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METHOD OF PERMEABLE ELEMENTS FOR SIMULATION OF POWDER METALS FORMING PROCESSES

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Abstract. For simulation of processes of compaction or forging of products from powder materials, a method of permeable elements is proposed. The essence of the method is to use elements whose shape is regulated in advance, and, unlike the finite element method, where the elements coincide with the material volumes and their masses are unchanged, here the masses elements are variable, and material can flow between adjacent cells. Examples of using the method for modeling the processes of forging of porous preforms in closed and open dies, as well as in a closed die with a compensation cavity, are presented.

Keywords: powder materials, simulation, numerical methods, forging, porous body, plasticity criterion.

1. Introduction

Among the numerical methods for solving problems associated with finding the stress and strain fields that arise both in compact and in porous materials, the finite element method (FEM) has become widespread. Its advantages include convenience in terms of algorithmization and programming, as well as the relatively high accuracy of the resulting solutions [1-4].

However, in the study of technological processes during which large deformations develop, the use of FEM is sometimes difficult due to excessive distortions of the finite element mesh. In particular, with large axial deformations, local flattening of the cells is possible, which is especially important for bulk compressible materials, since it may not be possible to calculate the density distribution. From a purely technical point of view, this regularization necessitates the regeneration of the grid every few counting steps, significantly increases the counting time, and significantly complicates the solution algorithm.

To solve the technological problems of this type, it seems more appropriate developed in Institute for problems of materials science NAS of Ukraine so called method of permeable elements (MPE). Its essence consists in the use of elements whose shape is regulated in advance, and, unlike the FEM, where the elements coincide with the material volumes and their masses are unchanged, here the masses of the elements are variable - material can flow between adjacent cells.

2. Objects and research methods

When constructing an algorithm for modeling the processes of compaction and deformation of porous preforms during their processing by pressure using the method of permeable elements, we used the model of an ideal rigid plastic porous body. The physical and mathematical formulation of the problem is based on the following basic assumptions.

A porous preform is an isotropic compressible body to which the basic equations of continuum mechanics are applicable. In the framework of this approach, a porous body is considered as a chaotic mixture of solid and emptiness. In accordance with the basic concepts of continuum mechanics, it is also assumed that the behavior of the elements under consideration can be described using the strain rate tensor e_{ij} , stress tensor σ_{ij} , relative density ρ or porosity θ and a number of other parameters [5].

The proposed method is designed to solve problems in a quasistatic formulation and its implementation can conditionally be divided into the following three stages:

- specifying a sampling structure and determining the corresponding form functions for the velocity field;

- determination of the velocity fields all over the processed material;

- determination of the field of densities, deformations, stresses and other deformation parameters.

The first of these stages is characterized by the fact that, unlike the FEM, the movement of the mesh in the MPE (if this is realized) is not associated with the movement of particles of the pressed material, but is independent of it and is determined based on the convenience of analyzing the results. The latter is due to the use of the motion of the deformable material as the basic Eulerian idea. In this regard, the mass of each element can be variable, and the elements themselves are permeable.

The grid of permeable elements and the evolution of their shape during processing are set a priori based on known data on the nature of the movement of the processing tool, the convenience of the analytical representation of the functions of the velocity fields, and also on the basis of the possibility of further experimental verification. The foregoing also constitutes one of the advantages of the MPE, since it simplifies the procedure for comparing the results obtained by experimental and computational methods; there is no need to use averaging operations that reduce the accuracy of the obtained solution.

At the same stage, by analogy with the FEM, kinematic parameters are determined as a function of shape through their unknown values at the grid nodes. In the general case, the MPE grid can be constructed as in the FEM, however, the movement of the nodes is no longer controlled by the velocity field of the material flow.

As a basic model for describing the processes of hot deformation of porous materials, the model of a plastic porous body [5] is used based on the assumption that there is some plasticity criterion F , which relates the components of the stress tensor with porosity:

$$F = \frac{p^2}{f_1(\theta)} + \frac{T^2}{f_2(\theta)} - \sigma_s^2 = 0 \quad (1)$$

where $f_1(\theta)$ and $f_2(\theta)$ are some functions of the material porosity θ , which determine its rheological properties; σ_s is the yield strength of the base material for these temperature-strain conditions.

The basic algorithm for solving problems using the method of permeable elements, based on the application of variational methods, includes the following operations. For the investigated volume of the deformable billet, some functions of the velocity field corresponding to the boundary conditions are set in the form of polynomials:

$$v_k = a_1\varphi_1(k) + a_2\varphi_2(k) + \dots + a_n\varphi_n(k) \quad (2)$$

where k is the generalized coordinate; $\varphi_i(k)$ are some coordinate functions; a_i - numerical coefficients - unknown variable parameters, the value of which at each step of deformation is selected using one of the known optimization methods in such a way that it minimizes the value of the functional J :

$$J = \sum_{k=1}^K \iiint_V \sqrt{e^2 \cdot f_1(\Theta) + H^2 \cdot f_2(\Theta)} \cdot dV + \sum_{m=1}^M \sqrt{3} \iint_F \mu \sqrt{f_2(\Theta) \cdot (v_1^2 + v_2^2)} \cdot dF; \quad (3)$$

where $e = e_{ij}\delta_{ij}$ is the first invariant of the strain rate tensor characterizing the speed of uniform comprehensive compression (or tension), which can be defined as

$$e = \frac{1}{\Theta} \frac{d\Theta}{dt} = \frac{1}{V} \frac{dV}{dt};$$

H_i - shear strain rate intensity; dV is the elementary volume of the deformable body; μ is the coefficient of friction; v_1 and v_2 are the components of the velocities of the particles of the deformable body along the contact surfaces (at the workpiece – tool interface); dF is the elementary area of the contact surface.

Having determined the values of the varied parameters a_i , we can find the velocity field functions (2) and calculate the components of the strain rate intensity H_i and the compaction rate e , which allows us to solve the problems of final shape change and determine the force and energy parameters of the process.

The density distribution field is found from the law of conservation of mass, which for any element of a continuous medium is formulated as:

$$\frac{dm}{dt} = \int_S v_n \rho \cdot dS \quad (4)$$

where m is the mass; t is the time; v_n is the projection of the flow velocity vector of the material of a given permeable element onto the normal to the surface S bounding this element.

When assuming the existence of velocity discontinuities in a deformable body, it is also necessary to take into account the energy expenditure on deformation on the discontinuity surfaces in the main energy equation. In view of the foregoing, functional (4) takes the form [6]:

$$J = \sum_{k=1}^K \iiint_V \sigma_{ij} \cdot e_{ij} \cdot dV + \sum_{m=1}^M \sqrt{3} \iint_F \mu \sigma_s \sqrt{f_2(\Theta) \cdot (v_1^2 + v_2^2)} \cdot dF + D; \quad (5)$$

where D is the power dissipation on the discontinuity surfaces of kinematically possible displacement velocities. The rupture surface is considered as the limiting position of a thin transition layer of thickness Δn at $\Delta n \rightarrow 0$, in which the displacement velocity undergoes a fast but continuous change.

The magnitude of power dissipation on the surface of the velocity gap can be determined as [6]:

$$D = \int_{\Sigma} \sigma_s \sqrt{(\Delta v_n)^2 f_1(\Theta) + \left[\frac{4}{3} (\Delta v_n)^2 + (\Delta v_t)^2 \right] f_2(\Theta)} \cdot d\Sigma \quad (6)$$

where $d\Sigma$ is the elementary surface of the velocity gap; Δv_n and Δv_t are the normal and tangent components of the discontinuity velocity vector.

If to take the values of the functions $f_1(\Theta)$ and $f_2(\Theta)$ in the form of dependencies:

$$f_1(\Theta) = \frac{4}{9} \cdot \frac{(1-\Theta)^4}{\Theta}; \quad f_2(\Theta) = \frac{(1-\Theta)^3}{3}; \quad (7)$$

then we can write:

$$D = \int_{\Sigma} \frac{(1-\Theta)^{3/2}}{\sqrt{3}} \sigma_s \sqrt{\frac{4}{3\Theta} (\Delta v_n)^2 + (\Delta v_t)^2} \cdot d\Sigma; \quad (8)$$

3. Results and their discussion

The developed general MPE algorithm was successfully used, in particular, to simulate the processes of closed and open hot forging and forging of porous preforms in a half-open die, compaction of bimetallic porous preforms with a horizontal separation plane, precipitation of an annular porous preform in a closed die with a free centripetal radial flow of material, consolidation of step products such as a sleeve with a shoulder, etc.

In particular, we give examples of the use of the considered method for studying the mechanics of material flow during open hot forging of porous preforms (Fig. 1, a) and closed forging in a die with a compensation cavity (Fig. 1, b).

The effectiveness of the application of hot forging methods for powder products in dies with open volumes at which additional shear deformations are realized along with axial ones is shown in a number of works [7-9].

At the same time, hot forging of porous preforms in an open die, a distinctive feature of which is the presence of a height-changing section of the preform with a free lateral surface throughout the deformation process, was not widely used, since uncontrolled extrusion of the material being compacted is possible in the flake in the early stages of deformation, which may cause under-compaction of the forging material.

In this regard, questions of studying the mechanics of the flow of the workpiece material during forging in dies having additional compensation cavities are of practical interest.

The calculation model for describing the flow of a porous material during hot open forging and closed forging in dies with a compensation slit was constructed on the basis of the variational principle, while material hardening, temperature effects and inertia were neglected, and the method of permeable elements was used as a numerical method for implementing the model.

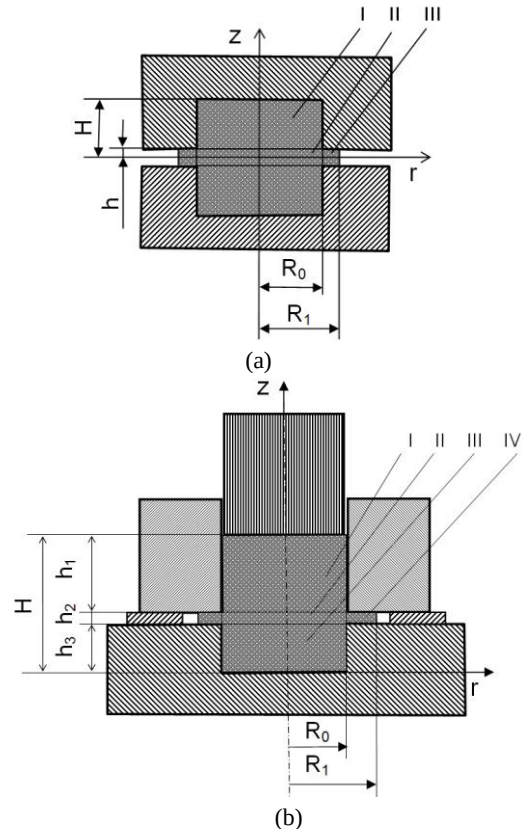


Fig. 1. The design models for the processes of open forging (a) and closed forging in a die with a compensation gap (b)

Taking into account the symmetry of the deformable workpiece relative to the vertical axis, the calculation was made for half of its cross section, which was conditionally divided into three design zones (permeable elements) for open forging (Fig. 1, a), and four zones for closed forging (Fig. 1, b). Inside each of the calculated elements, the distribution of density and strain rates at different compaction times is considered homogeneous.

The boundary conditions for the velocity components v_z and v_r can be expressed as:

$$v_z|_{z=H} = -v_0; \quad v_r|_{z=H} = 0; \quad v_z|_{z=0} = 0; \quad v_r|_{r=0} = 0; \quad v_z|_{z=h} = -v_0;$$

where v_0 is the velocity of the upper half-matrix.

Assuming that the velocities in each of the forgings calculated zones are linear functions of the r and z coordinates, taking into account the boundary conditions, we take the following expressions describing the velocity field of the material flow in each of the zones:

open forging:

$$\text{zone I: } v_z = -v_0 \frac{z}{H}; \quad v_r = 0;$$

$$\text{zone II: } v_z = -v_0 \frac{z}{H}; \quad v_r = \frac{v_0}{h} a_1 r v; \quad (9)$$

zone III:

$$v_z = -v_0 \frac{z}{h}; \quad v_r = \frac{v_0}{h} a_1 R_0 v (1 + a_2 \frac{r - R_0}{R_1});$$

closed forging:

$$\text{zone I: } v_z = v_{12} - \frac{v_0}{H} (z - h_2 - h_3); \quad v_r = 0;$$

$$\text{zone II: } v_z = v_{23} - \frac{v_0}{H} (z - h_3); \quad v_r = \frac{v_0 r}{Ha_1 v};$$

$$\text{zone III: } v_z = -\frac{v_0 z}{H}; \quad v_r = 0; \quad (10)$$

$$\text{zone IV: } v_z = 0; \quad v_r = \frac{v_0 R_0}{Ha_1 v} \left[1 + a_2 \frac{(r - R_0)}{R_1} \right];$$

where a_1 and a_2 are some variable parameters; v_{12} and v_{23} are the linear flow rates of material between zones I-II and II-III respectively (closed forging).

After the corresponding transformations, taking into account dependences (9) and (10), the form of the components of the functional J was determined and, taking the parameters σ , μ and v_0 as constant throughout the deformation cycle, at each calculation step we found the values of a_1 and a_2 that minimize the functional (5).

The calculation was carried out using the step loading method, in accordance with which the entire deformation period was divided into equal time intervals Δt , during which the workpiece is upsetting at $\Delta H = v_0 \Delta t$. At each i -th loading step, the parameters a_1 and a_2 were determined that minimize the functional J ; from (9) and (10) a new value of the flash radius R_1 was found from the dependence:

$$\Theta_k'' = 1 - \frac{m_k \pm \frac{dm_k}{dt} \Delta t}{V_h^k \gamma} \quad (11)$$

where Θ_k'' , V_h^k , and m_k are the porosity, volume and mass of the k -th zone of the preform, respectively, after the next step of deformation, the current values of porosity in individual zones of the preform were calculated.

Comparison of the simulation results of the hot forging process in an open die with similar data for forging in a closed die with compensators showed that while during forging in an open die the displacement of material in the flash starts already at the initial stages of deformation (Fig. 2), then in a closed die with

compensators - only after the workpiece reaches a certain threshold, sufficiently high density level (at $\theta = 2 \div 5 \%$) (Fig. 3), which leads to a decrease in the final value of the flash size during forging in a die with a compensator.

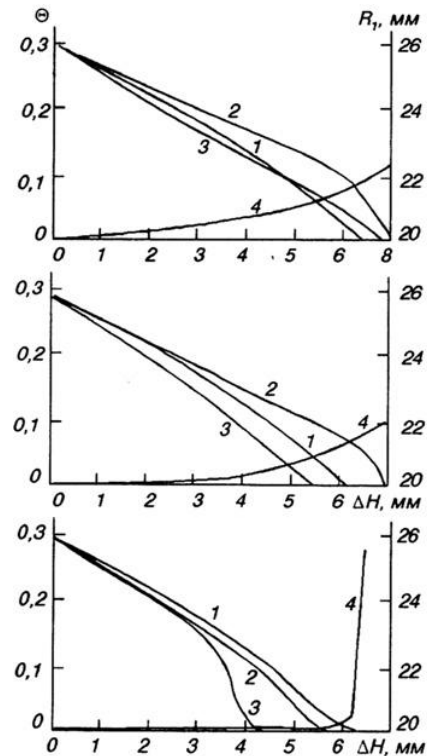


Fig. 2. The dependence of the current porosity of the material in the zones: I (1), II (2) and III (3) and the radius of the flash (4) from axial deformation at $h_0 = 11$ (a), 9 (b) and 7 (c) mm; $\theta_0 = 0.3$

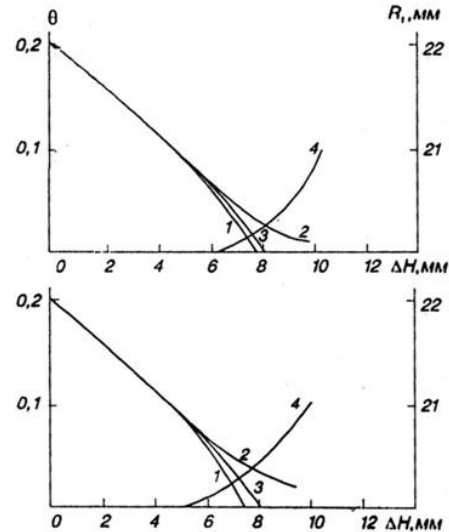


Fig. 3. The dependence of the current porosity of the material in the calculation zones I (1), II (2), III (3) and the radius of flash (4) at the axial strain ΔH for $h_2 = 3.0$ (a) and 4.0 (b) mm; $\theta_0 = 0.2$

At the same time, a sharp increase in the resistance to metal outflow in the flash at the final stage of open forging due to the gradual closure of the half-matrices, contributes to the compaction of the material in the second zone, while at closed forging with a compensator, due to the constant gap between the half-matrices, the resistance to outflow to the store changes slightly, which determines the presence of some residual porosity in zone II of the forging.

It should be noted also that, in contrast to open forging, in which the choice of optimal porosity, the ratio of the size of the workpiece and the cavity of the matrix has a significant effect on the density distribution in different parts of the forging and the final

value of the flash, then when implementing a closed forging scheme with compensators, the dependence of the qualitative characteristics forgings from the specified structural and technological parameters are significantly lower. This fact, along with the fact that the flash value is reduced during forging in a closed die with a compensator, *ceteris paribus*, allows us to conclude that the scheme under consideration is preferable to open forging. At the same time, it should be noted that the structural complexity of die tooling for the implementation of the closed forging process with compensators somewhat limits the possibility of its wider use in industry.

Information on the power parameters of the processes under consideration is essential when choosing a deformation scheme and brand of a press, as well as in calculating die tooling. Let us consider, along with the above schemes, the traditional closed powder forging scheme without compensation slots, traditional for powder metallurgy.

The forging forces for the three considered deformation schemes were calculated using the basic equation of the energy method.

Analysis of the simulation results, which are presented in Fig. 4 shows that the use of a closed forging scheme necessitates the application of significantly higher forces to obtain high-density forgings. The smallest deformation force is with open forging. These results allow us to conclude that with an increase in the rigidity of the loading circuit, the strain force increases. It is noteworthy that the difference in effort increases with increasing degree of axial deformation, and hence the average density of forgings. At the initial stages of compaction, the difference in the efforts for the considered deformation schemes is small.

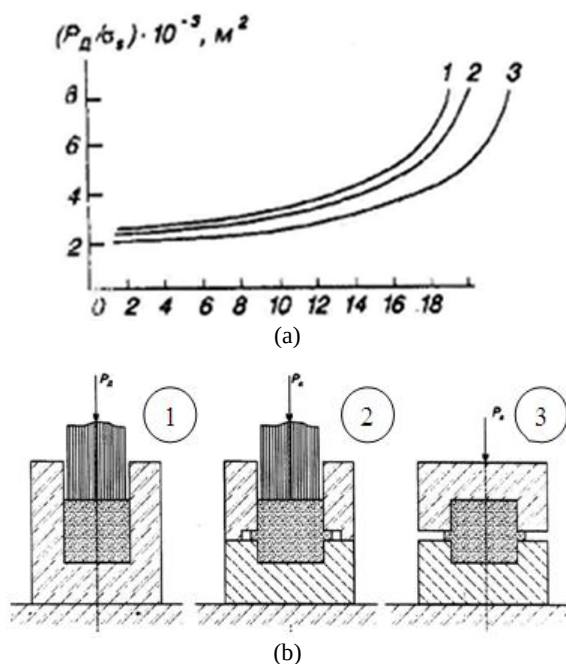


Fig. 4. The dependence of the relative value of the forging force on axial deformation (a) for deformation schemes (b): 1 - closed forging; 2 - forging in a closed die with a compensator; 3 - open forging

It should be noted that experimental studies of the processes of hot forging of powder samples lead to similar results [8-10]. The use of compensation slots allows to obtain high-density forgings with a homogeneous structure at a lower strain pressure compared to traditional closed forging.

Thus, the presented results allow us to conclude that the traditional scheme of hot forging of porous preforms in a closed die is the least preferred in terms of energy and process parameters. This indicates the advisability of using less rigid deformation schemes with partial extrusion of the workpiece material into the compensation cavities.

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PROCESSING OF BIMETALS IN THE ELECTROLYTE PLASMA

ОБРАБОТКА БИМЕТАЛЛОВ В ЭЛЕКТРОЛИТНОЙ ПЛАЗМЕ

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Abstract: Electrolyte plasma processing is environment-friendly and high-effective method to treat surfaces of metals and alloys and can be used for heat or thermochemical treatment as well as for cleaning or polishing of metal surfaces. Modes of processes in the electrolytic cell for different metals can partially overlap. So it is possible to perform the same or different processing for different metals (including components of bimetal) simultaneously. This article describes modes to be established at the active electrode during the anodic process, and application of these modes to various types of treatment of bimetals. It is shown that there exists an option of separate or simultaneous heat or thermochemical treatment, as well as polishing of one or both surfaces of bimetals.

KEYWORDS: BIMETAL, ELECTROLYTE PLASMA, HEAT TREATMENT, POLISHING.

1. Introduction.

Bimetals are modern composite material, including several (usually two) metallic layers jointed by adhesion or chemical bonding. Practically all industries use bimetals due to their unique complex of functional characteristics, which are not inherent to components. For example, bimetals can have high corrosion resistance combined with high strength and manufacturability or with high heat strength, electrical and heat conductivity.

Usually the jointing is the final procedure of the bimetals manufacturing. However, there are cases, in which it is necessary to produce heat or thermochemical treatment of one component without changing of properties of another. In other cases, it is required to polish both the surfaces of the bimetal.

There exist a lot of methods for heat or thermochemical treatment of metals, in which the entire material is subject to heating. In these cases, a localization of the thermochemical action is carried out by protecting the non-machined areas of the part surface by galvanic tinning, coppering, nickeling or by applying various pastes and coatings (e.g. [1]). However, these methods are not usually applicable for bimetals due to undesirable changes of mechanical or other functional properties of the second component, which are resulted from the heating due to the heat conductivity. Similar phenomena are observed during heating of separated areas of large-sized products by gas burners or by high-frequency currents [2]. In addition, it is not possible to process surface areas having a complex configuration by these methods.

Other problems arise during polishing of the surface of bimetallic products, since there are differences in the physicochemical and mechanical properties of each of the bimetal surfaces. The most popular methods of the metal polishing are [3]: mechanical or abrasive polishing; chemical polishing; electrochemical or galvanic polishing. Unfortunately, these methods have such drawbacks that do not allow (or significantly complicate) the polishing of the bimetals surface.

Mechanical properties of the bimetal components are usually strongly different and thus their simultaneous abrasive polishing is impossible. For example, the polishing of non-ferrous metals is usually much simpler (and thus quicker) operation than the polishing of steels. Chemical and galvanic polishing differs by the reagent or electrolyte compositions for various metals and alloys. For example, the electrolyte for Al polishing usually includes H_3PO_4 and H_2SO_4 , for Cu and carbon steel – H_3PO_4 and CrO_3 ; for stainless steel – H_3PO_4 with a little addition of H_2SO_4 . Besides that, electrolytes may include other additions. It is also important that the used reagents are ecologically hazardous and difficultly recycled.

These problems can be solved by the electrolyte plasma processing (EPP) of bimetals at anodic process (AP) [4-6].

Processes in the electrolytic cell at the active anode can be divided onto five modes depending on electric parameters [7]. These modes are: 1 – low voltage electrolysis (classical

electrochemistry) (LVE) – $0... (15\div 25)$ V; 2 – switch mode (Wehnelt's breaker) (SM) – $(15\div 25) ... (70\div 100)$ V; heating mode (HM) – $(70\div 100) ... (180\div 290)$ V; electro-hydro-dynamic mode (EHDM) – $(180\div 290) ... (300\div 360)$ V; contact electric discharges mode (CEDM) – higher $(300\div 360)$ V. Real values of the modes voltage boundaries depend on the metal and composition of the electrolyte.

Each of these modes can be used to treat surface of metals. The LVE results to anodic dissolution of the metal surface according to Faraday's law. A current output during this mode is depending on the metal type and varies from $15\div 20$ % for W to $95\div 98$ % for Cu [3]. This mode is used in various industries for polishing, deburring, etc.

The SM stimulates rapid dissolution of the active electrode both by pulse discharges and by intensive chemical reactions in a gas-vapor shell [8]. This mode can be used to produce needle-like tips from hard-to-process materials such as W, Mo, Ir, etc. or chemical compounds.

The anode specific erosion during the HM or EHDM of the AP can be significant [4, 6]. In these modes there are narrow ranges of the voltages with the maximal dissolution rate. And the current output is much higher than that during the LVE. Besides that, the classical electrochemistry distinguishes insoluble (inert) electrodes (Pt, Ir, graphite) and soluble ones, but after the plasma shell formation this difference is not actual. Particularities of the electrolyte plasma influence on the active electrode surface provide ability to treat metals in such electrolytes where they don't dissolve during LVE or where the current output is very little. These processes are used to polish metals in the electrolyte plasma [4, 5, 9].

The water solutions of salts are usually using to polish metals by EPP. For example, we have used water solution of fluoride and citrate of ammonium at the electrolyte temperature of $60\div 90^\circ\text{C}$ to polish Ti or Ag [10].

2. Experiments

EPP of bimetals were carried out during AP using experimental equipment described in [11]. Water-cooling bath was 2 l in volume. Second electrode (cathode) was made from lead or stainless steel. Cell voltage and current was varied in the ranges of $0\div 450$ V and $0\div 50$ A correspondingly. We used aqueous solutions with specific conductivity higher than $5\text{ Ohm}^{-1}\cdot\text{m}^{-1}$. The oxidation duration was varied from tens of seconds to 10 minutes. The electrolyte temperature was varied from 15 to 30°C for heat and thermochemical treatment and from 60 to 90°C for polishing. The rate of immersion of the anode into the electrolyte in all cases did not exceed 10 mm/s . After processing, the samples were washed by distilled water. The current density at the active electrode at the HM was $6\div 10\text{ kA/m}^2$ and at the EHDM $2\div 5\text{ kA/m}^2$, the temperature of the active electrode at EHDM is $90\text{--}100^\circ\text{C}$. The electrolyte temperature was measured by Hg thermometer. The active electrode

temperature at the heating mode was measured by “Testo 845” optical pyrometer. Morphology of the surface was studied by optical microscope Zeiss AxioScope A1. A reflection coefficient was measured using red laser light.

We have studied plated bimetal samples of various types produced by rolling with the layer thickness of 0.5...3 mm.

3. Results and discussion

3.1. Heat treatment of one component of bimetals at the heating mode of the anodic process

The HM of the AP is established when the gas-vapor shell completely covers the active electrode and the current density decreases to 10 A/m². This mode is used to heat the metal up to 400...1000°C. Followed EHDM is characterized by an increase of the gas-vapor shell thickness, intensive corona discharge, decreasing of the current density lower than 5 kA/m² and absence of high-temperature heating (the active electrode temperature is not higher than 100°C).

It is possible to choose the parameters for the AP to provide the HM on the surface of one component of bimetal (its temperature can be higher than 800°C) and the EHDM on the surface of the second component (it's temperature can be equal or less than 100°C). Thus, the heating of the bimetal will be selective. It is important that erosion will not appear on both the surfaces.

One example of such treatment is given by the following. We have studied the treatment of bimetal plate of steel AISI 321 and Al-based alloy 6063. Direct voltage electrolytic cell included bimetallic anode and lead cathode. The electrolyte was ammonium chloride water solution with specific electric conductivity of 34 Ohm⁻¹·m⁻¹ and the surface tension coefficient of 76 mN/m at temperature of 22°C. When the working voltage of 180 V was reached, the bimetallic electrode was immersed into the electrolyte. On the steel part of the anode, the HM was realized, and it's temperature was equal to 720°C. On the aluminum part of the anode, the EHDM was realized, and it's temperature was equal to 90°C. Cooling of the bimetal after the treatment was carrying out by turning off the current. Addition of corresponding reagents to the electrolyte [4] can provide nitration, carburization or carbonitration of the steel part of the bimetal.

Other bimetals were treated analogously. Modes and achieved temperatures are given in Table 1.

The use of this method to treat bimetals in electrolyte plasma allows the heat or thermochemical treatment of one metal to be carried out without heating the second metal above 100°C, and thus increases the quality of the processed bimetals and improves the manufacturability of the technological processes (the workpiece can have any configuration).

3.2. Polishing of bimetals at the electro-hydro-dynamic mode of the anodic process

EPP is the method to finishing conductive materials. This processing allows to remove the scale, to prepare the surface before covering by thin films and coatings [4, 5], to polish difficult profile surfaces of critical parts, to remove burrs, to blunten sharp edges and to produce decorative polishing of metal products. The EPP is largely devoid of the drawbacks inherent in mechanical and electrochemical polishing. Its advantages, in comparison with other methods of polishing, are high productivity and efficiency,

environmental cleanliness, high quality and rate of performed operations, as well as comparatively low cost.

To polish simultaneously surface of all bimetal product, it is necessary that EHDM was established at all surface. The voltage for EHDM establishing depends on the processed metal type and such molecular properties of the electrolyte as the surface tension coefficient and the kinematic viscosity: the greater these parameters, the lower the voltage, when this mode is set [4, 9].

The main characteristics of polishing quality are roughness and reflect ability. These characteristics depend from the chemical composition of processed material, voltage and duration of the EPP. The composition of processed material also determines composition and concentration of the electrolyte. Besides that, in some cases addition of such matters as glycerin, acetone, thrietanolamin, trilon-B, etc., which significantly influence on both coefficient of surface tension and kinematic viscosity of the electrolyte, can be used. These additions change conditions of gas-vapor shell formation and shift volt-ampere range of the EHDM [4].

The most important parameter which determines the polishing quality is working voltage. It was experimentally shown that when the voltage value decreases below a certain limit, the polishing quality significantly decreases. These minimal limits are: 220 V for stainless steels, 260 V for copper and copper-based alloys, 270...290 V for aluminum and its alloys, 280...290 V for argentine [4, 6]. However, an increase of the voltage over 360 V is unacceptable not only through economic reasons but through technologic ones. This increase can result in transition to CEDM of AP when the gas-vapor shell breaks down and the quality of the polished surface deteriorates [7].

The electrolyte temperature is one of the most important factors of the polishing quality. High quality of the polished surface can be achieved only if the electrolyte temperature is in 50...90°C range. When the latter is outside this interval, the surface quality is lower. Besides that, it should be borne in mind that electrolytes included ammonium salts, hydrochloric acid, some other components which can dissolve higher 85°C with formation of volatiles, and thus frequent adjustment of the solution composition will be necessary.

Electrolyte plasma polishing is usually carried out using water solutions of salts. For example, to polish Al or Al-based alloys, the water solution either of ammonium chloride or sodium chloride [5], or of potassium chloride, oxalic acid and glycerin [12] can be used, but, in order to polish stainless steels, the water solution of ammonium sulfate [13] can be used. So, processing schedules and electrolyte should be determined experimentally for each metal, alloy and especially bimetal or composite.

