniformity of the properties distribution (homogeneity) of the porous material under study. A more informative method for assessing the distribution of properties over the working surface of the PFM is a method based on measuring local gas flows passing through the sample at various points using a measuring head, followed by the calculation of local permeability coefficients [25]. The device used in this case is distinguished by its simplicity of execution and can be used in both laboratory and industrial conditions. As an example, information on the study of local permeability of three samples made from different fractions of tin-phosphorus bronze powder grade BrO10F1 is presented below: Figure 5 shows the measuring local permeability process [25], Figure 6 shows the measurement diagram, and Table 1 summarizes the results of calculating the permeability coefficients based on the measured local flow rates.



Fig. 4. Measurement process.

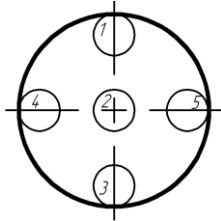


Fig. 5. Local permeability measurement scheme.

Table 1: Results of local permeability studies.

Research results	Powder particle size fraction, mm		
	(minus 0.315 + 0.20)	(minus 0.2 + 0.1)	(minus 0.1 + 0.063)
Local permeability coefficient, $\times 1013, m^2$, Point 1	179,9	137,1	63,8
Local permeability coefficient, $\times 1013, m^2$, Point 2	162,4	156,8	71,6
Local permeability coefficient, $\times 1013, m^2$, Point 3	175,2	150,5	66,8
Local permeability coefficient, $\times 1013, m^2$, Point 4	179,9	141,3	65,2
Local permeability coefficient, $\times 1013, m^2$, Point 5	175,2	151,6	66,0
Average local permeability coefficient	174,5	147,7	66,6
Standard deviation, σ	6,43	7,01	1,67
Variation coefficient, δ	0,04	0,05	0,03
Permeability coefficient, $\times 1013, m^2$	170,02	140,00	62,45

The presented results demonstrate that the permeability of the entire PFM can be estimated using the average value of local permeability coefficients. The greatest deviation of the average value of the local permeability coefficient from the sample permeability coefficient is less than 6.5%. A direct comparison of the uniformity of the distribution of properties of different PFMs is performed using the coefficient of variation.

Mechanical testing. As noted above, shear strength was chosen to evaluate the mechanical strength of the experimental specimens. The advantage of this characteristic is that it is determined not on reference specimens, but directly on the test specimens after evaluating the other properties. The shear test setup is shown in Figure 6.

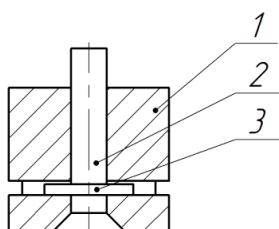


Fig. 6. Shear test diagram: matrix (1); punch (2); sample (3).

The shear strength was calculated using formula (3):

$$\sigma_{mid} = \frac{P}{\pi dh}, \quad (3)$$

where P is the shear force, N; d is the diameter of the cut surface, m; and h is the thickness of the test specimen, m.

A simple method for assessing the sintering quality of porous fiber materials has been developed [26]. Shear tests generally indicate the strength of the fiber material rather than the interparticle contacts; tensile tests are expensive, labor-intensive, and not justified when rapid and high-quality evaluation of the results of a particular operation is required. The apparatus design for implementing the developed method and the appearance of the test specimen are shown in Figure 7.

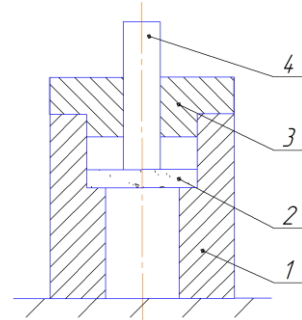


Fig. 7. A device for assessing the quality of sintering of fiber materials: matrix (1), sample (2), guide bushing (3), punch (4).

Distinctive features of the presented design include the implementation of a hole in the die with a variable diameter along its length and an excess of the hole diameter in the lower part over the punch diameter, which is used to apply force to the sample. The diameter of the hole in the upper part of the die is equal to the diameter of the sample, and its length is at least one and a half times the diameter of the sample, while in the lower part it is equal to 0.8 of the sample diameter. The ratio of the length of the upper part of the hole in the mold to the diameter of the sample allows for the installation of samples of a wide range of thicknesses and the absence of contact of the sample with the lower part of the guide sleeve during testing. The ratio of the diameters of the lower and upper parts of the hole ensures the maintenance of a stable sample position in the die during testing, and the excess of the hole diameter in the lower part over the diameter of the punch ensures the localization of areas of plastic deformation and subsequent failure in the zones of interparticle contacts (it should be noted that in this work the authors did not aim to analyze the stress-strain material state). To illustrate the effectiveness of the test method, Figure 8 shows photographs of the test results for three samples: two made of copper fiber, the sintering process of which was activated by fine copper powder particles, and, for comparison, a tin-phosphorus bronze sample sintered under optimal conditions. As the photographs show, the tests allow for a rapid assessment of the sintering quality of fiber materials.

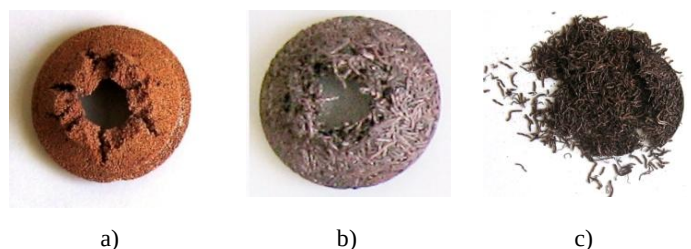


Fig. 8. Samples appearance after mechanical testing: from tin-phosphorus bronze powder (a), from powder-fiber material sintered at a temperature of 1050 °C (b) and from powder-fiber material sintered at a temperature of 950 °C (c).

To test the strength filter elements subject to low pressure differentials (up to 1 bar), a simple method was developed based on creating a vacuum in the sample cavity and sealing its outer surface with a polymer film. The purpose of the tests was not to determine the burst

pressure, but to quickly verify the filter element's ability to withstand a given pressure differential. The testing process is shown in Figure 9.



Fig. 9. Testing the filter element for its ability to withstand a given pressure drop (up to 1 bar).

It's worth noting that additive technologies make a significant contribution to increasing the efficiency of research: equipment for mounting samples when determining pore sizes and strength tests for the ability to withstand a given pressure drop, and a measuring head for measuring local flow rates were designed and manufactured using 3D-modeling and 3D-printing.

3. Conclusion

This article presents information on methods for evaluating the mechanical and filtration properties of permeable materials based on metal powders. It describes methods for determining the permeability coefficient and local permeability coefficient, pore size, filtration fineness, and shear strength of powder materials, assessing the sintering quality of composite permeable materials containing fibers, and testing for strength under operating pressure differentials (up to 1 atm). The role of 3D-modeling in improving the efficiency of research is highlighted, enabling the rapid development and manufacture of fixtures for hermetically sealed specimens as their shape and size change.

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On the structural characterization of thermo-reactive diffusion coated cast Ti6Al4V alloy

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Abstract: Thermo-reactive diffusion (TRD) coatings are formed on conventionally cast Ti6Al4V alloy using the pack cementation process to improve its surface characteristics. Aluminizing, siliconizing, and molybdenizing treatments are carried out at 700 °C, 1000 °C, and 900 °C, respectively, for 2 h using Al, Si, and Mo powder packs containing NH_4Cl as the activator and Al_2O_3 as the inert filler. The resulting coatings are systematically characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive spectroscopy (EDS) to investigate phase evolution, coating morphology, and elemental distribution. XRD analyses confirmed the formation of Ti–Al intermetallics (TiAl , TiAl_2 , TiAl_3), Ti–Si silicides (TiSi , TiSi_2 , Ti_5Si_3), and Mo-enriched Ti solid-solution phases within the coatings. Cross-sectional SEM observations have revealed continuous and well-adhered diffusion layers with thicknesses of approximately 20 μm for the aluminized coating and 50 μm for the molybdenized coating, while the siliconized layer (40 μm) exhibits a graded multilayer silicide structure. EDS elemental mappings have demonstrated inward diffusion of the deposited species and outward diffusion of substrate elements, confirming diffusion-controlled coating growth. The formation of metallurgically bonded reaction layers with compositional gradients is expected to substantially enhance the surface hardness, wear resistance, and high-temperature performance of the Ti6Al4V alloy. The results demonstrate that thermo-reactive diffusion by pack cementation provides an effective and controllable route for tailoring the surface microstructure of cast Ti6Al4V through appropriate selection of coating chemistry and processing conditions.

KEYWORDS: Ti6Al4V ALLOY, COATING, INTERMETALLICS, CHARACTERIZATION.

1. Introduction

The demand for titanium-based alloys as metallic engineering materials is undeniable across a broad spectrum of applications, particularly in the aerospace [1], biomedical [2], and chemical industries [3]. Through alloying strategies [4] and thermomechanical processing routes [5], significant improvements can be achieved in the physical, chemical, and mechanical properties of titanium alloys [6]. Consequently, owing to their unique combination of low density, moderate-to-high strength and toughness, and excellent corrosion resistance, these alloys have emerged as a preferred material choice for the manufacture of numerous critical components where multiple material selection criteria must be simultaneously satisfied, particularly as an alternative to conventional ferrous-based materials [7]. Among titanium alloys, Ti6Al4V has emerged as one of the most widely utilized engineering materials due to its ability to accommodate both α and β phases with varying phase fractions and morphologies, particularly as a function of heat-treatment conditions [8]. Although the alloy is regarded as a strong candidate for numerous engineering applications owing to its favorable room-temperature mechanical properties, its utilization in high-temperature environments remains relatively limited [9]. The mechanical performance of this dual-phase alloy is highly attractive and can be tailored through various manufacturing routes, including casting, forging, powder metallurgy, and additive manufacturing, as well as through post-processing heat-treatment schedules [10]. Nevertheless, Ti6Al4V exhibits inherent limitations in terms of tribological performance [11]. In this context, contemporary surface engineering technologies have become a decisive factor in extending the applicability of the alloy by enhancing its oxidation resistance, hot-corrosion resistance, and wear performance under demanding service conditions [12].

Thermo-reactive diffusion coatings significantly enhance the surface properties of metallic materials by forming a metallurgically bonded layer that improves wear resistance, corrosion resistance, and high-temperature stability [13]. These coatings are produced through a diffusion process where alloying elements are introduced into the substrate surface, creating a graded composition that reduces the risk of delamination under mechanical or thermal stress [14]. Among various diffusion coating techniques, pack cementation is widely preferred due to its simplicity, cost-effectiveness, and ability to produce uniform, adherent coatings with controlled thickness [15]. It is well established that diffusion coatings produced via the pack cementation process are highly effective in enhancing the surface properties of titanium alloys [16]. Through the formation of metallurgically bonded surface layers,

pack cementation treatments can significantly improve the oxidation resistance [17], hot corrosion resistance [18], wear behavior [19], and high-temperature durability of titanium-based materials, thereby extending their service life in demanding operating environments [20].

In surface engineering applications based on the pack cementation process, the pack composition, processing temperature, and time are key parameters that directly govern the type of intermetallic phases formed on the surface, as well as the thickness, microstructural uniformity, and overall integrity of the coating layer [21]. As a result, the surface-specific physical, chemical, and mechanical properties may vary considerably depending on the selected processing conditions. Therefore, although a wide range of metallic materials, particularly titanium alloys, can be successfully coated through thermo-reactive diffusion treatments, each finding within the process–structure–property relationship is of critical importance, as it ultimately determines the in-service performance and reliability of the coated material in practical applications. In the present study, thermo-reactive diffusion conditions are established on the surface of Ti6Al4V alloy to facilitate the formation of aluminum-, silicon-, and molybdenum-based intermetallic coatings. Through comprehensive structural characterization, the relationships between processing parameters, coating microstructure, and resulting properties are systematically investigated. Particular emphasis is placed on elucidating the mechanisms and process-structure-property interactions that contribute to the enhancement of the substrate material's performance in engineering applications.

2. Experimental procedures

Commercial cast Ti6Al4V alloy is employed as the substrate material. Prior to the coating, specimens with dimensions of 5x5x5 mm are ground and then polished in order to obtain a mirror-like surfaces. The pack composition contains metallic powder (Al/Si/Mo), activator (NH_4Cl), and inert material (Al_2O_3). The pack compositions and processing parameters (temperature and time) used for the aluminizing, siliconizing, and molybdenizing diffusion coatings are summarized in Table 1. As the compositions are prepared, a homogeneous mixture is ensured using a mini rotor for 2 h. The prepared pack mixtures are placed into alumina-based ceramic crucibles, and the specimens are fully embedded within the pack. Following the placement of ceramic lids, the crucibles are completely sealed using high-temperature-resistant refractory paste to prevent oxygen ingress during the diffusion treatments. After completion of the coating processes, metallurgical analyses are performed using X-ray diffraction (XRD, Rigaku Ultimate+) to

identify the phases formed on the coated surfaces. Cross-sectional specimens of the coatings are prepared through standard metallographic procedures and subsequently characterized using scanning electron microscopy (SEM, Jeol JSM 6060) to evaluate the coating morphology and microstructural features. In addition, elemental distribution and chemical composition analyses across the coating cross-sections are carried out by energy-dispersive X-ray spectrometer (EDS, IXRF).

Table 1: Pack compositions and process parameters used.

Process	Pack composition (wt.%)	Temperature (°C)	Time (h)
Aluminizing	40Al + 10NH ₄ Cl + 50 Al ₂ O ₃	700	2
Siliconizing	40Si + 7NH ₄ Cl + 53 Al ₂ O ₃	1000	
Molybdenizing	40Mo + 10NH ₄ Cl + 50 Al ₂ O ₃	900	

3. Results and discussion

Figure 1 shows the XRD patterns of the aluminized, siliconized, and molybdenized Ti6Al4V alloy. The diffraction results confirm the successful formation of diffusion coatings composed of Ti–Al, Ti–Si, and Ti–Mo based phases, indicating that intensive interdiffusion reactions occur between the deposited elements and the titanium substrate during the coating process. For the aluminized alloy, the coating predominantly consists of TiAl, TiAl₂, and TiAl₃ intermetallic compounds together with residual α -Ti reflections, suggesting the development of a multilayer aluminide coating with decreasing aluminum concentration toward the substrate. In contrast, the siliconized surface exhibits characteristic diffraction peaks corresponding to TiSi, TiSi₂, and Ti₅Si₃ silicides, accompanied by minor α -Ti and Al₂O₃ reflections. The coexistence of several titanium silicides indicates a sequential phase evolution governed by silicon diffusion and local compositional gradients across the coating thickness. The XRD pattern of the molybdenized alloy differs markedly from those of the aluminized and siliconized coatings, revealing predominantly α -Ti and β -Ti phases together with the metastable α' -Ti and α'' -Ti martensitic phases. These results demonstrate that molybdenum is mainly incorporated into the titanium lattice as a β -stabilizing alloying element, resulting in the formation of a Ti–Mo solid solution rather than discrete Ti–Mo intermetallic compounds. The retention of β -Ti together with metastable martensitic phases further suggests that molybdenum diffusion significantly modifies the phase stability of the Ti6Al4V substrate during the high-temperature treatment and subsequent cooling.

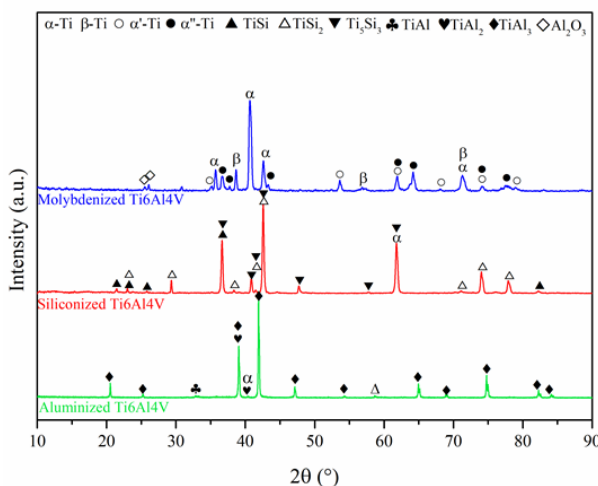


Fig. 1 XRD data of the coated alloys.

The SEM cross-sectional image of the aluminized titanium alloy coating is presented in Figure 2. Under the applied processing conditions (700 °C, 2 h), a continuous, homogeneous, and defect-free deposition layer ~ 20 μ m thick is successfully formed,

exhibiting a high degree of adhesion to the substrate material. EDS elemental mapping indicates that the coating layer contains a dominant aluminum content and suggests that inward diffusion of aluminum occurred under the processing conditions. Titanium and vanadium are also detected within the coating layer, revealing an outward diffusion tendency of these elements from the substrate composition. Furthermore, EDS point analyses performed from the outermost surface of the coating toward the substrate show a decreasing aluminum concentration gradient from the surface to the substrate, whereas titanium and vanadium exhibit the opposite trend, increasing toward the substrate material. Since Ti–Al intermetallic phases with progressively varying stoichiometries are formed from the surface toward the substrate, an improvement in the surface mechanical properties relative to the substrate is inevitable. It has been reported that Ti–Al intermetallic phases formed in the cross-section of Ti6Al4V alloy during low-temperature (700 °C) aluminizing processes can increase the hardness of the substrate (330–350 HV) to approximately 600–650 HV [22].

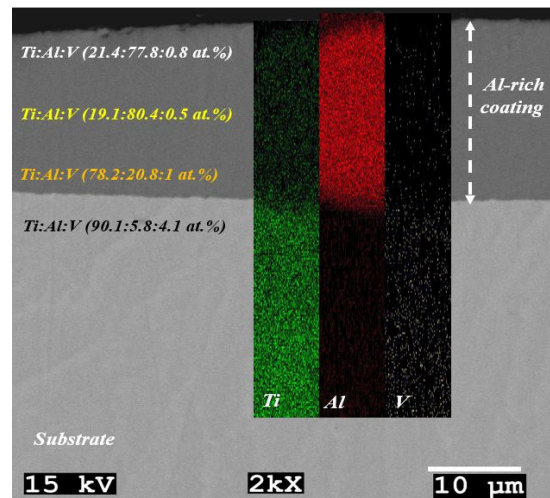


Fig. 2 SEM-EDS data for the aluminized alloy.