In this work we used to type of the steel/Al-based alloy bimetals. The first type consists of austenitic stainless steel AISI 321 and aluminum-based alloy 6063, the second type was made with austenitic stainless steel AISI 304 and aluminum-based alloy A92017. To polish above mentioned bimetals we used water solution including 2...6 mass % of potassium chloride, 1...4 mass % of oxalic acid and 1...2 mass % of glycerin. The voltage on the electrolytic cell was 320 V. We saw that in initial rolled state the both surfaces were frosted (Fig. 1) and the reflection coefficient was very small (0.5...2 %) (Fig. 2). The polishing during 2 min results in significant decrease of roughness (see Fig. 1) and the reflection coefficient increases up to 28 % for stainless steel and up to 21 % for aluminum alloy (see Fig. 2). Rapid decrease of the roughness is highly likely resulted from an active local

Table 1. Parameters of the EPP of bimetals.

Bimetal	Heated metal	Temperature in area of HM, °C	Temperature in area of EHDM, °C	Voltage between electrodes, V	Composition of the water-based electrolyte, mass %
Titanium / steel	Steel	620	80	300	10% NH ₄ Cl
Molibdenum / steel	Steel	630	90	300	15% NH ₄ Cl
Aluminum / steel	Steel	720	90	180	15% NH ₄ Cl
Aluminum / steel	Steel	850	90	220	15% NH ₄ Cl
Aluminum / brass	Brass	850	90	210	10%Na ₂ WO ₄ ·2H ₂ O

smoothing of the micro-relief in the places of the largest protrusions. Subsequent increase of the polishing duration results in a slower decrease in the roughness and an increase in the specular luster up to 41...42 % (see Fig. 2).

Obviously, it is easier to choose the electrolyte composition and technological parameters for polishing of metals than of bimetals. In this regard, for bimetals it is possible to achieve a greater reflection coefficient. For example, for parts made of aluminum alloy only, the reflection coefficient reaches 0.8 [4]. Thus, new additional studies are needed.

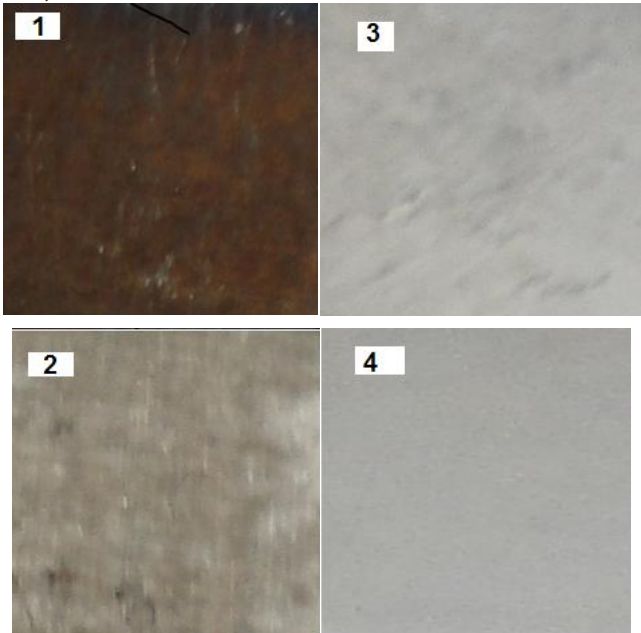


Figure 1. The surfaces of bimetal stainless steel AISI 321 (1, 3) / aluminum-based alloy 6063 (2, 4) before (1, 2) and after (3, 4) the polishing, respectively.

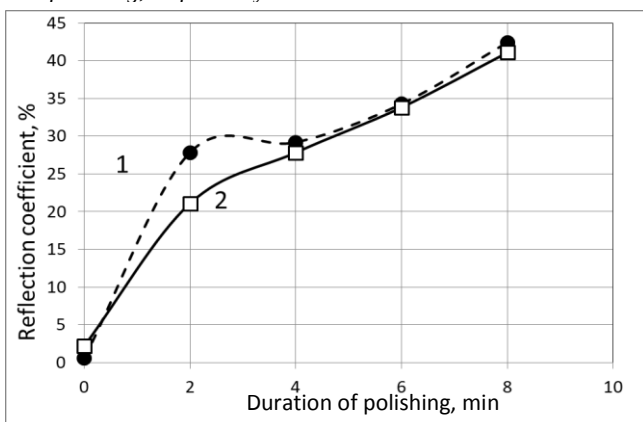


Figure 2. An influence of duration of the polishing on the reflection coefficient for bimetal stainless steel AISI 304 (1) / aluminum-based alloy A92017 (2)

4. Conclusions

1. This work shows possibility to carry out heat or thermochemical treatment of one component of a bimetal without heating of the second component over 100°C using electrolyte plasma processing. Treated bimetallic product may be of any configuration.

2. This work shows possibility of simultaneous electrolyte plasma polishing of both components of the bimetals, in particular bimetal of stainless steel and aluminum-based alloy. For this bimetal, polishing with 2 min duration significantly decreases the roughness and increases the reflection coefficient from less 2 % to more 20 % for both components. Subsequent increase of the polishing duration results to a slower decrease in the roughness and an increase in the specular luster over 40 %.

5. Acknowledgments

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THE EFFECT OF BALL MILLING SPEED ON THE PENETRATION OF Cr AND Zr INTO Cu FOR PRODUCTION OF Cu₁CrZr ALLOY POWDER

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Abstract: The copper alloys are preferred for their superior mechanical properties combined with high electrical conductivity for contact material applications. These properties should be a very good combination of high mechanical strength and enough electrical conduction properties as well as high thermal conductivity. To establish the electrical and thermal conductivity, the low resistive materials such as copper and their alloys to be chosen is a must. Copper can be alloyed with Cr and Zr in very low amounts down to 1 wt% and 0.15 wt%, respectively. These amounts can be found from phase diagrams of interest. The planetary ball milling was chosen as being a unique technique for powder processing to obtain mechanochemical activation and mixing of powders prior to sintering. The ball milling jar was ZrO₂ and ball to powder ratio was varied as 1:1, 5:1, and 10:1. The ball milling rotation was ranged from 400 and 500 rpm for different durations to be investigated. The SEM photos showed the Cr and Zr penetration onto Cu powders transformed to flakes of different sizes. The elemental composition maps showed the better and finer distribution of alloying elements at 500 rpm than 400 rpm.

KEYWORDS: CuCr₁Zr ALLOY, BALL MILLING, PENETRATION INTO Cu.

1. Introduction

Hard copper alloys can be industrially classified into three main groups; these are 1) aluminum bronzes having high strength and wear resistance, 2) beryllium containing, 3) beryllium free alloys for the applications that need electrical conductivity. But, during the last decade, the negative effect of beryllium on environment and health has become an issue and the alloy formation with beryllium was left. Instead of these alloys, a known CuCr₁Zr alloys has begun to be used [1].

From an industrial view point, copper is the first mentioned and very well known as high electrical conductive material with high abundance and being cheap. But the pure copper has very low hardness (49 HV) and yield strength (54 MPa) which makes the use of it impossible in point arc welding process. This problem is overcome by alloying the Cu. But many strengthening processes to improve the mechanical strength as solid solution strengthening, grain refinement, alloying and dispersion hardening are inversely proportional with electrical conductivity property. For instance, while the electrical conductivity decreases down to 5%-75% of IACS by alloying and plastic forming. The electrical conductivity of CuCr₁Zr varies between 76-90%IACS but the hardness increases up to 156 HB. By this study a unique, comparative and systematic approach will be carried out to partially fulfill the literature [2, 3].

Composites are of the materials with a 3D network as matrix and a reinforcement material. The superior properties of both materials as a new material can be achieved together as hardness from ceramics and toughness from metals, etc. As very well known, the electrical conductivity of metals is inversely proportional with the increasing mechanical strength. Since the alloying elements produce local strain and stress fields around the lattice, the mean free path of electrons become smaller by scattering from atoms of impurity elements. The decreasing mean free path of electrons decreases the conductivity while increasing the resistance. The dislocation interruptions are also obstacles for electrons to decrease the conductivity while increasing the strength by interfering with dislocation movement. Besides, following the plastic forming or strength improvement, the aging may recover the conductivity while decreasing the strength, but to a small extent. Cu, Cr and Zr will be planetary ball milled in appropriate weight ratios to produce CuCr₁Zr alloy. Also, after every process, the mechanical-electrical and thermal properties will be measured continuously to monitor the changes. The effect of process parameters on the physical properties will be investigated. The hard copper alloys are used in the application areas that need high

electrical and thermal conduction and mechanical strength as point electrode arc welding. The increasing energy demand worldwide and decreasing source of energy, by increasing energy prices makes the industry using intense energy input to develop high electrical-thermal stable and conductive as well as mechanically strong materials as automotive, panels, radiator fabrication systems.

As the pre-evolutionary result, the first part of the project, which is production of CuCr₁Zr powder alloy by mechanic activation, was presented.

2. Materials and methods

Pure spherical Cu (purity 99.8%, Dv50 25 µm), Cr (purity 99.9%, Dv50 3 µm) and ZrH₂ (purity 99.8%, Dv50 5 µm) powders were provided by Alfa Aesar as seen from Fig 1(a-c). The size of the metallic and compound powders was determined by FEG-SEM (Tescan Mira3 XMU, Czechia). XRD analyzes were performed using the RIGAKU D-MAX 2200 X-ray diffractometer. Microstructural analysis (grain structure and grain shape), phase formation and surface morphology of the produced CuCr₁Zr alloy powders were carried out by using FEG-SEM. Sonics VCX750 ultrasonic horn was used to produce homogeneous CuCr₁Zr powder mixtures. The powders were milled with Fritsch pulverisette 7 Premium line planetary milling device.

Since the ZrH₂ particle size is too coarse to be used in the studies, it has been subjected to a separate size reduction process. ZrH₂ powder with coarse particle size was subjected to wet milling in ethanol for 24 h and the particle size was reduced down to 3±1 microns (Fig. 1d).

Cu, Zr and Cr powders were dispersed in the separate containers using ethanol by ultrasonic homogenizer for 10 minutes. The separately prepared powders were then mixed in a clean beaker and subjected to ultrasonic stirring for 10 minutes using an 80% power at 5 s pulse durations. The prepared powders were placed in 80 ml zirconium oxide grinding jars. After adding zirconium oxide beads to the grinder jar in the determined proportions, the amount of ethyl alcohol was adjusted to 30 ml and the safety locks of the grinder jars were closed into the planetary mill. The milled powder in the planetary mill was separated from the balls using a clean wet sieve of -38 microns and then the powder-ethanol mixture continuously stirred rigorously at maximum of 60 °C to evaporate the ethanol. After that milling, SEM photographs were analyzed.

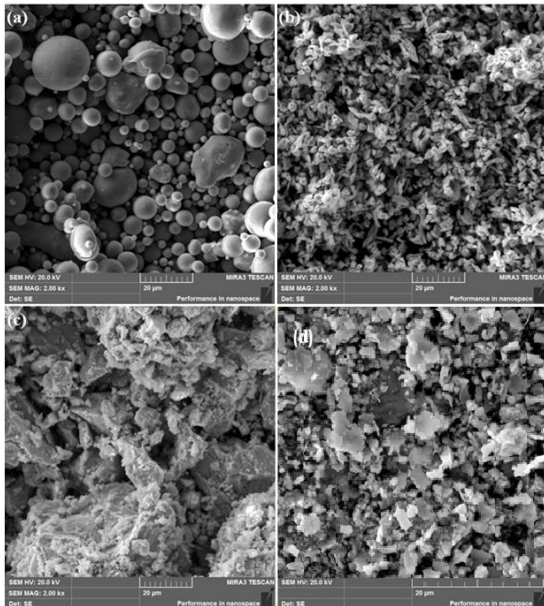


Fig. 1. The SEM images of (a) Cu, (b) Cr, (c) ZrH_2 , (d) after ball milling of (c).

3. Results and discussions

3.1. Preliminary Experiments of Mechanical Alloying

The centrifugal forces created by the movement of the rotating carrier disk and the movements of the chambers rotating about its axis in the device impact the balls and the metal particles. As seen in Fig. 2, the centrifugal forces act in similar and opposite directions since the chambers and the disk on the disk rotate in opposite directions. This causes friction by moving the balls downward on the inner surface of the chamber in which they are located. The friction effect continues when the metal particles and balls circulate freely in the cup jar and strike the inner surfaces of the jar.

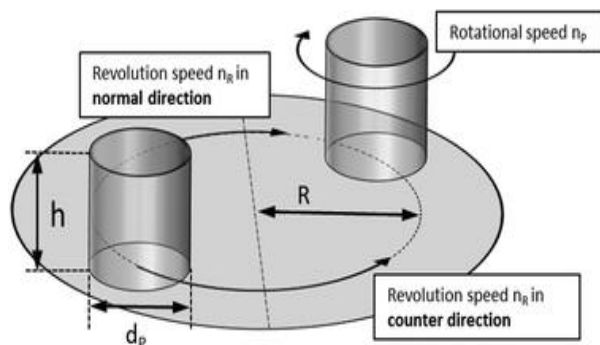


Fig. 2. Demonstration of movement direction of discs and chambers in planetary mill.

$Cu_{1Cr0.15Zr}$ composition (1% Cr, 0.15% Zr and 98.85% Cu) was kept constant in order to determine mechanical alloying conditions in mechanical alloying.

It is contemplated that the embedding of Cr and Zr particles into Cu grains can be quantitatively analyzed by powder shape change from spherical to flakes and stratification. In these experiments, it was determined that Cu grains were expanded as elliptical or flake by deformation. Increased Cu flakes indicate increased collision statistics and increased stress intensity. Fig. 3(a) shows the Cr and Zr distribution in Cu powders before milling while Fig. 3(b) indicates the distribution of finer Cr and Zr powders into Cu. Cr and Zr were not homogeneously distributed and embedded into Cu particles as shown the SEM images (Fig. 3-a). But they embed into Cu particles as finer particles after milling as shown the SEM images in the Fig.

3-b.

The main factor that indicates Cr and Zr are embedded into copper particles is that the spherical copper particles deform in the plate until the smallest distance between them remains. Thus, Cr and Zr impregnation on growing Cu flakes can be observed more effectively. For this reason, it has been tried to determine to what extent the mechanical alloying is successful by emphasizing the rate at which Cu powders were flaked, stratified and severely deformed. In order to convert the success of mechanical alloying into analytical data, it was decided to determine by conversion from sphericity to ellipse (CFSTE) (or deviation from sphericity) using linear intercept method. The details of this method are given below in Fig. 4 with an example.

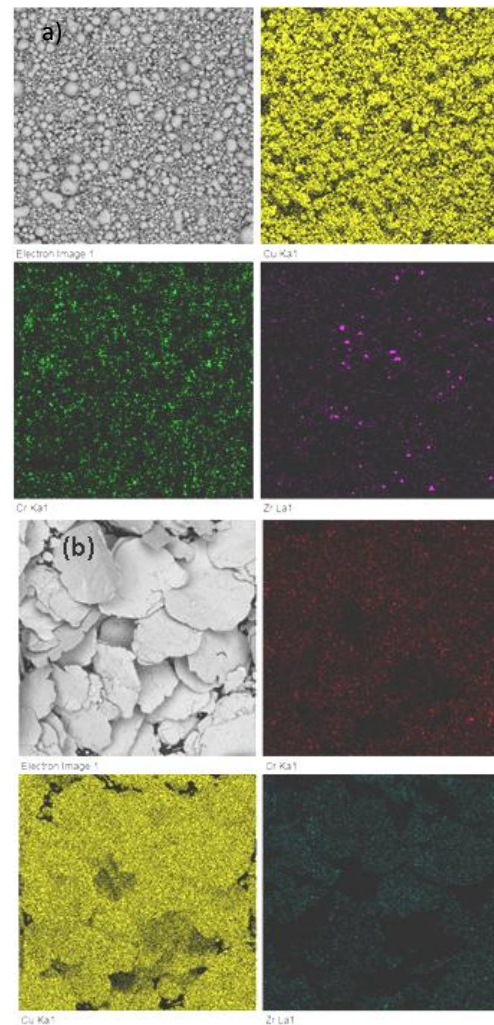


Fig. 3. SEM-EDX Elemental distribution maps a) before milling, b) after milling.

3.2. Percent Conversion from Sphericity to Ellipse Calculation

CFSTE ratio = ((X-axis length of ellipse/Y-axis length of ellipse)/1.5)*100

$$\dots\dots\dots (1)$$

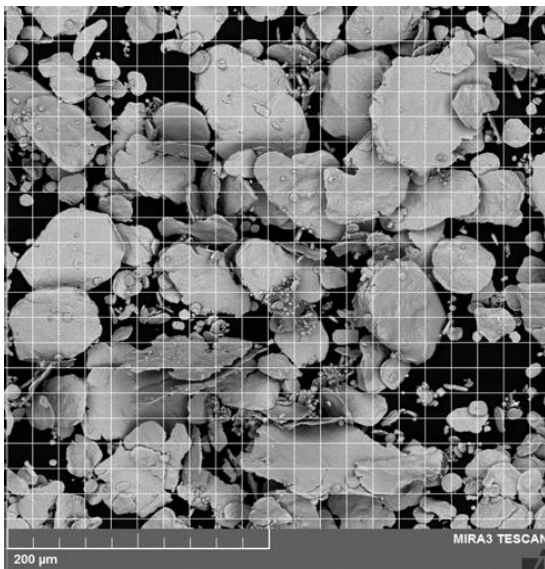
For the powder CFSTE measurement of powder $Cu_{1Cr0.15Zr}$ alloys; the X-axis length and Y-axis length of each ellipse particle were measured in microns (Table 1) using Nitro Pro from the SEM photographs (Fig. 4). The length obtained in micrometers was then divided by the bigger value over the smaller value to determine the CFSTE rate of each powder. For example, the lengths of the 32 powders on the X and Y axis of the SEM photograph given in Fig. 4 were measured with the help of the drawn squares and entered into

Table 1. The data used to determine the CFSTE rate of 32 grains in Figure 4.

Grain No	X (μm)	Y (μm)	X/Y or Y/X	Grain No	X (μm)	Y (μm)	X/Y or Y/X	Grain No	X (μm)	Y (μm)	X/Y or Y/X
1	19.5	17.5	1.114286	12	25	20	1.25	23	25	22.5	1.111111
2	65	58	1.12069	13	35	47.5	1.357143	24	19.5	17.5	1.114286
3	15	15	1	14	70	40	1.75	25	65	58	1.12069
4	32.5	30	1.083333	15	62.5	45	1.388889	26	15	15	1
5	74	62.5	1.184	16	69	75	1.086957	27	20	22.5	1.125
6	90	75	1.2	17	35	25	1.4	28	15	20	1.333333
7	75	60	1.25	18	82.5	75	1.1	29	27.5	35	1.272727
8	30	27.5	1.090909	19	51.5	35	1.471429	30	15	15	1
9	35	30	1.166667	20	47.5	35	1.357143	31	21.5	32.5	1.511628
10	75	87.5	1.166667	21	75	67	1.119403	32	27.5	29.5	1.072727
11	80	65	1.230769	22	47.5	40	1.1875				

X/Y Average = 1,219266 ; Standard deviation ± 0,161891

MS Excel as shown in Table 1. From these values, CFSTE rates (X/Y or Y/X values) of these 32 powders were calculated. Then, the average CFSTE ratio was determined for this SEM photograph of the sample by taking the arithmetic mean of the CFSTE rates calculated for these 32 values. Then, the same method was repeated for an SEM photograph of the same sample on a larger scale and the arithmetic average of the two ellipse rates obtained from these two photos by calculating the ellipse value of the sample using Equation 1. As mentioned previously, this value is considered as the successful measure of mechanical alloying. As the method was summarized from SEM photograph of a sample in Fig. 4, the lengths of 32 X and Y axes were measured (Table 1) and the average ellipse rate for this photograph was determined as 1.219266 from the above-described method. Then, the same process was repeated for 50 units in another photograph of the same sample at larger magnification and the ellipse rate was calculated as 1.22894 and the CFSTE rate of the sample was found as 1.224103 by taking the arithmetic average of these values. Using this value, the ratio of ellipse in mechanical alloying was determined as $(1.224103 / 1.5) * 100 = 81.61$. At the end of each mechanical alloy test, at least 2 SEM photographs were obtained.

**Fig. 4.** SEM photo of a mechanically alloyed sample used in the calculation of CFSTE %.

Chemical analysis of CuCrZr powder alloy by EDX after mechanical alloying showed that Zr contents of these powders increased. Since the planetary mill used in mechanical alloying was used by the zirconia mortar and the zirconia balls, it was decided to follow the Zr content of powder alloy in mechanical alloying studies

and after each experiment. SEM-EDX chemical analysis of the ground powders was carried out to determine the Zr content of the powder mixtures and to investigate the effect of alloying parameters on the Zr content of the powder alloy from zirconia mortar and ball. This increase in Zr content has been seen as a problem, since Zr passing from mortar or ball to powder alloy is in the oxide phase, which will reduce the electrical conductivity of the final product. Therefore, in the selection of alloying parameters, has been decided to choose the ones with the Zr content of the alloyed powder 0.15 wt% or the closest. Hence the initial Zr content of the powder mixture in these studies was 0.15% and this value was held constant in all experiments in this study. As a result, the effect of the parameters on the CFSTE ratio and Zr content of alloy powders was investigated and optimum mechanical alloying parameters were determined.

3.3. Milling at 400 rpm

Fig. 5 shows SEM (BSE) images of the powder mixture milled in a planetary mill for 1 hour at 400 rpm. Accordingly, the amount of deformation increases at 400 rpm with increasing ball to powder ratio. The increase in the amount of deformation was characterized by flattening of spherical Cu powders. With increasing ball to powder ratio, collision statistics and stress intensity increase, so that deformation imposed on Cu increases. However, as shown in SEM photo Fig 5(a), spherical powders are not suitable for Cr and Zr penetration, as they have a large surface area but do not tend to be flake superficially. It is thought that by using the deformation ability of Cu, increasing plating, ex-foliation and hard and brittle powders such as Cr and Zr will be broken and fragmented and buried on Cu surface. As a result of planetary milling for 1 hour at 400 rpm is not sufficient for embedding of Cr and Zr into Cu powders for all ball/powder ratios.

Table 2. Effect of ball/powder ratio on CFSTE rate and Zr content of powder mixture. (1 h of milling at 400 rpm).

Ball/Powder ratio	% CFSTE ratio	Zr %
1/1	80,10	0,261
5/1	82,03	0,187
10/1	78,61	0,158
20/1	75,88	0,129

3.4. Milling at 500 rpm

In Figure 6, SEM images of alloy powders milled at 500 rpm for 1 hour are available. As can be seen, even in the 1: 1 ball/powder ratio, effective results were found better than 400 rpm, and the deformation intensity of the powders increased and the foliation was observed. However, again at this rate and within 1 hour, it was found that there were still powders close to the sphere in the mixture, so that there was not enough plate surface for Cr and Zr penetration.

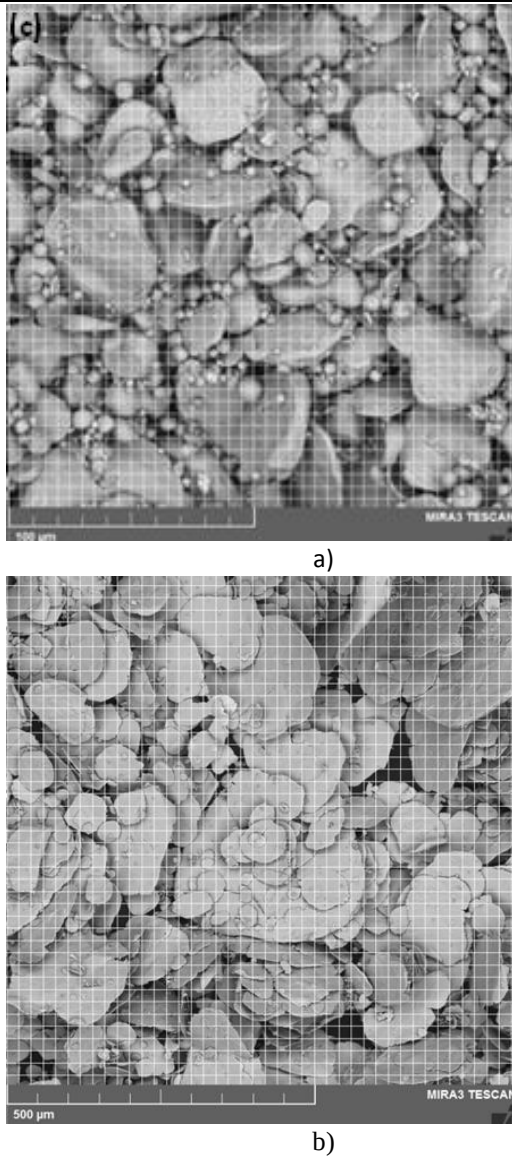


Fig. 5. SEM images of a) 10/1, b) 20/1 ball/powder ratios.

Table 3 shows the effects of ball/powder ratio on CFSTE ratio and Zr wt% determined by EDX analysis. The increase in ellipse shows that the deformation increases, the platened powder size becomes more brittle and tends to break, as well as thickening of the plates by stratification. One can conclude that a value of 1.3182 (87.88%) is one of the upper boundaries of ellipse as to be said that the ellipse value is close to 1.5 and the exact ellipse is the limit of deformation. ZrO_2 transport from balls to the powder mixture which seems to increase with the increasing ball/powder ratio is also thought to be increased with the statistics, number and intensity of collisions. According to these results, milling at 5/1 ball/powder ratio 500 rpm for 1 hour or milling at 1/1 ball/powder ratio 500 rpm for 5 hours is suitable for Cr and Zr penetration.

Table 3. Effect of ball/powder ratio on CFSTE rate and Zr content of powder mixture.

1 hour milling at 500 rpm		
Ball/Powder ratio	% CFSTE ratio	Zr %
1/1	86.73	0.104
5/1	83.17	0.147
10/1	82.21	0.185
5 hour milling at 500 rpm		
1/1	82.05	0.167
5/1	86.19	0.257
10/1	87.88	0.309

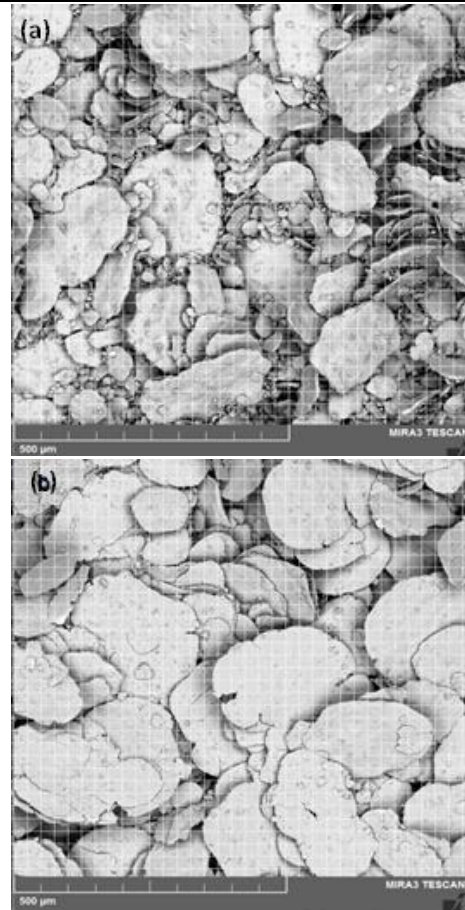


Fig. 6. SEM images of a) 5/1, b) 10/1 ball/powder ratio.

Fig. 7 shows the plating, Cr-Zr penetration and folding of Cu powder at a speed of 500 rpm and different durations. At low deformation times, the layers have just begun to overlap, but surface cracks have also occurred, and the penetrating Cr is partially retained in the grain shape, and the Cu surface is still smooth. At increasing speed, it is seen that the smoothness of the Cu surface is deteriorated and the shape of the Cu powder becomes close to the circle due to deformation. The effect of increased deformation on the stratification of the Cu plate can also be observed as folding or tearing. As can be understood from this, as the deformation increases, Cu can be folded and deposited on itself, leading to agglomeration of Cr and Zr penetration between them and subsequent plate breakage.

4. Conclusions

The ZrO_2 content of the powder mixture increases with increasing ball / powder ratio. Therefore, high ball / dust ratio should be avoided, especially at high speeds and long grinding times. Otherwise, the electrical conductivity of the composite material will decrease due to ZrO_2 . Milling at 5/1 ball/powder ratio, 500 rpm for 1 hour or milling at 1/1 ball/powder ratio, 500 rpm for 5 hours can be suitable for Cr and Zr penetration into Cu particles.

Acknowledgments

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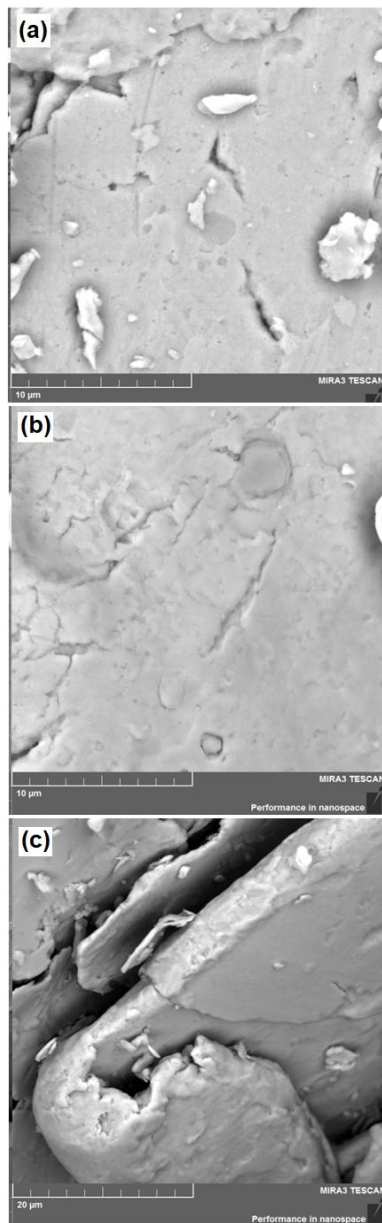


Fig.7. (a) Cr-Zr embedding morphology in powder rotated at 10: 1 ball / powder ratio at 500 rpm for 3 hours, (b) Cr-Zr embedding morphology in powder rotated at 10: 1 ball / powder ratio at 500 rpm, (c) stratification and excessive deformation of the plates of powder rotated with a 10: 1 ball / powder ratio at 500 rpm for 5 hours.

Al MATRIX FGM HAVING 5, 15 AND 25 % B₄C_p REINFORCED LAYERS

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Abstract: Functionally graded materials (FGM) containing different amounts of B₄C particles within 4 layers in an aluminum matrix were produced and characterized. The layers contained B₄C particles at amounts of 0, 5, 15 and 25 volume %. In order to form the composite structure, pure aluminum (<10 microns), copper (1 micron) and B₄C powders (<10 microns) were utilized. After mixing, functionally graded materials were compacted and shaped in a rectangular cross sectional geometry, having 6.4 mm thickness, 12 mm width and 33 mm length, by cold pressing at 600 MPa pressure. The parts were pressureless sintered at 610 °C for 30 min in high purity nitrogen atmosphere. The properties of the FGM sample were compared with those of the sample which did not contain B₄C particles, and which contained 25% B₄C particles throughout the whole sample.