The siliconizing of Ti6Al4V at 1000 °C is fundamentally a reaction-diffusion process coupled with high-temperature phase transformations. The treatment temperature places the substrate in the β -Ti field, enhancing atomic mobility and facilitating rapid silicon ingress. Because silicon exhibits very limited solubility in titanium, supersaturation is quickly relieved through the nucleation and growth of titanium silicides, predominantly Ti₅Si₃. Coating growth proceeds via inward Si diffusion and outward Ti diffusion, producing a graded multilayer structure controlled by thermodynamic stability and diffusion kinetics. Figure 3 shows the SEM image of the coating cross-section of the siliconized titanium alloy (~ 40 μ m), and local EDS analyses reflect the silicon content from the surface to the center. Considering the XRD data, the compositional gradient of silicon from the surface to the center in the coating section directly explains the existence of various Ti–Si intermetallics like TiSi₂, TiSi, and Ti₅Si₃. In coatings containing such phases, the surface hardness can reach approximately 1200 HV, thereby providing the material with enhanced surface resistance, particularly under tribological conditions [23].

During molybdenizing of Ti6Al4V, a series of diffusion-controlled metallurgical reactions occur between the deposited Mo species and the titanium substrate. The overall process can be divided into several stages: (i) formation and deposition of Mo rich compounds, (ii) interdiffusion between Mo and Ti, (iii) formation of a β -Ti(Mo) solid solution layer, (iv) formation of Ti–Mo intermetallics etc. It is fact that the dominant metallurgical phenomenon during pack-cementation molybdenizing of Ti6Al4V at 900 °C is the diffusion-driven formation of a Mo-enriched β -titanium layer, accompanied by possible Ti–Mo intermetallic formation and the establishment of a graded metallurgical bond

between the coating and the substrate. The SEM image of the molybdenized titanium surface section shown in Figure 4 indicates the presence of a continuous Mo-enriched β -titanium layer approximately 50 μm thick. Depending on the processing temperature and time, diffusion-controlled layer growth can be achieved, and the surface hardness can reach approximately 1400 HV owing to the formation of a Mo-rich solid-solution layer together with the partial presence of Ti–Mo intermetallic phases [24].

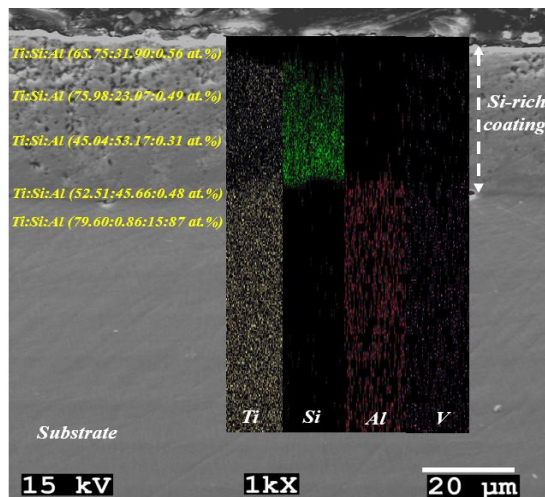


Fig. 3 SEM-EDS data for the siliconized alloy.

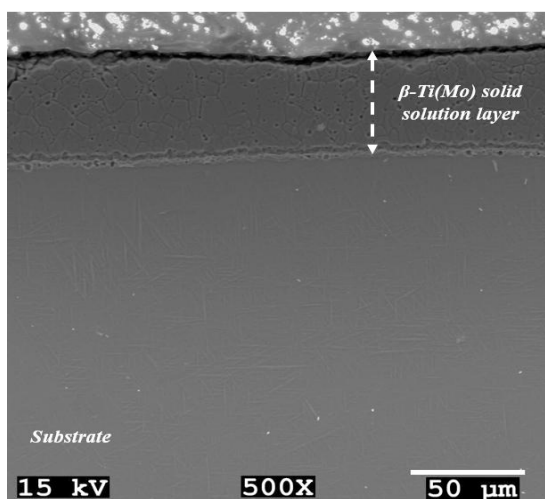


Fig. 4 SEM-EDS data for the molybdenized alloy.

4. Conclusion

Thermo-reactive diffusion treatments based on pack cementation successfully produced metallurgically bonded aluminide, silicide, and molybdenum-enriched surface layers on cast Ti6Al4V alloy. Structural characterization confirmed that the processing conditions promoted the formation of Ti–Al, Ti–Si, and Ti–Mo reaction products with continuous and adherent coating morphologies. The aluminizing process performed at 700 °C for 2 h generated a homogeneous defect-free coating approximately 20 μm thick, whereas siliconizing at 1000 °C resulted in the formation of multiple titanium silicide phases through diffusion-controlled growth throughout the 40 μm coating thickness. Molybdenizing at 900 °C produced the thickest coating (~50 μm), consisting predominantly of a Mo-enriched β -Ti solid-solution layer with possible Ti–Mo intermetallics. SEM-EDS analyses have revealed well-defined elemental concentration gradients across all coating cross-sections, confirming inward diffusion of the deposited species and outward diffusion of substrate elements, which are characteristic of diffusion-controlled coating growth. XRD analyses further verified the successful formation of the intended Ti–Al, Ti–

Si, and Ti–Mo phases. Based on the identified phase constituents and previous literature, the developed coatings are expected to provide significant improvements in surface hardness and, consequently, enhanced wear resistance and high-temperature performance. The results demonstrate that thermo-reactive diffusion processing offers an effective and controllable approach for tailoring the surface microstructure of cast Ti6Al4V alloy through appropriate selection of coating chemistry and processing temperature.

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Intelligent Brazing Materials

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ABSTRACT. The paper presents the research carried out for the development of brazing materials which, in a single pass, deposit two layers in the brazing joints: a buffer layer with high diffusion potential and increased silver content, and a filling layer with high strength and reduced silver content. The brazing performance of Ag20-type coated rods was improved by introducing a percentage of Ag40-type powder into the deoxidizing coating. The research was completed by establishing and patenting both the product formulation and the manufacturing process. The new product was successfully tested for producing brazed joints in copper pipes, copper-to-steel joints, and steel-to-tungsten carbide joints. The results of the mechanical tests met the required specifications, while the metallographic analysis of the joints showed an increase in diffusion of approximately 18.5% compared with the diffusion obtained using Ag20-type rods. The advantages of the brazing process using the new products consist of increased brazing productivity, improved quality of the brazed joints, and reduced environmental emissions from the resulting slag.

1. Introduction

The aim of the research is to develop coated rods for brazing with high efficiency and technological facilities for intelligent single-pass deposition of two layers, a buffer with high diffusion potential and rich in silver and a brazing joint filler with low silver content and high mechanical strength [1].

The new material [2] meets the technological condition, imposed for flame brazing and coated rods, of melting the deoxidizing coating before melting the metal rods, which allows increasing the deposition efficiency by introducing into the coating mass system, initially consisting of a mixture of deoxidizing substances, a portion of a metal alloy with melting temperatures at the level of the deoxidizing flux and high diffusion potential in the base materials.

The innovative principle, mentioned above, for obtaining coated rods for brazing with silver alloys with diffusion properties at the level of the Ag 40 type and mechanical strength of the Ag25 type. The experiments were carried out with materials that comply with environmental protection standards [3] and sustainable development principles for the circular economy [4].

2. Experiments

The experimental program was carried out in three main directions: improving the diffusion potential of the brazing material in the base materials; developing, optimizing and finalizing the product recipe and adapting and finalizing the coated rod manufacturing process to the new requirements for producing high-yield coated rods.

2.1 Improving diffusion potential

Previous research conducted by the authors highlighted the possibility of modifying the deposition yield by adding metal precursors to the deoxidizing coating, an idea also extended to increase the diffusion potential by adding metalloids, post-transition metals, non-metals with growth effect, the wetting and deoxidation properties of the melts which can be influenced from a structural point of view, after solidification by alloying and/or micro-alloying in the metal bath.

In order to determine how to influence the diffusion potential, a series of combinations of copper with zinc, silver, antimony, phosphorus, tin, carbon were performed, which were developed in the form of briquetted precursors (Fig. 1).

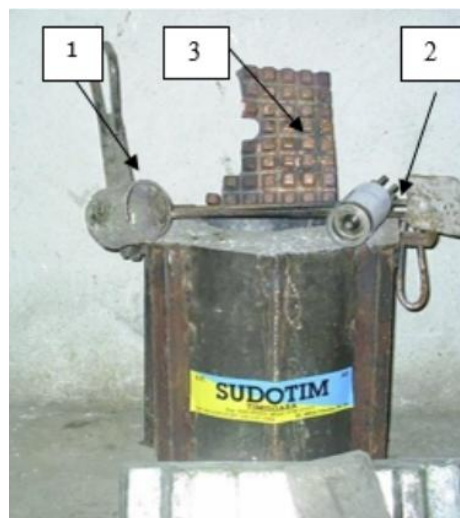


Fig.1. Charge melting stand with gas flame and compressed air.

1-Furnace crucible; 2-liquefied petroleum gas burner; 3-Briquette precursors.

The precursors were ground and introduced into the coating to determine the effects on brazing. An example is shown in Fig. 1b.

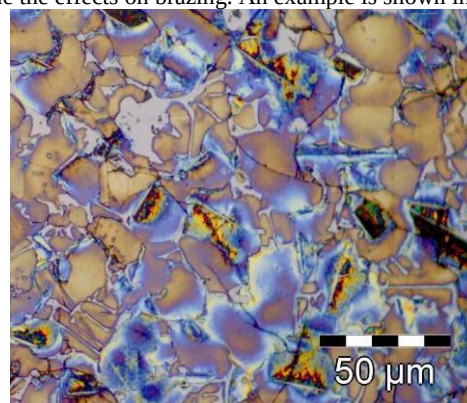


Fig.2. Microstructure of cast 85%Cu-5%P-10%Si alloy . Magnification 500x .

2.2 Development and finalization of the product recipe for VIAg25SnSiPR coated rods

The aim was to produce reasonably priced coated rods for high-efficiency brazing of pipe-to-copper pipe and steel pipe-to-copper pipe joints. The product recipe optimized on the minimum cost, maximum diffusion criterion of the VIAg25SnSiPR rods is presented in Table 1.

Table 1. Optimized product recipe of VIAg25SnSiPR rods.

Raw materials and materials	Participation in the product [% by mass]	Constituents [% by mass]
Naked rods SR EN ISO 17672:2010	65%	Ag125; $\Phi 2 \times 500 \text{ mm}$
Deoxidizing flux FH SR EN 1045:1999	25% powdery material, sort 0.15mm	35% boric frit (1/3 boric acid + 2/3 borax) 26% boric acid hydroxide 20% fluorite frit 19% binder+plasticizer
Activating precursors of the Cu-Sn-P-Si and Ag-Cu-Zn-Sn types	10% nanopowders	6%[Ag140/ENISO17672:2010] 4%[50%Cu-40%Sn-8%P-2%Si]

2.3 Development and implementation of the manufacturing process of VIAg25SnSiPR.

The introduction of metallic precursors ground in planetary mills into the coating modified the extrusion parameters of the coating mass on the rod, which led to a series of experiments to optimize and adapt the classic process of manufacturing extruded coated rods (Figure 2) to the new conditions.



Fig.3 . Manufacturing line rods WRAPPED for brazing .

The process parameters optimized on the minimum cost criterion are presented in Table 2.

Table 2. Parameters of the manufacturing process of VIAg25SnSiPR rods.

Process parameters	Optimized values
Dry homogenization time	8 ± 2 minutes
Wet homogenization time	6 ± 2 minutes
Briquetting pressure	20 ± 0.5 MPa
Extrusion pressure	20 ± 1 MPa
Rod mass flow rate	approx. 5.9 kg/hour
Coating mixture mass flow rate	approx. 2.5kg/hour
Wrapped rod take-up belt speed	approx. 9kg/hour

The problems of bare rod buckling, pressure jump extrusion and cracking of the coating were solved by modifying the rod feed train,

increasing the wet mixing temperature and controlling the humidity in the drying chamber.

The product recipe and the production process have been patented.

3. Results.

The production process presented above was used to produce a test batch, used to characterize the VIAg25SnSiPR product, in terms of deposition and technological performance.

3.1 Characterization of deposits with VIAg25SnSiPR rods.

The coated rods from the test batch were used to create, by melting with an oxyacetylene flame, a sample of deposited metal which was subjected, according to the regulations in force, to chemical analysis on a laboratory spectrometer, metallographic examination to highlight the microstructure and to hardness tests to confirm the structural analysis.

analysis was also performed on the bare rods under the same conditions, for comparison. The results are presented in Table 3.

Table 3 Chemical composition of MD and MV

Sample mark	Composition [% by mass]						
	Ag	Wt h	Zn	CD	Sn	An d	P
MV	24.	39.	31.	-/	1.5	-/	-/
Ag125 SR	0	0	0	0.01	...	-/-	0.0
EN ISO	...	...	...	0	2.5		08
17672/2010	26.	41.	35.				
0 prescribed	0	0	0				
Determine d MV	25.	40.	32.	0.00	1.8	0.1	0.0
	2	5	7	8		2	06
MD	25.	40.	31.	0.00	2.1	0.2	0.0
	6	7	2	7		5	2

Ag, Sn, and P additions are observed in the deposition (MD) compared to the composition of the rod (MV) which will be found in the bonding process in the buffer layer. The deposition defects and structural constituents were determined by metallographic analysis on the deposited metal sample, etched with ammoniacal cupric chloride, figure 3.

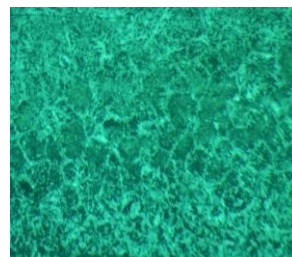


Fig. 3. a. MD microstructure [100X].

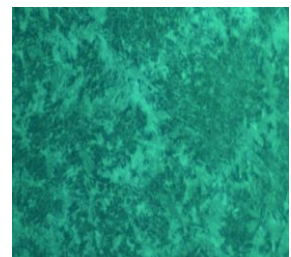


Fig. 3.b. MD microstructure [500X].

The metallographic analysis revealed a biphasic structure of alpha and beta solid solutions without defects such as micro cracks or imperfections such as hydrogen diseases and/or oxygen pores. The metallographic analysis was accompanied by sclerometric tests which revealed an average VICKERS-HV_{0.1} hardness of 184.6, confirming the observed structure.

3.2 Checking the brazing behavior

The brazing behavior was verified under industrial conditions, practically during the manufacture of refrigeration installations, through 2 concrete cases of joining: copper pipe to copper pipe and steel pipe to copper pipe, in capillary joints. The joints presented above were made until the date of the experiments with Ag40 type alloys, which are replaced by the new product. The 2 types of joints,

in batches of 24 pieces for each type, were made by oxyacetylene brazing with neutral flame with VIAg25SnSiPR rods. The batches thus obtained were subjected to pressure testing, in accordance with the regulations in force, without pressure losses or material deformations. The longitudinal cross-section investigation of the joints, figure 4, revealed for all joints a complete penetration and diffusion of the brazing alloy into the brazing materials at the level of the alloys with 40% Ag, probably determined by the silver content in the buffer layer.



a. **Fig. 4.** Steel pipe joint design in copper pipe .
a) joint design ; b) joint design

Figure 5 shows the critical case of the steel pipe to copper pipe joint. , The microscopic examination was performed according to SR 5000-97 and STAS 5500-74 . The structural analysis was performed on the MeF2 optical microscope, at 100X.

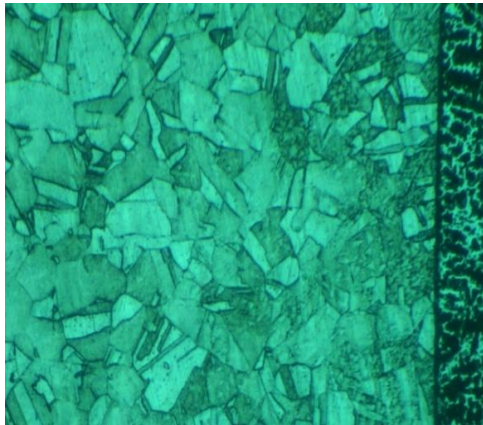


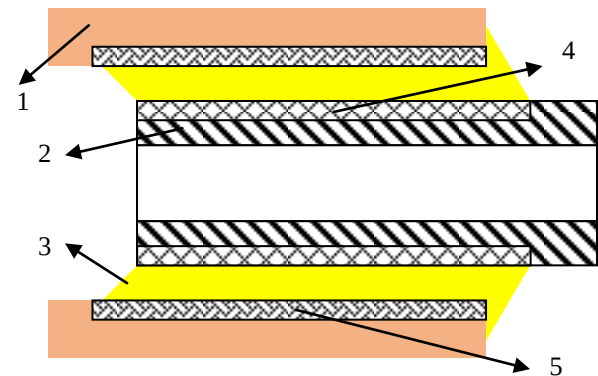
Fig. 5. [E1 attack , 100X].

The test joints were subjected to tensile rupture testing and the results were in accordance with the requirements, all tested samples either ruptured in the base material Figure 6.

The high strength of the joints is explained by the design of the figure 7.



Fig. 6. Tensile test specimen for steel pipe-to-copper pipe joint.



b) **Fig.7.** Test batch of joints. a) samples made; b) brazed assembly
1-MB1(copper,brass); 2-MB2(steel);3-soldering alloy; 4-ZIT MB2; 5-ZIT MB1

Conclusions

The research conducted was completed with the following results:

- a method for obtaining coated rods for brazing with high efficiency and deposition of 2 layers in a single pass was presented and experimented.

- a new generation of coated rods for brazing was created, by equipping the coating with adjuvant metallic systems and/or process catalysts, VIAg25SnSiPR.

- the classic manufacturing line of coated rods for brazing was adapted to the conditions of equipping the deoxidizing coating with metallic systems.

- the product recipe of the new generation of smart brazing materials has been patented.

- two high-efficiency and reasonably priced brazing technologies were developed, characterized and qualified according to SR EN13134:2002.

- the idea listed in the preamble has been confirmed through experimental research and AI-assisted simulation.

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Innovative Nanomaterials for the Conservation and Sustainable Protection of Cultural Heritage

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Abstract: Cultural heritage objects represent an essential component of human identity, history, and collective memory. However, monuments, artworks, manuscripts, archaeological artefacts, and historical buildings are continuously exposed to environmental deterioration processes, including humidity fluctuations, pollution, biological colonization, salt crystallization, and mechanical degradation. Conventional conservation methods often provide effective protection but may involve invasive procedures, limited durability, or the use of environmentally harmful chemicals. In recent decades, nanotechnology has emerged as a promising approach for sustainable conservation due to the unique physicochemical properties of nanomaterials, including high surface area, enhanced reactivity, photocatalytic activity, and the possibility of designing multifunctional protective coatings. This review discusses innovative nanomaterials used in cultural heritage conservation, including silica nanoparticles, titanium dioxide, zinc oxide, calcium hydroxide nanoparticles, graphene-based materials, and nanocomposites. Their applications in consolidation, cleaning, protection against pollutants, antimicrobial treatments, and sustainable preservation strategies are examined.