Microstructural examinations were performed by an optical microscope. 3- point bending tests were conducted by a universal testing machine. The sample that did not contain B₄C particles presented a 3-point bending strength of 380 MPa, whereas the sample that contained 25 % B₄C had a bending strength of 140 MPa. The FGM sample had a bending strength of 190 MPa. However, the failure of the FGM sample was composed of steps. The stepwise failure of the FGM sample was due to delamination and fracture of the layers containing 15 and 25 % B₄C. Microhardness values of each layer the samples were determined by Vickers micro hardness measurements.

Keywords: FUNCTIONALLY GRADED MATERIALS, ALUMINUM MATRIX, B₄C

1. Introduction

Continuous advancements in the modern industry have brought about the request for unconventional materials and properties. This necessitated a renovation of materials from their monolithic forms to composites, properties of which could be tailored as a function of their constituent's type and ratio [1-3].

Functionally graded materials (FGM) in a sense can be thought to be in the category of composite materials. They exhibit a graded structure and gradually changing properties, and they are constructed to provide a greater performance [1-3].

The graded structure of the FGMs can depend on variation in factors such as density, grain size, chemical composition or other physical properties. The change in the structure, brings about the variation in the properties, on an axis of the FGMs. The change in the structure or properties may be abrupt in passing from a layer to a subsequent layer, or it may be gradual. The gradual transition in the structure and properties eliminates the incompatibility that would otherwise take place in an abrupt transition [1-3].

The most common example on the benefits provided by the FGM structure is the elimination of thermal stresses. For example, in the combination of two dissimilar materials such as a metal and a ceramic, the thermal stress caused by the different amount of expansion of the joined materials leads to crack or delamination of the joint. Thus, a gradual change in thermal expansion coefficient, created by the FGM structure, could be useful to prevent the thermal stresses and delamination in the material [1-3].

Powder metallurgy, which involves the formation of articles by starting from their particles, has the advantage of microstructural and compositional control not only in the whole body of an article, but also as a function of position on an axis. It is a widely used technique which provides production with net shape. It is sometimes more economical and faster than other manufacturing methods. Therefore the formation of compositional gradients is mostly easier with powder metallurgy, than other production techniques. Formation of layers as thin as 1 mm is possible with this method by piling layers on one another [1,3-5].

In literature, B₄C, SiC, Al₂O₃ and TiB₂ have been used as reinforcement phase in aluminum matrix in FGM structure [2]. Centrifuge casting is utilized in most of the studies in literature on FGM [2].

In the study of Canakci et al. [4], Al-10%B₄C and Al-5%-10%B₄C FGMs were prepared by powder metallurgy. It was reported that hardness of the layers increased with increasing B₄C content.

In the present study, powder metallurgy was employed for formation of FGM which consists of layers having 5, 15 and 25 % B₄C reinforcement particles in an Al-%4Cu matrix.

2. Experimental Procedure

The unreinforced Al-%4Cu sample, sample reinforced with 25 vol.% B₄C having Al-%4Cu matrix and the FGM sample were produced by powder metallurgical techniques. The first layer of the FGM was unreinforced Al-%4Cu alloy. The other layers of the FGM were essentially individual metal matrix composites, having 5, 15 and 25 vol. % B₄C reinforcement particles in Al-%4Cu matrix. Utilized powders were pure aluminum powder (Alfa Aesar, purity >99.5%, particle size <10 microns), Copper (Merck, purity >99.7%, dendritic, -63 micron), B₄C (Alfa Aesar, purity >99, <10 microns).

Al, Cu and B₄C powders were weighed in proper amounts, separately for the 4 layers of the FGM. The amounts of the constituents of each layer are presented in Table 1. The theoretical thickness of each layer was calculated to be 1.6 mm and the total thickness of the FGM was planned as 6.4 mm. The width of the sample was 12 mm and length was 33 mm. These dimensions are suitable for 3-point bending sample according to ASTM B528 standard entitled: Standard Test Method for Transverse Rupture Strength of Metal Powder Specimens. The unreinforced sample contained Al-%4Cu of 7.768 g. The sample having 25 % B₄C in its whole body was composed of 5.826 g Al-%4Cu and 1.762 g B₄C.

Table 1: Amounts of the constituents of each layer in FGM.

Layer No	B ₄ C Vol. %	Wt. Al-%4Cu (g)	Wt. B ₄ C (g)
1	0	1,94	0
2	5	1.8449	0,0881
3	15	1.6507	0,2643
4	25	1.4565	0.4405

Al, Cu and B₄C powders were mixed separately for each layer and the mixtures were placed on top of each other in a tool-steel die in layered fashion. The mixtures were lightly pressed after each layer. The parts of the steel die were covered with stearic acid, in order to reduce die friction and to prevent the sticking of the pressed article to the die. After weighing and measuring the dimensions of the samples, they were placed in the hot zone of a tube furnace. Samples were sintered at 610 °C for 30 min in high purity nitrogen atmosphere. After sintering, they were subjected to microstructural examinations by optical microscope (Nicon Eclipse, LV150),

microhardness measurement in HV0.1 scale and 3-point bending tests (Shimadzu AG-IC 50 kN).

3. Results and Discussion

After sintering FGM sample and unreinforced Al-4%Cu sample presented a slight distortion in the form of bending in one direction. Therefore a slight curvature formed on one side of the samples. This effect was more pronounced in the FGM sample as compared to unreinforced Al-4%Cu sample (Fig.1). Two reasons can be proposed for this behavior. One is the uneven green density of the article, origination from uni-axial pressing in the die. The second is the uneven green density of the article, originating from the different amount of B_4C particles in the layers of the FGM. On the contrary to other samples, this effect was not observed in the 25 % B_4C reinforced Al-4%Cu sample, and that sample was flat.

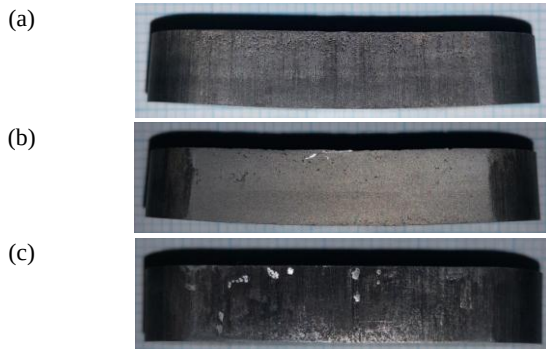


Fig. 1 Macro images of the samples after sintering. (a) unreinforced Al-4%Cu, (b) FGM, (c) 25 % B_4C reinforced Al-4%Cu.

Micrographs of the layers in the FGM structure containing 5, 15 and 25 % B_4C are presented in Fig.2. It can be seen that B_4C particles are in general homogenously distributed in the microstructure. The amount of B_4C in the microstructure increases in accordance with the reinforcement content of the layers.

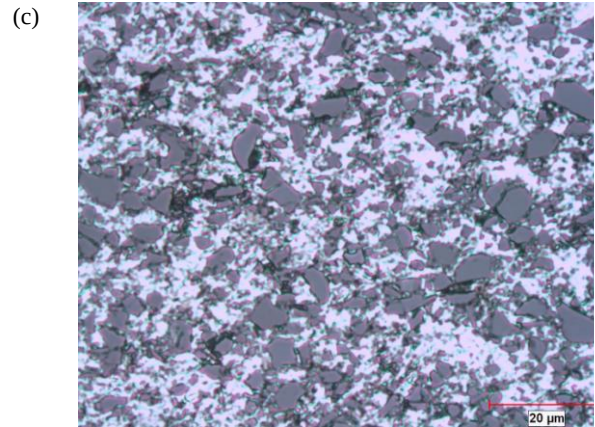
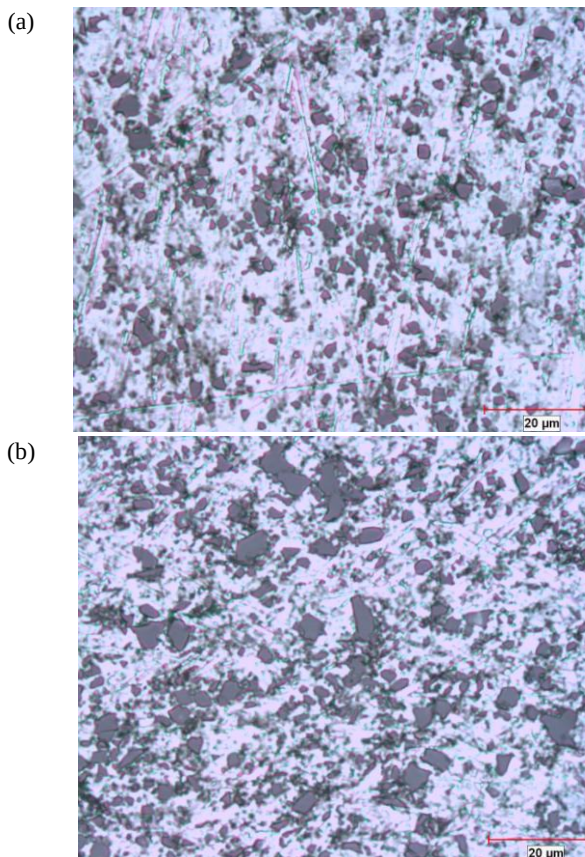


Fig. 2 Optical microscope images of the layers after sintering, containing (a) 5% B_4C , (b) 15% B_4C FGM, (c) 25% B_4C .

It can also be seen that porosity (black regions in the micrographs) increases in the structure with the increase in the amount of B_4C particles. The pores appear to form among the groups of B_4C particles (Fig. 2(c)). Higher sintering temperature or pressure assisted sintering may be effective in reducing these pores.

Microhardness values of the layers of the FGM sample is presented in Fig.3 as a function of B_4C content. Hardness of the unreinforced layer was 60.0 ± 4.14 HV0.1. Hardness increased steadily with the increase in the amount of B_4C reinforcement particles in the layers. Hardness of the layer containing 25 % B_4C was 115.7 ± 1.87 HV0.1.

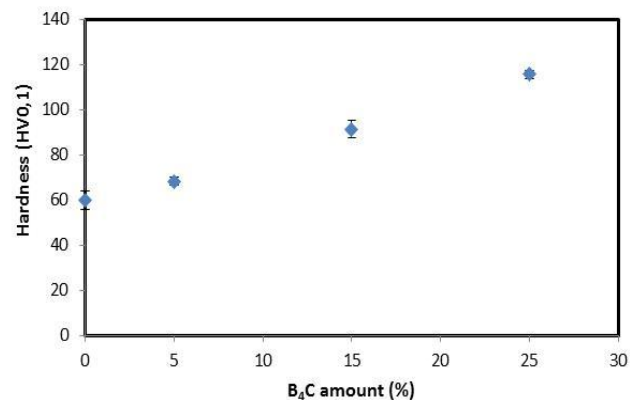


Fig. 3 Vickers microhardness values of the layers in FGM as a function of B_4C content

Stress-strain curves of the samples obtained by three point bending tests are presented in Fig.4. The unreinforced sample exhibited a bending strength of 375 MPa, with about 25 % strain. Therefore the unreinforced sample presented a quite ductile behavior, with high amount of plastic deformation. The macro image of the unreinforced sample given in Fig. 5(a) supports this outcome.

The composite sample containing 25 % B_4C in its whole body had a bending strength of 150 MPa, with about 3.6 % strain. This results indicates that the sample reinforced with 25 % B_4C fails in a brittle manner. Indeed, the macro structure of this sample presented no indication of plastic deformation (Fig. 5(c)). The reason for the brittle behavior may be due to the discontinuity of the matrix phase due to the presence of B_4C particles and the pores.

The FGM sample, on the other hand, presented a behavior that is in between the unreinforced sample and the sample reinforced with 35 % B_4C , in terms of bending strength and strain. Bending strength of the FGM sample was 150 MPa and strain was 10.7%.

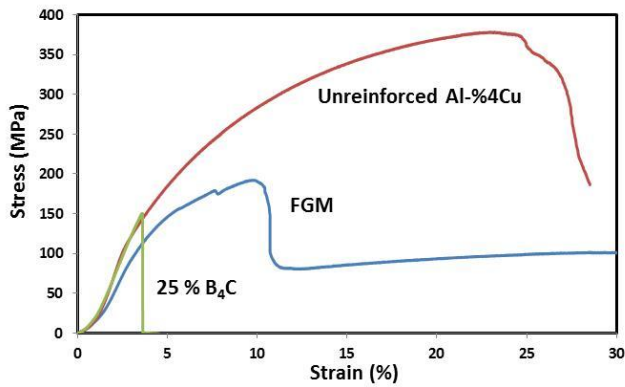


Fig. 4 Stress-strain plots of the samples obtained by three point bending tests.

The fracture of the FGM sample exhibited steps, in which the stress on the sample decreased during the bending test. The first step in the stress-strain plot of the FGM sample took place due to interlayer separation during the bending test. The interface between the layer containing 5 % B_4C and 15% B_4C was seen to delaminate horizontally during the test at this point. This delamination indicates the poor bonding of the matrix alloy between these layers during sintering. The poor bonding may have resulted due to pressing after laying each layer in the die.

The second and larger drop in the stress value that took place at about 10.7 % strain was due to vertical cracking of the layers. The cracking occurred in the top 2 layers of the FGM, which contained 15 and 25 % B_4C . These layers are expected to be less ductile than the other layers of the FGM. The vertical cracks in these layers can be seen in Fig.5(b). The bottom two layers of the FGM did not exhibit vertical cracking, indicating their ductile behavior. They presented high amount of plastic deformation.

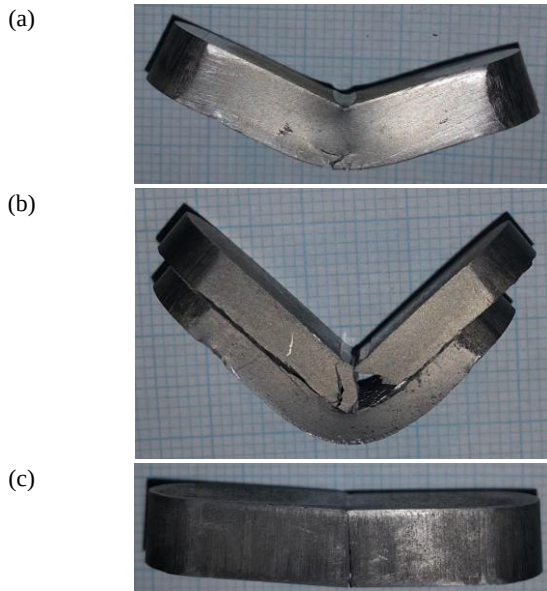


Fig. 5 Macro images of the samples after 3 point bending test. (a) unreinforced Al-4Cu, (b) FGM containing 0, 5, 15, 25 % B_4C reinforced layers, (c) 25 % B_4C reinforced Al-4Cu.

4. Conclusion

Functionally graded materials (FGM) containing B_4C particles at amounts of 0, 5, 15 and 25 volume % within 4 layers in an aluminum matrix were produced and characterized. The plain sample had a 3-point bending strength of 380 MPa, whereas that of the 25 % B_4C composite was 140 MPa. The FGM sample presented a bending strength of 190 MPa. The failure of the FGM sample was composed of steps which were suggested to originate due to delamination and fracture of the layers containing 15 and 25 % B_4C . It can be proposed that in order to obtain better mechanical properties from the FGM, a better bonding between the interlayers is crucial.

Acknowledgements

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FINITE ELEMENT SIMULATION ON TENSILE CREEP BEHAVIOUR OF UNDERGROUND SUPPORT LINER

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Abstract: Polymer-based products are widely used as load-carrying components in different structural applications due to the ease of manufacture, installation, and long lifetime properties. In mining and tunnelling industry, fast-setting, thin polymer-based products are in demand as an underground support liner. Researchers have agreed that the time-dependent material properties of underground support liners have significance for short to long term applications. Although some creep tests were performed in literature, there is no available study on the numerical analysis/simulation of the creep behaviour of underground support liners. In this study, viscoelastic mathematical models developed for two different underground support liners were analyzed in ABAQUS finite element numerical modelling software with a developed subroutine. After the implementation, tensile creep test specimens were modelled to verify the new subroutine. There are two main purposes of the simulation of experiments; Firstly, to verify that the subroutine works in accordance with the actual behaviour of the material, secondly, to obtain realistic creep behaviour results for cases where experiments were not performed. As a result, a good agreement was obtained between the mathematical model predictions and numerical results for different stress levels. The proposed subroutines may create a basement for future numerical studies.

Keywords: CREEP, THIN SPRAY-ON LINER, MINING, UNDERGROUND SUPPORT, FEM

1. Introduction

Support elements and systems have a crucial role to stabilize excavations and to reduce inward movement of the rock mass. Advancing deeper in underground mining has led to the necessity of different types of support systems by the help of the technological improvements¹. Researches have extensively focused on the issues of developing support systems. These studies have assisted in the increase of various support systems with different functions. The product “Thin Spray-on Liner” (TSL) is developed as an alternative of widely used surface support materials, mainly shotcrete, in the beginning of 1990s. TSL’s are fast-setting multicomponent polymeric materials applied on rock surface with a few mm thickness that have fairly high tensile strength, adhesion, and elongation capabilities. They are recently still being used only at around 150 mines and underground openings all around the world, as the support mechanism and time-dependent material properties are not fully understood yet. Laboratory tests and numerical studies have significant importance to understand the support mechanism and to make a comparison available for TSL products. Many different laboratory test setups are available in the literature. Those are creep, tensile strength, uniaxial compressive strength, punch, bonding strength, double sided shear strength, plate pull, linear block support, gap shear load, and coated core compression tests. In addition to laboratory studies, numerical studies are widely used tools in the last decade to simulate TSL laboratory testing and field conditions. These studies are summarized in Table 1.

Researchers have agreed that creep properties of TSL’s being important design parameters and that creep might become a serious problem due to the polymer ingredients. Further research is needed to evaluate the performance of polymer liners under sustained loading conditions².

Although some creep tests were performed in literature^{3,4}, there is no available study on the numerical analysis or simulation of the creep behaviour of underground support liners..

In this study, viscoelastic mathematical models for two different underground support liners were developed and analyzed in ABAQUS finite element numerical modelling software with a developed subroutine. After the implementation, tensile creep test specimens were modelled to verify the new subroutine. There are two main aims of this study. The first one is to verify the subroutine acts in accordance with the actual behaviour of the material. The second is to obtain realistic creep behaviour results where the experiments were not performed.

Table 1: Numerical TSL Studies in the Literature.

Authors	Model purpose	Numerical method
Tannant and Wang (2003) ⁵	Tensile, Block Punch tests and tunnel model	Discrete element
Malan and Napier (2008) ⁶	Fractured underground excavation (square)	Boundary element
Richardson et al. (2009) ⁷	Bending, Double sided shear test	Finite difference
Dirige and Archibald (2009) ⁸	Underground excavation (horseshoe)	Finite difference
Nater and Mena-Cabrera (2010) ⁹	Underground excavation (square)	Distinct element
Guner and Ozturk (2016) ¹⁰	Tensile testing	Discrete element
Guner and Ozturk (2017) ¹¹	Tensile and compression testing	Finite element
Li et al. (2017) ¹²	Gas permeability	Finite difference
Komurlu and Demir (2017) ¹³	Linear block support	Finite element
Lee et al. (2018) ¹⁴	Linear block support	Finite element

2. Materials and Creep Model

Numerical models were conducted for two widely used underground support liner products. Product A has two components; the liquid-component is a resin latex and the powder-component is a hydraulically curing powder based on special cement. Components are mixed with 2:1 liquid-powder ratio by weight. The tack-free time of the product was recorded as 30 minutes. Product B is a single component polymer-based powder in spray applications on rock faces for surface support purposes. The suggested mixing ratio is 2:1 powder/ water by weight. The tack-free time of the product was recorded as 5 minutes. It should be noted that Product A has cured for 1 day, on the other hand, Product B has cured for 2 days.

Depending on the loading conditions and environmental effects, polymeric materials can show different mechanical responses. They generally exhibit strong viscoelastic behaviour at room temperature.

Creep is a time and temperature-dependent behaviour and many different theoretical models have been used to describe viscoelastic behaviour of the material. Most of them are based on simple models consisting of springs and/or dashpots and try to describe the viscoelastic behaviour of various polymeric materials.

In this study, the nonlinear multi Kelvin–Voight modelling approach, which can be obtained from rheological models, was followed. The model is represented by Kelvin elements arranged in series, and a single spring. The single spring provides the elastic response and Kelvin elements provide the viscous response as shown in Fig. 1.

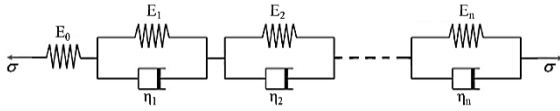


Fig. 1 Multi Kelvin (Voigt) model

Each Kelvin element in the series represent time intervals. Therefore, this model approach can be considered as suitable for longer testing periods. Based on the relationship between the constitutive elements, creep behaviour can be calculated as follows:

$$\varepsilon_t(t) = \frac{\sigma_n}{E_0} + \sum_{i=1}^n \frac{\sigma_n}{E_i} [1 - \exp(-t/\tau_i)] \quad (1)$$

where σ_n is the applied constant normal stress level, E_0 is the instantaneous elastic modulus, η_i and E_i are the dashpot viscosity and the spring stiffness of i th Kelvin element. η_i/E_i is the retardation time (τ_i). To simplify calculations, researchers generally take retardation times (τ_i) as constant values for different models. In this study, τ_1 was taken as 0.2 min and following retardation times were taken as 10 times of the previous Kelvin unit.

The experimental tensile creep test results and creep models used in this work have been reported by Guner and Ozturk (2019)⁴. Equation (1) was used to fit corresponding experimental creep curves using a computer routine providing a Levenberg-Marquardt algorithm. The 4 nonlinear viscoelastic models were generated for each product. Table 2 shows the model coefficients of the Multi Kelvin (Voigt) model.

Table 2: Model Coefficients for Creep Models⁴

Product A				
Stress (MPa)	K*	E0	E1	E2
1.06	4	21	1.45E+08	21
0.79	4	38	1.95E+07	72.07
0.53	6	52	9.18E+01	221.46
0.26	6	65	6.57E+01	40.7
Stress (MPa)	E3	E4	E5	E6
1.06	10.5	1.3	-	-
0.79	17.49	4.19	-	-
0.53	26.04	15.87	159.1	16.3
0.26	15.1	20.9	3590	20.4
Product B				
Stress (MPa)	K*	E0	E1	E2
1.46	4	28	5.10E+01	1.25E+02
1.10	4	116	6.59E+01	9.47E+01
0.74	3	140	1.77E+02	4.14E+01
0.37	3	205	3.26E+07	1.41E+02
Stress (MPa)	E3	E4	E5	E6
1.46	1.94E+00	-	-	-
1.10	7.78E+00	-	-	-
0.74	1.31E+01	1.23E+01	-	-
0.37	1.65E+01	5.68E+01	-	-

*Number of Kelvin Elements

This multi Kelvin model was represented as a subroutine in ABAQUS.

3. FEM Implementation of the Multi Kelvin Model

In ABAQUS software, 5 different creep laws are available. These laws are the power, the hyperbolic-sine, the Anand, the Darveaux, and the double power. Although, these default creep models provide significant convenience to the user, in some practical cases material creep behaviours are typically of very complex form to fit experimental data. Therefore, the creep laws are frequently user-defined via the use of user subroutine CREEP and included in a generic time-dependent, material formulation. Multi Kelvin or Voigt model is not standard model in ABAQUS and hence needed to be incorporated via the CREEP user subroutine.

In order to create the user-defined creep model in ABAQUS, the elastic material behaviour should be defined first. In the creep experiments, the instantaneous strain change that occurs when the samples are sustained under constant load is called elastic strain (ε_e). This elastic strain value also depends on the stress applied in the experiments performed under the same curing time. The parameter which controls the elastic strain behaviour is called the instantaneous elastic modulus (E_0), as mentioned in the previous section. E_0 values, presented in Table 2, depend on the applied stress levels. The relationship between E_0 and applied stress is presented in Fig.2.

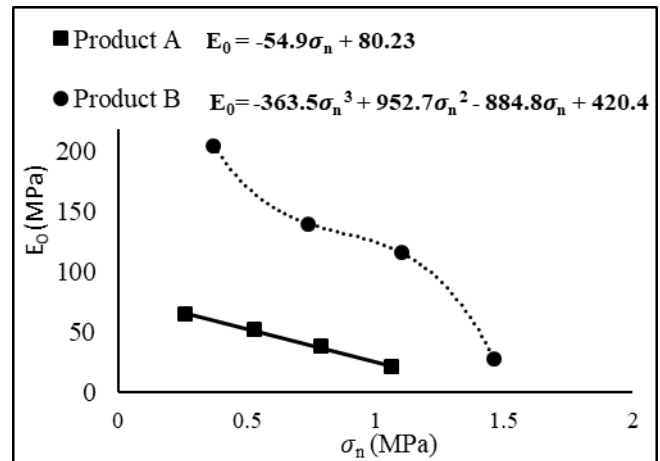


Fig. 2 The relationship between E_0 and σ_n

In this context, to specify the predefined field variables (E_0) called “UFIELD” in the software, the user subroutine was prepared by using the relationship presented in Fig 2.

Since the instantaneous strain change occurs when the samples are sustained under constant load, creep is not active in this step. It is recommended that small times step compared to the creep time be used in these steps. Therefore, this time step in which the sample takes instantaneous strain was taken as 0.01 minutes.

CREEP user subroutine defines the increments of inelastic creep strain as functions of the solution dependent variables, such as the deviatoric stress, pressure, temperature, and time increment Δt ¹⁵. A general creep equation (equation 1) is required to be discretized to fit available integration schemes. The derivative of the proposed Multi-Kelvin model has been defined on the assumption that the subroutine will be used with implicit integration. A user subroutine CREEP was coded in Fortran by using the obtained model coefficients. The validation of the developed creep model and the previously extracted parameters was performed using a tensile creep model.

The mesh structure of the tensile creep sample formed in this context is presented in Fig. 3. The meshed geometry consists of rectangles with 20 nodes and 1488 elements (C3D20R). The C3D20R element is a general-purpose quadratic brick element, with reduced integration (2x2x2 integration points).

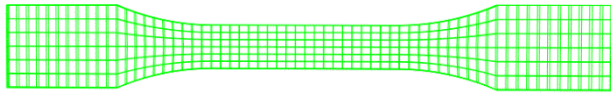


Fig. 3 The mesh geometry of the specimen

In FE creep test validation, first of all, the simulations of experiments were carried out for 4 stress levels in which the experiments were performed. It should be noted that validations were carried out with different subroutines for each product. At the end of the analysis, the experimental data were compared with the Multi-Kelvin model and finite element model results and highly consistent results were obtained. The software outputs showing the total strain values and strain distributions at the moment of rupture in the Finite Element validation models prepared for two different TSLs are presented in Fig. 4 and Fig. 5.

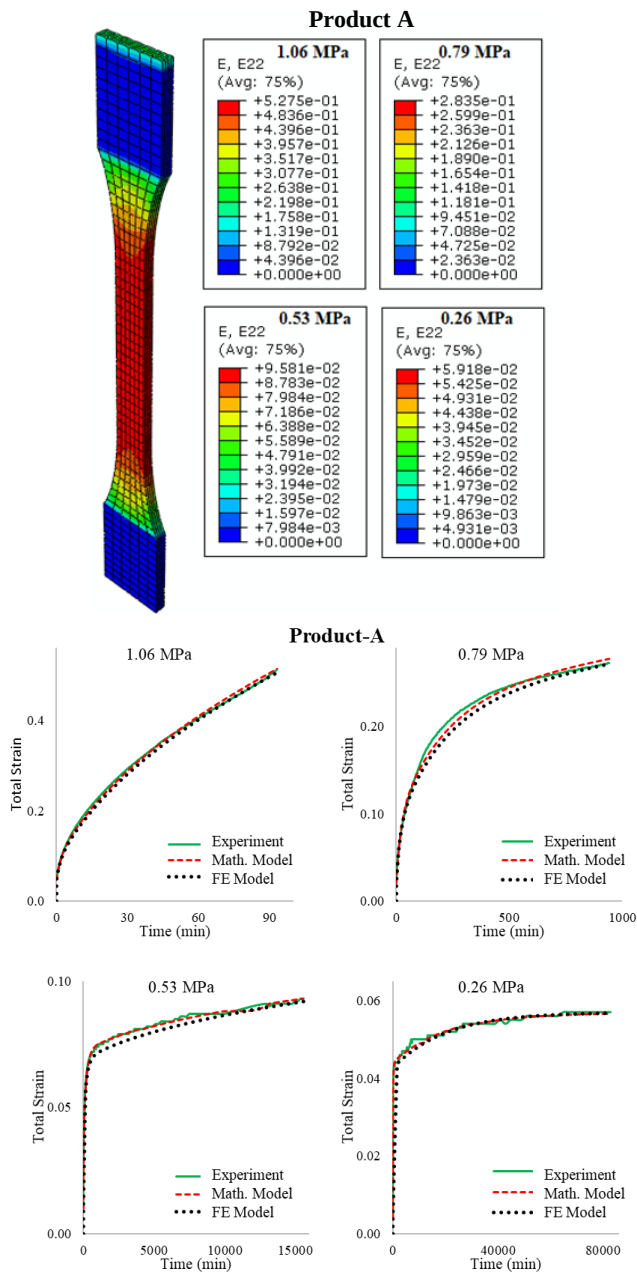


Fig. 4 FE creep modelling comparisons for Product A

The second aim of this study is to obtain realistic creep behaviour results for cases where experiments were not performed. Therefore, in addition to the model validation studies, additional validation models for Product B were performed to ensure that the creep behaviour was consistent in the intermediate stress levels.

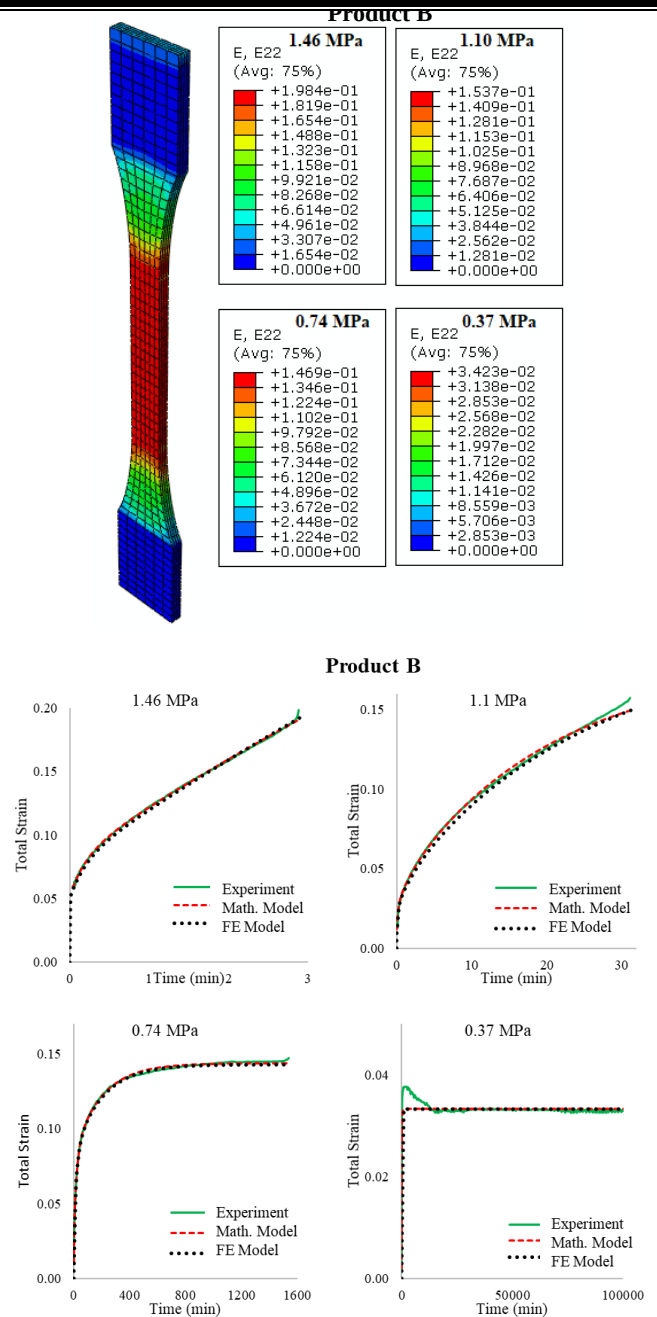


Fig. 5 FE creep modelling comparisons for Product B

For additional validation models, constant stress values of 1.27, 0.92, and 0.56 MPa were applied to the sample, respectively. The strain-time graphs of the models under these stress values are presented in Fig. 6. As testing duration varies from a few minutes to 2 months, for the better understanding of the behaviour, the first 100 minutes and the whole graph (log scale) are presented separately. Besides, the failure envelope obtained at the end of the experiments can be seen in the graphs.