The report is under project No. KII-06-H90/6 dated 10.12.2024 on the topic: "Ecologically sustainable conservation strategy for written heritage" from the Scientific Research Fund, Competition for funding of fundamental scientific research - 2024. Supervised by Chief Associate Professor, PhD, MSc Eng. Iskra Tsvetanska.

KEYWORDS: NANOMATERIALS; CULTURAL HERITAGE CONSERVATION; NANOPARTICLES; PROTECTIVE COATINGS; SUSTAINABLE RESTORATION; NANOTECHNOLOGY.

1. Introduction

The preservation of cultural heritage is a multidisciplinary challenge involving chemistry, materials science, conservation science, and environmental engineering. Historical materials such as stone, ceramics, paper, wood, metals, and wall paintings undergo progressive deterioration due to interactions with atmospheric pollutants, moisture, microorganisms, and human activity. [4] Climate change has further intensified these challenges by increasing temperature variations, humidity instability, and the frequency of extreme weather events affecting heritage sites worldwide. [12]

Traditional conservation approaches have relied on organic polymers, inorganic consolidants, and chemical cleaning agents. Although many of these materials have demonstrated effectiveness, they can suffer from problems such as incompatibility with original substrates, ageing, poor reversibility, and environmental toxicity. [1] Consequently, there is growing interest in developing sustainable conservation technologies capable of providing long-term protection while minimizing damage to cultural objects.

Nanotechnology offers new possibilities by exploiting materials with dimensions typically below 100 nm. At this scale, materials exhibit unique optical, mechanical, chemical, and biological properties that differ significantly from their bulk counterparts. [11] Nanomaterials can penetrate porous structures, enhance mechanical stability, control chemical reactions, and provide protective functionalities such as self-cleaning or antimicrobial activity. [3]

2. Role of Nanotechnology in Cultural Heritage Conservation

Nanotechnology has introduced innovative solutions for several major conservation problems:

- consolidation of fragile materials;
- removal of unwanted deposits and pollutants;
- protection against water infiltration;
- inhibition of microbial growth;

- development of environmentally friendly protective coatings.

The effectiveness of nanomaterials is mainly related to their high surface-to-volume ratio, which increases chemical interaction with heritage substrates. Furthermore, nanoscale engineering allows researchers to tailor particle size, morphology, surface functionality, and compatibility with specific historical materials. [7]

One of the major advantages of nanotechnology is the possibility of creating multifunctional materials. For example, a single nanocomposite coating may simultaneously provide hydrophobic protection, UV resistance, and antimicrobial properties, reducing the need for repeated restoration interventions.

3. Silica Nanoparticles for Consolidation and Protection

Silica nanoparticles (SiO_2) are among the most widely investigated nanomaterials for cultural heritage conservation because of their chemical stability, transparency, and compatibility with mineral substrates. They are particularly useful for consolidating deteriorated stone materials, especially limestone and sandstone.

The consolidation mechanism is based on the penetration of nanosilica dispersions into porous structures, followed by gel formation and strengthening of weakened regions. Compared with traditional silica-based consolidants, nanosilica offers deeper penetration and better control over the treatment process. [14]

Studies have demonstrated that silica nanoparticles can improve mechanical strength without significantly altering the appearance or permeability of stone surfaces. [9] This is particularly important because conservation treatments must preserve the original aesthetic and physical characteristics of historical materials.

However, challenges remain regarding aggregation of nanoparticles, long-term stability, and optimization of particle size distribution. Surface modification of silica nanoparticles with organic or inorganic functional groups has been proposed to improve compatibility and performance. [7]

4. Titanium Dioxide Nanoparticles and Photocatalytic Conservation

Titanium dioxide nanoparticles (TiO_2) have attracted considerable attention due to their photocatalytic activity, chemical stability, low toxicity, and relatively low cost. Under ultraviolet

radiation, TiO_2 generates reactive oxygen species capable of decomposing organic pollutants, including atmospheric contaminants and biological residues. [6]

In cultural heritage conservation, TiO_2 coatings have been investigated for self-cleaning surfaces on architectural monuments. Their photocatalytic properties allow the degradation of organic pollutants such as hydrocarbons deposited from urban pollution. [8]

Additionally, TiO_2 nanoparticles exhibit antimicrobial properties by producing oxidative stress that can damage bacterial and fungal cells. This makes them attractive for protecting stone monuments and historical surfaces affected by biological colonization.

Despite these advantages, TiO_2 applications require careful evaluation because excessive photocatalytic activity may potentially accelerate degradation of organic components in artworks, particularly pigments and binders. Therefore, controlled formulations and protective strategies are necessary.

5. Zinc Oxide Nanoparticles in Heritage Protection

Zinc oxide nanoparticles (ZnO) represent another important class of functional nanomaterials used in conservation. ZnO possesses antimicrobial activity, UV absorption capability, and photocatalytic properties.

The antimicrobial effect of ZnO nanoparticles results from the generation of reactive oxygen species and the release of zinc ions, which interfere with microbial metabolism. [13] These properties make ZnO suitable for applications in protecting paper, textiles, wood, and stone materials.

Furthermore, ZnO -based coatings can provide UV shielding, reducing photo degradation of sensitive historical materials. However, possible interactions between ZnO nanoparticles and pigments require detailed assessment before practical application.

6. Calcium Hydroxide Nanoparticles for Lime-Based Materials

Calcium hydroxide nanoparticles ($\text{Ca}(\text{OH})_2$ nanoparticles) have become one of the most successful examples of nanotechnology applied to cultural heritage conservation.

Traditional limewater treatments have historically been used for consolidating carbonate-based materials. However, their effectiveness is limited because calcium hydroxide has low solubility and poor penetration ability. Nanocalcium hydroxide overcomes these limitations due to its extremely small particle size and increased reactivity. [2]

After application, nanoparticles penetrate cracks and porous structures, reacting with atmospheric carbon dioxide to form calcium carbonate, which restores cohesion within deteriorated limestone, frescoes, and lime-based mortars.

Nanocalcium hydroxide has been successfully applied in the conservation of wall paintings and architectural surfaces because it provides chemical compatibility with original carbonate materials while minimizing visual alteration. [7]

7. Titanium Dioxide Nanoparticles and Photocatalytic Conservation

Graphene and graphene oxide have emerged as advanced materials with exceptional mechanical strength, electrical conductivity, and barrier properties. Their application in cultural heritage conservation is still developing but shows significant potential.

Graphene-based coatings may provide protection against moisture, oxygen diffusion, and chemical pollutants due to their highly impermeable structure. [10] Additionally, graphene oxide can be functionalized with nanoparticles to create hybrid materials with enhanced antibacterial or protective properties.

Nanocomposites combining graphene derivatives with silica, titanium dioxide, or metal nanoparticles represent a promising direction for developing multifunctional conservation materials.

8. Environmental Sustainability and Future Perspectives

The development of sustainable conservation technologies requires balancing effectiveness, safety, and environmental impact. Nanomaterials contribute to sustainability by enabling:

- longer-lasting protective treatments;
- reduced frequency of restoration interventions;
- lower consumption of chemical cleaning agents;
- minimally invasive conservation procedures.

Nevertheless, potential risks associated with nanoparticle release, toxicity, and environmental accumulation must be carefully evaluated. Future research should focus on green synthesis methods, biodegradable nanomaterials, and comprehensive life-cycle assessments. [5]

The integration of nanotechnology with digital monitoring systems, artificial intelligence, and advanced characterization techniques may further improve preventive conservation strategies.

9. Conclusion

Innovative nanomaterials represent a transformative approach for the sustainable conservation of cultural heritage. Materials such as silica nanoparticles, TiO_2 , ZnO , calcium hydroxide nanoparticles, and graphene-based nanocomposites provide new opportunities for consolidation, protection, cleaning, and antimicrobial treatment of historical materials.

Although significant progress has been achieved, responsible implementation requires careful evaluation of compatibility, durability, and environmental safety. Future developments in green nanotechnology are expected to provide increasingly efficient and sustainable solutions for preserving cultural heritage for future generations.

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Influence of non-metallic inclusions and products of corrosion on the wear particles formation on the railway wheel and rail treads

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Abstract. The effect of inclusions and corrosion products on the mechanism of wear of railway wheels made of R7 steel and also of rails made of R7 steel M76. was studied. The research was carried out by several methods: metallographic, electron microscopy, X-ray, micro-X-ray spectral analysis. The features of the formation of wear particles near nonmetallic inclusions and corrosion products related to structural changes near the treads of railway wheels and rails as well as to the interaction of steel with the surrounding atmosphere during operation were investigated. The influence of various factors on the role of nonmetallic inclusions and corrosion products in the formation of wear particles is shown, which makes it possible to predict the nature of wear of the rolling surface of railway wheels and rails under various operating conditions. The main sources and the mutual influence of various factors on the formation of wear particles near nonmetallic inclusions and corrosion products have been established, which makes it possible to predict the nature of wear of the rolling surface of railway wheels and rails under various operating conditions.

KEYWORDS: RAILWAY WHEEL, RAIL, WHEEL STEEL, RAIL STEEL, WEAR, WEAR PARTICLES, NON-METALLIC INCLUSIONS, CORROSION.

1.Introduction.

The reliability and durability of railway wheels and rails depend on the technological process of their production, the chemical composition, microstructure and mechanical properties of steels, the geometry of the tread profiles, and the conditions of interaction of the wheel-rail friction pair [1]. When considering the wear characteristics of the tread surfaces of railway wheels and rails, it is necessary to take into account the tribological interaction in the contact zones of the friction pair, which occurs in their surface layers under the influence of high contact stresses, cyclic loads and temperatures, abrasive particles, and active atmospheric and industrial environments. During operation, the wheel and rail are in a complex stress state determined by a system of contact, dynamic and cyclic stresses [1], which cause elastic-plastic and thermal phenomena in the wheel and rail, contribute to fatigue processes in the rim and disk, crest undercutting and destruction of the tread surface. *The goal* of the work is to study influence of non-metallic inclusions and products of corrosion on the wear particles formation on the railway wheel and rail treads during operation.

2. Materials and Procedures.

The wear mechanism of railway wheels made of R7 steel and rails made of M76 steel was investigated. The studies were conducted using several methods: metallographic analysis (Neophot-31), X-ray diffraction (DRON 2.0), electron microscopy (JSM-35), and micro-X-ray spectral analysis (Nanolab-7).

3. Results and discussion.

During a visual inspection of wheels and rails with heavily worn rolling surfaces (treads), defects such as sliding, metal drag, delamination, metal build-up from the rolling surface

onto the outer side face of the rim, and fatigue-corrosion wear were discovered (Fig. 1, a, b).

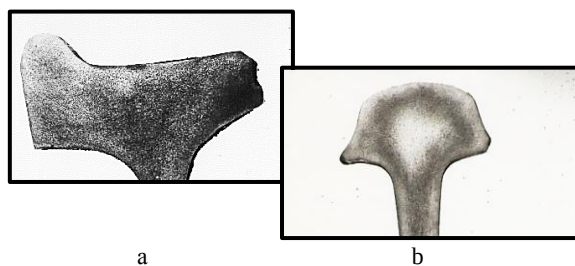


Fig. 1. The microstructure of railway wheel (a) and rail (b) after operation.

The microstructure of steel surfaces near the treads of railway wheels and rails is characterized by the presence of a zone of deformed grains and "white layer" areas (Fig. 2, a, d) [1, 2]. Wear particles formed as a result of the development of intense plastic deformation near the treads are flakes or plates of varying thickness (Fig. 2, b, e). They are characteristic of normal wear conditions [3]. According to the theory of "flaking" wear [3], during friction, the maximum dislocation density occurs not directly on the surface, but at a certain depth, where extended microcracks appear, growing to a critical size as a result of plastic flow of steel. The second source of wear particles

formation on the treads of wheels and rails is the so-called "white layer", which has increased brittleness (Fig. 2, c, f) [1,2]. Near the "white layer" areas, localized deformation of the wheel rim surface layer or of rail tread is detected, which contributes to stress concentration and chipping of the "white layer." Wear particles appear as sharp-edged, irregularly shaped fragments (Fig. 2, g, h). They typically occur under very high pressures and their appearance can be associated with the formation of small fatigue cracks in the surface layers of the rims and rail tread [3].

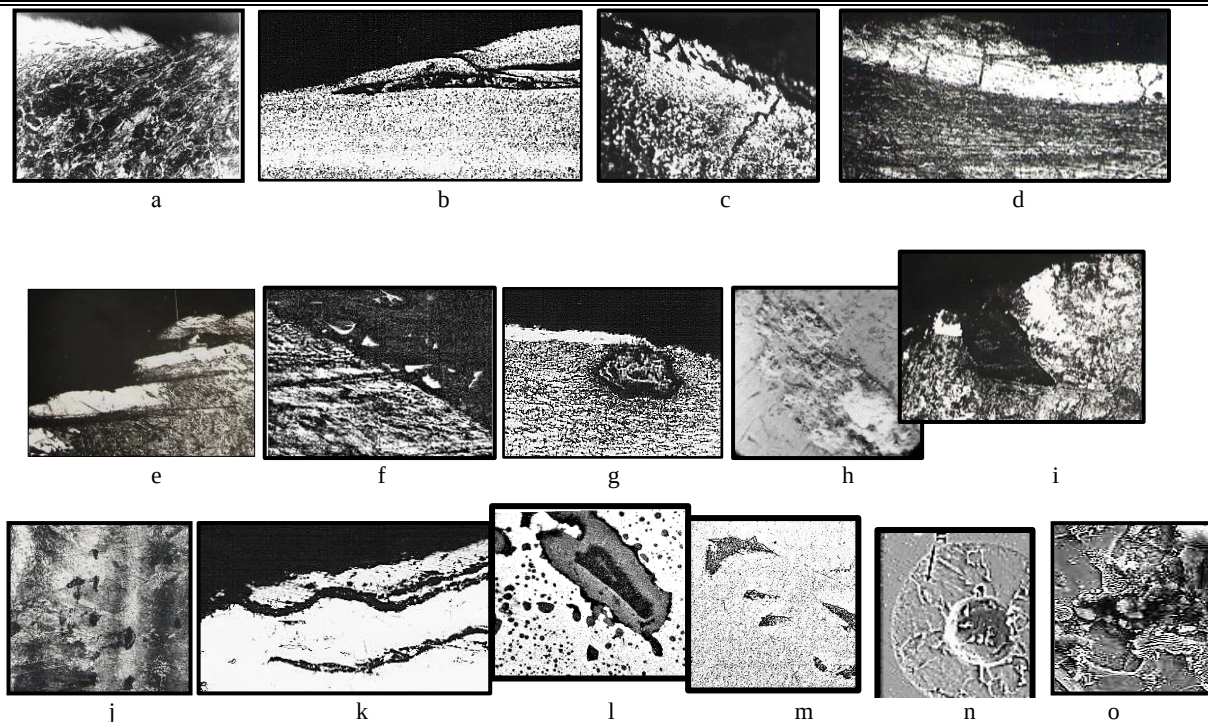


Fig. 2. Microstructure (a, d), wear particles on the tread of railway wheels (b, c, k) and rails (c, f) and areas of corrosion damage of the treads of wheels (g, h, l) and rails (i, j, m - o); x400.

A comparative analysis of the microstructure and wear particles of the treads of railway wheels and rails indicates the uniform nature of the processes occurring in the friction pair, despite some differences in the chemical composition of wheel and rail steels. When considering the specific features of plastic deformation development in steel near the wheel and rail treads, one should take into account their interaction with the surrounding atmosphere. It is known that the initial stage of corrosion includes the adsorption of atoms of elements from the surrounding humid environment, which can cause the effect of adsorption facilitation of plastic deformation near the wheel and rail treads (the P.A. Rebinder effect) [4]. For the formation of surface defects (wear particles), it is necessary to achieve the limiting state of strain strengthening of steel, and this stage of deformation is accelerated by the action of a surface-active environment [4]. With the formation and detachment of wear

particles, new "fresh" surfaces of developing cracks appear, on which adsorption of active elements of the surrounding humid environment occurs.

Thus, a humid environment, also containing various lubricants, contaminants, etc., has a negative effect on the fatigue strength of the wheel and rail treads due to the adsorption and corrosion effects. Spots of corrosion damage were found on the wheel and rail treads (Fig. 2, g - j). The process of corrosion destruction of wheel steel was studied in detail in works (Fig. 3, a, c) [5], where the chemical and phase composition of the corrosion products were determined by various methods. A study of the corrosion products of rail steel showed similar results, which are inclusions of complex oxides. In addition, as a result of adsorption processes on the surface of wear particles formed in areas of intense plastic flow, corrosion develops, therefore, oxidation products of steel are often visible on them (Fig. 2, k).

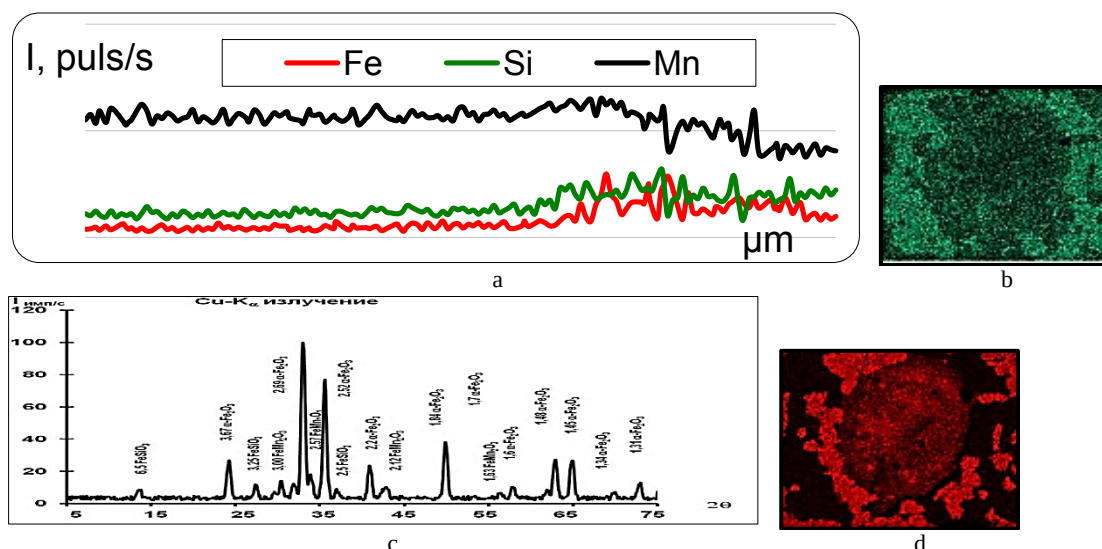


Fig. 3. Distribution of chemical elements in oxide phases of corrosive origin (a, cur. 1 - Fe, cur. 2 - Si, cu. 3 - Mn), the phase composition of corrosion products of the wheel steel (c), and the corrosion zone near the non-metallic inclusion - distribution of Fe (b) and O (d).