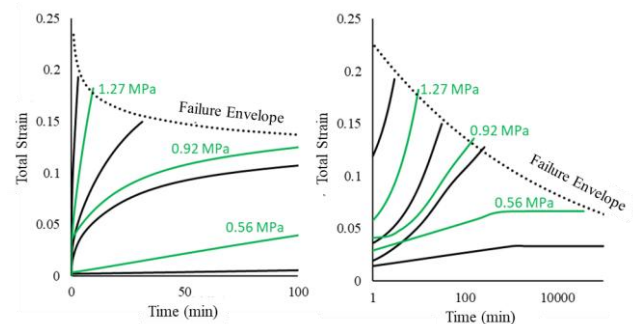


Fig. 5 FE creep modelling for intermediate stress levels

As a result of the validation models, The correlation between the experimental creep strain, Multi Kelvin creep model, and numerical creep strain obtained using ABAQUS Finite Element Analysis were found to be adequate.

4. Conclusions

This study presents the numerical model implementation of the generated viscoelastic creep mathematical models for two different underground support liners. Multi Kelvin mathematical models were introduced to ABAQUS finite element numerical modelling software with a developed subroutine. After the implementation, tensile creep test specimens were modelled to verify the new subroutine. Since the instantaneous elastic strain part depends on the applied stress levels, user subroutine UFIELD was used in this section. The creep component was successfully modelled in CREEP user subroutine employing a Multi Kelvin model. The developed subroutines may be used as an input parameter in future large scale numerical studies of time-dependent behaviour of surface support liners.

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FAILURE CHARACTERIZATION OF ROCK ANCHOR BOLTS BY THERMAL CHANGE DETECTION UNDER TENSILE STRENGTH TESTING

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Abstract: Rock bolts are widely used in tunnels, underground openings, and also rock slopes to provide support and attach blocks to the rock mass in various engineering fields. They have a significant role for the stability of underground structures and natural rock slopes both in the mining and civil engineering industry. The understanding of the rock bolt behavior under stress conditions plays a key role in the long term stability and sustainability of rock slopes, nearby structures, and underground openings. The detection of the failure type and the investigation of failure behavior can be supported by thermal imaging of certain regions on the anchor bolt during tensile testing. In this context, anchor bolt behavior under tensile testing with a force controlled testing environment was analyzed with a LWIR (Longwave Infrared) thermal camera. The heat dissipation during experiments was recorded by a thermal camera together with the force reading from the hydraulic press data logger. As a result of the experiments, it was seen that there is a relation between the temperature change and bolt failure behavior. Therefore, it is expected that this study will provide an opportunity to detect the failure characteristics of the anchor bolts under tensile strength test by the help of infrared thermal camera.

Keywords: ROCK BOLT, ANCHOR BOLT, TENSILE STRENGTH TEST, PULL-OUT TEST, INFRARED THERMAL CAMERA, FAILURE CHARACTERISTICS OF BOLT

1. Introduction

The main purpose of mining activities is extracting the resources for energy generation and raw material requirement. Because of the decreasing availability of the shallow resources, importance of operating underground operations has increased and will become more crucial in the near future. For this reason, new resources are explored consistently. The exploration of new resources aims underground mining operations that could be operated safely and more efficiently. Rock mechanics is a field of research that focuses on the analysis of rock behavior under stress and tension conditions. It has a crucial role for underground activities. Therefore, integrating new technologies such as thermal cameras, acoustic emissions, high speed cameras, etc. to mining industry, especially rock mechanics, plays a key role for safer and more efficient mining.

Rock bolts are defined as long anchor bolts and are used for stabilizing rock excavations for engineering purposes. The main aim of using of rock bolts is to transfer the load from the unstable object to a stable object. The correct design of the rock bolts is very substantial for safety and sustainability of mining operations (Hoek, Kaiser & Bawden, 2000; Peng & Tang, 1984). To ensure the continuity, capacity of bolts, and possible failure types should be known properly (Ballarini, Shah & Keer, 1986). This knowledge can be achieved by performing experiments in laboratory environment and on field.

To determine the strength of the rock bolts, some experiments can be carried out. In laboratory testing, tensile strength tests can be performed to determine the yielding force and the maximum tensile force that the material can withstand. As a result of such experiments, the structures can be designed in the light of information regarding the strength of the required support. On site, pull-out tests are carried out to determine the maximum tensile force to pull the bolt from the rock surface regarding the testing equipment used. This study involves the tensile testing and pull-out tests of rock bolts for a specific site. As a result of the experiments, the capacities of bolts and failure types were compared.

Laboratory tests were monitored by a longwave infrared thermal camera to detect the failure region of the bolts during loading stages. The reason of utilizing the thermal camera instead of a video camera is to track the change in the temperature along the failure region of bolt and try to related it with the failure region.

Researchers in this field commonly worked on finding out the failure modes of rock bolts by experimental testing. These failure

types are investigated for bolts under tensile loading, bolt-grout interface failures where the bolts are pulled out of grout, slipping of the bolt with grout from the rock, and failure of the rock (Brown, 2015; Yang, Thota & Zhao, 2015). There are suggested design methods for each type of these failure modes. To prevent the failure, possible failure region of the bolt should also be well defined. By considering the failure region, more reliable and critical bolt selection and design can be performed. Figure 1 shows the failure modes of rock bolts.

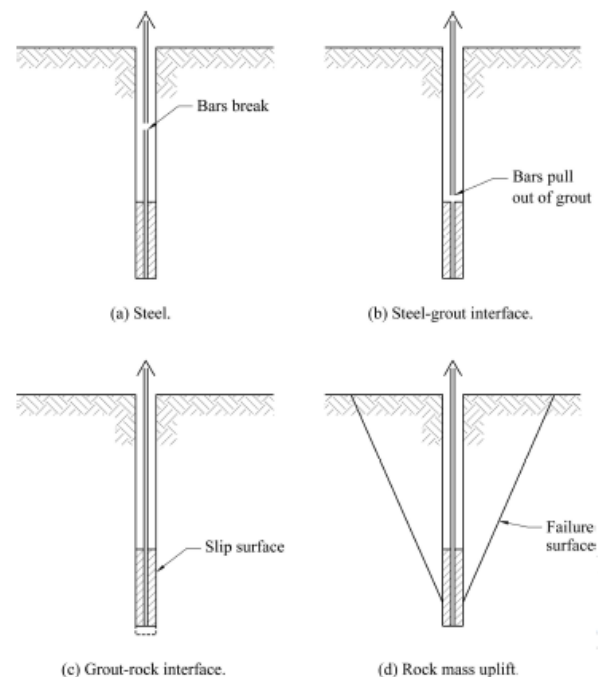


Figure 1: Failure modes of rock bolts under tension (Pease & Kulhawy, 1984)

Based on these reasons, this article aims to provide an application of thermal imaging to detect failure type and failure behavior of rock bolts under tension. The results are based on laboratory testing and also pull-out tests conducted on site. This provided an insight to the field conditions of the rock mass that is planned to be supported by rock bolts. The isolated environment of the rock mechanics laboratory might cause deficiencies in representing the surface conditions of the rock mass and also the impact of weathering and other environmental conditions.

2. Objectives and Methodology

The samples used in the experiments were steel rods of two different types, which are non-ribbed and ribbed type. Non-ribbed steels have rather a flat surface whereas rods with protruding surface are named as ribbed steel. Ribbed steels have greater tensile strength compared to non-ribbed steels. They provide better interlocking properties when used together with rock minerals. By these ribs, the displacement of the steel material within the rock mass becomes more difficult. Ribbed steel types of rods were used for the experiments within the scope of this study. According to the technical specifications about the steel, their minimum yielding strength is 420 MPa and the minimum tensile strength is about 500 MPa with a minimum final elongation is 10%. These values were compared with the experimental results.

The reason of using thermal imaging technology in rock mechanics experiments is that an essential engineering field should always be improved by integrating new technologies to achieve more reliable data at critical time intervals. The current devices used for rock mechanics testing can be misleading because of the weak remote sensing capability of the equipment. Hence, some inferences must be made for interpreting the results of experiments. By integrating new technologies to this research field, these inferences can be minimized to a certain extent and can be based on more information.

The objectives of this study can be summarized as to achieve more detailed and visual data about the failure moment of rock bolts and to compare field and laboratory test results. It was planned to investigate the concept whether stress concentration points are related with temperature increase on the tested sample or not. Regarding this problem, some laboratory experiments on the bolt samples were performed at the rock mechanics laboratory with a load controlled equipment. One of the tests was monitored by a longwave infrared thermal camera to collect thermo-images. The load controlled testing machine was used for tensile stress determination. In addition, some field experiments were performed to compare the laboratory and field test results. Figure 2 and Figure 3 represent the experimental setup and the infrared thermal camera that was used, respectively.



Figure 2: Experimental Setup, Force controlled test machine



Figure 3: Uncooled longwave infrared thermal camera

Methodology of this study covers the field tests and laboratory tests of rock bolts. On site, ten pull out tests were conducted and the results were recorded. Different types of grout and epoxy were used to test the adherence of the contact surface between the rock mass and the rock bolt. In laboratory testing, tensile strength tests of rock bolts were performed. One of the tests was monitored by an infrared thermal camera. The infrared thermal camera was placed in front of the sample at about 1 meter distance for safety precautions. The temperature changes recorded on the rock bolt could be followed by the software of the thermal camera. In this software, an average temperature curve on selected lines and a histogram plot can be visualized in real-time and also as a recording. The maximum and minimum temperature values on selected lines can also be recorded as separate profiles. These values were monitored during the laboratory experiments. As a result of this thermal camera recording, the difference between the maximum and the minimum temperature provided the temperature increase on certain regions of the sample. Hence, certain relations could be examined between stress concentration points and temperature increase. The graphical user interface of the infrared thermal camera software can be seen in Figure 4.

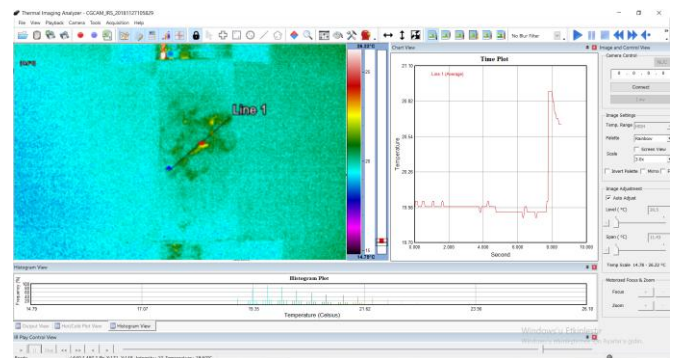


Figure 4: Interface of the infrared thermal camera software

There are research questions related to the utilization of thermal cameras, such as the aim of using this equipment or the available technology. According to Fematek (2018), the reason of utilizing this equipment is that thermal energy is observed at the range of infrared, which cannot be seen with naked eye by human beings. Thermal cameras convert the readings about heat to images and figures that can be interpreted easily. The long wavelength infrared thermal cameras work in 8-14 micron band, mid wavelength infrared thermal cameras work in 3-5 micron band, and short wavelength infrared thermal cameras work efficiently in ranges up to 1.5 micron. At room temperature, the wavelength emitted from an object is at most about 10 microns. At 300° C temperature, the wavelength emitted by an object is approximately 5 microns. The sun wavelength emits around 0.5 microns. Therefore, the most ideal thermal camera setup for laboratory testing of samples is commonly suggested as a long wavelength infrared thermal camera as it was chosen for this study. The reason of utilizing an uncooled thermal camera system is that the cooled systems are much more expensive and require maintenance

frequently because of the separate cooling apparatus. Cooled systems are preferred mostly for long distances whereas uncooled systems do not operate together with cooling systems of the detectors. These detectors are named as micro bolometers and the temperature change in the object is converted to electrical signals. Uncooled systems have to work with longwave infrared thermal cameras whereas cooled systems can be operated with middle wave and longwave infrared thermal cameras. Resolution of the thermal camera used in this study is 640 x 480 pixels which provides a comparably better detail in images than cameras with 160 x 120 and 320 x 240 resolution. The first stage of the determination of tensile strength of rock bolts was based on the pull out tests on site.

3. Experimental Results

The experimental studies were divided into two stages, pull-out tests and laboratory tests of the rock bolts.

• Pull-out tests

Pull out tests were performed on site. Five samples with 90 cm length and five samples with 120 cm length were tested by using different chemicals as bonding material between the inner surface of the drilled hole in the rock mass and the rock bolt. The results are summarized in Table 1.

Table 1: Pull-Out Test Results

Sample Nr.	Anchor Length (cm)	Chemical	Test Result (kN)
K1	90	Epoxy	285.09
K2	90	Grout	380.12
K3	90	Epoxy	380.12
K4	90	Epoxy	380.12
K5	90	Grout	380.12
U1	120	Grout	380.12
U2	120	Epoxy	142.55
U3	120	Epoxy	380.12
U4	120	Grout	380.12
U5	120	Epoxy	380.12

The results in Table 1 show that the maximum force that can be withstand by the rock bolt was approximately 380 kN. However, this should not be misinterpreted as the samples did not fail at this load. The samples can withstand more, but due to safety precautions of the testing team on site and the capacity of the testing equipment the experiments were finalized before the material yielded completely. By considering the technical considerations of the project where the bolts were planned to be used, 250 kN was determined as a minimum threshold value defined for bolt capacity. Hence, only 1 sample result did not satisfy the required capacity stated in the technical specifications. When the different chemical types were analyzed, it was seen that bolts used together with grout achieved a stronger adhesion than samples which were used together with epoxy. Although the pull-test results were successful in representing the site conditions in a more realistic manner, the technical capacity of the testing equipment limited the tests. Therefore, a set of experiments were also performed in the rock mechanics laboratory.

• Tensile Strength tests

Tensile strength tests were performed at the rock mechanics laboratory where four samples with dimensions as seen in Figure 5 were tested.

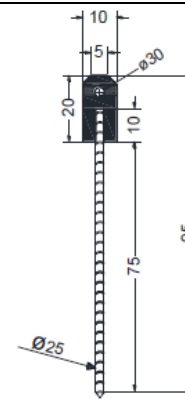


Figure 5: Dimensions of the anchor bolt

The samples were attached to the testing machine which has 500 kN capacity. The test results can be seen in Table 2.

Table 2: Tensile Strength Test Results

Sample Nr.	Anchor Length (cm)	Yielding Force (kN)	Max. Tensile Force (kN)
S1	95	248.40	308.19
S2	95	226.75	295.30
S3	95	234.47	293.60
S4	95	226.80	291.60

When the test results are evaluated, all samples have more bearing capacity than the required technical specifications. Although they have enough capacity, test results do not give exact information about failure type and failure region. At this point, requirement of new technologies arises. In this context, one of the experiments was monitored by an infrared thermal camera. By recording this test, possible failure regions and failure type of the rock bolt were investigated.

4. Thermal Response

When the test result was monitored by the thermal camera, it was seen that temperature increase on the failure region is related with failure type and failure region of the sample. Figure 6 shows the relation between the temperature increase at certain regions and the force that is applied on the bolt.

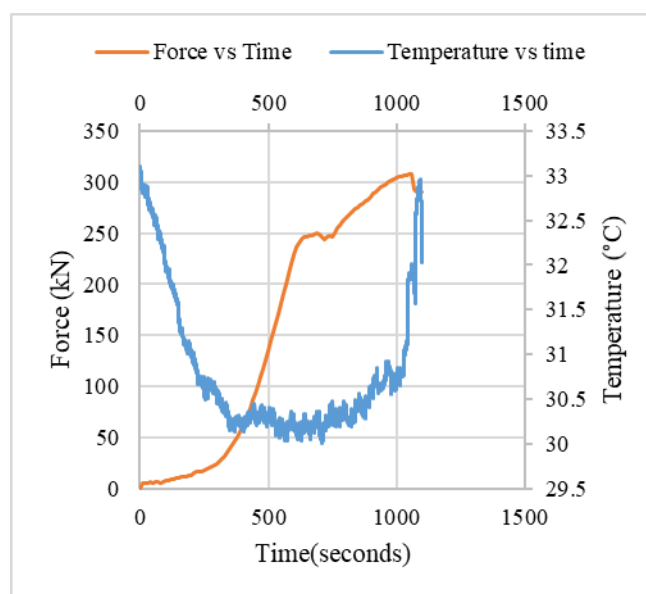


Figure 6: Relation between force and temperature increase on the failure region

It can be seen that the temperature decreased to 30.5°C until yielding point. After that, it remained constant at approximately 30.5°C. When the force started to increase again until failure, where the maximum tensile force was recorded, the temperature started to increase again within this region. Finally, temperature achieved the peak value which was recorded as 33°C.

As a result of the laboratory experiments, it can be said that the temperature increase in certain regions of the rock bolt is directly related with the possible failure region. Thermal images also give a clue about the failure type. Figure 7 and Figure 8 show the thermal image of the bolt at yielding point and failure point, respectively. The temperature range is represented by a color range defined in the thermal camera software.

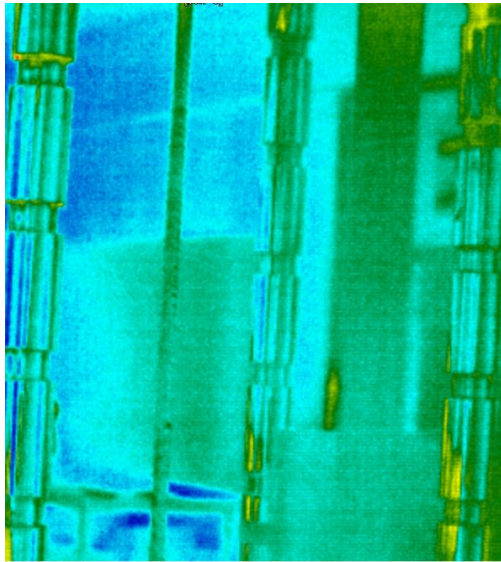


Figure 7: Thermal image of the anchor at yielding point

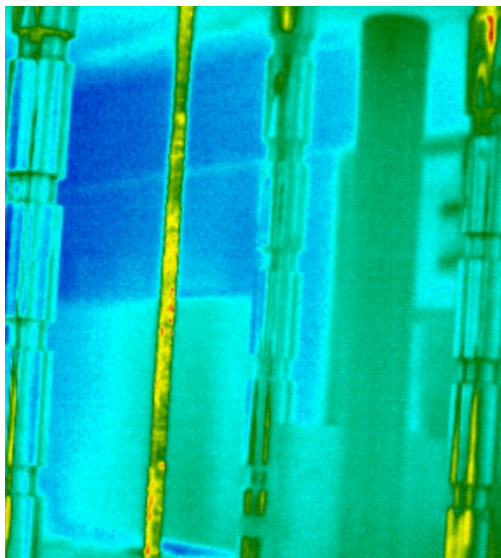


Figure 8: Thermal image of the anchor at failure point

As it can be seen, the thermal response of the rock bolt is directly related with failure region of the sample. Regions that are highlighted by the green color turn to yellow and red color at yielding period. The red color represents the maximum temperature reading in the thermal image whereas the lowest temperature can be seen in blue color. In addition to the failure region, failure type can also be estimated from the views. For example, possible failure type for this sample was estimated to be the breaking of the anchor from the middle since temperature increase was observed in the middle. This shows also stress concentration points on the sample.

5. Results and Discussion

Ten anchor bolts were tested on site by pull-out tests with different chemical types. The results showed that bolts have enough capacity by considering the technical specifications and the capacity also depends on the used chemical. Rock bolts with grout could withstand more force than bolts applied with epoxy. Therefore, selection of the chemical is a very important criterion for field tests. Since the bolt is placed inside the rock mass by means of a drilled hole, the pull-out tests could not be monitored by a thermal camera.

Four anchor bolts were tested at the rock mechanics laboratory by tensile strength tests. As a result of the experiments, all bolts have enough capacity by considering technical specification. As for the thermal behavior, there is an expected increase of temperature at the failure region of the sample. Tests recorded with the thermal camera showed that there is 2.5°C temperature increase at failure region.

Hence, there is a relation between the failure characterization and temperature response, the integration of new technologies will provide more information about failure behavior of the samples in the near future.

6. Conclusion

Rock mechanics plays a significant role for the stabilization of structures and is therefore essential for mining and civil engineering industry. The detection of the possible failures of engineering material must be performed in detail. In this context, new technologies such thermal cameras, high-speed cameras, acoustic emission systems can be integrated to field. This will help to detection of the failure behavior of samples easily. These devices provide more reliable and detailed data about failure behavior of the samples. This study provided a preliminary implementation of a thermal camera setup for tensile strength testing in the laboratory where a temperature increase of 2.5°C was recorded at the failure moment. Although the capacity of the load controlled testing equipment was also not sufficient, similar to the pull-out test equipment, it was possible to record the thermal behavior during testing.

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FEATURES OF WEAR OF RAILWAY WHEELS TREAD

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Abstract. Causes of wear particles formation on railway wheels tread were investigated. Structural factors connecting with plastic deformation, formation of “white layer”, and also with non-metallic inclusions and corrosive products of wheel steel, defining wear of railway wheels tread during operation were fixed. Repeating heat-mechanical action on railway wheel tread by interaction between wheel and rail promotes incandescence of stresses and defects (microcracks, separations) that promote wear particles formation having different shape, source and mechanism of nucleation. Results of analysis of microcracks, spalls and wear particles and also of mechanisms of their formation was shown that wear of railway wheel tread is very composite phenomenon. It passes by few mechanisms (fatigue, adsorbtional, corrosive, under friction) and is multifactors process.

KEYWORDS: WEAR PARTICLES, RAILWAY WHEELS, TREAD.

1. INTRODUCTION.

Complex approach for wear mechanism of railway wheels includes not only investigation of structural changes happening in surface layers of wheel rims [1-6], but also analysis of wear particles and establishment of mechanism of their formation. The variety of working conditions of friction pairs suggests that the general approach may be the idea of the fatigue nature of the destruction of the surface layers [7,8]. Interest for investigation of wear mechanism connects not only with necessity of cutting down of wear losses but also with development of effective methods of prediction of wheels service life, and with ensuring of their operation especially in extremal conditions. Wear mechanism of railway wheels tread presents assemblage of mechanical, thermo-physical and chemical effects. It connects with wear particles and microcracks formation in areas of intensive plastic deformation and “white layer”, near non-metallic inclusions and corrosive products of wheel steel [1-6,9,10]. Influence of some structural changes on the wear of tread was discussed [1-6] but analysis of these processes on mechanisms of wear particles formation on railway wheels tread during operation was not conducted.

The goal of this work was investigation of mechanisms of wear particles formation on railway wheels tread during operation.

2. MATERIALS AND METHODS OF INVESTIGATION

Mechanism of wear of railway wheels made of steel R7 and operated about 5 years under passenger rolling stock was researched. Wheels were taken off operation by limit wear of rims. Microstructure of wheel steel and defects were investigated by a few methods: metallographical, micro X-ray spectral, X-ray diffraction analysis, electron microscopic.

3. INVESTIGATION RESULTS AND DISCUSSION

Analysis shown that along the whole railway wheels tread microcracks and separations resulting in formation and spalling of wear particles and brittle fracture of cove zone were discovered. Formation of wear particles has different causes. Shape of wear particles depends on conditions of their formation.

During the initial stages of the railway wheel operation, this effect promotes burnishing of roughnesses on the wheel and rail surfaces allowing the wheel profile to change, which accelerates the so-called useful wear necessary to speed up the process of running in the wheel-rail system. Further it is necessary improvement the limit condition of deformational strengthening of steel for the formation of surface defects (wear particles). This stage of deformation is quickened under action of surface-reactive medium [11]. In the time of formation and spalling of wear particles the new “fresh” surfaces of cracks are appeared

One of main cause of wear particles formation is plastic shears proceeding near tread with big degree of deformation which has heterogeneous character [1-6]. On the boundaries of zones with different degree of deformation and also in areas of intensive and turbulent deformation the microcracks (fig. 1, a) and wear particles (fig. 1, b) are formed. In cove zone the group of wear particles was formed that evidences about considerable localization of plastic deformation and results in fracture of crest.

Wear particles formed on account of development of intensive plastic deformation near tread have the shape of scales or plates with different thickness. They are typical for normal conditions of wear [7]. According to wear theory by “spalling” [1-6] maximum dislocation density under wear origins is not near surface but on the some depth. Just microcracks growing to critical size in the results of plastic flow of steel are formed (fig. 1, c, d). Plastic flow of steel are localized between microcracks and wheel tread and particles having shape of scales are formed and spalled. Formation of wear particles happens in the result of ductile spalled of metal in the time of coalescence of these microcracks and are accompanied of plastic flow of steel near wheel tread.

Data by definition of dislocation density on and near wheel tread citing in [1-6] evidence about development of intensive plastic deformation during operation of railway wheels. Dislocation density was $10^{10} \dots 10^{11} \text{ cm}^{-2}$ on wheel tread, but on the depth of 100-200 μm from wheel tread it was higher about in 2...3 time as compared with wheel tread. Such difference of dislocation density connects with going out of moving dislocations on free surface during plastic deformation. In addition to it is known the fact of “braiding” influence of surface (in our case it is wheel tread) on moving dislocations. Wear stresses for wear particles with definite thickness near wheel tread were defined [1-6].

Considering the features of plastic deformation development in wheel steel near wheel tread it is essentially to take into account interaction of this surface with ambient air bring to formation of corrosive damages. It is known the elementary stage of corrosion includes adsorption of atoms of elements from damp ambient air that can to evoke effect of adsorption to facilitate plastic deformation near wheel tread (Rehinder effect) [11].

and these surfaces adsorb atoms of elements from damp ambient air active. Adsorption penetration of surface-reactive elements into cracks happens with high rate [11] that result in fall of surface energy of cracks (and work of their formation) and also makes easier their development in process of plastic deformation of steel. Thus, a wet environment, which also contains various lubricants, impurities, etc., has a negative effect on the fatigue strength of the wheel surface due to its adsorption and corrosive effects.

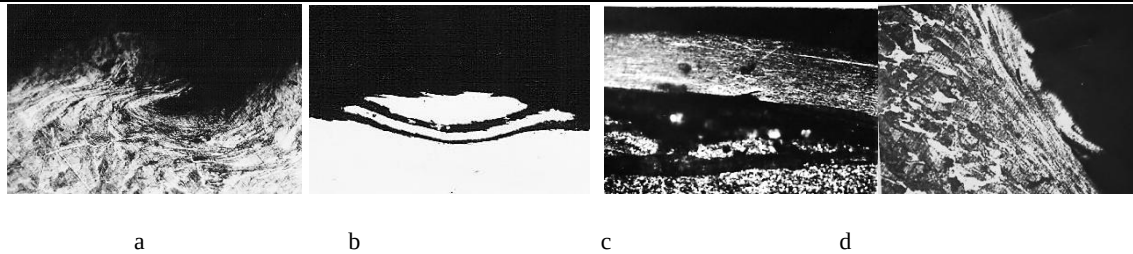


Fig. 1. Wear particles near tread in zone of intensive plastic deformation; a – $\times 100$, b – d – $\times 200$

According to the data of [8], wear particles, which are flakes or plates of different thickness, are formed with a uniform distribution of dislocations over the thickness of these flake particles. After the formation of these flaky particles, some of them fall between the two sliding surfaces of the wheel and the rail, as a result they can be broken in shape or destroyed, or remain the same size and "flat" shape.

Wear of the wheel surface, accompanied by the development of intense plastic deformation, occurs in layers and each layer consists of a large number of scales - wear particles. Their number N in each layer is proportional to the number of microcracks formed during plastic deformation. The rate of fusion of microcracks and the critical degree of deformation required for the formation of free wear particles depend on the depth of the plastic deformation zone with the maximum density of dislocations. The overall wear of the tread during the development of plastic deformation, the size and level of roughness of wear particles, the wear rate of the tread have been determined. Obviously, the wear rate decreases when the intensity of plastic deformation in the surface layer of the wheel rim decreases.

The second source of the formation of wear particles on the tread of wheel is the so-called "white layer", which has an increased brittleness [1-6]. The localization of deformation of the surface layer of the wheel rim is detected near the "white layer"

areas, which contributes to stress concentration and chipping of the "white layer". Wear particles have the shape of splinters with sharp edges of irregular shape (Fig. 2 b). They usually occur at very high pressures and their appearance can be associated with the formation of small fatigue cracks in the surface layers of the rims [7]. It should be noted that under the brittle "white layer" steel is plastically deformed and repulsive forces appear at the border of these structural components acting on moving dislocations [8]. This can lead to the appearance of microcracks both along the border of the "white layer" - deformed steel structure, and at some distance from it, similar to those shown in fig. 1c and parallel to the rolling surface of the wheel (Fig. 2 b). For the transformation of such microcracks into wear particles, the reasoning given above is valid.

The third reason for the formation of wear particles are non-metallic inclusions that are of metallurgical origin and are stress and strain concentrators in the wheel steel [12,13]. The formation of cracks and wear particles also contributes to the oxidation and corrosive destruction of the tread, and these processes lead to the formation of coarse oxides of coarse inclusions directly on the tread. The presence of inclusions and corrosion products significantly increases the rate of formation of microcracks and their combination, which increases the intensity of wear (both fatigue and friction) [12,13].

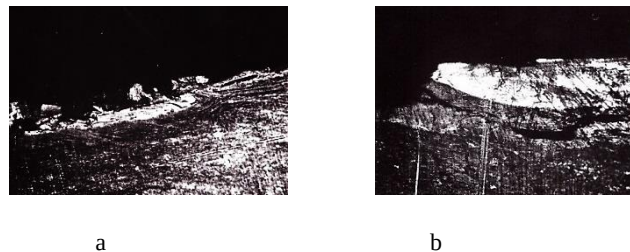


Fig. 2. Wear particles in areas of the "white layer"; $\times 1000$

Analysis of the microstructure of the wheel steel near treads of the wheels showed that non-metallic inclusions and corrosion products of the wheel steel contribute to the non-uniform development of deformation, the appearance of turbulent

The behavior of non-metallic inclusions located near the tread under the influence of acting stresses depends on their type [12]. They are plastic (sulphides) or not subject to plastic deformation (oxides, silicates, titanium carbonitrides). Non-metallic inclusion, located near the wheel tread, is in a complex stress state determined by a system of contact, dynamic and cyclic stresses. The temperature gradient arising during the operation of the wheel near the tread leads to a non-uniform distribution of deformations in the steel matrix and the forces acting on the inclusion.

Under conditions of high pressures and cyclically varying temperatures, the interaction of the contact surfaces of the inclusion and the steel matrix occurs by establishing mechanical contact and the development of diffusion processes. Mechanical contact is established as a result of smoothing the irregularities of the surfaces of the inclusion and the matrix of the wheel steel by developing the contact friction of their surfaces relative to each other, preventing their relative movement. Such

flow zones (Fig. 3 a) and also contribute to the formation of zones with increased degree of deformation.

friction is a kinematic dry sliding friction [12,14]. The conditions of friction on the interphase boundaries of non-metallic inclusion-steel matrix have been analyzed [9]. Contact friction enhances the non-uniformity of deformation of the wheel steel matrix near the inclusions, which can have an impact not only on the level of stresses acting on the inclusion, but also on the stress state scheme [12,15]. In the process of contact interaction of inclusions and the steel matrix, the inclusions can rotate (Fig. 3a), which causes momentary stresses and contributes to local plastic rotations in the steel matrix as a result of its vortex flow.

In the process of joint deformation of the inclusion-matrix system near the tread of the wheel, a characteristic fibrous grain structure of the wheel steel is formed (Fig. 3a). The fibers of the matrix round the inclusion, gradually "bumping" on it (Fig. 3 b - d), and stratification along the interfacial interfaces of the inclusion-matrix is possible. In this case, at the boundaries of the inclusion-matrix, compressive and shear stresses are concentrated, which can destroy the inclusion if they exceed the

tensile strength of the inclusions under compression. Similar processes occur near corrosion products (Fig. 3 e, f).

Under accelerated braking under conditions of a sharp rise in temperature, the possible role of slipping along grain boundaries in austenite and on the interfacial boundaries in the development of deformation near the tread of the wheel should

be considered [1-6]. It should be noted that non-metallic inclusions are often the centers of local decarburization of steel (see Fig. 3 c), which causes structural heterogeneity and entails a heterogeneous development of plastic shears, as well as the formation of microcracks and wear particles.

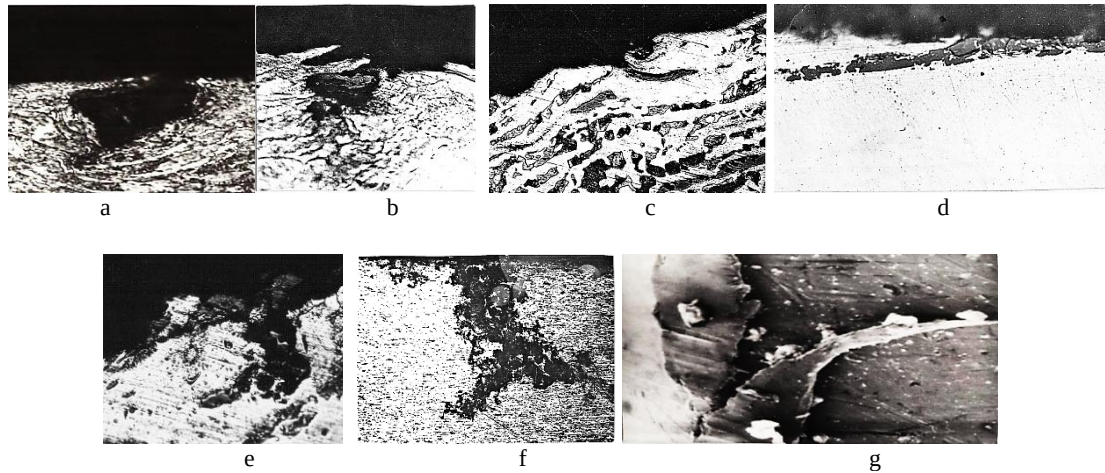


Fig. 3. Localization of deformation (a) and formation of wear particles near non-metallic inclusions and corrosive products of wheel steel (b – g) on railway wheel tread: a, b, g – x400, c – f – x200

It is necessary to consider another aspect of the influence of low-melting non-metallic inclusions on the formation of wear particles near tread of railway wheels 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 [12-16]. Then the manifestation of local sulphide red brittleness is possible [16], when the ductility and strength of wheel steel are sharply reduced. If the tread of the wheel heats up to high temperatures, the sulfide inclusions melt (Fig. 4 a), there is a sharp localization of the steel deformation and a lot of cracks occur, that is, the steel is destroyed when relatively low degrees of deformation are reached. Liquid interlayers are easily drawn out parallel to the tread of the wheel (Fig. 4 b), cracks

propagate between them, contributing to the formation of wear particles.

Near non-metallic inclusions and corrosion products of wheel steel, wear particles have the form of loops, spirals, similar to cutting chips during cutting (Fig. 3 g). They take this form due to residual stresses associated with the inhomogeneous distribution of dislocations over the thickness of wear particles formed near the inclusions [8]. According to [9,10], such wear particles precede damage and are found mainly on the tread before local destruction. In the case of local sulphide red brittleness of steel in areas of the tread, where sulphide inclusions melted, there are many wear particles of both spiral and compact irregular shapes (Fig. 4c), which is caused by the presence of many microcracks in these zones, as well as crushing of particles wear due to strong localization of deformation.

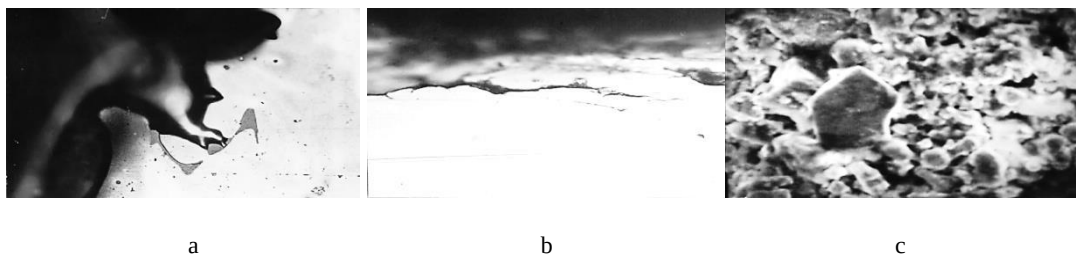


Fig. 4. Formation of wear particles in zones of sulphide red brittleness on railway wheels tread; x600

Considering the effect of corrosion products on the formation of wear particles, one more aspect of the influence on the development of fatigue failure on the tread of the external environment should be taken into account. It was noted above that the initial stage of corrosion involves the adsorption of atoms of elements from the environment, which causes an effect of adsorption lowering the strength of the product (the Rehbinder effect) [11]. Plastic shears arising in the surface layer of the rim (near tread), thermal cycling during braking and also interaction with the surrounding atmosphere create conditions for the intensive development of diffusion processes that contribute to more intensive adsorption of elements from the environment. Adsorption-fatigue phenomena occur on the tread surface of the railway wheel, leading to the nucleation of fatigue cracks and wear particles and accompanying their growth and detachment [7]. The presence of

interphase boundaries non-metallic inclusion-steel matrix with their defect structure and interphase stresses [17], as well as the formation of microcracks in them undoubtedly contributes to the localization of the effect of the adsorption decrease in the tread strength of the railway wheel near non-metallic inclusions, which is energetically characterized by a decrease in the formation of new solid surfaces in the process of deformation and destruction under the influence of the formation of an adsorption layer on them [11]. Apparently, for the interphase boundaries of the inclusion-matrix, the force interpretation of the effect of the adsorption decrease in strength is particularly relevant, due to the occurrence of a wedging effort within these boundaries, contributing to the penetration of the adsorption layer into the depth of the tread [10], which in turn contributes to the detachment of inclusions from steel matrix and the formation of wear particles.

4. CONCLUSIONS.

Repeating heat-mechanical action on railway wheel tread by interaction between wheel and rail promotes incandescence of stresses and defects (microcracks, separations) that promote wear particles formation having different shape, source and mechanism of nucleation:

- More wide-spread wear particles are the scales or plates with different thickness. Formation of such wear scales happens in the result of ductile spalling of metal. They are typical for normal conditions of wear and their appearance connects with plastic deformation near wheel tread.
- By brittle fracture of areas of "white layer" wear particles are formed with shape of fractions with sharp edges. Their appearance one can to connect with formation of fatigue microcracks in surface layers of wheel rims. Spreading of fatigue microcracks to surface is the beginning of wear particles formation.

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- Wear particles near non-metallic inclusions and corrosive products of wheel steel have shape of loops or spirals. They are formed by localization of deformation near inclusions and galling of metal matrix on the inclusions and also in the results of separation of interphase boundaries non-metallic inclusion-steel matrix. Such wear particles are discovered on wheel tread before local fracture.

- In the cause of local sulphide red brittleness a great number of wear particles with spirals and compact irregular shape were observed. This connects with presence of a great number of microcracks in these areas and also with grinding of wear particles by tough localization of deformation.

Results of analysis of microcracks, spalles and wear particles and also of mechanisms of their formation was shown that wear of railway wheel tread is very composite phenomenon. It passes by few mechanisms (fatigue, adsorbition, corrosive, under friction) and is multifactors process.

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THE PHENOMENON OF REVERSION IN AGING MAGNESIUM ALLOYS WITH THE RARE-EARTH METALS

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Abstract: The reversion in magnesium binary alloys with rare-earth metals (Nd, Sm, Y, Gd, Tb, Dy, Ho) at a temperature of 300°C has been studied. The phenomenon of the reverse dissolution of rare-earth metals in solid magnesium has been established to begin in all alloys under study already after short-term annealing at 300°C for 15 min. As the annealing time at 300°C further increases, the reverse dissolution of rare-earth metals in solid magnesium first continues and then the magnesium solid solution becomes again depleted of rare-earth metals because of their precipitation. The degree of reversion has been found to generally increase with an increase in the atomic number of the yttrium-group rare-earth metal in accordance with its position in the lanthanum row: Gd, Tb, Dy, and Ho. The degree of reversion in the alloys with cerium-group metals (Nd, Sm) with atomic numbers below those of Gd, Tb, Dy, and Ho is substantially smaller than that is in the alloys with yttrium-group metals.

Keywords: MAGNESIUM ALLOYS, RARE-EARTH METALS, REVERSION, SOLID SOLUTION DECOMPOSITION, STRUCTURE.

1. Introduction

Magnesium alloys are used in modern industry as light structural materials. The addition of rare-earth metals to magnesium can substantially improve their strength characteristics at both ambient and elevated (200-300°C) temperatures [1 - 4]. However, upon short-term holdings at not-too-high temperatures, the magnesium alloys age-hardened to high strength exhibit the phenomenon of reversion, which can lead to weakening because the REM-containing hardening particles precipitated upon aging undergo a reverse dissolution in the magnesium solid solution [5 - 7]. The reversion process should lead to a decrease in strength properties, especially at elevated temperatures. The paper presents the results of the study of reversion at a temperature of 300°C in the magnesium alloys containing rare-earth metals of cerium (Nd, Sm) and yttrium groups (Y, Ho, Dy, Tb) after age hardening at 200°C for the maximum strength.

2. Experimental

The alloys for the study were prepared by melting in an electric resistance furnace in iron crucibles under the standard VI2 flux containing 38-46% MgCl₂, 32-40% KCl, 5-8% BaCl₂, 3-5% CaF₂, which was used for the protection of the molten magnesium from ignition. Magnesium Mg95 (>99.95% Mg) and rare-earth metals such as ItM-1 yttrium, NM-1 neodymium, SmM-1 samarium, GdM-1 gadolinium, TbM-1 terbium, DiM-1 dysprosium, and GoM-1 holmium at least 99.83% pure (hereinafter, the compositions are given in wt.%) were used as the starting materials. The rare-earth metals were added into the Mg melt upon melting as master alloys. The rare-earth metals for the study were chosen because, in binary magnesium alloys, they provide a substantial strengthening effect caused by the decomposition of the supersaturated magnesium solid solution upon aging [3]. The alloy compositions were chosen with allowance for the binary phase diagrams in such a way that the content of each rare-earth metal was somewhat below its solubility in solid magnesium at the homogenization temperature. Table 1 shows the alloy chemical compositions analyzed by inductively coupled plasma atomic emission spectroscopy with an Ultima 2C Jobin-Yvon instrument.

The alloys were cast into a stainless steel mold heated to ~300°C to obtain cylindrical ingots of 15 mm in diameter and 90 mm long. The ingots were cut into blanks, which were subjected to homogenization annealing for 6 hours at temperatures given in Table 1. The blanks after homogenization annealing were quenched in room-temperature water in order to obtain a supersaturated magnesium solid solution.

Table 1: Alloys compositions, homogenization temperatures, and aging time at 200°C for achievement of the maximum hardness

Alloy composition	Homogenization temperature, °C	Aging time at 200°C for the achievement of maximum hardness
Mg-8.3% Y	540	128
Mg-2.2% Nd	530	4
Mg-3.5% Sm	520	16
Mg-15.5% Gd	520	32
Mg-17.7% Tb	540	32
Mg-19.5% Dy	540	128
Mg-21.4% Ho	540	128

Samples for the hardness and electrical resistivity measurements were made from the quenched blanks of the alloys.

The hardness was tested by the Brinell method with a TSh-2M tester using a ball of 5 mm in diameter at a load of 2500 N. Electrical resistivity of the samples of 6 mm in gage diameter and 23.7 mm in gage length was measured with a unit based on the BSZ-010-2 microohmmeter. The error of the electrical resistivity measurement was about 0.7%. The microstructure of the alloys was examined with an M 24 Reichert metallographic microscope. The polished samples were etched with 0.5% nitric acid solution in ethanol or 30% orthophosphoric acid solution in ethanol.

3. Results and discussion

At the first stage of the study, the alloy samples after homogenization and quenching were subjected to isothermal aging at 200°C for up to 128 hours with the view to follow the process of age hardening upon decomposition of the supersaturated magnesium solid solution by measuring hardness and to establish the aging time, at which the maximum hardness is reached. The established aging times required to achieve the maximum hardening at 200°C in the alloys are given in Table 1. Then the alloys were subjected to isothermal annealing at 300°C for 0.25 - 128 hours to detect reversion. The results of the hardness and resistivity measurements for the alloys aged for maximum hardening at 200°C and then annealed at 300°C for the study of the reversion are shown in Figs. 1 and 2.

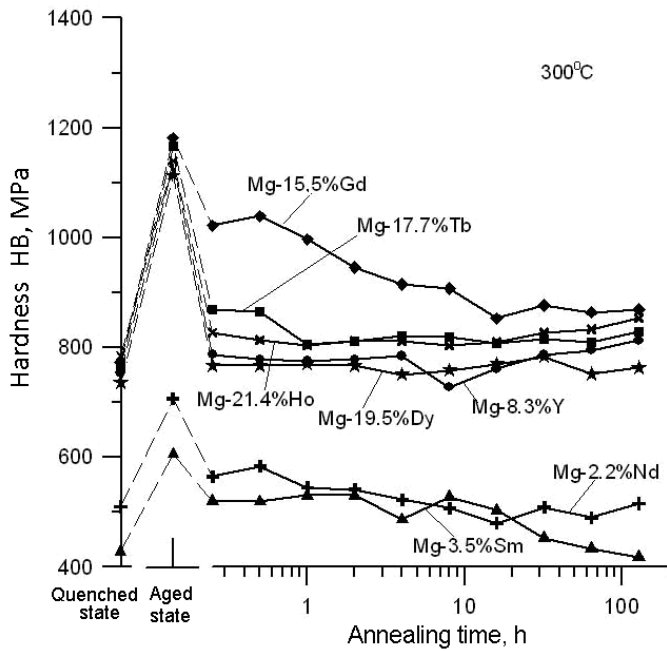


Fig. 1. Hardness of the REM-containing magnesium alloys quenched and aged at 200°C for the maximum hardening as a function of time of subsequent isothermal annealing at 300°C.

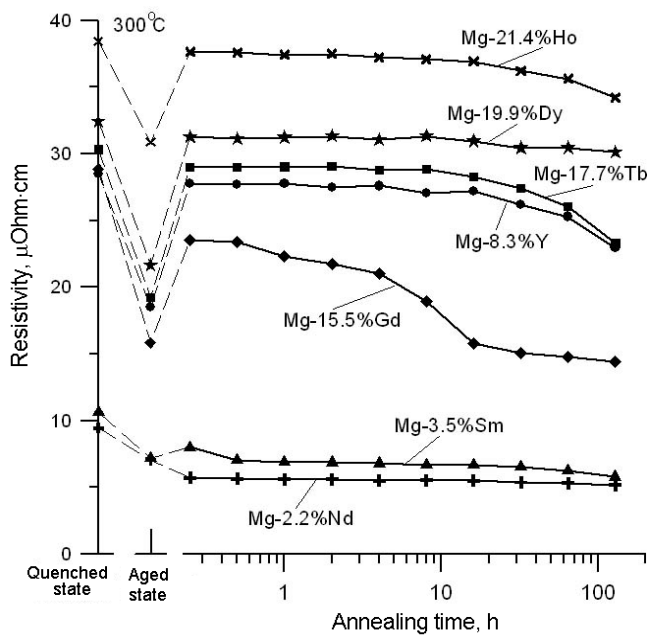


Fig. 2. Electrical resistivity of the REM-containing magnesium alloys quenched and aged at 200°C for the maximum hardening as a function of time of subsequent isothermal annealing at 300°C.

According to the data presented in Fig. 1, the hardness of the aged magnesium alloys with rare-earth metals is abruptly reduced even after the shortest (15 min) annealing at 300°C, especially in the alloys with yttrium group rare-earth metals (Y, Gd, Tb, Dy, Ho). This suggests the possibility of the reversion phenomenon, i.e., the reverse dissolution of rare-earth metals in solid magnesium. Such reversion effect presumed according to change in hardness becomes proven by the observed increase in resistivity after annealing by the same regime (Fig. 2). The resistivity data show that, with a further increase in the annealing time at 300°C, first the reverse dissolution of rare-earth metals in solid magnesium remains unchanged, and then the magnesium solid solution becomes again depleted because of the precipitation of rare-earth metals. The degree of reversion is substantially smaller in the magnesium alloys with cerium-group

rare-earth metals, which have been studied for comparison, and can be even absent in the magnesium alloy with neodymium.

The degree of reversion was evaluated by the formula: $S = (\rho_{\text{ann}} - \rho_{\text{ag}}) / (\rho_{\text{q}} - \rho_{\text{ag}})$, where S is the degree of reversion, and ρ_{ann} , ρ_{ag} , and ρ_{q} are the resistivities of the alloy in the states after annealing at 300°C for the maximum reversion, after aging to the highest hardening at 200°C, and after quenching, respectively. The results obtained are given in Table 2. Note that the degree of reversion in the magnesium alloys with yttrium group rare-earth metals of the lanthanum row (Gd, Tb, Dy, Ho) increases with increasing their atomic number. The degree of reversion of magnesium alloy with yttrium, which does not belong to the lanthanum row, is close to that of the magnesium alloys with the lanthanum row metals such as dysprosium and holmium. The reversion after aging in the alloys with cerium group metals is substantially smaller than in the alloys with yttrium group metals and can be even absent, as in the alloy with neodymium. An increase in the degree of reversion with increasing atomic number of yttrium group rare-earth metals of the lanthanum row (Gd, Tb, Dy, Ho) was also observed in the same alloys upon annealing at a temperature of 250°C [7].

Table 2: Degree of reversion in aged magnesium alloys with rare-earth metals after annealing at 300°C.

Atomic number of REM	Alloy	Degree of reversion, %
60	Mg - 2.2% Nd	0
62	Mg - 3.5% Sm	24.1
64	Mg - 15.5% Gd	58.8
65	Mg - 17.7% Tb	88.8
66	Mg - 19.5% Dy	89.9
67	Mg - 21.4% Ho	89.5
39	Mg - 8.3% Y	92.3

The microstructure examination of the alloys by optical microscopy did not reveal any signs of the decomposition of the supersaturated magnesium solid solution after aging to the maximum hardness and after reversion after short-term annealing at 300°C. This can be explained by very fine precipitates from the magnesium solid solution and by the retention of the coherence between the crystal lattices of the precipitates and the magnesium solid solution. However, after long-term annealing following the reversion at 300°C, optical microscopy revealed the decomposition of the magnesium solid solution in the structure of the alloys in the form of the particles precipitated at three planes of the crystal lattice of the magnesium solid solution and at grain boundaries (Fig. 3). With allowance for the results reported in [3, 8, 9], we can assume that the increase in hardness up to the stage of the maximum strengthening is due to the formation of a metastable β' phase, which is typical for the decomposition of the magnesium solid solution in the magnesium alloys with yttrium group rare-earth metals. This phase has a base-centered orthorhombic crystal lattice.

4. Conclusions

1. It is established that the binary magnesium alloys with yttrium group rare-earth metals (yttrium, gadolinium, terbium, dysprosium, and holmium) after age hardening can undergo a substantial reversion with the reverse dissolution of the particles precipitated upon the decomposition of the supersaturated solid solution.

2. The degree of reversion sequentially increases with increasing atomic number of the rare-earth metals belonging to

yttrium group of the lanthanum row from Gd to Tb, Dy, and Ho. The degree of reversion of the magnesium alloy with yttrium, which does not belong to the lanthanum group, is close to those of the alloy with holmium and dysprosium. The reversion in the magnesium alloys with cerium group rare-earth metals is substantially smaller than that in the magnesium alloys with yttrium group metals and can be even virtually absent.

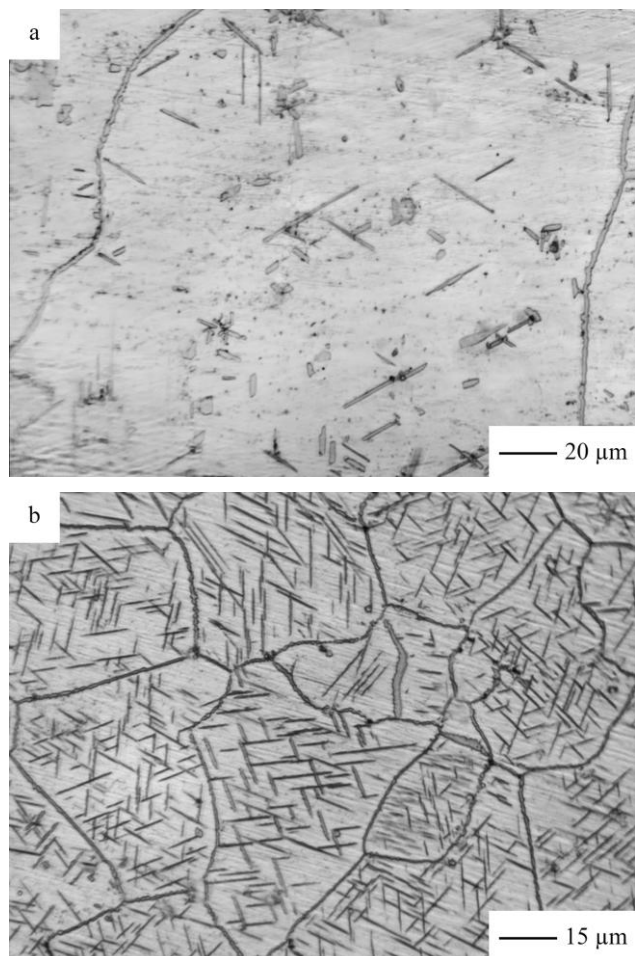


Fig.3 Typical microstructures of the alloys: Mg - 8.3% Y (a), and Mg - 17.7% Tb (b) after quenching + aging at 200°C + annealing at 300°C for 128 h.

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EFFECT OF AGING ON ZAMAK 3

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Abstract: Zamak 3 is a zinc-aluminum alloy with a composition of 3.7-4.3 %Al, <0.05 % Cu, 0.02-0.06 % Mg, rest is Zn. It is well suited for pressure die casting and it results in sufficient mechanical properties and a good surface finish. Zamak 3 is utilized in applications automotive industry, household appliances, in structural applications of buildings, etc. It was reported in literature that the mechanical properties of the zamak alloys deteriorate in time at room temperature. In the present study, this effect was investigated by artificial aging of zamak 3 alloy.

Zamak 3 samples were produced by cutting and melting of Zamak 3 ingots at 500 °C, and casting into steel molds. Aging experiments were conducted by keeping the Zamak 3 samples in a muffle furnace for 24 h at temperatures of 85 °C, 105 °C and 130 °C. Optical and scanning electron microscopy was employed for investigating the microstructure of the samples. Hardness values of the samples were measured by a Brinell hardness tester. A universal testing machine was used for conduction three point bending tests of the samples. It was found that both hardness and bending strength of Zamak 3 alloy decreased with increasing aging temperature. Hardness and strength values of Zamak 3 were 89 HB10 and 490MPa, respectively. After aging at 130 °C hardness and strength values were found to fall to 75 Brinell 10 and 415 MPa, respectively.

Keywords: ZAMAK 3 ALLOY, AGING, MECHANICAL PROPERTIES

1. Introduction

Most of the zinc alloys are appropriate for pressure die casting due to their low melting point and good castability. Zamak 3 is a member of the zinc-aluminum alloy family. The composition of Zamak 3 is 3.7-4.3 %Al, <0.05 % Cu, 0.02-0.06 % Mg and rest is Zn. As compared to Zamak alloys that contain higher amount of coppler, such as Zamak 5, Zamak 3 exhibits lower hardness and strength, whereas it has higher ductility and toughness [1-3].

Zamak 3 presents adequate mechanical properties and a good surface finish of the as cast products. It is meets the requirements for use in frames and handles and other parts of PVC window systems, machine parts, automotive engine parts, household appliances [1,4].

It was reported in literature that the mechanical properties of the zamak alloys deteriorate in time at room temperature [5,6]. It was also stated that 24 h aging at 105 °C results in the changes in the properties of the zamak alloy that would take place in 1 year at room temperature [5,6]. In the present study, this effect was investigated by artificial aging of Zamak 3 alloy.

2. Experimental Procedure

Melting of the Zamak 3 samples prior to casting were conducted in a clay bonded graphite crucible, in a muffle furnace at 500 °C, after cutting pieces from Zamak 3 ingots.

After melting, samples having the dimensions and geometry for 3-point bending tests were cast in permanent steel molds.

Aging experiments were conducted by keeping the Zamak 3 samples in a muffle furnace for 24 h at temperatures of 85 °C, 105 °C and 130 °C. These temperatures and durations were selected according to the literature.

Unaged and aged samples were subjected to microstructural examinations with an optical microscope (Nicon Eclipse, LV150) and scanning electron microscope. Before that, samples were subjected to metallographic surface preparation. After cutting the samples they were lapped on emery paper having 600, 1200, 3000 and 4000 grit. Final polishing was performed with 1 micron polycrystalline diamond paste.

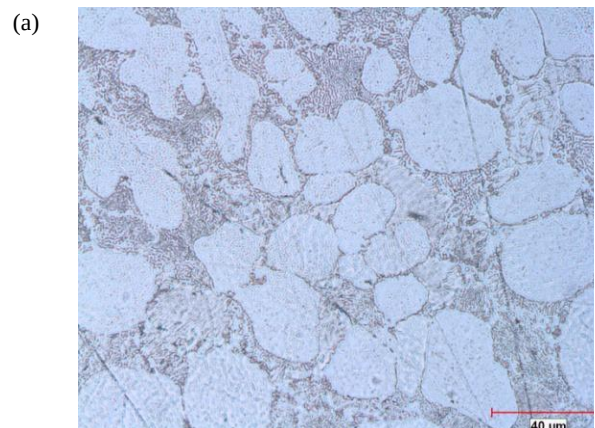
Samples having 25 mm span length, 6 mm thickness and 12 mm width were used for 3-point bending tests. These tests were conducted with a universal mechanical testing machine (Shimadzu AG-IC, 50 kN).

A Brinell hardness tester was utilized for hardness tests. Brinell 10 scale was used in the hardness measurements. Tests were conducted with a 2.5 mm carbide ball and 62.5 kg load.

3. Results and Discussion

Optical micrographs of the unaged sample and samples aged at 85, 105 and 130 °C are presented in Fig. 1. The white globular grains in these micrographs are the alpha phase, which is rich in zinc. They are formed before the eutectic transformation, in the Zn-Al phase diagram. In the eutectic transformation, beta+eta forms. The gray regions are alpha+eta phase mixtures, which are formed by the eutectoid transformation. Alpha phase is rich in aluminum. The beta phase in the eutectic beta+eta, transform into alpha+eta in the eutectic transformation. Therefore, at room temperature, eta + alpha phases are present.

Amounts or morphology of the phases did not present a noticeable change after aging at different temperatures. There are some differences in the contrast of the alpha phase, due to etching process.



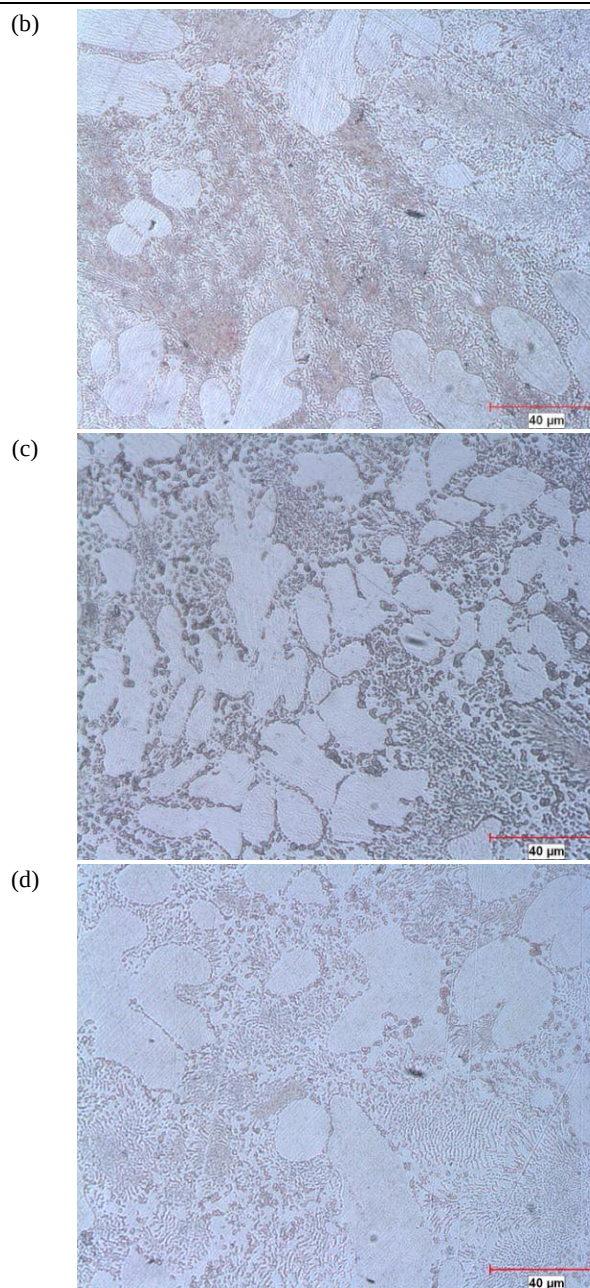


Fig. 1 Optical microscope images Zamak 3 alloy samples after (a) casting, (b) aging at 85 °C, (c) aging at 105 °C, (d) aging at 130 °C for 24 h.