Corrosion damage often occurred near non-metallic inclusions of metallurgical origin (Fig. 2, l-o, 3, b, d). The

influence of inclusions on the local corrosion process is associated with both the local dissolution of the metallic matrix

and the activating action of their dissolution products. In addition, inclusion-matrix interphase boundaries are the centers of initiation of corrosion damage due to their defective structure and electrically stimulated processes of redistribution of interphase dislocations, the appearance of high current densities at the boundaries, and the development of adsorption of surface-active elements, which “shakes” these boundaries [6]. The leading role of inclusion-matrix interphase boundaries in increasing the microchemical heterogeneity of steels and the development of adsorption and corrosion processes is also obvious, which is due to the influence of the defective structure of these boundaries, as well as the appearance of a significant potential gradient and the presence of high current densities at these boundaries. Penetration of a corrosive environment into inclusion-matrix boundaries promotes the formation of corrosion products, the volume of which typically exceeds the volume of

the boundaries in which they formed, transforming adsorption processes into corrosion processes. Corrosion induces additional stresses at the inclusion-matrix boundaries due to the wedging action of the resulting corrosion products, which facilitates the development of fatigue cracks from non-metallic inclusions in the steel matrix.

The formation of wear particles is caused by non-metallic inclusions of metallurgical origin located near the treads of wheels and rails, which act as stress and strain concentrators in wheel and rail steel (Fig. 4, a) [6]. Furthermore, the formation of cracks and wear particles is also facilitated by oxidation and corrosion of the treads, which lead to the formation of coarse inclusions of complex oxides directly on the rolling surface [5]. The presence of inclusions and corrosion products significantly increases the rate of microcrack formation and their coalescence, which increases the intensity of wear (fatigue and friction).

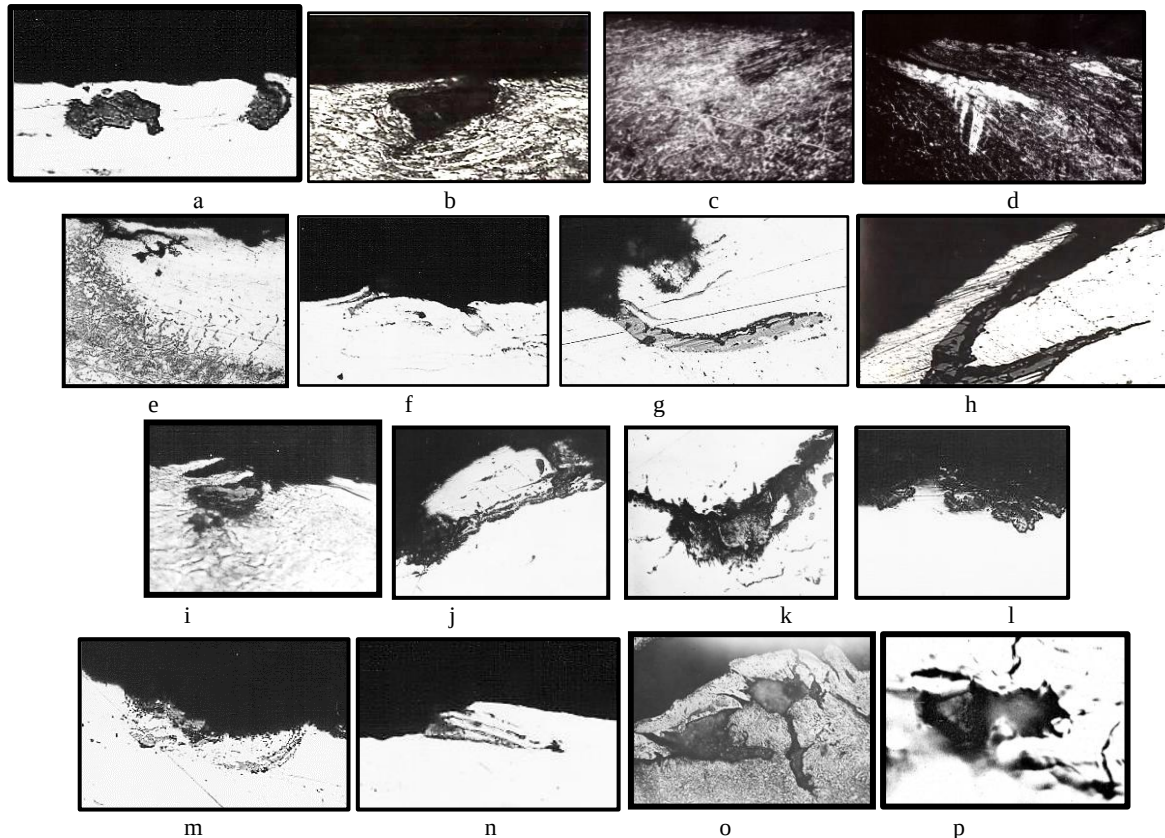


Fig. 4. Localization of deformation (a) and formation of wear particles near corrosion products of wheel and rail steel (b – h) and non-metallic inclusions (i – o) on the treads of railway wheels (b, c, e – l, n – p) and rails (a, c, d, m): x 400.

An analysis of the microstructure of wheel and rail steels near the treads of wheels and rails has shown that non-metallic inclusions and corrosion products contribute to the non-uniform development of deformation, the appearance of turbulent flow zones (Fig. 4, b – d) and promote the formation of zones with an increased degree of deformation. The behavior of non-metallic inclusions located near the treads under the influence of acting stresses depends on their type. They are plastic (sulfides) or not subject to plastic deformation (oxides, silicates, titanium carbonitrides) [6]. A non-metallic inclusion located near the treads of a wheel or rail is in a complex stress state determined by a system of contact, dynamic and cyclic stresses [6]. The temperature gradient arising near the treads during wheel and rail operation leads to a non-uniform distribution of deformations in the steel matrix and the forces acting on the inclusion.

The steel matrix fibers envelop an non-metallic inclusion (of metallurgical or corrosive origin) on one side if it directly extends onto the tread, or on both sides if it is located near the tread. In the first case, the matrix fibers gradually “run

Under conditions of high pressures and cyclically changing temperatures, the interaction of the contacting surfaces of the inclusion and the steel matrix occurs through the establishment of mechanical contact and the development of diffusion processes [6]. Mechanical contact is established as a result of smoothing the uneven surfaces of the inclusion and the wheel or rail steel matrix through the development of contact friction of their surfaces relative to each other, which prevents their relative movement. Contact friction increases the heterogeneity of the deformation of the wheel or rail steel matrix near the inclusions, which can affect not only the level of stresses acting on the inclusion, but also the stress state diagram [6]. During the contact interaction of the inclusions and the steel matrix, rotation of the inclusions is possible (see Fig. 4, b), which causes moment stresses and promotes local plastic rotations in the steel matrix as a result of its vortex flow.

into” the inclusion (Fig. 4, e – h), which acts as a brake for the moving fibers, bending and often twisting into loops or spirals (Fig. 4, i). In the second case, the matrix fibers envelop the inclusion on both sides, which may result in delamination along

the inclusion-matrix interphase boundaries and non-uniform deformation (Fig. 4, j - m). Compressive and shear stresses are concentrated at the inclusion-matrix boundaries, capable of destroying the inclusion if they exceed the tensile strength of the inclusions (of metallurgical or corrosive origin) under compression (Fig. 4, n, o).

During accelerated braking under conditions of a sharp increase in temperature, the possible role of slippage along grain boundaries in austenite and inclusion-matrix interphase boundaries in the development of deformation near the wheel or rail treads should be taken into account [1]. In addition, under conditions of emergency braking and local heating of the tread to temperatures corresponding to the austenitic region, when melting and even melting of low-melting iron-manganese sulfides and sulfide eutectics can occur [6]. Then, local sulfide red brittleness may appear (Fig. 4, p), a sharp localization of steel deformation occurs and multiple cracks appear, i.e., steel failure occurs when relatively low degrees of deformation are reached. Liquid layers are easily stretched parallel to the wheel or rail tread, cracks develop between them, facilitating the formation of

wear particles. Liquid layers are easily drawn out parallel to the rolling surface of the wheel or rail, and cracks spread between them, facilitating the formation of wear particles.

Near non-metallic inclusions and corrosion products of the wheel and rail steels the wear particles have the form of loops and spirals, similar to chips during cutting (Fig. 5, a - c). They take this shape due to residual stresses associated with the non-uniform distribution of dislocations across the thickness of wear particles formed near the inclusions [9, 10]. According to the data of work [9], such wear particles precede damage and are found mainly on the tread before localized destruction. In the case of localized sulfide red brittleness of steel, in areas of the tread where melting of sulfide inclusions has occurred, numerous wear particles of both spiral shape and compact irregular shape are observed (Fig. 5, d, e), which is caused by the presence of numerous microcracks in these zones, as well as fragmentation of wear particles due to strong localization of deformation.

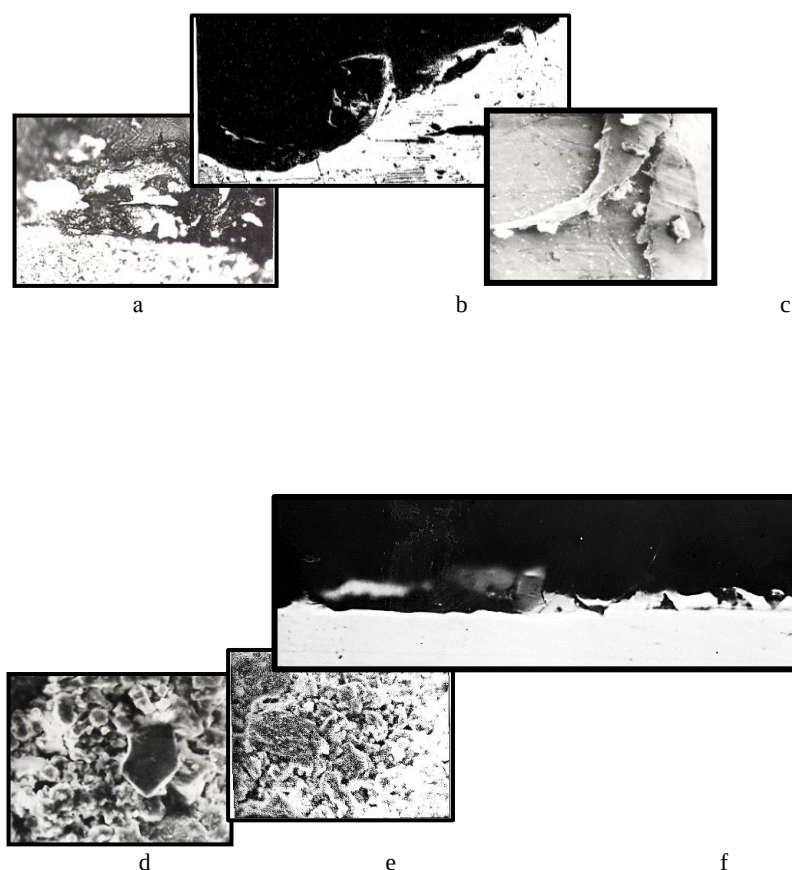


Fig. 5. Wear particles near non-metallic inclusions and corrosion products on the tread of railway wheels (a, c, d) and rails (b, e, f); x600.

Abrasive wear is possible in the wheel-rail friction pair, which is associated with mechanical destruction of the treads, resulting from the cutting or scratching action of solid particles from outside (various contaminants, particles of transported bulk materials, etc.). It results from shearing and plastic deformation

4. Conclusions.

Near non-metallic inclusions and corrosion products of wheel and rail steel, wear particles take the form of loops and spirals. They form due to localized deformation near the inclusions and the entrainment of the matrix metal onto the inclusions, as well as due to delamination of the inclusion-matrix interphase boundaries. Such particles are found primarily on the treads of wheels or rails before localized failure. In the case of

of the surface roughness (Fig. 5, f). In these cases, wear particles have an irregular, acute-angled shape, depending on the conditions of their formation (the ratio of particle hardness to steel, the speed of the train, the nature of plastic processes near the rolling surfaces, etc.).

localized sulfide brittleness, numerous spiral-shaped and compact, irregularly shaped wear particles are formed, caused by the presence of numerous microcracks in these zones, as well as the fragmentation of wear particles due to severe localization of deformation. Corrosion processes influence wear particle formation in zones of intense deformation near the treads. In the case of abrasive wear, wear particles have an irregular, acute-angled shape, depending on the conditions of their formation/

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Thermodynamic Properties and Phase Equilibria in the Fe–W–C System

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Abstract: By utilizing the tools of the PyCALPHAD library and a thermodynamic database, Python-based software components were developed to calculate the energetic characteristics of phase transitions, enabling the prediction of Fe–W–C system behavior under varying composition and temperature. According to the CALPHAD calculation, it was established that within the temperature range of 500–2000 K for the composition of 43 at.% Fe, 43 at.% W, and 14 at.% C, an intermediate Fe₃W₃C phase is formed, which melts incongruently at 1770 K.

Keywords: THERMODYNAMICS, CALPHAD, Fe-W-C SYSTEM, PHASE EQUILIBRIUM, THERMODYNAMIC PROPERTIES

1. Introduction

Iron-based alloys are widely used in various fields of science and technology. The quality of products made from them depends on the methods of production, which can be improved based on physicochemical properties. Steel matrix composites are formed by adding reinforcing materials (such as nitrides, carbides, borides, oxides and intermetallics) to the steel matrix, which allows the production of materials with improved performance characteristics [1–5]. The synthesis of such composites is an effective approach to improve the mechanical properties of steel: strength, hardness, durability and wear resistance [4]. Tungsten carbide (WC) is a common ceramic material that is widely used as an agent in nickel- and iron-based alloys [6]. WC is well suited for such materials due to its high melting point, thermal stability, strength and hardness, good wettability and thermal expansion coefficient similar to iron.

Since most methods of producing steels and alloys are associated with their melting, the thermodynamic properties of iron alloys in the liquid state are of great importance. Studies of phase equilibria are key to understanding the behavior of materials and alloys at high temperatures. At the same time, the determination of the liquidus and solidus surfaces in a wide concentration range is of great importance for the development of new alloys and the optimization of existing technologies. Experimental work remains one of the main research methods in this area at present. But studies of the thermodynamic properties of melts and phase equilibria in alloys of the Fe–W–C system have not been conducted at present. Given the complexity of high-temperature experiments to determine the thermodynamic properties of melts, it is advisable to calculate them from similar data for limit binary systems according to the “analytical” Redlich–Kister–Mujian model. Therefore, we will consider and analyze the thermodynamic properties of melts of limit binary subsystems first.

2. Results and discussion

Fe–W system. Thermochemical properties of iron-tungsten melts were determined by calorimetry [7–10]. The results of these works are compared in Fig. 1 and given in Table 1.

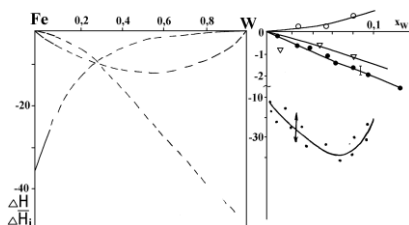


Fig. 1 Enthalpies of mixing of melts of the system:

— [7], 1873 K
 - - [8], 1863 K;
 . . , - [9], 1960 K

It can be seen that there is a very good agreement between the data of [8–10], and [7] do not coincide with them even in sign. This discrepancy is difficult to explain, since the results of the mixing enthalpies of the binary systems Fe–Cr(V, Ta, Mo, Nb) of [7] agree with the data of other studies. But since the data of three independent studies of Fe–W melts [8–10] coincide, it can be stated that they correctly reflect the energy of interparticle interaction in melts of the Fe–W system.

Table 1: Thermochemical properties of melts of the Fe–W system, in kJ/mol.

x W	-ΔH [9] T=1960 K	-ΔH _W	-ΔH [9] T=1960 K	-ΔH [8] T=1863 K	x W	ΔH [7] T=1873 K
0	0	10±1	0	0	0.05	0.42
0.02	0.25±0.05	22±2	0.35±0.05	0.2	0.02 5	0.38
0.04	0.6±0.05	31±3	0.7±0.05	0.5	0.07 5	0.8
0.06	1.0±0.1	35±3	1.1±0.1	0.8		
0.08	1.6±0.1	36±3	1.7±0.1	1.1		
0.10	2.0±0.2	27±3	2.1±0.2			
0.11	2.4±0.2	20±2	2.5±0.2			

The EMF method in the concentration range 0.95 < x_{Fe} < 1.0 at 1870 K investigated [11] the activities of tungsten in liquid alloys of the Fe–W system using a concentration chain of the form:

W–WO₂/ZrO₂–Y₂O₃/ Fe–W, WO₂.

The obtained EMF values and the calculated tungsten activities in melts of the iron-tungsten system at 1870 K are given below.

Table 2: Values of calculated EMF

x W	E 10 ³ , B	a _{w(sp.)}	a _{w(lp.)}
0,068	70,8	0,1724	0,1711
0,0188	58,9	0,2317	0,2270
0,0298	44,5	0,3313	0,3208
0,03541	32,1	0,4507	0,4338

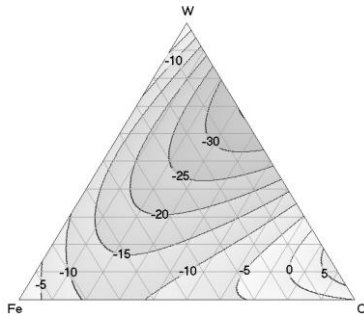
It was found that the tungsten activities of alloys of the Fe–W system are characterized by very large positive deviations from Raoult's law and correlate with the data [12].

Fe–C system. Thermodynamic data of melts of this system have been studied in many works, since iron-carbon alloys are very important for the development of ferrous metallurgy. The enthalpies of mixing and dissolution of melts of the Fe–C system have been determined at 1873 K and 1973 K [13,14], and the activities of the components - [15, 16]. The most reliable thermodynamic properties at 1900 K of Fe–C melts are given in Table. 3.