SEM micrographs of the unaged sample and sample aged at 105 °C for 24 h are presented in Fig. 2. In higher magnification than the optical micrographs, eta+alpha phase layers in the eutectic formation can be visualized in more detail in the SEM micrographs.

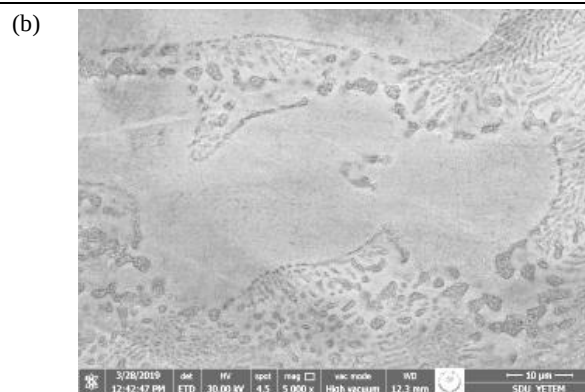
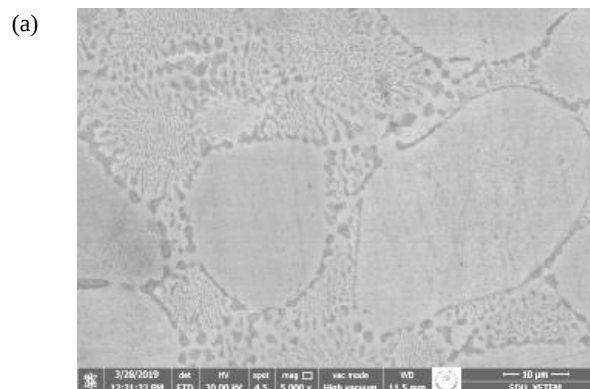


Fig. 2 SEM images Zamak 3 alloy samples after (a) casting, (b) aging at 105 °C for 24 h.

Aging temperatures, and hardness, bending strength and bending strain values of the samples according to aging temperature are given in Table 1. The changes of these properties are presented in the following figures.

Table 1: Aging temperatures, and hardness, bending strength and bending strain values of the samples according to aging temperature.

	Aging Temperature (°C)			
	Unaged	85	105	130
Hardness (HB10)	88.7	85.1	79.1	75.4
Bending strength (MPa)	490.4	459.7	454.7	414.1
Bending strain	4.8	4.5	5.7	5.0

Change in the hardness of Zamak 3 alloy samples is presented in Fig. 3 as a function of aging temperature. Hardness of the unaged sample was 88.7 HB10. Hardness was seen to decrease with the increase in the aging temperature. Hardness of the Zamak 3 sample which was aged at 130 °C for 24 h was 75.4 HB10.

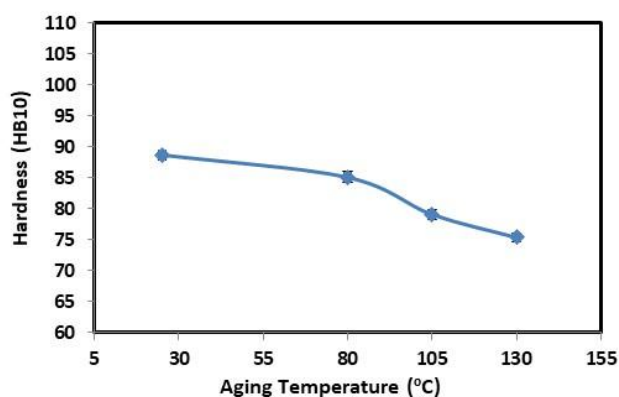


Fig. 3 Hardness values of Zamak 3 alloy samples as a function of aging temperature.

Change in the bending strength of Zamak 3 alloy samples is presented in Fig. 4 as a function of aging temperature. Bending strength of the unaged sample was 490.4 MPa. Bending strength was seen to decrease with the increase in the aging temperature. Strength of the Zamak 3 sample which was aged at 130 °C for 24 h was 414.1 MPa.

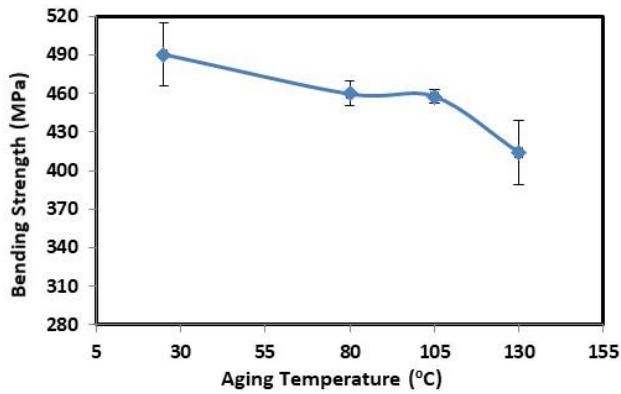


Fig. 4 Bending strength values of Zamak 3 alloy samples as a function of aging temperature.

Change in the three point bending strain values of Zamak 3 alloy samples is presented in Table 1 as a function of aging temperature. It was seen that the strain values were in 4.8-5.7. Strain values were seen not to be dependent on the aging temperature.

4. Conclusion

It was found that both hardness and bending strength of Zamak 3 alloy decreased with increasing aging temperature. Hardness and strength values of Zamak 3 were 89 HB10 and 490 MPa, respectively. After aging at 130 °C hardness and strength values were found to fall to 75 Brinell 10 and 415 MPa, respectively.

The high hardness of the unaged Zamak 3 samples was attributed to the presence of regions that contain elements higher than the equilibrium composition, due to the effect of fast cooling during casting. The decrease in hardness and bending strength of the Zamak 3 samples a result of aging may be attributed to the elimination of the regions that contain elements above the limit, by diffusion during aging.

Acknowledgements

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FORMATION OF ZrB_2 -AlN POWDER MIXTURE BY SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS

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Abstract: Formation of ZrB_2 -AlN mixed powder by self-propagating high-temperature synthesis (SHS) was investigated. Powders of Zr, B, BN and Al (purity >99 %) was used as the starting materials. Two initial mixtures were prepared and mixed at specific ratios. The first mixture was made of Zr and B, which were weighed at stoichiometric amounts to form ZrB_2 . The second mixture contained Zr, BN and Al; amounts of which were calculated so as to form ZrB_2 and AlN.

The starting powders were mixed in a mortar and pestle, and then the reactant mixture was slightly pressed in a steel die. The SHS reactions were conducted in high purity argon atmosphere, in an SHS chamber. The reactant pellet was ignited from one end with a tungsten wire. The reaction products were examined with scanning electron microscopy and they were subjected to X-ray diffraction analyses.

It was found that when the reactants contained 40 % mixture-1 and 60 % mixture-2, according to the XRD results of the products, the peaks related to ZrB_2 were dominant. The product contained some AlN and ZrN. When the amount of mixture-2 was increased to 90 %, the amounts of AlN and ZrN both were observed to increase. When Al amount in mixture-2 was increased by 30 % and mixture-2 was added as 90% into mixture-1, amount of AlN in the reaction products increased and amount of ZrN decreased. According to SEM examinations, ZrB_2 particles were seen to be mixed with AlN particles in the reaction products. Size of ZrB_2 particles were about 1 micron and AlN particles were larger.

Keywords: ZAMAK 3 ALLOY, AGING, MECHANICAL PROPERTIES

1. Introduction

Aluminum nitride (AlN) is important for electronic industry due to its good properties such as high electrical resistivity, thermal conductivity and low thermal expansion. In addition, its corrosion resistance and oxidation resistance make it a suitable material for high temperature boats that are used in various metal melting and similar processes [1].

Zirconium diboride (ZrB_2) is in the class of ultra-high temperature ceramics. It has also high thermal conductivity and thermal shock resistance. It is used in high temperature applications and as high temperature boats [2].

Self-propagating high-temperature synthesis (SHS) is a procedure that utilizes the exothermic character of the reactions for sustaining the formation of the products. One chief benefit of this practice is that it requires little amount of energy, which is necessary to initiate the reaction. Low equipment cost, uncontamination of the products, fast production rate are a few of the other rewards [3].

AlN was prepared through combustion synthesis by heating Al powder in high pressure nitrogen atmosphere. AlN was used as diluent. It was reported that reaction did not take place when the Al content of the mixture was less than 20 wt % [1].

For high temperature applications, there are some disadvantages of single phase ceramic materials, such as low ablation resistance. Therefore, they are combined with other ceramics. Addition of secondary or tertiary compounds enhances their properties and density [2]. Addition of SiC to ZrB_2 increases its strength and addition of MoSi_2 to ZrB_2 increases its high temperature strength [2].

In the study of Liu, ZrB_2 based ceramics were produced by reactive spark plasma sintering [4]. Zr, ZrH_2 , Si, B_4C , BN were used as the starting materials and ZrB_2 and SiC or AlN or ZrN containing ceramics were obtained.

In the study of Zhang et al. [4], boron carbide and boron nitride were used as reactants for producing boride containing ceramic composites. ZrB_2 and AlN ceramics were obtained by in situ reactive hot pressing technique.

In this study, formation of ZrB_2 -AlN mixed powder by self-propagating high-temperature synthesis (SHS) by using of Zr, B, BN and Al powders, was investigated.

2. Experimental Procedure

Formation of ZrB_2 -AlN mixed powder by self-propagating high-temperature synthesis (SHS) was investigated. Powders of Zr, B, BN and Al (purity >99 %) was used as the starting materials. Two initial mixtures were prepared and mixed at specific ratios. The first mixture (mixture-1) was made of Zr and B, which were weighed at stoichiometric amounts to form ZrB_2 according to Reaction (1).



The second mixture (mixture-2) contained Zr, BN and Al; amounts of which were calculated so as to form ZrB_2 and AlN according to Reaction (2).



Three samples were produced by using different amounts of starting materials. The first composition was contained 40 wt % of mixture-1 and 60 wt % of mixture-2. The second composition contained 10 wt % of mixture-1 and 90 wt % of mixture-2. The third composition contained 10 wt % of mixture-1 and 90 wt % of mixture-2, but the amount of Al in mixture-2 was 30 % in excess of the stoichiometric amount (Table 1).

Table 1: Produced samples and amounts of the reactants powders in them

Sample	Amounts of Reactants (g)			
	Zr	B	BN	Al
Sample-1 (%60M1-%40M2)	0.4525	0.0575	0.1146	0.1247
Sample-2 (%10M1-%90M2)	0.37	0.0134	0.1604	0.1746
Sample-3 (%10M1-%90M2 (Al 30%xs))	0.37	0.0134	0.1604	0.2275

The starting powders were mixed in a mortar and pestle, and then the reactant mixture was slightly pressed in a steel die. The SHS reactions were conducted in high purity argon atmosphere, in an SHS chamber. The reactant pellet was ignited from one end with a tungsten wire. The reaction products were examined with scanning electron microscopy and they were subjected to X-ray diffraction analyses.

3. Results and Discussion

After self-propagating high-temperature synthesis reaction the pellets preserved their shapes. Some swelling was observed, along with increase in the porosity. In addition, the pellets had a lamellar morphology, a typical result of the SHS reaction. The pellets were ground in a mortar and pestle into powder form.

XRD Analyses

The ground samples were subjected to XRD analyses at a rate of 2 °/min. XRD patterns of the product obtained from the sample containing 40% mixture-1 and 60% mixture-2 (Sample-1) is presented in Fig. 1(a). The peaks related to ZrB₂ were dominant. The product contained some AlN and ZrN. The intensity of AlN peaks are much lower as compared to that of ZrB₂ peaks. The intensity of the AlN peaks are believed to be not directly proportional with the amount of AlN phase in the products.

The reaction was conducted in argon atmosphere. Thus, the only source of nitrogen in the system is BN. There was no sign of unreacted aluminum or boron nitride in the system, indicating that the reaction was completed. In the present study, BN was used to provide nitrogen, whereas in literature AlN production by combustion synthesis is frequently performed in high pressure nitrogen [1].

When the amount of mixture-2 was increased to 90 % in the reactants (Sample 2), the amounts of AlN and ZrN both were observed to increase (Fig 1(b)). Mixture-2 contains BN, which is the nitrogen source in the system, and the products of Reaction (2) are ZrB₂ and AlN. Therefore AlN is expected to increase in the products, as a result of increase in the amount of mixture-2 in the reactants. On the other hand, amount of ZrN also increased in the products, due to increase in the amount of BN in the reactants.

In the third sample, Al amount in mixture-2 was increased by 30 % and mixture-2 was added as 90% into mixture-1, (Sample 3). It was seen that the amount of AlN in the reaction products increased and amount of ZrN decreased (Fig. 1(c)). It can be deduced that in sample-2, there was a loss of aluminum during the SHS reaction. Adding excess aluminum into the reactants may have compensated for the loss of aluminum from the system and led to formation of more AlN. In addition, this decreased the formation of ZrN. According to XRD pattern given in Fig. 1(c), there was trace amount of unreacted aluminum in the reactants, when 30 % excess aluminum was used in mixture-2.

Microstructure

Scanning electron micrographs of the products are given in Figs 2-4. These images were obtained in the backscatter electron mode. Therefore, compositional contrast was obtained. The white particles are believed to be ZrB₂ (or ZrN in some cases), with higher average atomic weight. Gray particles are believed to be AlN (or Al), with lower average atomic weight.

According to SEM examinations, ZrB₂ particles were seen to be mixed with AlN particles in the reaction products. The microstructure is very similar to the ZrB₂-AlN system synthesized by in situ reactive hot pressing [5]. Size of ZrB₂ particles were about 1 micron and AlN particles were larger.

Amount of AlN phase was seen to be higher in Sample-3 (Fig. 4), as compared to other samples.

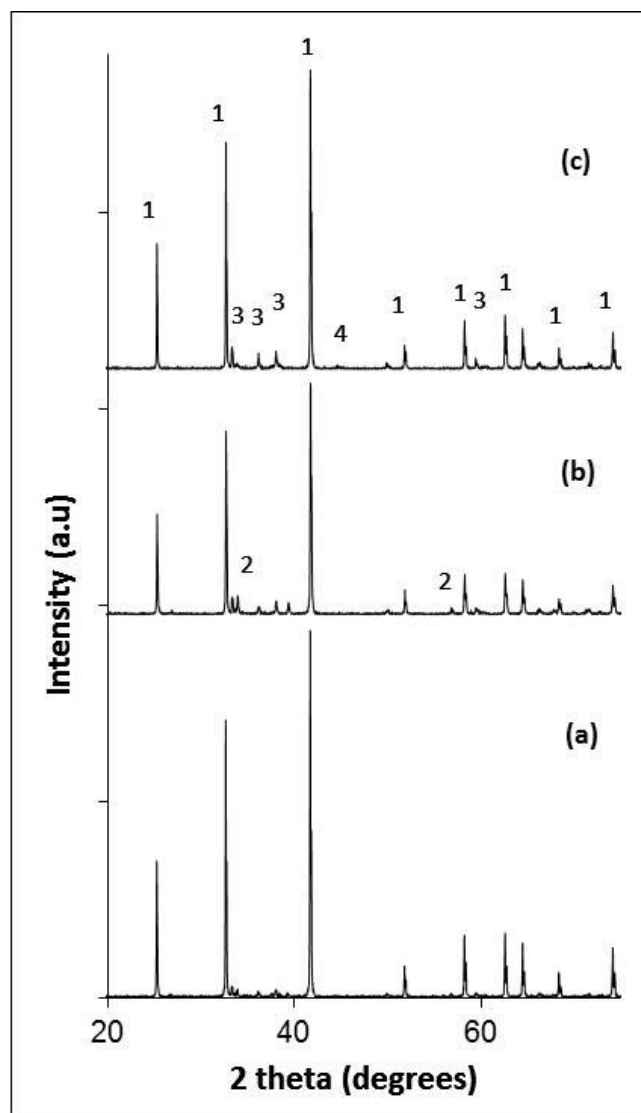


Fig. 1 XRD patterns of the products (a) S1 (%60M1-%40M2) (b) S2 (%10M1-%90M2) (c) S3 (%10M1-%90M2 (Al 30%xs)), 1. ZrB₂, 2. ZrN, 3. AlN, 4. Al

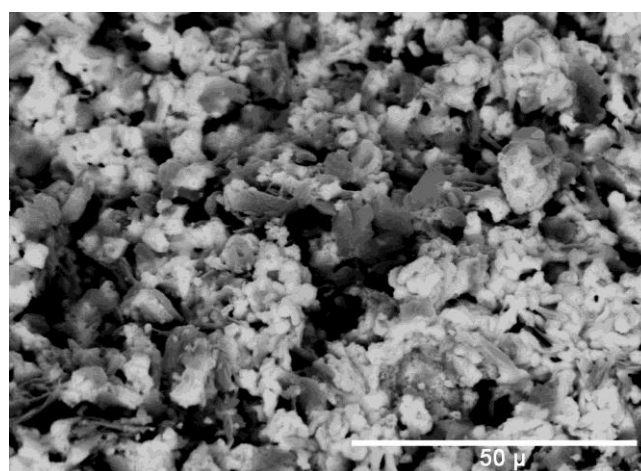


Fig. 2 Scanning electron micrograph of the products obtained from reactants containing 40% mixture-1 and 60% mixture-2 (Sample-1).

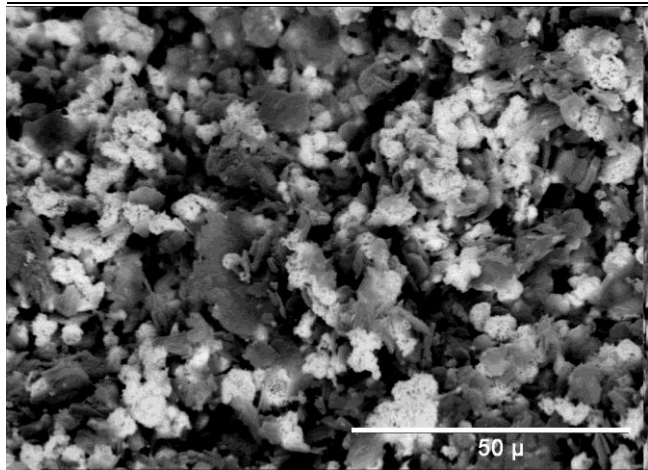


Fig. 3 Scanning electron micrograph of the products obtained from reactants containing 10% mixture-1 and 90% mixture-2 (Sample-2).

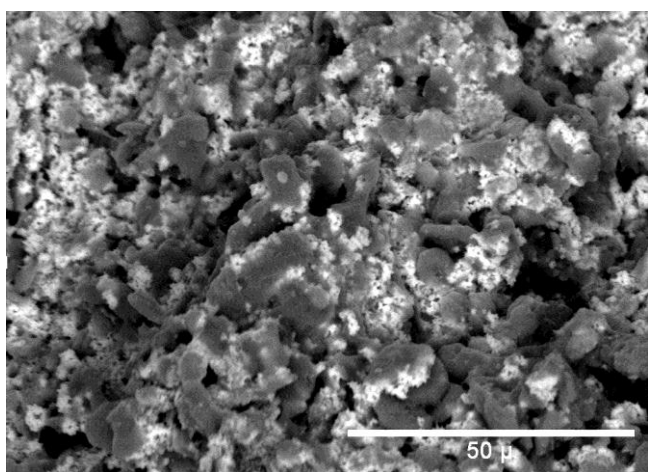


Fig. 4 Scanning electron micrograph of the products obtained from reactants containing 10% mixture-1 and 90% mixture-2 (Al 30%xs) (Sample-3).

4. Conclusion

ZrB₂-AlN powder mixture was produced from Zr, B, BN and Al reactant mixtures by self-propagating high-temperature synthesis. Increase in the amount of BN and Al in the reactants, resulted in an increase in the amount of AlN in the products.

ZrB₂ and AlN particles could be visualized by compositional contrast in SEM. ZrB₂ particles were seen to be mixed with AlN particles in the reaction products. Size of ZrB₂ particles were about 1 micron and AlN particles were larger.

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MATERIAL-SCIENCE ASPECTS OF FORMATION AND EVOLUTION OF FLAWS WHICH DEFINE THE RESOURCE EXPLOITATION OF ALUMINUM STRUCTURES OF AIRPLANES

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Abstract: The work is devoted to the study of evolution of flaws in aluminium alloys of Al-Zn-Mg-Cu, Al-Cu-Mg-Mn alloying systems and to the determination of their connection with structural factors of the material such as the size and composition of intermetallic phases conditioned by heat treatment tempers, and also to the study of the effect of big number of physical factors on the long-term behaviour of structural elements of aluminium alloys, and to the determination of the rate of formation of corrosion damages in structural elements of the aircraft wing

KEYWORDS: ALUMINIUM ALLOYS, CORROSION DAMAGES, INTERMETALLIC PHASES, COPPER, HEAT TREATMENT, MICROSTRUCTURE, CORROSION RESISTANCE, GRAIN BOUNDARIES, REGRESSION ANALYSIS

Introduction

It was studied of evolution of flaws in aluminium alloys of Al-Zn-Mg-Cu, Al-Cu-Mg-Mn alloying systems and to the determination of their connection with structural factors of the material such as the size and composition of intermetallic phases conditioned by heat treatment tempers, and also to the study of the effect of big number of physical factors on the long-term behaviour of structural elements of aluminium alloys, and to the determination of the rate of formation of corrosion damages in structural elements of the aircraft wing[1-3]. The work is aimed at securing the long-term safe operation of airplanes.

Materials for Production

The issue of evolution of flaws and metal-science aspects of their formation was examined for B93T1, B93пчT1, 1933T3 forging aluminium alloys of Al-Zn-Mg-Cu alloying system which is applied in the aircraft primary structural elements where we face large concentration and localization of stresses that can lead to their very rapid failure. In this case, origination and propagation of a failure can occur even without the previous effect of corrosion. Susceptible areas are strictly regulated and they are first of all subject to monitoring during scheduled inspection of the airplane. Using the techniques of electronic microscopy and X-ray chemical microanalysis, it was determined that during industrial heat treatment of B93 alloy using T1 temper: quenching 450-465°C and two-stage phase ageing 115°-125°C (6-10 hours), 165°-175°C (4-8 hours), chains of the phase with increased content of copper precipitated at the grain boundaries during quenching simultaneously with strengthening phases (fig. 1 a). The size of some links of the chains reached 1-5 mcm. Their presence stipulates for concentration of stresses which contribute to corrosion cracking. Precipitation of inclusions of intermetallic phases containing copper at the grain boundaries does not correspond to the statements of developers of the alloy.

It was proved that change of heat treatment temper to coagulation ageing (T3 temper: quenching 450-465°C and two-stage ageing 115°-125°C (6-10 hours), 180°-190°C (6-10 hours)) contributed to higher corrosion resistance of B93пчT3 alloy compared to B93T1 alloy, that was conditioned by much smaller content of copper in precipitation of intermetallic phases after quenching, formation of strengthening phases after ageing which did not contain copper, higher fragmentation of particles of intermetallic phases, which precipitated during quenching.

During coagulation ageing of 1933T3 alloy, which differed from B93пчT3 alloy with the zirconium admixture, precipitation of particles of strengthening phases occurred mainly at the stage of coagulation ageing. At the grain boundaries we observed particles of $MgZn_2$, $Al_2Zn_3Mg_3$, Mg_2Si phases connected with the matrix in a coherent way. Precipitation of particles of intermetallic phases with copper content at the grain boundaries did not occur, that obviously stipulated for the highest corrosion resistance of the alloy (fig. 1 b).

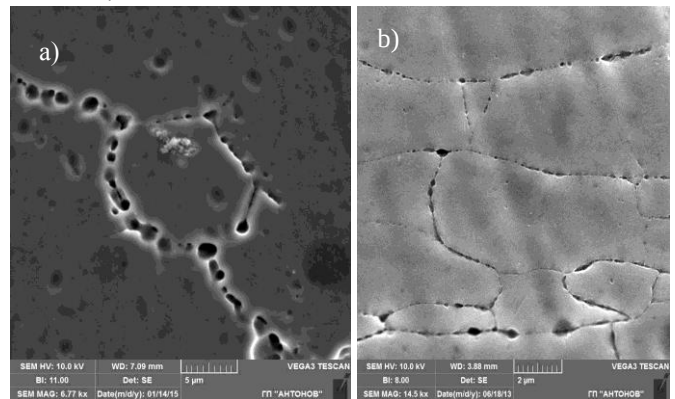


Fig. 1. Precipitations of intermetallic phases along the boundaries of grains in the alloy B93T1 (a) and in the alloy 1933T3 (b)

Such differences in the structure of alloys, conditioned by heat treatment, contributed to evolution of flaws during transition from B93T1 alloy to 1933T3 alloy and the increase of corrosion resistance, yield and plasticity while maintaining high strength.

Application of X-ray microanalysis techniques allowed to determine the presence of coarse inclusions of insoluble phases with the length of 10 to 300 mcm in the grain body of all three alloys, which presence contributes to the development of fatigue failure during long-term operation.

Monitoring of flaws which contributed to premature failure of parts showed that B93T1 alloy had low resistance to alternate and static loads, and was susceptible to corrosion cracking even under the absence of aggressive environments. 1933T3 alloy appeared to be the most resistant to static, dynamic and alternate loads, and not susceptible to corrosion cracking.

Further examination of the issue of evolution of flaws and their monitoring in service was continued for wing upper and lower panel skins which are the most responsible structural elements of the airplane though they are much less loaded if compared to structural elements produced of forging alloys. For the upper panels, which are in compressed state during the flight, we apply B95T1 high-strength alloy of Al-Zn-Mg-Cu alloying system, quenched and aged to the maximum strength. And for the lower panels, which experience tensile loads during the flight, we apply Д16Т long-life alloy of Al-Cu-Mg-Mn alloying system. The skins are produced of long extruded or rolled products: sheets or plates, in which the direction of elongated grains is perpendicular to the acting loads (the area of action of the load coincides with the transversal-longitudinal area of fibres in the material). Taking into account the design features: considerable length of joints, big number of fastener holes and areas of permanent condensation in the torsion boxes of the wing centre section (closed cavities), the

most probable areas of origin of corrosion damage are the grain boundaries located in problem areas perpendicular to the direction of application of loads. A critical factor which determines the life of skins is thinning of their crosscut due to corrosion damage. Stated features of evolution of flaws and also relatively easy access to damaged areas change the strategic approach to enhancement of durability of these structural elements. An emphasis is made on the increase of latent period of formation of corrosion damage and fatigue cracks that is achieved at the first stage by applying protective coatings and restoring them after certain interval of operation. However, this effective method does not ensure 100% guarantee from appearance of corrosion spots in the metal matrix, that is why there is another and not less important way to delay the appearance of critical flaws called scheduled maintenance which ensures detection and elimination of flaws on external and internal surfaces of the wing. In this case, a critical condition is to provide the optimum amount of scheduled maintenance which from one side would ensure an accident-free operation and from the other side would minimize capital expenditures.

The paper addresses the main operating factors which influence the origin and growth of flaws on the surface of the aircraft wing skins, and determines characteristic areas which are susceptible to corrosion. The largest amount of statistical data is accumulated for two versions of An-24 and An-26 airplanes that allowed evaluating the damage growth rate in the centres of corrosion which appeared on upper and lower panel skins of B95T1 and Д16Т alloys respectively. As a criterion there was taken the change of the maximum depth of damage on flawed surface areas which appeared in the interval between two successive inspections of panels, it means not considering the latent period of damage formation. It was shown that linear relation exists between the geometrical parameters of corrosion flaw and the time. The rate of corrosion is determined by the tangent of the angle of slope of the straight line (fig. 2).

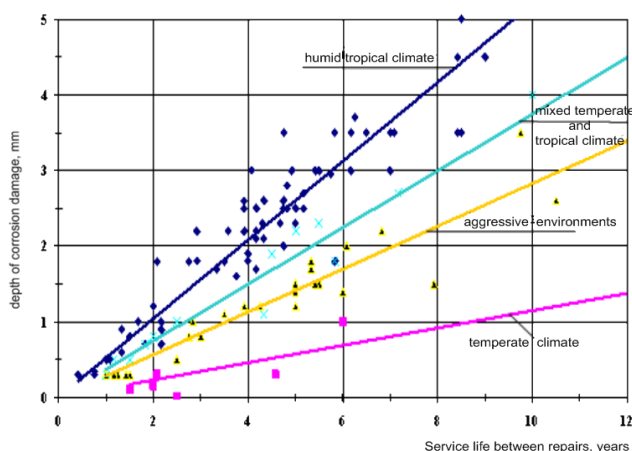


Fig. 2. Dependence of the depth of corrosion damage from time between technical surveys in different climatic zones.

Basing on the results of statistical analysis of a huge amount of data from inspections performed in certain climatic zones such as the size of corrosion damage, accumulated life, service life, intensity of flights, number of overhauls, etc., it was determined that climatic conditions and inspection intervals had the maximum effect on the corrosion damage growth rate (unlike the total time of operation).

Using the method of regression analysis, there were obtained the equations of regression which determined the corrosion damage growth rate for different climatic zones, and there was determined the maximum damageability of the aircraft wing skins during one year of operation: this data is shown below.

Lower panel skins of Д16Т alloy:

Moderate climate - $y=(0.12\pm0.015)x_1$;

Continental climate - $y=(0.23\pm0.042)x_1$;

industrial zones - $y=(0.33\pm0.035)x_1$;

maritime climate - $y=(0.34\pm0.045)x_1$;

mixed operating conditions - $y=(0.28\pm0.037)x_1$;

humid tropics - $y=(0.47\pm0.056)x_1$.

where x_1 is the life between overhauls.

Upper panel skins of B95T1 alloy:

moderate climate - $y=(0.12\pm0.028)x_1$;

maritime and industrial zones - $y=(0.27\pm0.014)x_1$;

mixed tropical and moderate climate - $y=(0.37\pm0.025)x_1$;

humid tropical climate - $y=(0.51\pm0.022)x_1 + (0.00024\pm0.00018)x_2$ - where x_1 is the life between overhauls; x_2 is the intensity of flights.

There by, the increase of the time between overhauls by 1 year leads to the increase of the maximum depth of corrosion damage on the lower panel skins of the wing centre section by ~0.12 mm in moderate climate zones; by ~0.23 mm in continental climate zones; by ~0.33 mm in industrial zones; by ~0.34 mm in maritime climate zones; by ~0.28 mm under mixed operating conditions; by ~0.47 mm in humid tropics zones.

Using the similar procedure, there was determined the depth of corrosion damage of the external surface of the wing upper panel skins of B95T1 alloy. In this case we shall expect the increase in depth by ~0.12 mm in moderate climate zones; by ~0.27 mm in the zones of influence of maritime and industrial environments; by ~0.37 mm in the zones of mixed tropical and moderate climate; by ~0.58 mm in the zones of humid tropics with the intensity of flights of 300 flights/year.

Basing on the obtained results of the analysis, technical documentation was developed for ultrasonic inspection of the thickness of lower panels of the wing centre section of An-24 and An-26 airplanes and there were issued the recommendations for timely detection of damages of different structural elements. This will make it possible to operate these airplanes without disassembly of panels during not less than 10 years after preliminary inspection and despite the climatic zone where the airplane is based.

The results of corrosion growth rate analysis for different structural elements of the wing were used to adjust the inspection intervals and to determine the optimum time for renovation of anticorrosion protection of the analyzed area and group of airplanes, as well as other areas, groups and types of airplanes for which this area and group can be considered a prototype.

General scheme of acquisition and analysis of corrosion damage data based on service experience of the available aircraft fleet allows solving both the problems of its continued airworthiness, and the problems of reliable anticorrosion protection of newly designed aircraft based on available data.

Research of the microstructure of industrial aluminium alloys in terms of susceptibility to corrosion cracking is taken into account when evaluating the life of a separate element and that of the entire structure, and it is also used for successful selection of materials with required set of features for existing and future aircraft structures.

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ПОЛУЧАВАНЕ И ОХАРАКТЕРИЗИРАНЕ НА МАНГАНОВИ ОКСИДИ И БИРНЕЗИТИ, ПРИЛОЖИМИ КАТО ЕЛЕКТРОДНИ МАТЕРИАЛИ В ЙОННИ БАТЕРИИ

PREPARATION AND CHARACTERIZATION OF MANGANESE OXIDES AND BIRNESITES, APPLIED AS ELECTRODE MATERIALS IN ION BATTERIES

гл. ас. д-р Златанова З, Кирева М.

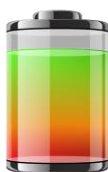
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Abstract: Since the first clay batteries were used, constant efforts have been made in searching of new anode and cathode materials with better properties. The aim is obtaining materials with better properties such as energy density, capacity, charge/discharge regimes, safety and of course cost. Our work is focused on Na – Mn and Mg – Mn oxides of birnesite type as new promising cathode materials. The results, give us a reason to conclude that replacement Na- ions with Mg-ions has a positive effect.

KEYWORDS: BIRNESITE, MANGANESE OXIDE, CATHODE, MATERIAL

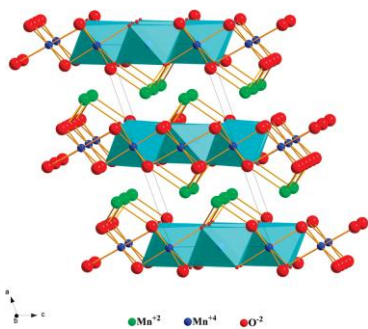
1. Увод

Търсенето на нов тип презарежими батерии, които да са безопасни, безвредни за околната среда и евтини дава нови перспективи пред редица учени. Различните възможности за съхранение на енергия в различни по състав, структура и свойства материали провокира научно-изследователската мисъл и дава нов тласък за разработване на все по-нови и по-нови подходи. Възможността за съхранение на енергия чрез замяна на йони и промяна на вида на електродните материали провокира мисълта на научните среди и обуславя ново научно течение, търсещо оригинални методи в постигането на целите си.



Разнообразието в подходите, структурите, способите за синтез и свойствата на материалите е огромно. Търсенето на най-подходящ и съвместяващ в себе си всички необходими качества електроден материал с годините придобива все по-широк аспект. Основните усилия се насочват към конкретни групи съединения, като например: сплави, метални оксиди, халкогениди, полимери, въглеродни материали, които могат да се разделят в различни структурни подгрупи - слоести, шпинелни и такива с оливинов тип структура.

В последните години широк интерес за редица учени представляват съединенията със слоеста структура на основата на различни химични елементи.



Фиг.1 Слоеца структура

Химичният елемент манган е представител на VII Б група на периодичната система, част от групата на така наречените

преходни елементи. Характерно за тях е, че електроните в електронните обвивки на атомите им са разположени на d- и f-орбитали. Всички те са метали, а името преходни идва от мястото им в периодичната система. Образуват редица химични съединения с разнообразни свойства и приложения. Мангановите оксиди са изключително важни материали с широко приложение, като катализатори или като електродни материали за батерии. Голямата им значимост и интригуващи качества до голяма степен се дължат на техните структура и физични свойства, които не са характерни за съединения на други преходни метали [1]. Кристализират обикновено с редица други слоести материали, като различното съдържанието на мангана и неговите различни степени на окисление (+2, +3 или +4) са причина за разнообразното приложение и свойства на тези материали [2]. Широко изучени са слоести структури от типа на бирнезитите, които също представляват съединения на основата на манганови оксиди [3]. Интригуващото в материали от типа на Mn_5O_8 е смесеният валентен характер на елемента манган в състава на едно и също съединение. Характера на химичната връзка в този бинерен манганов оксид допринася за интереса към него, като потенциален проводник на йони за горивни клетки или батерии [4,5].

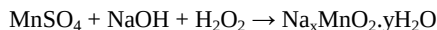
Струва си да се отбележи, че макар и с няколко метода за синтез, чрез които могат да се получат този тип съединения са проведени редица системни проучвания върху свойствата и приложимостта им [6 – 10].

2. Резултати и дискусия

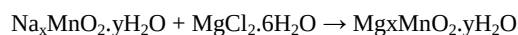
Всички използвани за синтеза химикали ($MnSO_4$, $NaOH$, H_2O_2 и $MgCl_2$) са с висока чистота и не бяха подлагани на допълнителни процедури с цел пречистване.

За синтезирането на магнезиев бирнезит първо беше необходимо получаването на натриев такъв. Натриевият бирнезит беше получен от 50% разтвор на $MnSO_4$ и разтвор на 2.16 g $NaOH$ в 90 ml дестилирана вода и 10 ml водороден пероксид. За $MnSO_4$ (50%) бяха необходими 5.377g $MnSO_4$, които се разтворят в 50 ml дестилирана вода при интензивно разбъркване до получаването на бледо розов разтвор. Вторият необходим за синтеза разтвор получаваме, като разтворим 2.16 g $NaOH$ в 90 ml дестилирана вода и към тях прибавим 10 ml водороден пероксид. След като двата разтвора са готови следва смесването им. Мангановият разтвор беше поставен на електромагнитна бъркалка с умерено темпо на разбъркване и към него на малки порции, бавно прибавихме разтвора на натриева основа и водороден пероксид. Още при първата порция се получи наситено кафяво оцветяване, характерно за получаващият се натриев бирнезит. След прибавянето а последната порция от втория разтвор, оставихме полученият

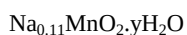
материал да се разбърква при същите обороти за 2 часа. След определеното време спряхме разбъркването и оставихме разтвора да „стареє“ в продължение на 24 часа. На следващият ден, филтрувахме разтвора под вакуум. Полученият бирнезит промиваме двукратно в дестилирана вода и един път с етанол още до като е в бюхнеровата фуния. Извадихме вече готовата маса и оставихме да се суши за едно денонощие при 50 °C.



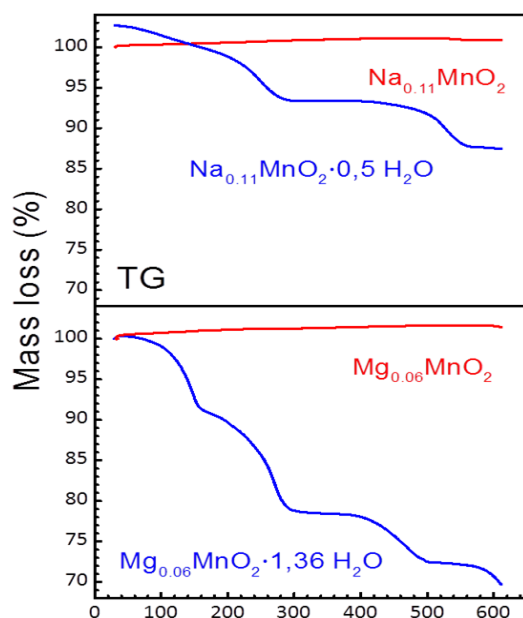
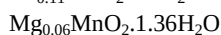
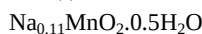
От така получения натриев бирнезит следва получаването на магнезиев такъв. За целта 0.5 g от готовият бирнезит смесихме със 100 ml предварително приготвен 1M разтвор на магнезиев хлорид. (За получаването на 1L, 1M разтвор на MgCl_2 , разтворихме 203 g $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ до 1L с дестилирана вода.) Новият разтвор поставихме на електромагнитна бъркалка за 24 часа при умерено разбъркване и стайна температура. На следващият ден готовият вече магнезиев бирнезит се филтрувахме със същата процедура – двукратно промиване с вода и еднократно с етанол. Полученият материал сушихме на 50 °C за една нощ.



Получените материали бяха подложени на редица изследвания с цел пълно охарактеризиране на състава и структурата им. С цел изясняване стехиометрията на двете получени съединения проведохме атомна абсорбционен анализ, за определяне количествата на натрий и магнезий:

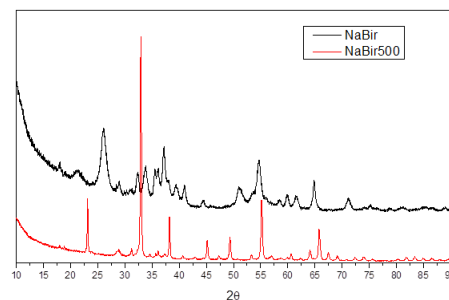


и термогравиметрия, за определяне на стехиометричното количество вода:

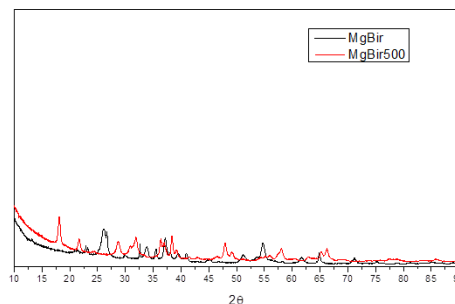


Фиг.2 Термогравиметрични криви на натриев и магнезиев бирнезит, показващи загубата на вода при отгряване до 600°C; синя крива

Рентгенов анализ, характеризиращ микроструктурата на бирнезитните материали показващ сравнение между отгрят и неотгрят при 600°C натриев бирнезит. Ясно се вижда, че в процеса на нагряване пробата кристализира, доказателство за което са ясно изразените пикове на кривата показваща отгрятия материал- червената.



Фиг.3 Рентгенограма на натриев бирнезит; изходен – черна крива; отгрят – червена крива

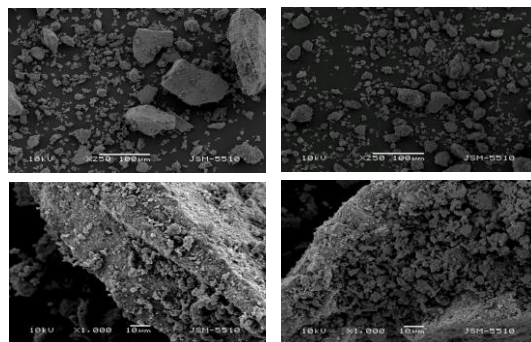


Фиг.4 Рентгенограма на магнезиев бирнезит; изходен – черна крива; отгрят – червена крива

При рентгеновата дифрактограма на магнезиевият бирнезит ясно се вижда, че в процеса на нагряване пробата не претърпява големи промени в своята структура.

Като цяло между структурите на двата бирнезита има голяма разлика, дължаща се преди всичко на начина, по който те са синтезирани, а именно – единият е произведен на другия. Получаването на магнезиев бирнезит от натриев такъв, чрез йонно-обменна реакция за протичането на която са необходими 24 часа, води до промени в структурата на материала. Непрекъснатото разбъркване в процедурата на синтез помага за видимото от рентгенофазовият анализ изситняване на материала.

Резултатите от сканиращата електронна микроскопия (SEM), ни дадоха информация за морфологията на двете проби. При по-ниските увеличения се наблюдава приблизително еднаква структура на бирнезитите, но както ясно се вижда при по-високите увеличения магнезиевият материал е с много по-пореста структура, което се дължи на размера на магнезиевия катион.



Фиг.5: SEM – изображения на материалите; горе, лявои дясно – натриев бирнезит; долу, ляво и дясно – магнезиев бирнезит

3. Заключение:

На база проведените изследвания и получените от тях резултати става ясно, че заместването на натриеви с магнезиеви йони в състава на бирнезитите има благоприятно влияние върху търсените от нас структурни характеристики на материалите.

Този факт, придружен с редица други съществени доказателства за положителния ефект от наличието на магнезий ни дава основателна причина да продължим с изследванията и търсенето на още по-подходящи структури и комбинации от такива. Такива материали в бъдеще биха могли да бъдат използвани, като катода в йонни батерии. Натриево-мангановите оксиди и магнезиево-мангановите оксиди с бирнезитов тип структура са ново и многообещаващо поколение източници на енергия, отговарящи на всички изисквания – екологичност, възобновяемост, добри параметри на действие и ниска цена.

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EFFECT OF STRUCTURE AND TEXTURE ON THE MECHANICAL CHARACTERISTICS OF MAGNESIUM ALLOYS PROCESSED BY EQUAL-CHANNEL ANGULAR PRESSING

ВЛИЯНИЕ СТРУКТУРЫ И ТЕКСТУРЫ НА МЕХАНИЧЕСКИЕ ХАРАКТЕРИСТИКИ МАГНИЕВЫХ СПЛАВОВ, ПОЛУЧЕННЫХ РАВНОКАНАЛЬНЫМ УГЛОВЫМ ПРЕССОВАНИЕМ

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Abstract: ECAP was carried out with a gradual decrease in temperature and an increase in the number of passes on two medical magnesium alloys: WE43 (Mg-3.56%Y-2.20%Nd-0.47%Zr) and ZX10 (Mg-1.0%Zn-0.3%Ca). It was shown that ECAP leads to a significant refinement of the alloys structure. For ZX10 alloy, the average grain size after ECAP decreased from $\sim 105 \mu\text{m}$ in the initial state to $8 \pm 0.18 \mu\text{m}$ in the longitudinal section and to $4 \pm 0.19 \mu\text{m}$ in the transverse one. For the WE43 alloy, the average grain size was changed from $70 \mu\text{m}$ to $0.69 \pm 0.13 \mu\text{m}$ and the precipitation of particles of the $\text{Mg}_{41}\text{Nd}_5$ phase with an average size of $0.45 \pm 0.18 \mu\text{m}$ was also discovered. At the same time, the grain refinement led to an increase in the strength characteristics of the both alloys (including fatigue strength), and increased prismatic slip activity (along with the formation of an inclined basal texture in ZX10 alloy) led to an increase in their ductility. The alloy structure formed during the ECAP process does not lead to a decreasing in resistance to chemical corrosion.

KEYWORDS: MAGNESIUM ALLOYS, EQUAL CHANNEL ANGULAR PRESSING, ULTRAFINE GRAINED STRUCTURE, TEXTURE, MECHANICAL PROPERTIES, FATIGUE LIFE

1. Introduction

Magnesium alloys have been actively studied in many countries for two decades as medical materials [1-3]. Currently, the most studies are aimed at obtaining the optimal composition and properties of these materials. The development of the compositions of such alloys is complicated by the fact that the selected alloying elements must have good biocompatibility and fulfill their basic properties: strengthen the alloys and increase its corrosion resistance. Therefore, a limited number of elements are now used (Ca [4], Zn [5], Ag [6], Li [7], Sr [8], Mn [9], some rare-earth metals [10-12]), which fall in their characteristics under both criteria. But just only alloying is not enough to obtain optimal properties. Therefore, alloys usually are deformed to improve their mechanical properties. In this sense, severe plastic deformation [13], and in particular equal-channel angular pressing (ECAP) [14], which allows significantly strengthening magnesium alloys by formation an ultrafine grained (UFG) structure, is especially attractive. In addition, the cases of increasing the corrosion resistance of magnesium alloys after formation of UFG structure are known [15, 16]. Therefore, the effect of alloying and ECAP on the structure and properties of two medical magnesium alloys (WE43 and ZX10) was studied in this study.

2. Materials and Methods

Two magnesium alloys were investigated in the work: WE43 (Mg-3.56%Y-2.20%Nd-0.47%Zr) and ZX10 (Mg-1.0%Zn-0.3%Ca). In the initial state, the WE43 alloy was homogenized at 525°C for 8 hours and then air cooled. The ZX10 alloy was also subjected to homogenization at 450°C for 3 hours and then water quenched. The Bc ECAP route was carried out on the machine with a channel intersection angle of 120° with a gradual decrease in the deformation temperature and an increase in the number of passes. For WE43 alloy, the ECAP temperature decreased from 425 to 300°C with a step of 25°C and 2 passes at each temperature ($N_\Sigma = 12$). For the ZX10 alloy, stepwise ECAP was performed with a decrease in temperature from 400 to 300°C with a step of 25°C and also 2 passes at each temperature ($N_\Sigma = 10$) (Figure 1).

The microstructure was examined using an Axio Observer D1m Carl Zeiss optical microscope and an electron microscope JEM-1400 (Jeol, Japan) operating at a voltage of 120 kV . Foils for TEM analysis after ECAP were cut in the longitudinal direction,

mechanically ground up to $180 \mu\text{m}$ and then subjected to ion-bombardment on a GATAN 600 unit. The size of the microstructural units was estimated by the method of random sections using the Image ExpertPro 3 software.

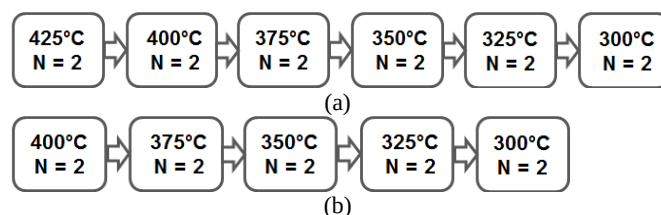


Figure 1 - The ECAP processing regimes employed (N denotes the number of passes at a given temperature)

Texture analysis was carried out using X-ray diffraction in a Rigaku Ultima IV diffractometer with the $\text{CuK}\alpha$ radiation in the reflection mode. Five complete pole figures $\{00.4\}$, $\{20.2\}$, $\{10.2\}$, $\{10.3\}$, $\{11.0\}$ were obtained with a maximum inclination angle $\alpha_{\text{max}} = 70^\circ$ and a step of 5° in the radial angle α and the azimuth angle β on a pole figure. The orientation distribution functions (ODFs) were calculated from the measured pole figures presented as a superposition of a large number (1000) of standard distributions with a small scatter. The centers of standard functions were located on a regular three-dimensional grid in the orientation space [17]. With these ODFs, complete pole figures were also calculated. The volume fractions of the major orientations were estimated using the ODFs as described in [17]. Using the Euler angles and the volume fractions of the orientations, the generalized Schmid factors for the existing deformation systems and the inverse orientation factors were calculated, as described in [18].

The mechanical properties were evaluated through uniaxial tensile tests carried out at room temperature in an Instron 3382 testing machine with an extension rate of 1 mm/min on 33 mm long dog bone-shaped specimens with a gauge length of 15.0 mm and diameter of 3.0 mm . The fatigue tests were carried out under cyclic tension using an ElectroPulsTME3000 machine (testing frequency of 30 Hz , stress ratio $R = 0.1$).

The corrosion properties were investigated by the weight loss method upon immersion of specimens in a 0.9% NaCl solution at

37°C for 4 weeks. Before weighing the specimens after immersion, the corrosion products were removed according to ASTM_G1-03-E.

3. Results and Discussion

The study of the alloys microstructure in the initial state showed that after homogenization in both cases a uniform structure with equiaxed grains is formed (Figure 2 a, b) (an average grain size of $\sim 70 \mu\text{m}$ for the WE43 alloy and $\sim 105 \mu\text{m}$ for the ZX10 alloy). The formation of smaller grains in the case of the WE43 alloy, even despite the longer homogenization duration, is probably associated with the presence of Zr in its composition, which use as a modifier for refinement the structure. In addition, due to the low diffusion rate of rare-earth metal atoms in the case of WE43 alloy, more thermal energy is required to dissolve the phase. ECAP leads to a significant refinement of the structure of both alloys. An ultrafine grained structure with an average grain size of $0.69 \pm 0.13 \mu\text{m}$, as well as globular particles of the $\text{Mg}_{41}\text{Nd}_5$ phase [19] with an average size of $0.45 \pm 0.18 \mu\text{m}$ are formed after ECAP in the WE43 alloy (Figure 2 c). At the same time, ECAP in the ZX10 alloy leads to the formation of a rather inhomogeneous structure elongated along the pressing direction with an average grain size of $8 \pm 0.18 \mu\text{m}$ in the longitudinal section (Figure 2 d) and $4 \pm 0.19 \mu\text{m}$ in the transverse one. The formation of larger grains, in comparison with the previous alloy, is probably associated with a smaller number of ECAP passes, as well as a lower temperature recrystallization of the alloy. The second phase particles were not found in this alloy after ECAP.

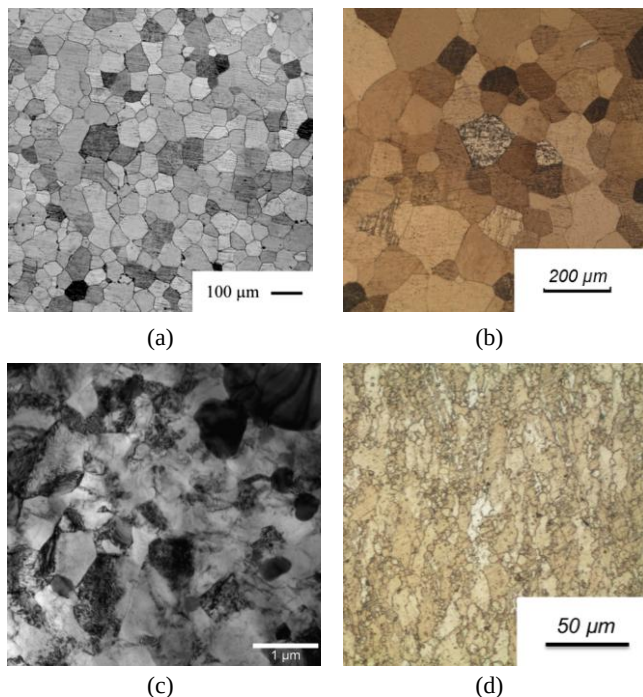


Figure 2 – The microstructure of the WE43 alloy (a, c) and the ZX10 alloy (b, d) in homogenized (a, b) and deformed (c, d) states

The study of the mechanical properties of the alloys showed that the formation of a more dispersed structure after ECAP in the case of the WE43 alloy most significantly strengthened the alloy. So in the initial state, a yield stress (YS) of the WE43 alloy was 161 MPa, an ultimate tensile strength (UTS) - 234 MPa, and an elongation (EL) - 9%. But these characteristics increased to 260 MPa, 300 MPa, and 12.4% after ECAP, respectively. The yield stress was 92 MPa, the ultimate tensile strength - 194 MPa, and the elongation - 12.8% in the case of the initial state of the ZX10 alloy. After ECAP, the ductility is almost doubled to 23.9%, while the strength remains almost unchanged (the yield stress is 106 MPa and the ultimate tensile strength is 215 MPa) (Table 1).

Table 1 – Mechanical properties of WE43 and ZX10 alloys in the initial state and after ECAP processing

Conditions		UTS, MPa	YS, MPa	El, %
ZX10	Initial state	194	92	12.8
	ECAP	215	106	23.9
WE43	Initial state	234	161	9.0
	ECAP	300	260	12.4

As noted earlier, the increase in strength characteristics is associated with the grain refinement. However, for example, for ZX10 alloy, grain refinement after ECAP more than 10 times does not lead to a significant increase in strength. In addition, for both alloys, despite the refinement of the structure, an increase in ductility is observed. That is, it can be assumed that there are additional factors affecting the final properties. In magnesium alloys, in addition to structure, the transformation of texture also contributes to the change in mechanical properties. Therefore, to assess the contribution of the texture to the change in the mechanical properties of materials, direct pole figures of the alloys were constructed before and after ECAP, and the orientation factors of the main deformation systems and twinning were calculated (Figure 3 and Table 2).

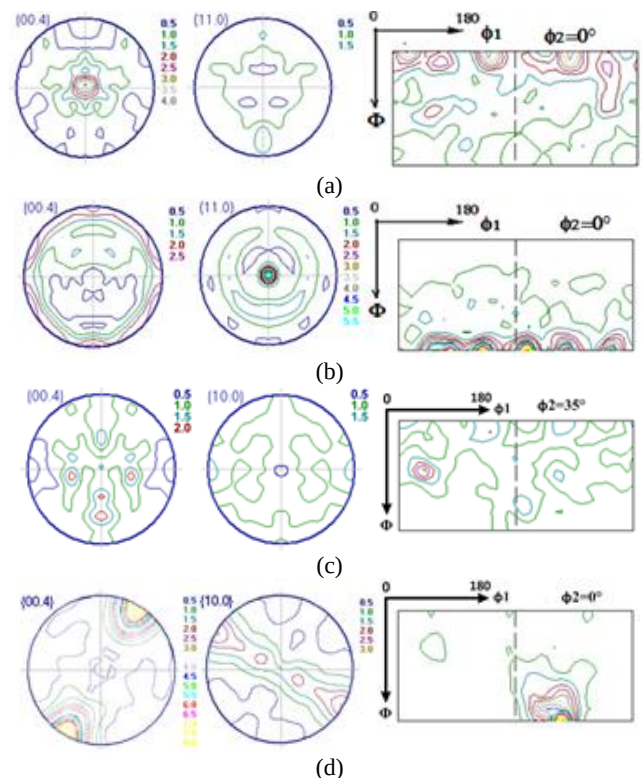


Figure 3 - {00.4} and {11.0} pole figures and ODF sections at $\phi_2 = \text{const}$ for the WE43 (a, b) and ZX10 (c, d) alloys in the initial states (a, c) and after ECAP processing (b, d)

The texture analysis showed that a cardinal change of texture type occurs from a sharp basal texture in the initial state to a sharp prismatic texture in the case of ECAP of the WE43 alloy (Figure 3 a, b). It is known that activation of prismatic slip in magnesium alloys leads to an improvement in their ductility, which we observe for the WE43 alloy. At the same time, the magnesium alloys after ECAP are also characterized by the formation of an inclined basal texture, which has a beneficial effect on the ductility, but does not improve (and often worsens) their strength [19, 20]. We observe a similar situation in the case of the ZX10 alloy, where ECAP leads to the transformation of a diffused basal texture into a sharp basal texture inclined by $80-85^\circ$ (Figure 3 c, d). At the same time, in the case of alloys with rare-earth metals, fine particles are deposited in the basal planes, which make it difficult to slip dislocations along

them [21]. This leads to partial inhibition of the basal slip and activation of the prismatic one, which does not lead to deterioration in ductility and strength. We observe a similar situation for the WE43 alloy. Moreover, a decrease in the values of orientation factors for prismatic slip planes also indicates an increased likelihood of prismatic slip in the alloys after ECAP (Table 2). The probability of basal and pyramidal slip activity, as well as twinning in the case of WE43 alloy, is reduced. Twinning activity in the case of the ZX10 alloy remains unchanged.

Table 2. Orientation factors for WE43 and ZX10 alloys in the initial state and after ECAP processing

Conditions	Basal {0001} <1120>	Prismatic {1010} <1120>	Pyramidal <c+a>	Twinning {1012} <1011>
Initial state (WE43)	4.6	6.2	4.1	4.0
ECAP (WE43)	6.0	4.3	5.0	5.0
Initial state (ZX10)	4.4	5.1	4.7	4.8
ECAP (ZX10)	5.9	3.9	4.9	4.8

The Figure 4 shows the results of the fatigue life study of the both studied alloys before and after ECAP.

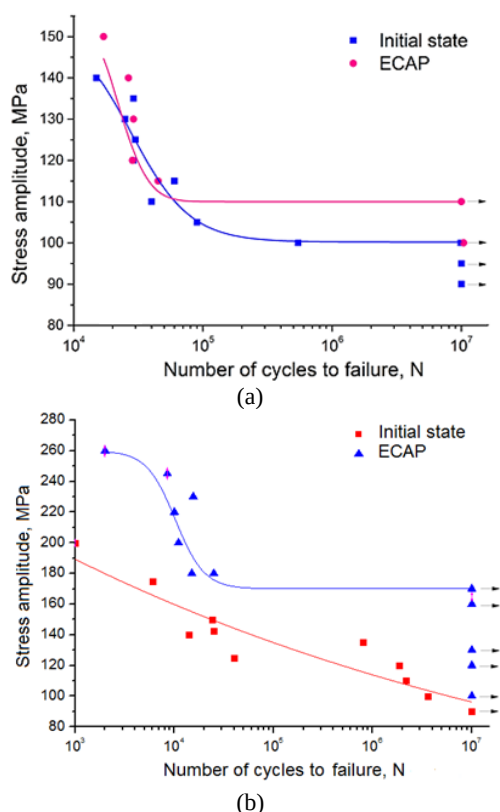
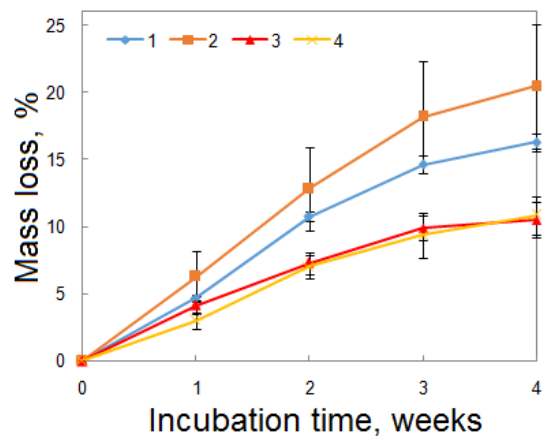


Figure 4 - Fatigue behaviour of the ZX10 (a) and WE43 (b) alloys in the initial states and after ECAP

It was shown that the grain refinement after ECAP leads to an increase in the fatigue limit of both studied alloys. In the case of the ZX10 alloy, the increase in fatigue limit is small, probably due to the formation of an unfavorable texture. So the fatigue limit of the alloy in the initial state was 100 MPa, and after ECAP - 110 MPa. For the WE43 alloy, the increase in the fatigue limit is more substantial: from 90 MPa in the initial state to 170 MPa after ECAP. One of the most important in-service properties of the magnesium medical alloys is their corrosion resistance. The corrosion resistance studies were carried out in a physiological saline solution of 0.9% NaCl at 37 °C in this work (Figure 2).



1 – ZX10 alloy in initial state; 2 – ZX10 alloy after ECAP;
3 – WE43 alloy in initial state; 4 – WE43 alloy after ECAP
Figure 5 – The study of corrosion resistance of the alloys

It was shown that the WE43 alloy has a higher corrosion resistance compared to the ZX10 alloy both in the initial state and after ECAP, apparently, due to the presence of rare-earth metals in the composition. Moreover, ECAP does not lead to a decrease in the corrosion resistance of both alloys. The total mass loss over 4 weeks of testing was 16.3 ± 0.6 and $20.4 \pm 4.7\%$ for the homogenized and deformed states of the ZX10 alloy and 10.5 ± 1.3 and $10.8 \pm 1.5\%$ for the homogenized and deformed states of the WE43 alloy, respectively.