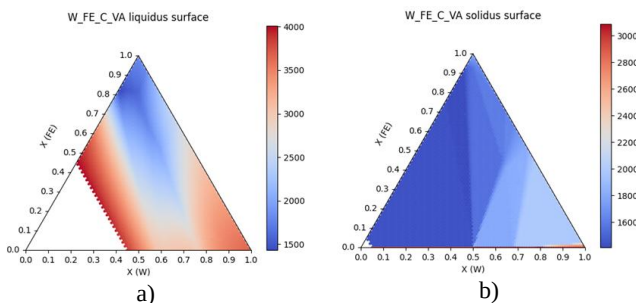
W–C system. W and C form the compounds WC and W₂C, and their enthalpies of formation (ΔH_f) are determined to be: – 38 and – 46 kJ/mol. However, the thermochemical properties of melts of the W–C system have not been investigated, so we have taken them equal to the enthalpies of formation. Using the molar enthalpies of melts of binary boundary systems, similar data were calculated for ternary melts of the Fe–W–C system according to the “analytical” Redlich–Kister–Mujianu model (Fig. 2). It can be seen that the extreme value of the integral molar enthalpy of melts of this ternary system is –46 kJ/mol and falls on the W_{0.66}C_{0.34} melt, i.e. the main contribution to the interaction energy between dissimilar atoms of these ternary melts is made by the boundary W–C system.

Table 3: Thermodynamic properties of Fe-C melts (standard state - liquid iron and liquid supercooled carbon) at 1900 K

x_C	$-\Delta_m \bar{H}_C$	$-\Delta_m H$	$\Delta_m \bar{S}_C^{exc.}$	$-\Delta_m S^{exc.}$	$\Delta_m \bar{G}_C^{exc.}$	$-\Delta_m G^{exc.}$
	kJ/mol		J/(mol·K)		kJ/mol	
0	83,3±15,1	0	-	0	57,7±13,4	0
0,02	67,2	1,5	-6,9	0,2	54,0	1,1
0,04	59,9	2,7	-5,0	0,03	50,4	2,2
0,06	58,2±15,3	3,9±0,9	-6,0±4,0	0,4±0,1	46,8±13,5	3,1±0,9
0,08	59,4	5,1	-8,4	0,6	43,4	4,0
0,10	61,4	6,3	-11,2	0,8	40,2	4,8
0,12	62,2±15,7	7,5±1,8	-13,2±4,5	1,0±0,4	37,1±13,4	5,6±1,7
0,14	60,1	8,7	-13,6	1,3	34,2	6,3
0,16	53,8	9,9	-11,7	1,6	31,5	6,9

**Fig. 2.** Isoenthalpies of mixing of melts of the Fe-W-C system, calculated using the “analytical” Redlich–Kister–Mujianu model

Studies of phase equilibria are key to understanding the behavior of materials and alloys at different temperatures. However, despite their significant applied importance, the liquidus and solidus surfaces of the Fe–W–C system have not been studied due to the very high smelting temperatures of both Fe, W, C, and their compounds. Therefore, thermodynamic modeling is an important tool for studying such phase diagrams. An important aspect is the modeling of the energy characteristics of phase transitions, which allows predicting the behavior of systems when changing composition and temperature. To calculate the liquidus and solidus surfaces (including multidimensional ones), software components were created in Python, using the PyCALPHAD library tools and thermodynamic databases. According to CALPHAD calculations of phase equilibria in the temperature range of 500–2000 K for the composition 43% Fe 43% W, 14% C (at.%), an intermediate phase Fe_3W_3C is formed, which melts incongruently at 1770 K. The images of the liquidus and solidus projections of the Fe–W–C system obtained from the calculation data are shown in Figure 3.

**Fig. 3.** Projections of the liquidus and solidus surfaces of the Fe–W–C system according to CALPHAD calculations: a) liquidus; b) solidus

On the liquidus surface, the W–C-enriched region has clear eutectic depressions and maxima due to the formation of stable tungsten carbides. Approaching the iron angle smoothes these features, signaling a transition to iron-based crystallization, and a zone of reduced liquidus temperatures extends inward from the Fe

angle. In this case, the Fe_3W_3C compound melts incongruently and affects the topology indirectly through adjacent phase equilibria. The solidus surface is relatively smooth in the carbon-rich corner, but along the W–Fe direction it has steps and discontinuities, successive solid-phase transformations involving carbide phases.

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Mechanical Properties of Dissimilar Welded Joints Between Duplex Stainless Steel and Structural Steel

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Abstract: Dissimilar welding between duplex stainless steel and structural steel is increasingly used in the offshore, petrochemical and energy industries due to the combination of high corrosion resistance and good mechanical strength. This study presents a comparative investigation of two welding procedures for which they were used as filler metal: a BÖHLER Q G 309L-Si solid wire (GMAW 135 process), a FOX core 2209-T1 flux-cored wire (FCAW 136 process) and M21 shielding gas (82% Ar + 18% CO₂). The butt joint was performed between 10mm thick plates made of duplex stainless steel UNS S32205 and structural steel S355J2. During tensile testing, failure occurred in the base material, confirming the high strength and good quality of the welded joints. Charpy impact testing revealed high toughness in the weld area for the GMAW 135 process, while excellent toughness was obtained for the GMAW 136 process. Hardness measurements showed a more uniform hardness distribution for the flux-cored wire welds and macrostructural analysis confirmed full penetration of both welds.

Keywords: MECHANICAL PROPERTIES, DISSIMILAR WELDING, DUPLEX STAINLESS STEEL, CARBON STEEL

1. Introduction

Dissimilar welded joints are increasingly used in offshore structures, pressure vessels, petrochemical plants and energy systems, where the combination of corrosion-resistant materials and high-strength structural steels provides both economic and technical advantages [1-2], [3]. One of the most common combinations is duplex stainless steel UNS S32205 and structural steel S355J2. Duplex stainless steel UNS S32205 is a widely used engineering material characterized by a dual-phase microstructure consisting of approximately equal proportions of ferrite and austenite. This combination provides high mechanical strength, excellent resistance to stress corrosion cracking and superior corrosion resistance compared to conventional austenitic stainless steels [4-5]. In these steels, it is desired to maintain the phase proportion of approximately 40% to 60% ferrite, with the remainder being austenite, by adding austenite stabilizers, such as Ni, C, and Mn, in balance with ferritizing elements, such as Cr and Mo.

The effects of nitrogen on stainless steels are multiple, promoting both increased corrosion resistance (extension of the passive potential) and austenite content or mechanical strength. The microstructure in the heat-affected zone during welding is influenced by the thermal regime through the heat input in relation to the material thickness, the preheating temperature or the interlayer temperature. In other words, the maximum value of the exposure temperature controls the proportion of dissolved austenite and the diameter of the ferrite grains. The increase in the size of the ferritic grains has the effect of delaying the formation of austenite and, therefore, disturbing the phase equilibrium in the steel [6-8].

Structural steel S355J2 is commonly employed in welded constructions due to its good weldability, adequate mechanical properties and economic advantages. The combination of S32205 duplex stainless steel and S355J2 structural steel is frequently used in offshore, petrochemical and energy applications where both corrosion resistance and structural strength are required [2, 5-6].

The welding of these dissimilar materials presents significant metallurgical challenges because of differences in chemical composition, thermal expansion coefficient and phase transformation behaviour during welding thermal cycles [2], [5]. Improper control of welding parameters may lead to undesirable microstructural changes, residual stresses or deterioration of mechanical properties [1], [9].

The selection of filler material and welding process plays a crucial role in achieving sound dissimilar joints with adequate mechanical and metallurgical properties [9]. Gas Metal Arc Welding (GMAW) is widely employed in industrial applications because of its high productivity, good deposition rates and versatility [2].

Among the various GMAW variants, process 135 utilizes solid wire electrodes, while process FCAW employs flux-cored wire

consumables, each providing different thermal and metallurgical conditions during welding. Several studies have demonstrated that welding parameters, filler metal composition and heat input significantly influence the microstructure, toughness, hardness distribution and overall performance of duplex stainless-steel weldments [1],[5]. However, comparative data regarding the behaviour of dissimilar joints between S32205 duplex stainless steel and S355J2 structural steel produced using GMAW and FCAW processes remain limited.

The objective of this study is to perform a comparative evaluation of the mechanical and metallurgical behaviour of dissimilar welded joints produced using GMAW processes. The investigation has included non-destructive testing, tensile testing, bend testing, Charpy impact testing, Vickers hardness measurements and macrostructural examination to identify the most suitable welding procedure for industrial applications involving dissimilar materials.

2. Materials and Methods

2.1. Materials

In the experimental study two dissimilar welding joints have been performed between duplex stainless steel UNS S32205 and structural steel S355J2 plates, with 10 mm thickness.

The chemical composition of S32205 steel contains (wt.%): 0.03 C; 2 Mn; 1 Si; 21 – 23 Cr; 2.5 – 3.5 Mo; 4.5 – 6.5 Ni; 0.08 – 0.2 N. Based on this chemical composition, a PREN corrosion index value of 34 results, which is an average value in the duplex steel class. It has a tensile strength of 755 MPa, the yield strength of 582 MPa, Rockwell hardness HRC of 28 and elongation of 35%.

The chemical composition of S 355 J2 is (wt.%): 0.15 C; 1.38 Mn; 0.1 Si; 0.1Cr; 0.04Al; 0.1Ni. The main mechanical properties of this steel are: Tensile strength of 550 MPa, yield strength of 405 MPa, Brinell hardness of 170HB and elongation of 23%.

From the analysis of the property values of the steels that were welded, it is observed that problems can arise due to the diffusion of carbon from S 355 J2 towards the weld and the failure of the joint in the component made of this steel. The samples were machined for “V” butt –bevel shape, having an angle of 60°, depth of root face of 1.5 to 2mm, root gap of 2 to 4 mm (Fig. 1) [10]. Before welding, a layer of 309L-Si stainless steel was cladded on the joint area of the carbon steel part. The use of this filler material over the entire section of the joint required complex management of the rutile slag layer, a factor that decisively influenced the volumetric integrity of the weld, as observed during radiological control (Fig. 1b).

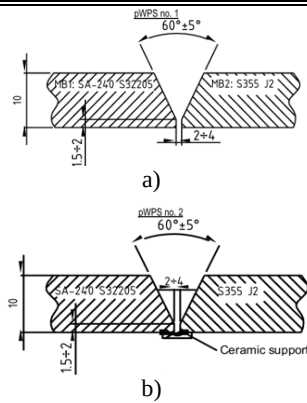


Fig. 1. Joint geometry and layers configuration for welded samples: a) GMAW 135 process; b) FCAW 136 process [9].

In both welding processes, shielding gas type M21 (18 %CO₂ and 82% Ar) was used. To prevent oxidation of the root weld of the sample welded with solid wire, root protection was performed using a separate cylinder with Argon (I1) shielding gas with a purity of 99.996%, that is the specific method applied in the case of stainless steels welding. To make the sample welded with tubular wire, the root was protected with a ceramic support. Two Gas Metal Arc Welding (GMAW) procedures were investigated in the study.

Welding process GMAW 135 has been performed using a solid wire electrode (BÖHLER Q G 309L-Si), while FCAW (Flux-Cored Arc Welding) 136 process utilized a flux-cored wire electrode (FOX core 2209-T1), both of them having 1.2mm diameter. BÖHLER Q G 309L-Si wire has an austenitic steel with the chemical composition of (wt.%): 0.02C; 0.9 Si; 1.6 Mn; 23.1 Cr; 0.1 Mo; 13.3 Ni; 0.1 Cu. FOX core 2209-T1 cored wire is projected for welding of duplex steels, with the chemical composition of (wt.%): 0.03C; 0.69 Si; 0.96 Mn; 23.25 Cr; 3.05Mo; 8.97 Ni; 0.09 Cu; 0.14 N.

2.2. Methods

Welding was performed with LINCOLN ELECTRIC SPEEDTEC 405 SP inverter multi-process equipment, equipped with advanced arc monitoring functions. The preliminary Welding Procedure Specification documentation (pWPS) was developed in accordance with the requirements of the standard SR EN ISO 15609-1[11].

The influence of the welding process on the mechanical behaviour, hardness distribution and structural integrity of the welded joints was subsequently evaluated through destructive and non-destructive testing methods [11-18].

The welding parameters (presented in Table 1) and the interlayer temperature were monitored during the execution of both welding procedures. Technological parameters such as the welding regime, the filler materials used, the type of shielding gas, as well as the thermal conditions for making the joint were defined for each technology. The temperature was monitored throughout the welding process to ensure that the maximum interlayer temperature of 150°C was not exceeded. To counteract the effect of thermal stresses and avoid angular deformations, the two plates were positioned and rigidly fixed on a metal alignment support (Fig. 2).

Table 1. Welding parameters

Layer	Current, A	Voltage, V	Travel speed, cm/min	Heat input, kJ/mm
Sample 1				
Root	110	16	24	0.40
L2	160	24	28	0.73
L3	180	26	30	0.80
Sample 2				
Root	130	22	25	0.69
L2	150	24	34	0.67
L3	170	25	22	0.98
L4	180	26	40	0.59

In this way, the start and completion of the weld could be done outside the welding area, thus avoiding the appearance of specific imperfections when starting and ending the electric arc burning process.

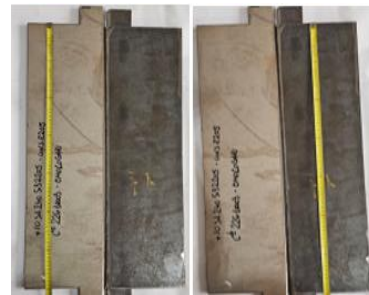


Fig. 2. The samples prepared for welding.

Upon visual inspection [12], both welded samples presented a satisfactory appearance after removal of welding spatter. The surface of the weld was clean, with regular geometry and uniform deposition of the filler material. When checking with penetrant liquids [13], it was observed that the welded samples had a compact surface, lacking relevant indications according to the acceptability criteria for quality level B (Fig. 3).

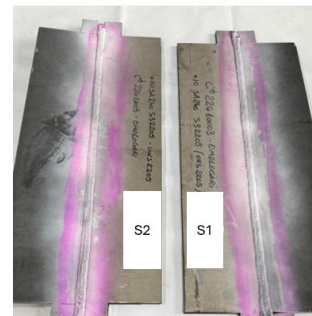


Fig. 3. Overview of welded samples after completion of liquid penetrant inspection.

Following the radiographic examination (RT), the solid wire welded sample did not present any unacceptable defects according to the criteria specified in the applicable standards [14].

The radiographic image obtained revealed a homogeneous structure of the weld, without major discontinuities such as cracks, inclusions or lack of penetration beyond the permitted limits, the sample being declared acceptable. In the case of the sample 2, welded with tubular wire, non-conforming internal discontinuities were highlighted, which required excavation and re-welding operations of the defective areas. The microstructure analysis was carried out in accordance with the requirements of the standard [15], using selective chemical attack to highlight the geometry and heat-affected zones. The Tensile tests and bending tests have been

performed using Universal tensile test machine OPTIMUM MB4P (12 t) [16] and bending tests have been performed using a hydraulic press (30 t) with bending rollers (diameter of 40mm), to verify ductility and inter-layer fusion [17]. Charpy impact tests were carried out at a temperature of -20 °C and the dimensions of the samples were 10x10x55mm, using a Wolpert PW 30/15 pendulum [18].

The measurement of hardness was performed by the Vickers method (HV0.5), according to the SR EN ISO 6507-1:2023 standard [19], using Shimadzu HMV 2T apparatus, for the evaluation of the local variation of mechanical properties in the characteristic areas of heterogeneous welded joints: base metal (S355 J2 and Duplex 2205), heat-affected zone (HAZ) and deposited metal. The measurements were performed using a force of 4.9 N (HV0.5) and a holding time of 10 s, according to the standardized methodology.

3. Results and discussion

Representative specimens were cut from the welded joints, processed according to international standards, for the following determinations:

between the joined materials.

Table 2. Comparative results of transverse tensile tests

Sample	Notch location	KV values, J			Average KV, J
Sample 1	Weld	122.5	100	112.5	112
	FL-CS	90	90	80	87
	FL-SS	122.5	92.5	147.5	121
Sample 2	Weld	72.5	75	62.5	70
	FL-CS	70	115	77.5	88
	FL-SS	182.5	122.5	147.5	151

FL-CS: fusion line from Carbon Steel; FL-SS: fusion line from Stainless Steel.

Table 3. Impact energy values (KV) obtained for samples of standard size

Sample	Width, mm	Thickness, mm	Surface, mm ²	Force, N	Rm, MPa	Failure zone	Result
T1.1	11.26	9.92	111.70	69.18	619.34	Base metal	Approved
T1.2	11.62	9.95	115.62	69.99	605.35	Base metal	Approved
T1.3	11.09	9.78	108.46	66.68	614.79	Base metal	Approved
T2.1	11.73	9.97	116.95	71.54	611.71	Base metal	Approved
T2.2	12.08	9.86	119.11	74.05	621.69	Base metal	Approved
T2.3	11.83	9.88	116.88	71.77	614.05	Base metal	Approved

Regarding the impact resistance test results, as shown in Table 3, it is observed that the fracture energy values are higher in the case of welded sample 2, especially in the fusion line with the stainless steel. However, the fracture energy values are lower in the welded interface with the structural steel. However, the lowest values of breaking energy were recorded in the weld made with tubular wire (sample 2), of maximum 75J, while in the sample welded with solid wire (sample 1) it was of minimum 87J.

The preparation of the specimens followed a rigorous protocol, using high-grain finishing equipment available in the laboratory to prevent alteration of the two-phase microstructure of the duplex steel. Macroscopic analysis of the fracture surfaces for both series of samples was carried out. As can be seen in the macroscopic analysis of sample 1 (Fig. 4a), it shows complete penetration and good fusion at both interfaces (duplex and carbon steel), from which it is found that it does not present macroscopic defects.

- 3 specimens from each sample were subjected to transverse tensile testing, to determine the ultimate strength (Rm) and tensile strength (Rp). The values obtained were targeted in the range of 470–630 MPa, to ensure compatibility with the strength limits of the base materials joined [4] (Table 2).
- 4 specimens taken from the samples for transverse bending tests: 2 specimens for root bending (TRBB) and 2 for face bending (TFBB).
- 3 sets of specimens for the impact bending tests (weld, duplex base material heat-affected zone and carbon base material heat-affected zone) (Table 3).
- one specimen from each welded sample for macroscopic examination (fig. 4).

In the case of Sample 1 (welded using solid wire electrode) most of the specimens showed lateral plastic deformations and a fibrous-looking fracture, suggesting high toughness.

For Sample 2 (welded using flux-cored wire electrode), the fracture surfaces indicated a mixed behaviour, highlighting the influence of the flux in the cored wire on the crystallinity of the weld metal.

In the case of bending tests, the good results are associated with the failure of the S355 J2 base material, without affecting the weld itself, thus highlighting a difference in mechanical behavior

Macroscopic analysis of sample 2 (Fig. 4b) highlights the stability of the cored wire welding process.

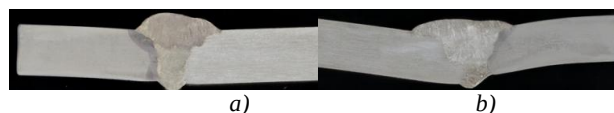


Fig. 4. Cross-sections through the welded samples.
a) sample 1; b) sample 2.

A uniform and well-defined morphology of the weld beads is noted, as well as a metallurgical continuity without discontinuities (Fig. 5a). No internal defects were identified, but an axial rotational deformation occurred on the weld axis during the operation to remedy internal defects detected during the X-ray penetration test. In the case of sample welded using flux-cored wire electrode, the appearance of areas of partial mixing between the 2 alloys (filler

metal and S 355J2) as well as dispersed spherical inclusions is observed (Fig. 5b). The hardness distribution shows high values in the duplex base metal (331–333 HV0.5) and lower values in the S355 J2 carbon steel (212–214 HV0.5), corresponding to the differences in microstructure.

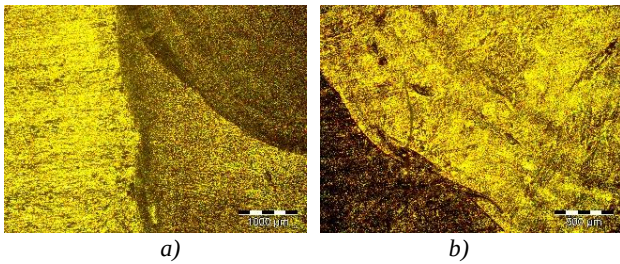


Fig. 5. Optical microstructure of cross section on fusion line. a) sample 1; b) sample 2.

In the Heat-Affected Zone (HAZ) of the S355 steel, an increase in hardness (265–270 HV0.5) is observed, determined by the microstructural transformations induced by the welding thermal cycle. The HAZ of the duplex steel shows high values (318–319 HV0.5), indicating the maintenance of the two-phase structure. The weld metal shows intermediate values (203–233 HV0.5), characteristic of a predominantly austenitic structure, which leads to a difference in hardness compared to the duplex steel. In the case of sample 2, the hardness values for the base metal remain similar to those of sample welded using solid wire electrode, but significant differences are observed in the deposited metal area.

The weld metal shows high values (351–355 HV0.5), close to those of duplex steel, indicating superior mechanical compatibility. This is determined using duplex filler material. The heat-affected zone of the carbon steel shows lower values (225 HV0.5), indicating a different influence of the thermal cycle. Sample welded using flux-cored wire electrode presents a more uniform distribution of hardness and a more gradual transition between zones, which indicates a superior mechanical compatibility. The results are consistent with the literature on the influence of the filler material on the mechanical properties of dissimilar welded joints [5–8].

4. Conclusion

The analysis of the mechanical test reports highlights that both samples meet the acceptance criteria for the tensile and impact tests, confirming good mechanical behavior of the dissimilar welded joints. For sample 1, all tensile specimens were accepted, and the rupture occurred in the base material, which indicates a superior resistance of the weld bead. In the case of bending tests, the “failure” results are associated with the failure of the S355 J2 base material, without affecting the weld itself, thus highlighting a difference in mechanical behavior between the joined materials. For sample 2, the results indicate a mixed behavior in bending, with specimens accepted on the weld face and rejected on the root, which highlights a different stress distribution and a more pronounced influence of technological parameters on the quality of the joint. The high fracture energy values (121 and 151 J in fusion line area) confirm an excellent toughness of the joint, especially at the interface with stainless steel, being in line with the microstructural observations on the metallurgical compatibility.

The experimental results highlight good compatibility near the fusion line with the duplex steel and a stable dendritic structure in the weld area. For sample welded using flux-cored wire electrode, the results indicate a mixed behavior in bending, with specimens accepted on the weld face and rejected on the root, which highlights a different stress distribution and a more pronounced influence of technological parameters on the quality of the joint.

The high fracture energy values (up to 150 J in the duplex area) confirm an excellent toughness of the joint, especially at the interface with stainless steel, being in line with the microstructural observations on the metallurgical compatibility.

The microstructure shows good compatibility with the fusion line at interface with duplex steel and a stable dendritic structure in the weld area. The Vickers hardness analysis highlights a differentiated distribution of mechanical properties between the characteristic areas of the joint, sample welded using flux-cored wire electrode presenting a higher homogeneity and hardness values closer to the duplex steel, which indicates a superior mechanical compatibility of the weld metal.

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Effect of carbide-forming components on phase and structure formation in the Fe-Ga system and corrosion properties of the obtained materials

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Abstract The high-temperature self-propagating synthesis (SHS) of carbides and borides in powder systems (30, 50, 70) wt.% (Fe-Ga)-(Ti-C) and (30, 50, 70) wt.% (Fe-Ga)-(Ti-B₄C) was investigated at a temperature of 1200 °C in a neutral argon atmosphere. It was established that the introduction of a master alloy (Fe-55 wt.% Ga) into the initial mixture intensifies the SHS process. The appearance of a liquid phase at lower temperatures in gallium-containing systems is likely one of the main reasons for the activation of transport mechanisms for mass transfer to the chemical reaction front. Corrosion tests in a simulated seawater environment (3% NaCl solution) showed that simultaneous alloying of iron with gallium and boron significantly increases the corrosion resistance of iron-based materials. Specifically, the material containing 30 wt.% (Fe-Ga)-(Ti-B₄C) demonstrated high protection (ASTM G-1, Good) in an environment close to seawater.

KEYWORDS: FE-GA SYSTEM, POWDER GALFENOLS, SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS (SHS), TITANIUM CARBIDE, TITANIUM BORIDE.

1. Introduction

In the line of the latest smart materials, which have been actively developed and used in the last two or three decades, iron-based materials still occupy a significant number of positions due to the unique properties that are added to them by doping with components that are “untraditional” for them, in particular, such as rare-earth metals or gallium [1]. In particular, magnetostrictive materials of the Fe-Ga system, the so-called galfenols, have become an alternative to alloys based on rare-earth metals of the composition (e.g., Terfenol-D: Tb_{0.3}Dy_{0.7}Fe₂), which, in addition to their high cost and limited availability, are also characterized by a low set of mechanical characteristics (e.g., the tensile strength of Terfenol-D is about 27 MPa). Galfenols are obtained mainly by casting methods followed by cold and hot rolling [2]. From a technical point of view, rolling cast Fe-Ga alloys is quite difficult due to their tendency to brittle fracture during deformation even at elevated temperatures [2]. In the last decade, scientific and technical proposals have appeared on the use of powder galfenols and composite materials containing them using powder metallurgy methods [3, 4]. This allows avoiding problems arising during thermomechanical processing of cast alloys and expanding the scope of application of galfenols in particular, for three-dimensional printing, or fillers of composite materials for various functional purposes. Currently, galfenols work effectively in the field of hydroacoustic, in particular in surface and underwater communication devices, but their corrosion resistance and the influence of alloying impurities on this property remain insufficiently studied. There are separate publications in the literature on the positive effect of elements C, B, and some carbides on the magnetic and mechanical characteristics of single-crystal galfenols [5, 6], which makes them promising alloying additives for galfenols. At the same time, it is known that a powerful strengthening effect in iron-based materials can be created by titanium carbides and borides (TiC and TiB₂). In addition, the introduction of these compounds into the iron-containing matrix of the powder material under certain conditions can increase its corrosion resistance. The electrochemical type of corrosion is a very common phenomenon during the operation of various types of products, and in the case of using multi-component materials, it can be characterized by a complex multi-stage process of their gradual or rapid destruction.

In this regard, the work conducted studies of the thermal synthesis of titanium carbides and borides in-situ during the sintering of powder mixtures of the systems (Fe-Ga)-(Ti-C) and Fe-Ga-(Ti-B₄C) depending on the ratio of iron-containing and reactive components, and determination of the corrosion resistance of the obtained materials in a salt solution that simulates sea water.

2. Thermodynamic conditions of thermal synthesis of titanium carbides and borides in systems with iron

High-temperature self-propagating synthesis (SHS) is a typical mechanism of the titanium carbide reaction, which is characterized by significant heat release ($Q = 3480$ kJ/kg, $T_{ad} = 3290$ K). However, the SHS process in a mixture of elementary powders depends on many factors and is a process that is difficult to control [7]. Therefore, to determine the optimal temperature conditions for the formation of carbides and borides in the Fe-Ti-C(B) system, it makes sense to assess the thermodynamic probability of the thermal synthesis of titanium carbides and borides in a system with iron under experimental conditions. Calculations based on the temperature dependences of partial pressures and activities of Ti and C for titanium carbides of different stoichiometry, carried out according to the method [7], showed that in the temperature range of 1100-1500 °C (1173-1773 K), the stoichiometric carbide TC is characterized by the most negative values of ΔG^0_T , which indicates a high probability of its formation in the Ti-C system (Fig. 1).

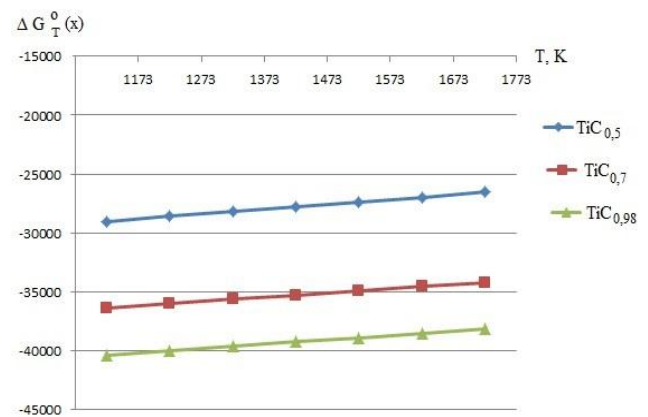


Fig1. Temperature dependence of ΔG^0_T for titanium carbides of various stoichiometry.

Unlike binary systems, which are defined in detail in a wide temperature range, the ternary system Fe-Ti-C(B) are less studied from the point of view of phase equilibria. Increase in the carbon concentration in the system leads to the formation of cementite (Fe₃C), and excess free carbon is not observed. The quasi-binary Fe-Ti-C intersection is a diagram of a simple eutectic type with the reaction $L(\text{liquid}) = \text{Fe} + \text{TiC}$ at 1450 °C [8]. A comparative analysis of all possible reactions between the components in the Fe-Ti-B₄C system showed that among all possible reactions, the most probable is the reaction $3/4\text{Ti} + 1/4\text{B}_4\text{C} = 1/2\text{TiB}_2 + 1/4\text{TiC}$, since ΔG for it has the largest modulus value. In addition, with high probability, the reactions

$2/3\text{FeTi}+1/4\text{B}_4\text{C} = 3/4\text{Fe}+1/2\text{TiB}_2+1/4\text{TiC}$ and $2/3\text{Fe}_2\text{Ti}+1/4\text{B}_4\text{C} = 3/2\text{Fe}+1/2\text{TiB}_2+1/4\text{TiC}$ are possible, i.e. the interaction of intermetallics FeTi, Fe_2Ti and pure Ti with B_4C with the formation of borides TiB_2 and TiB , and titanium carbide TiC . The obtained data are consistent with the results of calculations carried out in [9], where the features of phase and structure formation in the Ti-B-Al (N,C) systems were studied. The authors showed that there is a high probability of the formation of titanium boride TiB in multicomponent systems, through the formation of intermediate boride Ti_2B . The quaternary systems Fe-Ga-(Ti-C(B)) have not yet been studied, and therefore their behavior cannot be predicted. It can only be assumed that since Ga does not chemically interact with carbon, in the structure of the materials it will be contained in the matrix iron-containing phase within the limits of its solubility in the bcc lattice of Fe, and its excess will form, first of all, intermetallic phases of the composition Fe_6Ga_5 and Fe_3Ga_4 [10]. However, it is not excluded that in materials of the systems Fe-Ti-C and Fe-Ti- B_4C , when doping them with Ga, an additional factor stimulating the processes of thermal synthesis is a decrease in the temperature of the liquid phase and an increase in its volume during the thermal synthesis of carbides.

3. Starting materials

The starting materials used were iron powder (99.95% wt. Fe) and titanium (99.8% wt. Ti) after screening at $<100\text{ }\mu\text{m}$, graphite powders (size $<63\text{ }\mu\text{m}$ not less than 65 wt. %), Ti and B_4C (Zaporizhzhia Abrasive Plant, Ukraine), Ga-ingots (99.98% wt. Ga), was previously crushed. The technological scheme provided for obtaining a ligature of composition Fe-45% wt. Ga and mixing it with iron powder in a proportion corresponding to the composition Fe-21,5% wt. Ga. The production of ligature alloy is a necessary stage of obtaining samples, since obtaining a uniform mixture of components directly from iron and gallium powder causes significant technological difficulties due to the low melting point of gallium ($\sim 29\text{ }^\circ\text{C}$). Thermal synthesis was carried out in the temperature range of $1200\text{--}1250\text{ }^\circ\text{C}$ in an electric resistance furnace, using a container filled with ingredients that form a glassy layer on the surface during sintering, thereby preventing oxidation of the mixture components inside the container.

4. Influence of gallium on phase and structure formation in the Fe-Ti-C(B) systems

For experimental studies of the possibilities of thermal synthesis of carbides and borides in the Fe-Ga system, 2 batches of mixtures were prepared, in which the reactive component of the initial charge contained (Ti-C) or $(\text{TiH}_2\text{-B}_4\text{C})$. In each batch, the proportion of the ligature alloy (Fe-Ga) was 30, 50 and 70 wt. %. The ratios of components in the reaction component were constant, determined from the calculation of the complete passage of the reactions $\text{Ti}+\text{C}\rightarrow\text{TiC}$ and $\text{Ti}+\text{B}_4\text{C}\rightarrow\text{TiB}_2+\text{TiC}$ and were 80Ti:20C and 72,2Ti:27,8 B_4C , respectively. Samples for research were obtained by pressing the charge into cylindrical blanks, which were then sintered. Obtaining carbides and borides in the Fe-Ga material structure was assumed by thermal synthesis at a temperature of $1200\text{ }^\circ\text{C}$ in a pure Ar environment. The heating rate was 40 degrees/min for 2 hours, the duration of isothermal holding at the synthesis temperature was 2 hours, cooling was carried out together with the furnace. For metallographic analysis of the structure of the samples, sections were made in a standard way, which were studied by optical and scanning electron microscopy methods using The presence of Ga in the system may become an additional factor that can activate diffusion processes in Fe-Ti-C(B) materials, due to the formation of low-melting phases, which significantly accelerate the processes of substance transport to the reaction front. To test this hypothesis, differential-thermal analysis (DTA) of the Ti-Fe and Ti-Fe-Ga systems was performed. Mixtures of compositions were

investigated, in which the mass ratio of Ti to Fe powders and Ti to the Fe-Ga alloy was the same and was 43:57, which corresponds to the atomic composition of 46Ti-54Fe and 49Ti-24Fe-27Ga, respectively. DTA of the mixtures was performed at a heating rate of 20 and $40\text{ }^\circ\text{C}/\text{min}$, in the temperature range of $20\text{--}1270\text{ }^\circ\text{C}$ in a stream of pure argon. The DTA results are presented in Fig. 2.

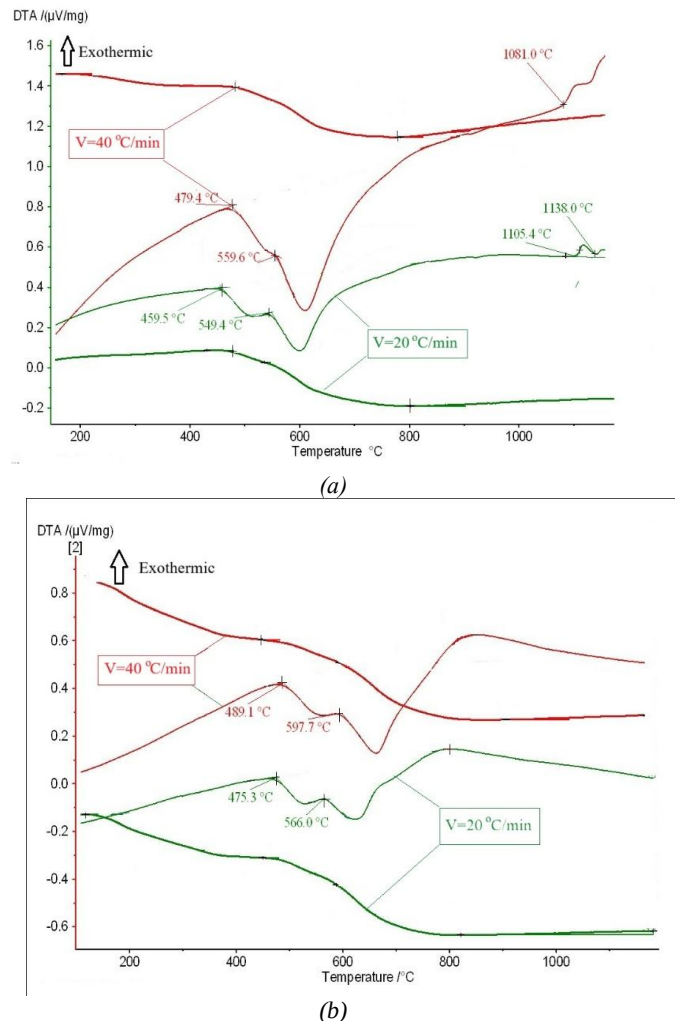


Fig. 2. DTA curves of the mixtures: 46 % wt. Ti-Fe (a) and 49 % wt. Ti-24Fe-27Ga (b).

The obtained data indicate that in the studied temperature range in both systems, structural changes are observed, causing significant thermal effects, and the rate of temperature change within $20\text{--}40\text{ deg/min}$ does not significantly affect their position relative to the temperature scale. When heating both mixtures, the first endothermic peak is observed in the temperature range of $600\text{ }^\circ\text{C}$, which in the equilibrium Ti-Fe system corresponds to the reaction of the eutectoid transformation: $\alpha\text{Ti}+\text{FeTi}\leftrightarrow\beta\text{-Ti}$. The fixation of the eutectoid reaction with the formation of a $\beta\text{-Ti}$ solution from two phases – the intermetallic FeTi and $\alpha\text{-Ti}$, shows that in both cases the formation of the intermetallic FeTi takes place in the solid phase during heating to the specified temperature. The next phase transformation in the sample of the dual composition is recorded at a temperature of $1080\text{--}1100\text{ }^\circ\text{C}$ and corresponds to the eutectic reaction: $\text{FeTi}+\beta\text{-Ti}\leftrightarrow\text{L}$. The difference in the behavior of the system in the presence of Ga begins to appear at a temperature of $800\text{ }^\circ\text{C}$. At higher temperatures, an endothermic peak is observed in the Ga system, which probably accompanies the appearance of a liquid phase in significant quantities. Thus, it can be concluded that in iron-based systems, when doped with gallium, thermal synthesis of titanium carbides can be intensified due to the activation of

diffusion processes and changes in the chemical potential of carbide components at the interface with the melt.

The obtained materials had the appearance of a sponge with a developed coarse-porous structure, which is quite typical for materials obtained by HPS-mechanism. According to the X-ray diffraction data, the structure of the samples consists of an iron-based matrix phase in which the products of thermal synthesis are dispersed - carbides and intermetallics, which have a micron and submicron size (Fig. 3). It is interesting to note that gallium is mainly concentrated in a solid solution based on iron within the limits of its solubility in the Fe-Ga binary system (8...12 wt.%), and in systems with its excess - in the composition of Fe_3Ga_4 inclusions. An increase in the proportion of the reactive component of thermal synthesis contributes to its activation, as a result of which the porosity in the sintered samples increases, and the pores themselves become spheroidized, and the crystalline inclusions slightly increase in size. Among the crystalline inclusions, titanium carbide of variable composition (TiC_x , $x=0.4...0.9$) prevails, in addition, FeTi and cementite (Fe_3C) appear in the structure. The structure of materials obtained from the charge with boron carbide is similar, while boron forms intermetallics with iron. We have established that in thermally synthesized materials of the Fe-(Ti-B₄C) system, boron is contained mainly in borocementite $\text{Fe}_3(\text{B,C})$ [11].

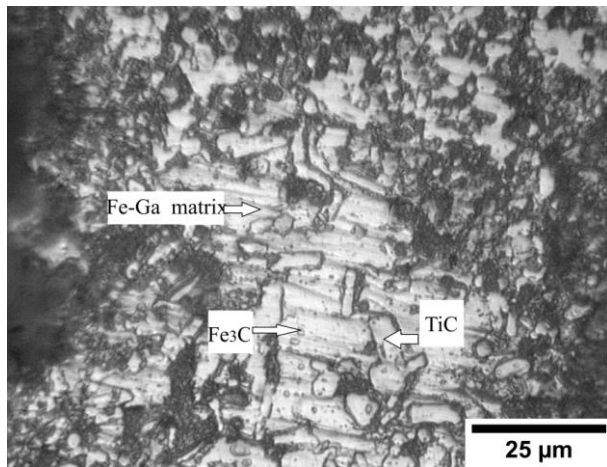


Fig. 3 Microstructure of sintered samples with the composition 30(Fe-Ga) – (Ti-C).

5. Corrosion tests and their discussion

Corrosion tests of materials (Fe-Ga)-(Ti-C(B)) were carried out in a saline solution of water (3% NaCl, $\text{pH} \approx 7$), which simulates the marine environment. For comparison, samples from materials of the Fe-(Ti-C) and Fe-(Ti-B₄C) systems with similar ratios of reactive to iron-containing components in the initial charge, which were obtained using the same technology as the samples of the research batches, participated in the corrosion tests. In general, the tests lasted for 121 h, during which the samples were periodically immersed in solutions for 30 min, followed by removal and exposure in air for 15 min. It is believed that such a regime prevents the development of stagnant phenomena at the interface of the surface of the samples with the solution, which inhibit oxidation processes. The temperature of the solution was 40 ± 5 °C, air 38 ± 5 °C. After the tests, the samples were visually inspected to determine the nature of the corrosion, and then dried and weighed. Then the samples were subjected to mechanical cleaning to remove the remains of corrosion products on their surface, followed by ultrasonic treatment in a solution of ethyl alcohol for 3-5 min, and drying in a vacuum drying oven for 30 min, and again weighed. The quantitative assessment of the material mass loss during corrosion was calculated using the corrosion index $K = (M_0 - M_1)/(S \cdot \tau)$, M_0 and M_1 are the mass of the sample before corrosion

tests and at their end, after cleaning the corrosion products from the surface of the material; S is the surface area of the sample; τ - time) and $D = 8.760 K / d$ (d - density). The corrosion index was assessed taking into account changes in the corrosion reaction area of porous samples. For this purpose, the parameter $S(\text{app})$ was used, which differed from the geometrically determined S (imaginary area), and was determined according to the results of quantitative metallography of sections, using data on the distribution of pores by size (Fere diameter), grouped into 3 main fractions. According to the obtained data, the studied materials, in terms of corrosion resistance in salt solution, belong to the groups of reduced and low-resistant (Fair and Poor) and corrosion-resistant (Good) (see table below).

Table. Corrosion properties of materials and qualitative assessment of corrosion resistance (according to ASTM G-1)

Composition, wt. %	K , $\text{g/m}^2 \cdot \text{h}$	D , mm/year	by ASTM G-1
70Fe-(Ti-C)	0,16	0,24	Fair
70Fe-(Ti-B ₄ C)	1,23	1,70	Poor
70(Fe-Ga)-(Ti-C)	0,9	1,2	Poor
70(Fe-Ga)-(Ti-B ₄ C)	0,63	0,84	Fair
50Fe-(Ti-C)	0,60	1,16	Poor
50Fe-(Ti-B ₄ C)	0,63	0,88	Fair
50(Fe-Ga)-(Ti-C)	0,42	0,56	Fair
50(Fe-Ga)-(Ti-B ₄ C)	0,10	0,14	Fair
30Fe-(Ti-C)	0,41	0,59	Fair
30Fe-(Ti-B ₄ C)	0,48	0,73	Fair
30(Fe-Ga)-(Ti-C)	0,71	0,98	Fair
30(Fe-Ga)-(Ti-B ₄ C)	0,03	0,05	Good

In general, the materials of the samples are characterized by increased corrosion resistance relative to technical iron and ordinary carbon steels, which are considered to be of low resistance in non-oxidizing salt solutions. The least corrosion-resistant materials were found to be those with the highest iron content (70 wt. % Fe), which is an expected phenomenon, given that iron-containing phases become anodic areas, where the processes of dissolution of the solid phase are actively developing. The introduction of Ga as an alloying agent increases the resistance of the material, moving it from the Poor to Fair group. A possible explanation for this effect is that Ga, according to PCMA, is mainly present in the composition of the iron-based solid solution, thereby shifting its potential towards more negative values, and reducing its solubility in the electrolyte. The maximum increase in corrosion resistance occurs with simultaneous alloying of B and Ga, which is most pronounced in the material of the composition 30(Fe-Ga)-(Ti-B₄C). According to X-ray diffraction data, the main corrosion product of all samples is the molecular complex $\text{Fe}(\text{OH})_3 \cdot n\text{H}_2\text{O}$, where $n=0.0-0.25$. Fe_3O_4 is also present in the deposits, but in a much smaller amount. In addition, the spectra of the corrosion products of samples containing boron contain lines of iron borides and gallium oxide. It should be noted that in the corrosion products of materials of the Fe-(Ti-C(B)) systems, weak peaks from ternary compounds of Fe-carbonyl group were

detected. This is not characteristic of materials containing Ga, which can be explained by the inhibitory effect of this element on the processes of Fe dissolution in the electrolyte. On the other hand, this may be due to the stoichiometry of titanium carbide particles in the structure of the materials. Our studies have established that titanium carbides formed in-situ during sintering of mixtures of the Fe-Ga-Ti-C systems are characterized by an increased carbon content (TiC_x , $x=0.7...0.9$) [in press].

5. Conclusions

Based on thermodynamic calculations, optimal conditions for the thermal synthesis of titanium carbides and borides in iron-containing systems were determined using Fe, Ti, C, or B_4C powder mixtures as starting materials. Experimental studies of the thermal synthesis of carbides and borides in Fe-Ga-(Ti-C) and Fe-Ga-(Ti- B_4C) powder systems were conducted at 1200–1250 °C in a neutral argon atmosphere. It was established that the introduction of a master alloy of equiatomic composition (Fe-55 wt.% Ga) into the initial charge promotes the intensification of synthesis via the SHS mechanism. A decrease in the liquid phase formation temperature to ~800 °C in the Ti-Fe-Ga system, compared to ~1000 °C in the Ti-Fe system (as recorded by DTA and TG), is likely one of the reasons for the activation of mass transport mechanisms to the chemical reaction front.

Corrosion tests of Fe-(Ti-C), Fe-Ga-(Ti- B_4C), (Fe-Ga)-(Ti-C), and (Fe-Ga)-(Ti- B_4C) materials were performed in a simulated seawater environment (3% NaCl solution). It was found that materials with 70Fe-(Ti-C) and 70Fe-(Ti- B_4C) compositions exhibit the lowest corrosion resistance (ASTM G-1, Poor). Alloying iron with gallium improves corrosion resistance, with the maximum increase observed in samples containing 30 wt.% (Fe-Ga)-(Ti- B_4C). This composition corresponds to a high level of protection (ASTM G-1, Good) in an environment approaching the C5-M category for marine conditions.

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Main casting defects at the interface between a cast iron ring insert and an al-si piston

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Abstract: The study identifies the main casting defects at the interface between a cast iron ring insert and an Al-Si piston and determines their principal causes. Industrial investigations of 1000 pistons showed that the Al-Fin process increases Fe content in the AlSi9 alfining alloy, promoting long plate-like β -Al₅FeSi phases and porosity in the joint region. Gas contamination by oxides and hydrogen further weakens the bond. The addition of approximately 0.05 wt.% Mn and intermediate re-refining of the AlSi9 alloy were found to improve the microstructure and reduce porosity, thereby increasing the quality of the insert-piston joint.

KEYWORDS: ALUMINIUM PISTONS, CASTING DEFECTS, IRON-RICH PHASES, POROSITY, AL-FIN PROCESS

1. Introduction

Depending on the type of internal combustion engine, different piston designs are used, consisting of multiple components. One of these is the ring zone, which accommodates the piston rings (usually 1 to 4 in two-stroke engines and 2 to 4 in four-stroke engines). Their functions include sealing the gases generated by combustion of the fuel-air mixture and preventing them from entering the lower part of the piston and the crankcase. To achieve this, the rings must conform to the shape of the cylinder bore over their entire diameter and width [1, 2].

To reinforce the piston-ring operating zone and dissipate heat from the upper part of the piston, the rings are seated in a cast iron insert in which a groove is machined for the appropriate ring. To obtain a sound bond between the Al-Si piston alloy and the cast iron ring inserts, the inserts are subjected to the so-called Al-Fin (alfining) process. This process consists of briefly immersing the insert in molten AlSi9 alloy in order to form a diffusion layer [3, 4]. After cooling, the inserts are placed in a permanent mould and overcast with the piston alloy. Unfortunately, the Al-Fin process causes the iron content and the level of other contaminants in the AlSi9 alloy to increase. These promote casting defects at the insert-piston interface, thereby increasing the proportion of defective products. Until recently, pistons with inserts were used only in heavily loaded diesel engines operating at combustion pressures of 2.5–3.5 MPa, compared with 1.0–1.5 MPa for gasoline engines. Currently, piston inserts are used in all types of internal combustion engines [5, 6]; therefore, identification of defects at the insert-piston interface is of major importance.

2. Prerequisites and means for solving the problem

The aim of the study was to identify the most important casting defects at the diffusion-bonded interface between a cast iron ring insert and a silumin piston casting, and to determine the principal causes of these defects and possible methods of limiting their occurrence.

The defects were identified using pistons for a 1400 cm³ gasoline engine (representing the largest share of orders), based on a population of 1000 randomly selected pistons.

The scope of the study included:

- identification of the main casting defects at the insert-piston interface,
- microstructural examination of the insert-piston joint region,
- identification of potential causes of insufficient adhesion between the insert and the piston,
- proposal of corrective solutions and analysis of their feasibility under industrial conditions.

2.1. Research problem

Statistical data from the Quality Control Department of Federal-Mogul Gorzyce sp. z o.o. indicate that casting defects, particularly those occurring at the insert-piston interface, constitute the most important group of causes of piston rejection. These nonconformities are detected only at the end of the manufacturing process during final piston quality inspection, which generates

additional losses of energy and time and reduces production-process efficiency.

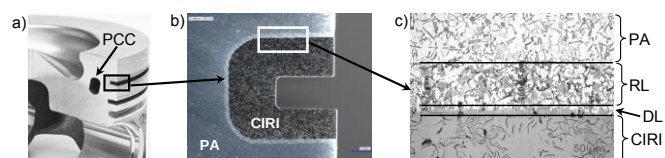
Preliminary investigations showed that these defects result from an improper insert-piston bond within the remelting and diffusion layers. They occur, among other reasons, when the permissible iron content in the AlSi9 alloy is exceeded as a result of:

- diffusion of iron atoms from the cast iron insert into the alfining alloy,
- accidental dropping of an insert into the molten alloy,
- lack of control of the iron content in the alloy during alfining, and
- excessive gaseous contamination of the AlSi9 alloy.

Reducing the formation of defects will lower costs, reduce machine utilisation and material consumption in subsequent production stages (machining and surface treatment of pistons). It is therefore essential to identify the causes of defect formation and implement corrective actions.

2.2. Materials and research methodology

The subject of the investigation was casting defects occurring at the interface between the insert and a gasoline-engine piston cast from AlSi11Cu4Ni3Mg alloy (Fig. 1). Identification of casting defects at the insert-piston interface and assessment of insert adhesion to the piston were carried out using the ultrasonic and penetrant testing methods employed at Federal-Mogul Gorzyce.



where:

- PCC – piston cooling channel,
- PA – AlSi11Cu4Ni3Mg piston alloy,
- RL – remelting layer,
- DL – diffusion layer,
- CIRI – cast iron ring insert.

Fig. 1. Object of the study: (a) fragment of a gasoline-engine piston; (b) insert-piston interface region; (c) correct microstructure of the insert-piston joint (property of Federal-Mogul Gorzyce sp. z o.o.).

According to the Federal-Mogul guidelines (standard TS1E-010-020), the bond between a ring insert made of Ni-Resist cast iron EN-GJLA-XNiCuCr15-6-2 according to TS1E-012-006 (DIN EN 13835:2012) and the piston casting around its circumference is considered unacceptable if at least one of the following conditions is met:

- the RL thickness is less than 70 μm or greater than 400 μm,
- the DL thickness is less than 5 μm or greater than 40 μm,
- the fraction of iron-rich phases in the RL exceeds 10% of the insert circumference,
- the presence of gaseous contamination, cracks, porosity and other defects is unacceptable.

The pistons were examined ultrasonically over the entire circumference of the insert while it was rotated about its own axis. Defects were examined on the upper and lower surfaces of the insert. One hundred percent of the products were subjected to quality-control testing of the insert-piston joint.

3. Results and discussion

Based on the investigations, the main causes of casting defects at the interface between the insert and the piston casting were found to be:

- an Fe content exceeding 0.5 wt.% in the AlSi9 alloy, resulting in the formation of iron-rich phases with an unfavourable morphology,
- gas contamination of the AlSi9 alloy, mainly by hydrides and oxides, causing porosity, and
- other casting defects between the insert and the piston cooling channel (PCC – Fig. 1).

Examples of microstructures of defective insert-piston joints containing unfavourable iron-rich phases are shown in Fig. 2; defects caused by excessive porosity are shown in Fig. 3, whereas other defects, mainly delaminations, are presented in Fig. 4.

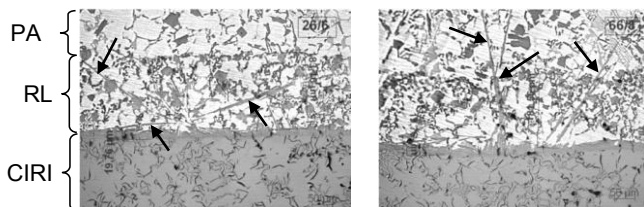


Fig. 2. Microstructures of an improper insert-piston joint containing iron-rich phases in the remelting-layer region (property of Federal-Mogul Gorzyce sp. z o.o.).

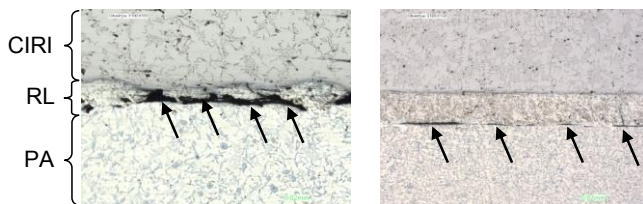


Fig. 3. Microstructures of an improper insert-piston joint showing porosity in the remelting-layer region (property of Federal-Mogul Gorzyce sp. z o.o.).



Fig. 4. Macrostructures of an improper insert-piston joint: (a) shrinkage cavities between the insert (CIRI) and the piston cooling channel (PCC); (b) porosity around the insert (property of Federal-Mogul Gorzyce sp. z o.o.).

3.1. Preventive measures

Microstructural analysis showed that the iron-rich phases present in the remelting layer are Al_5FeSi (the so-called β -Fe phase). To reduce their adverse effect, manganese was added to the AlSi9 alloy in the form of an AlMn50 master alloy (approximately 0.05 wt.% Mn). The literature analysis [7–9] indicates that manganese has a beneficial effect on the plate-like precipitates of the β -Fe phase. It was also found that after 3–4 h of alfining the inserts, the AlSi9 alloy should be subjected again to a refining treatment, for example with argon, after which the alfining process can be continued for a further 3–4 h. Examples of insert-piston joint microstructures after

the addition of manganese to the AlSi9 alloy and subsequent re-refining are shown in Fig. 5.

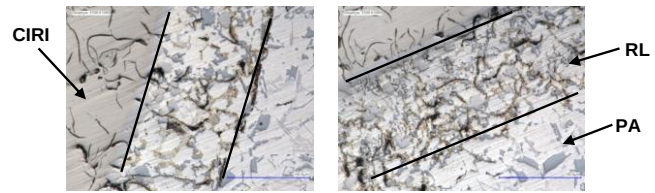


Fig. 5. Microstructures of a correct insert-piston joint after manganese addition and a repeated refining treatment (property of Federal-Mogul Gorzyce sp. z o.o.).

3.2. Discussion

The study was aimed at identifying the principal casting defects in the joint region between a cast iron ring insert and a piston casting made of AlSi11Cu4Ni3Mg alloy. Defects were identified on the basis of microstructural examinations of a population of 1000 randomly selected pistons for a 1400 cm³ gasoline engine. The investigations were carried out under industrial conditions at the Federal-Mogul Gorzyce sp. z o.o. foundry in Gorzyce. The investigations and observations showed that, during piston production, the most problematic operation is the Al-Fin treatment of the ring inserts. During this process, iron atoms from the cast iron inserts diffuse into the AlSi9 alloy, increasing its iron content. Consequently, long (500–800 μm), plate-like Al_5FeSi precipitates crystallise in the joint region between the cast iron ring insert and the piston casting (Fig. 2). These phases cause the insert-piston joint to become unacceptable from a quality standpoint, resulting in a higher scrap rate and associated losses.

Another problem during the Al-Fin treatment of ring inserts is that the process is carried out in open furnaces, which considerably increases the amount of non-metallic inclusions in the AlSi9 alloy, mainly oxides and hydrogen. This results in increased porosity in the insert-piston joint region (Fig. 3). The presence of pores weakens the material, first generating local stresses and then microcracks, which develop into cracks during piston operation. This occurs because long, needle-like β - Al_5FeSi precipitates whose major dimension is oriented perpendicular to the applied load undergo decohesion, whereas β - Al_5FeSi phases aligned parallel to the load crack along their arms and fragment [10]. These factors significantly reduce fatigue strength under long-term loading (more than 10^6 cycles), which is an important parameter for piston castings and contributes to a reduction in the strength of the cast iron ring–Al–Si piston joint.

Porosity between long β - Al_5FeSi phases reduces feeding capability by restricting the flow of liquid alloy between the dendrite arms of the $\alpha(\text{Al})$ solid solution. Such regions act as sites for microcrack initiation, reducing fatigue and static strength, impact toughness and ductility as expressed by elongation. In addition, the size of the needle-like β - Al_5FeSi precipitates, and particularly their highly unfavourable morphology, makes piston machining more difficult. Under the pressure exerted by the turning tool, β -Fe phase precipitates further reduce the durability of the insert-piston joint. Other casting defects were also observed (incorrect thickness of the remelting and diffusion layers, delaminations and shrinkage cavities – Fig. 4); however, their share was small.

To prevent these defects, two improvement measures were proposed:

- addition of manganese to the AlSi9 alloy, and
- an intermediate re-refining treatment of the AlSi9 alloy.

Microstructural examination of the AlSi9 alloy after manganese addition showed that, in addition to a small number of β - Al_5FeSi precipitates, the α - $\text{Al}_{15}(\text{Fe},\text{Mn})_3\text{Si}_2$ phase was present. Its morphology was dendritic or of the so-called “Chinese-script” type. The investigations also showed that an additional refining treatment of the AlSi9 alloy reduces the amount of gaseous contamination. This directly decreases porosity in the interface region between the

cast iron ring insert and the piston, thereby enabling a sound joint to be obtained (Fig. 5).

It should be emphasised that, in addition to the proposed measures aimed at improving the bond between cast iron ring inserts and the piston, further research should be continued, particularly with regard to further reducing iron contamination and non-metallic inclusions, especially oxides and hydrides.

4. Conclusion

Based on the investigations and analysis of the results, the following conclusions were formulated:

1. The process that generates the largest number of casting defects is the Al-Fin treatment of ring inserts. This process requires special supervision.
2. An excessive amount of morphologically unfavourable β -Al₅FeSi phases was identified in the AlSi9 alloy. Their size and fraction increase with increasing alfining time as the iron content rises. The presence of these phases is one of the principal causes of an unacceptable-quality bond between the insert and the piston.
3. Brittle β -Fe phases block interdendritic channels and impede the flow of liquid alloy, causing shrinkage porosity and shrinkage voids in the insert-piston interface region. Their presence increases the brittleness of the joint, while the resulting porosity produces numerous casting defects at the ring insert-piston interface.
4. Manganese has a beneficial effect on modification of the unfavourable β -Al₅FeSi phase morphology. Its action consists in 'binding' iron and promoting crystallisation of the Al₁₅(Fe,Mn)₃Si₂ phase. The addition of manganese to the AlSi9 alloy changed the morphology of the β -Fe phases into α -(Fe,Mn) phases.
5. Microstructural examination of the insert-piston joint region revealed the presence of gas pores. Their fraction can be reduced, for example, by introducing an intermediate alloy degassing treatment. Further investigations in this area should be continued.

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Effect of refining time on porosity and mechanical properties of low-pressure casting made of AlSi7Mg alloy

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Abstract: The study determines the effect of gas-bubbling refining time on porosity and mechanical properties of low-pressure AlSi7Mg alloy castings under industrial conditions. Refining was performed with an NH_2 mixture (90% N + 10% H_2) for 5.0–9.0 min. Porosity, density index, HB hardness, tensile strength R_m , 0.2% proof stress $R_{p0.2}$ and elongation A were evaluated. The optimum refining time was 7 min, giving minimum porosity and mechanical properties meeting customer requirements. Longer refining increased porosity and reduced selected mechanical properties.

KEYWORDS: ALUMINIUM ALLOYS, POROSITY, LOW-PRESSURE CASTING, MECHANICAL PROPERTIES.

1. Introduction

The mechanical properties of aluminium alloys depend, among other factors, on structural heterogeneity resulting from the manufacturing process. The course of this process affects the morphology and size of constituents such as $\alpha(Al)$ solid-solution dendrites, silicon crystals in the $\alpha(Al)+\beta(Si)$ eutectic, and intermetallic phases such as Mg_2Si . Individual production stages also influence casting defects, including shrinkage and gas porosity [1–4]. This is particularly important because, in many foundries, porosity is a major product-quality criterion that must meet customer requirements. This is because porosity adversely affects many service properties, especially the strength and ductility of castings.

The porosity of aluminium-alloy castings is affected by numerous technological, material and process-related factors. The most important include [5–10]:

1. hydrogen content – an excessive amount of hydrogen dissolved in liquid aluminium leads to gas porosity. During solidification, hydrogen is released in the form of gas bubbles,
2. hydrogen sources – e.g. moisture in the furnace atmosphere, wet tools and alloy contamination,
3. presence of oxides and inclusions – Al_2O_3 particles and other contaminants may initiate pore formation. Inclusions disturb solidification and facilitate hydrogen entrapment,
4. refining process – improper refining fails to remove hydrogen and oxides. A process that is too short or too intensive may be ineffective or even detrimental,
5. chemical composition of the alloy – some alloying additions increase susceptibility to porosity; for example, a high magnesium content increases the tendency towards oxidation and hydrogen absorption.

It follows from the above that most factors affecting porosity are related to the refining process; therefore, investigations were undertaken in this area.

2. Prerequisites and means for solving the problem

As already mentioned, for manufacturers of many cast products, porosity is the most common criterion for compliance with customer specifications and therefore determines whether a product can be sold. On the one hand, foundries should reduce the defect rate; on the other hand, because of corporate constraints, they cannot directly modify the technology or the type of casting alloy. Such changes require customer approval each time and involve costly pre-production tests. Therefore, solutions that improve profitability, are inexpensive, easy to implement and do not require customer approval are preferred. One such measure is optimisation of the AlSi7Mg alloy refining parameters towards reducing the process duration, since the customer also specifies the type of refining.

3. Solution of the examined problem

The aim of the study was to determine the shortest possible refining time for the AlSi7Mg alloy. The porosity fraction and mechanical properties were adopted as evaluation criteria.

Importantly, the effect of changes in refining time on selected output characteristics was investigated under industrial conditions. The scope of the study included:

1. gas-bubbling refining at different times followed by casting of test specimens,
2. microstructural examination with respect to porosity fraction,
3. measurement of the density index,
4. HB hardness and mechanical-property testing (R_m , $R_{p0.2}$, A).

3.1. Material and research methodology

AlSi7Mg alloy (chemical composition according to EN 1706:2020) was selected for the investigations. It is a standard alloy used for the production of automotive wheels by low-pressure casting (approximately 0.2 MPa, i.e. 2 bar). Refining tests, according to the experimental plan shown in Fig. 1, were carried out in a holding ladle with a capacity of 1.8 Mg (1.6 Mg of liquid metal). An NH_2 mixture (90% N + 10% H_2) was used for refining at a flow rate of approximately $18.5\text{--}19.5\text{ L}\cdot\text{min}^{-1}$. After refining, samples were taken from the central part of the ladle for metallographic examination, density-index measurement and mechanical-property testing. The previously used refining time was 9 min.

Microstructural images of the samples were acquired using an Olympus GX-51 optical microscope in bright-field mode at $50\times$ magnification. Metallographic specimens prepared according to the recommendations of the Buehler expert system were polished and not etched. For quantitative assessment of porosity, 15 images were acquired in randomly selected fields of view, giving a total analysed area of approximately 70.5 mm^2 . Image binarisation was performed using Otsu's method. Porosity was measured by quantitative metallography using ImageJ image-analysis software. The number and size of pores were determined. The density index was measured hydrostatically (Archimedes' principle) using a laboratory balance by weighing the specimen in air and in distilled water.

HB hardness was measured in accordance with PN-EN ISO 6506-1 using a Zwick ZHF hardness tester under a load of 187.5 kg, with a 2.5 mm diameter steel ball and a test duration of 35 s.

Mechanical properties were determined from a static tensile test at 20°C in accordance with PN-EN ISO 6892-1 using an Instron 3382 testing machine, with a 20:1 ratio and a constant crosshead speed of $4\text{ mm}\cdot\text{min}^{-1}$. Eight measurements were performed; the two extreme values were rejected, and the arithmetic mean of the remaining results was calculated and rounded to the nearest integer. The results obtained were compared with the customer requirements.

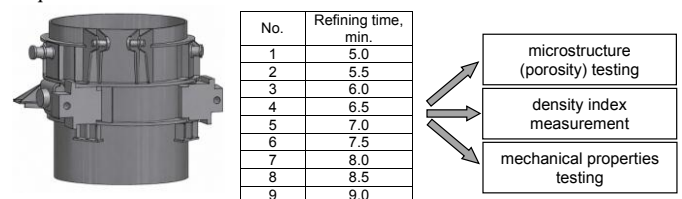


Fig. 1. Test stand and experimental scheme.

4. Results and discussion

4.1. Effect of refining time on porosity and density index of the AlSi7Mg alloy

Microstructural examination showed that samples 1, 6, 7, 8 and 9 were characterised by a high porosity fraction and are therefore not shown. The effect of different refining times on the AlSi7Mg microstructure with respect to porosity for the remaining samples is presented in Fig. 2. Selected quantitative metallography measurements of porosity parameters are shown in Table 1.

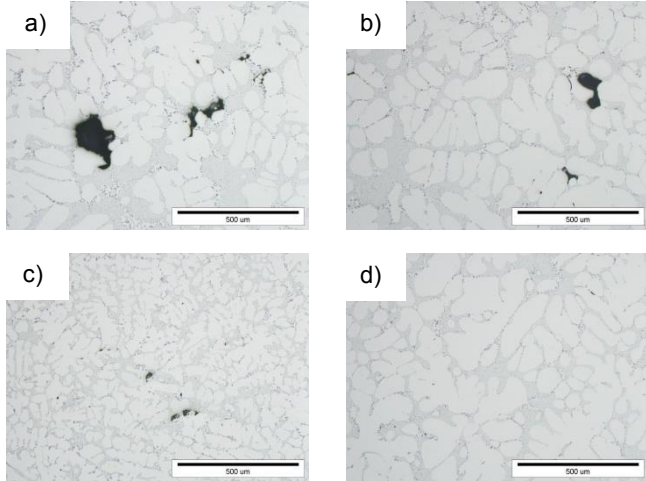


Fig. 2. Microstructure of the AlSi7Mg alloy after different refining times: (a) 5.5 min (sample 2); (b) 6.0 min (sample 3); (c) 6.5 min (sample 4); (d) 7.0 min (sample 5).

Table 1. Results of quantitative metallographic measurements of porosity parameters in the AlSi7Mg alloy.

Sample No.	NH ₂ mixture refining time, min.	Number of pores		Pore size	
		Volume fraction of pores, VV, %	Number of pores per 1 mm ² , NA, mm ⁻²	Mean area of planar pore cross-section A, μm ²	Mean Feret diameter of planar pore cross-section D, μm
1	5.0	1.230	4.97	3630	73
2	5.5	0.910	2.91	3162	52
3	6.0	0.512	2.14	1476	48
4	6.5	0.132	0.26	2378	42
5	7.0	0.002	0.04	466	30
6	7.5	0.873	1.02	2648	53
7	8.0	2.112	2.89	7681	71
8	8.5	3.160	2.96	11123	95
9	9.0	5.478	4.16	16433	110

Analysis of the quantitative metallography results presented in Table 1 shows that reducing the NH₂-mixture dosing time during refining of the AlSi7Mg alloy to approximately 7 min decreases the porosity fraction. For sample No. 5, the volumetric fraction of pores falls within the range of 0.002–0.132%. The number of pores per 1 mm² ranges from approximately 0.04 to 0.26 mm⁻², while the mean diameter of the planar pore cross-section is approximately 30–35 μm. When the refining time exceeds 7 min, the trend begins to reverse and porosity increases, both quantitatively and qualitatively. Taking the above into account, it can be concluded that refining approximately 1.6 Mg of AlSi7Mg alloy should last 7 min. Exceeding this value may increase porosity and consequently the proportion of casting defects in finished products. This is probably caused by the increasing hydrogen content in the alloy (10% H₂), which reduces alloy density [11]. This is consistent with the results reported in [12–14]; however, confirmation of this assumption requires additional investigations.

The results of the density-index measurements for the AlSi7Mg alloy are presented in Fig. 3.

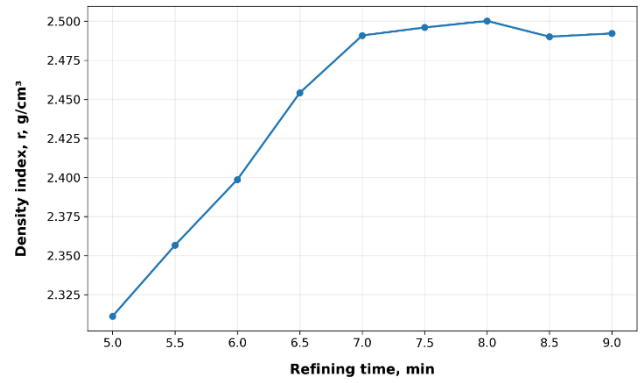


Fig. 3. Density index (ρ) of the AlSi7Mg alloy as a function of refining time.

In addition to porosity and density index, which are key quality criteria determining whether a product is approved for sale, hardness and mechanical properties (R_m , $R_{p0.2}$, A) are also important. Therefore, further investigations focused on the effect of NH₂-mixture dosing time on strength characteristics determined from the static tensile test.

4.2. Effect of refining time on the mechanical properties of the AlSi7Mg alloy

The effect of refining time with the NH₂ mixture on hardness and selected mechanical properties of the AlSi7Mg alloy is presented in Fig. 4.

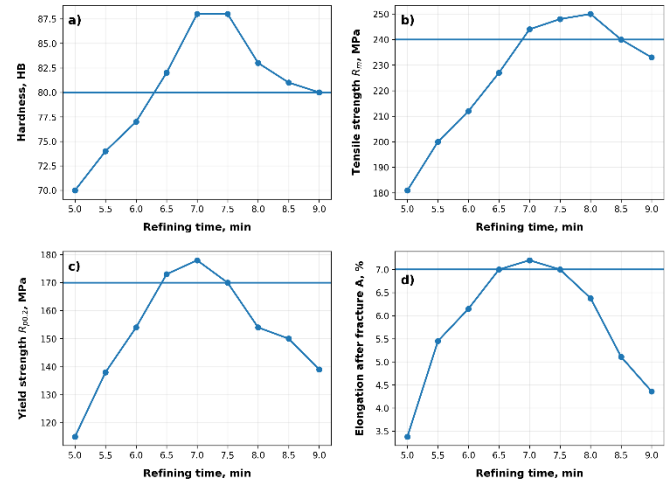


Fig. 4. Effect of NH₂-mixture refining time on: (a) HB hardness; (b) tensile strength R_m ; (c) 0.2% proof stress $R_{p0.2}$; (d) elongation after fracture A (the solid line indicates the minimum strength-property values specified by the customer).

The results presented in Fig. 4 show that both hardness and the mechanical properties (R_m , $R_{p0.2}$, A) change in a similar manner. The highest values are observed for samples No. 5 and 6 (NH₂-mixture dosing time of 7–7.5 min). It is worth noting that this refining time results in:

- hardness of approximately 88 HB (minimum required level: 80 HB),
- tensile strength R_m of 244–250 MPa (required level: 240 MPa),
- 0.2% proof stress $R_{p0.2}$ of approximately 178 MPa (required level: 170 MPa),
- elongation after fracture A of approximately 7% (meeting the required level).

At shorter refining times, or when the NH₂-mixture dosing time exceeds 7 min, hardness and mechanical properties decrease. It should also be noted that the hardness and mechanical-property results are consistent with the porosity results (Table 1) and the density index (Fig. 3).

5. Conclusion

Although many studies have already been conducted on the effect of refining on selected mechanical properties of aluminium alloys, the subject remains relevant. This is mainly because individual stages of aluminium-alloy melting and casting technologies, particularly pressure casting processes, continue to change, as do the requirements concerning the metallurgical cleanliness of castings, measured for example by the permissible porosity fraction.

The literature also reports numerous studies on the effect of microstructure on specific mechanical properties of aluminium alloys [11]. However, there is still no unequivocal answer as to whether the phase composition, stoichiometry and morphology of structural constituents, or the fraction of gaseous impurities removed by refining, plays the more significant role in determining specific mechanical properties of aluminium alloys.

Regardless of whether views on this issue differ [10–14], refining remains one of the main stages in preparing liquid alloy both for pressure casting and for gravity casting of other non-ferrous metal alloys [15].

Among the parameters of gas-bubbling refining, the density of the AlSi7Mg alloy is influenced mainly by the duration of introduction of the NH_2 mixture (90% N + 10% H_2). Therefore, the effect of refining time on porosity and mechanical properties was investigated for AlSi7Mg alloy used for automotive-wheel castings. It was found that the optimum refining time for AlSi7Mg alloy in a ladle with a capacity of approximately 1.8 Mg (1.6 Mg of liquid metal), with respect to limiting porosity and maintaining mechanical properties, is 7 min. Exceeding this value results in deterioration of porosity, both qualitatively and quantitatively, as well as hardness and selected mechanical properties (R_m , $R_{p0.2}$, A).

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