4. Conclusions

1. ECAP leads to significant structure refinement of both studied alloys. For the WE43 alloy an UFG structure is formed with an average grain size of $0.69 \pm 0.13 \mu\text{m}$, as well as $\text{Mg}_{41}\text{Nd}_5$ phase particles with an average size of $0.45 \pm 0.18 \mu\text{m}$ during ECAP. ECAP processing of ZX10 alloy gave rise to grain refinement from $\sim 105 \mu\text{m}$ in the initial state to $4.0 \pm 0.19 \mu\text{m}$ and $8.0 \pm 0.18 \mu\text{m}$ in transversal and longitudinal cross-sections of the billets.
2. The grain refinement caused by ECAP of WE43 alloy result in improved strength characteristics ($Y_S = 260 \text{ MPa}$ and $UTS = 300 \text{ MPa}$), while also raising its tensile ductility to 12.4%. ECAP of ZX10 alloy was shown to moderately increase the mechanical strength of the alloy, while doubling its tensile elongation. The increasing of ductility is believed to be associated with the activation of prismatic slip.
3. A further positive effect of ECAP is an increase of the fatigue limit by $\sim 10\%$ on the ZX10 alloy and $\sim 89\%$ on the WE43 alloy.
4. The above beneficial effects were achieved without ECAP impairing the chemical corrosion resistance.

Acknowledgments

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5. Literature

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THE STRENGTHENING AND RENWALING OF DAMAGER REINFORCED CONCRETE STRUCTURES BY HRESTRESSED FIBER REINFORCTD PLASTICS

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Abstract: *The results of experimental studies of the operation of reinforced concrete structures with surface reinforcement of fiber reinforced plastics for various types of stress state under static and dynamic loads are presented. Methodologies have been developed for the restoration of damaged reinforced concrete structures by pre-stressed fiber reinforced plastics. Examples are given of restoring the operational suitability of various objects.*

KEYWORDS: STRENGTHENING, RENEWALING, DAMAGER REINFORCED CONCRETE CTRUCTURE, PRESTRESS, FIBER, PLASTIC

1. Introduction

Due to the frequent change of purpose, reconstruction and renovation, the requirements for improving reliability and comfort, physical wear and damage from corrosion of buildings and structures, the need to strengthen building structures is becoming increasingly important. A particularly important role is played by strengthening and restoring buildings and structures in seismic areas of construction, in areas of natural disasters, after accidents and terrorist acts.

The main material of the supporting structures of buildings and structures of the present is monolithic or precast concrete, therefore, the problems of improving methods for reinforcing reinforced concrete structures are of particular relevance.

Traditional methods for reinforcing reinforced concrete structures are very laborious, require overburden, welded and concrete work, require considerable time to provide the required strength.

In recent decades, the scope of application of composite materials in construction has been rapidly expanding. The market of composites in construction is about 5 billion euros, and composite carbon materials occupy a leading place in them, which are used primarily for restoring bearing capacity and strengthening various engineering structures, industrial and civil buildings. Composite materials are used to strengthen building structures from various materials. More than 80% of reinforced concrete structures in the USA, Japan, Canada, Germany, England and other developed countries are made by external reinforcement systems using fibro-plastic materials [1], [2].

In the last decade, an active expansion of the production of beams, laminated plates, shells and sandwich structures from a composite with fiber reinforced fibers has begun. Working Group CEN / TC250) WG) 4 published a Report, which contains preliminary provisions of the FRP (Fiber-reinforced Fiber-Plastic Reinforced Structural Design Guide).

New, modern methods of reinforcing reinforced concrete structures are focused on the application for reinforcing composite fiber-reinforced plastic materials characterized by high strength, corrosion resistance and durability. The main component of these composite materials are layers of parallel-oriented fibrous mineral materials (carbon, basalt, aramid or glass), manufactured by nanotechnology.

Reinforcing fibers are located in a polymer matrix of various resins (epoxy, phenolic, polyester, vinyl ester or other types of organic resins). These products are manufactured in the factory and are supplied in the form of lamellas or laminate tapes having one directional fiber, as well as wrapping sheet coatings in the form of fabrics, nets or sheets with unidirectional or bidirectional fibers. The most common are carbon fiber reinforced materials for which

carbon fibers are obtained from synthetic and natural fibers based on cellulose, copolymers of acrylonitrile, oil and coal tar pitch, etc.

Carbon fibers have the following advantages:

- have high tensile strength superior to steel;
- have a low density, they are lighter than aluminum by 20%;
- withstand temperatures up to 16,000 C;
- are a good energy absorber.

The disadvantages of carbon fiber:

- high price;
- the complexity of repair or restoration in case of damage;
- electrical conductivity;
- is damaged in the sun;
- carbon fiber from carbon fiber and rubber can withstand the most powerful shock loads, but it breaks brittle.

The most common method of reinforcing reinforced concrete structures with fiber reinforced plastics is the use of surface reinforcement of building structures, for which a primer is used for surfaces, putty, adhesives, one or two-sided sheet fabric or laminate. Surface reinforcement of reinforced concrete structures is carried out by directly gluing reinforcing composite materials onto concrete surfaces and is characterized by high reinforcement efficiency, simplicity and high speed of execution, low laboriousness of work, and quick terms of acquiring the required strength.

2. Experimental research

2.1. Studies of the work of flexible structures

To study the features of the work of flexible concrete structures reinforced with a carbonate unidirectional laminate and nets, a complex was completed that included the following steps:

- study of the work of normal sections of flexible reinforced concrete beams reinforced with carbon fiber laminate tapes;
- study of the strength of inclined sections of flexible concrete beams reinforced with carbon fiber fibers of laminate tapes and nets;
- assessment of the effect of a few dynamic repeated loads on the work of flexible reinforced concrete beams reinforced with carbon fiber tapes of laminate tapes.

Experimental reinforced concrete samples of beams 2200 mm long, 120x200 (bxh) mm section, were reinforced with spatial knitted frames with asymmetric steel reinforcement of 2□12 class A-III in the lower zone, 2□10 class A-III in the upper zone and clamps □6 Class A-I.

The study of the work of normal sections was performed on three batches of prototypes:

- The first batch was reinforced with laminate tapes based on carbon fiber fibers of the S&H Laminate CFR 150/200 type with a section of 50x1.2 mm (BASF, Germany);
- The second batch was reinforced with ribbons of the FibArmTape 530/300 12/100 type lamella with a section of 100 × 1.2 mm (HC Composite, Russia);

- The third batch was reinforced with laminate tapes based on carbon fibers of the MBACE LAM CF 165/3000 100x1.4 100m type with a section of 100x1.4 mm (BASF, Germany).

Static tests of beams according to the scheme of a single-span beam, loaded in thirds of the span with two concentrated forces, were carried out in a power stand under phased loading with a transverse load created by a hydraulic jack and a manual pump station.

The destruction of beams that did not have reinforcement occurred in the zone of pure bending and was accompanied by crushing of the compressed zone of concrete at stresses in tensile reinforcement corresponding to the yield strength of steel, deflections of the beam were 1 / 27.5 of the span, and the crack opening width reached 2 mm or more.

Gluing one layer of S&P C FK 150/2000 laminate onto the stretched edge of the beam led to an increase in beam strength by an average of 76%. In this case, the deformations of the compressed zone of concrete and the tensile reinforcement did not practically change, and the deformations of the stretched laminate were about 6 ‰, the deflections of the beam and the width of the crack opening were more than halved.

Testing of the beams of the second batch reinforced with a FibArm-Tape 530/300 1.2x100 mm lamella showed that with one layer of glued lamella, normal cracks in the zone of clean bending appeared at a bending moment of 26.3 kNm, and later, until the fracture, the opening width cracks did not exceed 0.25 mm. The destruction of the beams was caused by the separation of the protective layer of concrete in the area of anchoring of the lamella and was brittle. The gluing of two lamella layers in the stretched zone did not lead to an increase in the strength of the beams, but was accompanied by a decrease in the deflection, a decrease in the width of the crack opening, and a decrease in the deformations in the stretched reinforcement. The beams turned out to be reinforced, their destruction was accompanied by a detachment of the concrete protective layer in the lamella anchoring zone and was brittle (Fig. 1).



Fig. 1 - Detachment of protective milking concrete in the area of anchor lamella

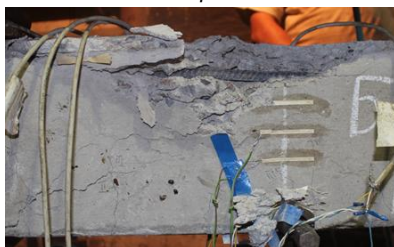


Fig. 2 - Destruction of the compressed zone of normal section

Beams of the third batch were reinforced in the stretched zone by a laminate of the MBACE LAMCF 165/3000 series 100x1.4 mm

and they occurred in the clean bend zone and were accompanied by the destruction of the compressed concrete zone and the peeling of the laminate from the stretched concrete zone, or by the separation of the concrete protective layer with a normal crack opening width of more than 1.2 mm. Longitudinal deformations along the height of the section are distributed almost linearly. The greatest deformations of concrete on the compressed face reach 3.0 ‰, and the deformations of the laminate were in the range of 2.6-3.7 ‰.

The features of the normal cross sections of bent beams under dynamic loading were studied by testing the samples with a hydrodynamic machine with double action jacks with an asymmetry coefficient of force change $\rho \approx 0.04$ and a loading frequency of 1.0 hertz.

In the process of a few repeated loads (15–25 loading cycles), the deflections of the non-reinforced beams of the first batch increased by 30–40%, and the deformations of the stretched steel reinforcement increased by 25–30%. Similar beam specimens reinforced in the stretched and compressed zone by S&P C FK 150/2000 laminate tapes withstood 15-65 repeated loading cycles with a maximum amplitude of 67-75% of the static strength of the twin samples. Dynamic

the strength of the reinforced samples was 45% higher than the dynamic strength of the samples without reinforcement, and the strains of the stretched laminate reached 3.0-3.52 ‰, the strains of the compressed laminate reached 2.6 ‰.

Samples of bending beams of the B-II series, reinforced in the lower zone with a single layer of FibArm Lamel 12x100 lamella, withstood 8-280 cycles of dynamic repeated loading with a maximum amplitude exceeding the static strength by 3-28%. In this case, the lamella deformations increased from 3.51 ‰ to 3.821 ‰ (by 10%), and the vertical deflections increased from 23 mm to 29 mm (26%). The destruction of the beam was accompanied by the formation and development of normal and inclined cracks, as well as the breaking of the protective layer of concrete in the zone of anchoring of the lamella. The dynamic strength of the reinforced beams exceeded the static strength by 33%. Strength at repeated loads is described by the following empirical dependence:

$$M_d/M_{cr} = 1.33 - 0.116 \lg n \quad (1)$$

Prototypes of reinforced concrete beams designed to study inclined sections were reinforced with spatial knitted frames with asymmetric metal reinforcement of 3-12A-III in the lower zone, 2-10A-III in the upper zone. The supporting zones of the beams are reinforced with a sticker of vertical and inclined fiber-reinforced polymer networks.

Three batches of prototypes were tested:

- The first batch was strengthened with grids based on carbon fiber fibers of the S&P C Sheet 240 type with a width of 50 mm (BASF, Germany);
- The second batch was reinforced with grids of the FibArmTape 530/300 type with a width of 300 mm (HC Composite, Russia);
- The third batch was reinforced with grids based on carbon fiber S&H Laminate 150/200 cross-section of 50x1.2 mm.

Gluing of unidirectional vertical meshes in the supporting zone led to an increase in the shear strength of the inclined sections of flexible concrete structures by 1.4-2.3 times, and the destruction of the beams was caused by the separation of the protective layer of concrete from the sides of the beam and is accompanied by brittle fragmentation of the concrete of the supporting zone.

The diagram of tensile stresses in the fibers of fiber-reinforced nets along the length of the inclined section is in the form of a convex arc with the greatest deformations up to 3-3.8 ‰.

2.2. Studies of compressed structures

The study of the operation of compressed concrete elements reinforced with fiber reinforced plastics was carried out on samples of concrete cylinders and prisms of various sizes, differing in the number of layers of reinforcement meshes, as well as the nature of the application of compressive load.

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Static tests were carried out on hydraulic presses IPS-200 and ALPHA 3-3000S. For this, the samples were subjected to axial compression with a gradual increase in the longitudinal compressive force at a rate of 0.3 MPa / s, up to the destruction of the sample. During the tests, longitudinal and transverse deformations of the experimental samples were measured using strain gauges with a base of 50 mm glued to the side surfaces.

Reinforcement of samples by wrapping (gluing) material of the FibArm Tape 530/300 series causes triaxial compression of concrete due to the limitation of transverse deformations of concrete caused by the presence of a holder from reinforcing grids, and leads to a significant increase in the strength of compressed samples, as well as longitudinal and transverse deformations. The strength of concrete when reinforced with one layer of nets increased by 1.79-3 times, by two layers - by 3.12-3.75 times and by three layers - by 3.5-5.53 times. This suggests that with increasing cross-sectional dimensions, the effect of increasing strength decreases depending on the percentage of reinforcement of the section with polymer networks.

Strengthening compressed concrete samples with fiber reinforced cages leads to a significant increase in longitudinal and transverse deformations of concrete.

Thus, the ultimate longitudinal deformations of concrete as compared to deformations of samples without reinforcement by clips increased 1.82–4.4 times, and transverse deformations increased 4.6–20 times. In this case, the ultimate transverse strains in magnitude approach the longitudinal strain.

In the process of increasing longitudinal compressive stresses in concrete reinforced with a cage made of polymer nets, microcracks are formed in concrete, and after exceeding the stresses corresponding to the exhaustion of concrete strength without cages, an accelerated increase in concrete volume begins, with stresses in reinforced concrete of about 70-80 % of destructive stresses, the volume of concrete is close to the initial volume (the volume of concrete before loading with longitudinal force), and before the strength of concrete reinforced with meshes is exhausted, an increase concrete volume can reach 0.55-0.65%.

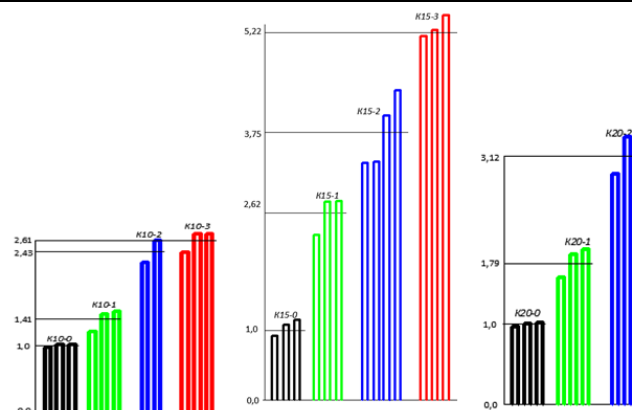


Fig. 3 Dependence of strength on the number of gain grids

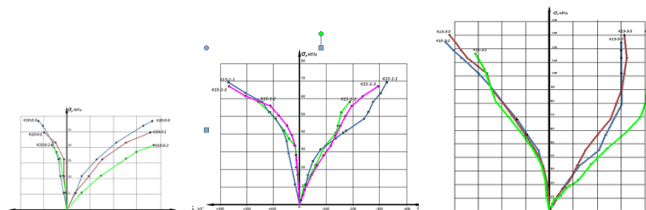


Fig. 4 Diagrams of longitudinal and transverse deformations concrete without reinforcement, with 1st and 3rd layers of reinforcement mesh



Fig. 5 - Photo of the destroyed samples without amplification, with 1st and 3rd gain grids

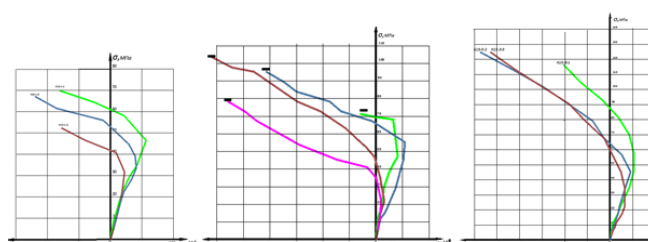


Figure 6 - Diagram of the increase in concrete volume at the 1st, 2nd and 3 layers of gain grids

In the process of increasing the longitudinal compressive force, an accelerated development of transverse deformations of concrete was observed, and before failure, a crack is heard from the rupture of individual fibers of fiber-reinforced plastics, then the net grids break, accompanied by crushing of the concrete and a sharp sound

The second batch of concrete samples consisted of cylinders with a diameter of 150 mm and a length of 300 mm, which were reinforced with clips of nets MBACE FIB CF 230 / 4900.530g / 5.100m. In general, the results of testing concrete cylinders of the second batch are similar to the results of testing the first batch of cylinders.

The effect of dynamic repeated compressive forces was studied on samples of concrete prisms under axial compression created using a hydraulic machine with an asymmetry coefficient of force change ($\rho = 0.0 \div 0.10$), a loading frequency of 1.0 hertz and a value of compressive stresses that ensure the destruction of experimental samples for $3 \div 100$ cycles. The experimental concrete samples were reinforced with bandages from one layer of the MBACE FIB CF 230 / 4900.530g / 5.100m mesh.

Characterizing the process of damage accumulation in concrete during repeated loading, two cases can be distinguished:

- With the number of repeated loads exceeding 50 cycles, after the first loading cycles, the concrete deformation diagram changes little over a considerable number of subsequent loads, and only a few cycles before the concrete breaks, an accelerating increase in inelastic deformation is observed, ending with the exhaustion of concrete strength.

- When the maximum compressive stresses are more than 90% of the concrete strength, which lead to the destruction of concrete in several cycles, with each subsequent loading cycle, an accelerating increase in inelastic deformation of concrete occurs.

The magnitude of the longitudinal deformations of concrete in the process of reloading was $146 \times 10^{-5} \div 175 \times 10^{-5}$ relative units, and the value of the residual deformations of concrete reached 100×10^{-5} relative units. The greatest value of transverse deformations of concrete was $33 \times 10^{-5} \div 56 \times 10^{-5}$ relative units. The following dependence of the strength on the number of repeated loads is proposed;

$$\sigma_n = 1.256.R_0(1 - 0.11 \lg n) \quad (2)$$

Dynamic tests of concrete prisms, reinforced with one, two and three layers of nets, showed that the average strength increase of the samples is, respectively, 43%, 55% and 75%; ultimate longitudinal deformations of concrete increased by 37%, 38% and 55%; and transverse strains - by 27%, 54% and 86%.

2.3.Features of strength calculation

When calculating the reinforcement of reinforced concrete structures with fiber reinforced plastics (FRP), it is recommended to observe the following provisions:

1. When calculating the strength of normal sections, the tensile diagram of the fiber reinforced plastic reinforcement is assumed to be linear. After strengthening to failure, the conditions for the compatibility of deformations are preserved. The selection of the cross-sectional area of the fiber reinforced plastic reinforcement is carried out by the iterative method by setting some of its area and further adjusting it according to the results of strength calculations in the right direction. When calculating, one should take into account the possibility of delamination of the fiber reinforced plastic reinforcement from concrete or the separation of the protective layer of concrete by this reinforcement.

2. The strength of the inclined section reinforced with fiber reinforced plastics is defined as the sum of the strength of the section without reinforcement and the additional transverse force that is perceived by the transverse fibro-plastic.

3. The strength of the columns under the action of longitudinal compressive force can be determined by the following relationships:

$$N = 0,85.[0,85R_{bf}(A - A_s) + R_s^1 A_s] \quad (3)$$

$$R_{bf} = R_b[2,25\sqrt{1 + 7,9\frac{\sigma_f}{R_b}} - 1,25 - 2\frac{\sigma_f}{R_d}] \quad (4)$$

3. Restoration of structures

During the construction and operation of buildings, often bent reinforced concrete structures receive unacceptable deflections or excessive crack opening. With surface reinforcement, fiber-reinforced plastics are included in the work of reinforced structures only with a further increase in load, although at the same time they reduce speed.

To restore the serviceability of damaged bent reinforced concrete structures, the use of prestressing reinforcing fiber reinforced plastics is proposed. A method has been developed to create prestress by creating a temporary building lift of damaged structures. This causes crack clamping and a decrease in deflections in the damaged elements. Then, surface reinforcement of the exposed areas of the overlap is performed, and after the adhesives are set, the raised structures are lowered and lowered in the process with the restored element, the pre-stress of the glued fiber-reinforced plastics occurs. Fiber reinforcing materials are included in collaboration with the restored element.

This provides a reduction in the strains of the stretched metal reinforcement, reduces the width of crack opening, eliminates excessive deflection of the structure and restores the serviceability of reinforced concrete floors. To restore the serviceability of damaged flexible concrete structures, it is proposed to use prestressing reinforcing fiber reinforced plastics.

This provides a reduction in the strains of the stretched metal reinforcement, reduces the width of crack opening, eliminates excessive deflection of the structure and restores the serviceability of reinforced concrete floors.

In Kazakhstan, several dozen large multi-story buildings received reinforcement and restoration of damaged reinforced concrete floors. The bending elements were lifted by telescopic racks, jacks or truss systems from ropes.

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ENERGY HEAT EXCHANGE IN THE ZONE OF CONTACT OF THE PROBE OF AN ATOMIC FORCE MICROSCOPE WITH THE SURFACE UNDER STUDY

ЕНЕРГЕТИЧНИЙ ТЕПЛООБМІН В ЗОНІ КОНТАКТУ ЗОНДА АТОМНО-СИЛОВОГО МІКРОСКОПА З ДОСЛІДЖУВАНОЇ ПОВЕРХНЕЮ

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Abstract: *The article studies the mechanisms of energy exchange and transformations, which occur in a measuring instrument (probe) of an atomic force microscope (AFM) in the process of studying solid surfaces of materials. Mathematical modeling of the heating process of individual elements of the measuring unit of the atomic force microscope at the preparatory, final stages and the scanning stage of the surface under study was carried out. At the same time, such energy components of the processes occurring in the AFM control unit were taken into account. By minimizing the factors (heat dissipation due to friction of the probe on the surface), which negatively affect both the results of the monitoring and the condition of the probe and the surface, stable operation modes of the AFM are established. The solution of the equivalent thermal scheme of an atomic force microscope is presented, which confirms the adequacy of the mathematical models obtained.*

KEYWORDS: ATOMIC FORCE MICROSCOPE, ENERGY HEAT EXCHANGE, HEAT LOSSES, MATH MODELING, EQUIVALENT THERMAL SCHEME

1. Introduction

Among the main analytical methods for studying the state of the surface and physico-mechanical properties, method of atomic force microscopy, of course, has significant advantages: high spatial resolution, efficiency, accuracy and objectivity [1-3].

At the same time, despite the obvious advantages of the method, the accuracy and adequacy of the results of the study of some nanostructured coatings and materials by atomic force microscopy (AFM) is questionable. This is due both to the wrong choice of research modes, and to external power and energy factors of interaction of the sensitive AFM element with the surfaces of these materials [4, 5].

Some attempts to carry out calculations and modeling of both power and energy factors were carried out by a number of both domestic and foreign scientists, such as: Suslov A., Garishin O., Konstantinov V., Chizhik S., Sviridova O., Moya S. and other [5-7]. However, the results of such modeling, which are given in the works of these scientists, have low accuracy and, in general, make it impossible to carry out operational calculations directly in the process of experimental research.

At the same time, if the factors of force interaction can be experimentally confirmed, the experimental confirmation of the mechanisms of energy heat exchange taking place in the measuring instrument (probe) of an atomic force microscope in the process of studying solid surfaces of materials is currently impossible.

Therefore, it is relevant to study these phenomena is a mathematical modeling of the heating process of individual elements of the measuring unit of an atomic force microscope.

The purpose of this work is to study the mechanisms of energy heat exchange in the zone of contact of the probe of an atomic force microscope with the surface under investigation by applying mathematical modeling techniques and their subsequent confirmation by the method of equivalent thermal scheme.

2. The modeling results and their discussion

The measuring unit of an atomic force microscope «NT-206» (manufacturer Microtestmachines Co., Belarus) was chosen as an object for modeling energy heat exchange, namely the systems: «cantilever → probe → object under study ← piezo scanner» and «laser positioner → probe → photo detector».

The simulation of the interaction of these systems consisted in the compilation of an adequate energy scheme of the device operation. At the same time, the main energy parameters of the scheme were divided into two groups: heat losses and energy impact.

Among the heat losses, the costs for mechanical friction of the probe over the surface under study, costs for the action of electromagnetic fields and electric charge in the sensor's coverage area, costs for bending and torsion of the cantilever during the scanning process and costs for the piezo scanner result from the device operation should be highlighted.

At the same time, the energy impact in the scanning area of the AFM is divided into: radiation energy, which is generated by the laser positioner and sent to the AFM cantilever, laser energy, which is reflected from the cantilever and sent to the photodetector, the energy that drives the piezoscanner, and also the useful work of the piezoscanner. It should be noted that the difference between the energy of the radiation generated by the laser positioner and the energy that is sent to the photodetector is almost completely converted to heat, which heats the cantilever.

For modeling, the energy scheme of the measuring unit of the AFM was proposed, which allowed determining the mechanism of thermal exchange between the individual elements of the measuring unit of the device (Fig.1).

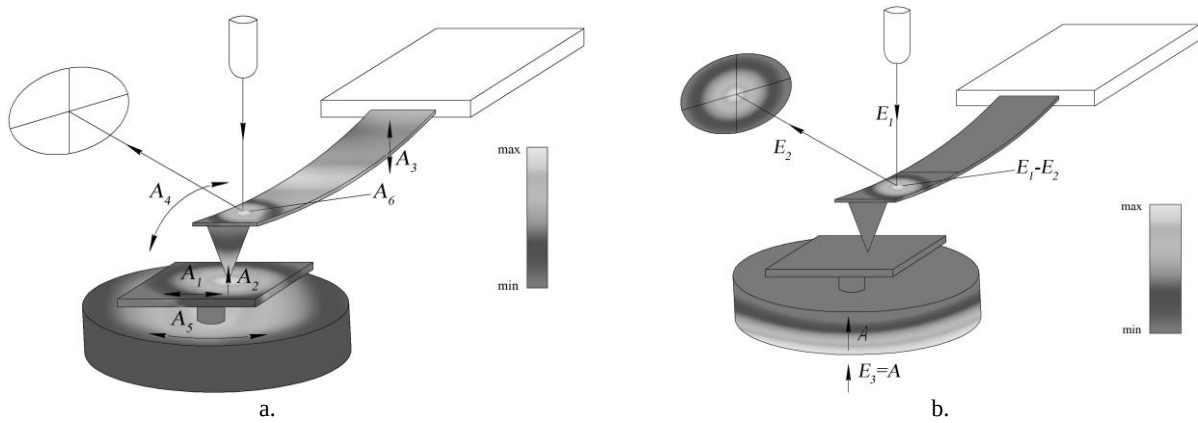


Fig.1. Energy scheme of the measuring unit of the atomic force microscope:

Heat losses (a): A_1 – on the mechanical friction of the probe on the test surface; A_2 – on the action of electromagnetic fields and electric charge; A_3 – on the cantilever bending; A_4 – on the cantilever torsion; A_5 – on the piezo scanner torsion.

Energy impact (b): E_1 – laser positioner radiation energy; E_2 – laser energy incident on a photo detector; E_3 – the energy that drives the piezoscaner; A – useful work piezo scanner

In the process of thermal calculations, a number of mathematical models of the heating process of individual elements of the AFM were compiled and analytically solved using the inverse Fourier transform method. In this case, only heat exchange of heat

transfer was taken into account (neither convective nor ray types of heat exchange were considered due to their irrelevance).

The basis of thermal calculations laid the equation of heat balance between the individual elements of the AFM [6]:

$$(r, t) = \left\{ T_0^{v+1} + \frac{(v+1)q_{n0}R}{\lambda_0} \times \left[\frac{3a_0^2 t}{10R^2} - \sum_{n=1}^{\infty} \frac{2R \sin\left(\mu_n \frac{r}{R}\right)}{\mu_n^3 \cos(\mu_n) R} e^{-\left(\frac{\mu_n a_0}{R}\right)^2 t} \right] \right\} \quad (1)$$

$$(z, t) = \left\{ T_0^{v+1} + \frac{(v+1)q_{n0}a_0}{\sqrt{\pi}\lambda_0} \int_0^t \frac{e^{-\frac{z^2}{4a_0^2(t-\tau)}}}{\sqrt{t-\tau}} d\tau \right\}^{\frac{1}{v+1}} \quad (2)$$

$$(z, t) = \left\{ T_0^{v+1} + \frac{(v+1)q_{n0}}{\lambda_0} \times \left[\frac{a_0^2 t}{H} + \frac{3z^2 - H^2}{6H} + \frac{2H}{\pi^2} \times \sum_{n=1}^{\infty} (-1)^{n+1} \frac{\cos\left(\frac{\pi n z}{H}\right)}{n^2} e^{-\left(\frac{\pi a_0 n}{H}\right)^2 t} \right] \right\}^{\frac{1}{v+1}} \quad (3)$$

$$T(r, z, t) = \left\{ T_0^{v+1} + \frac{(v+1)}{4\pi\lambda_0} \int_0^{+\infty} \left(1 - \frac{r^2 \xi^2}{4} + \frac{r^4 \xi^4}{64} - \frac{r^6 \xi^6}{2304} \right) \times \right. \\ \left. \left[p_{01} \left(1 - \frac{R_1^2 \xi^2}{8} + \frac{R_1^4 \xi^4}{192} - \frac{R_1^6 \xi^6}{9216} \right) + p_{02} \left(1 - \frac{\xi^2}{8} (R_2^2 - R_1^2) + \frac{\xi^4}{192} (R_2^2 - R_1^2) - \frac{\xi^6}{9216} (R_2^2 - R_1^2) \right) \right] \right\} \times \quad (4)$$

$$\begin{aligned}
 & \times \left[e^{-\frac{z}{2a_0\sqrt{t}}} \left(1 - \operatorname{erf} \left(\frac{z}{2a_0\sqrt{t}} - \xi a_0 \sqrt{t} \right) \right) - e^{-\frac{z}{2a_0\sqrt{t}}} \left(1 - \operatorname{erf} \left(\frac{z}{2a_0\sqrt{t}} + \xi a_0 \sqrt{t} \right) \right) \right] d\xi \left. \right\}^{\frac{1}{v+1}} \\
 T(r,t) = & \left[T_0^{v+1} + \frac{(v+1)q_{n0}R}{\lambda_0} \times \left(\frac{3a_0^2 t}{10R^2} - \sum_{n=1}^{\infty} \frac{2R \sin\left(\mu_n \frac{r}{R}\right)}{\mu_n^3 \cos(\mu_n)R} e^{-\left(\frac{\mu_n a_0}{R}\right)^2 t} \right) \right] \quad (5)
 \end{aligned}$$

Here, T_0 – model initial temperature, K; v – empirical coefficient, q_{n0} – external heat flux, Вт/м²; r , R – радіуси вістря та основи зонда відповідно, м; λ_0 – коефіцієнт теплопровідності, W/мK; a_0 – coefficient of thermal diffusivity, m²/s; t – heat exposure time, s; ξ – the variable of layer thickness is heated, m; z – probe penetration depth, m; H – plate thickness, m; p_{01} , p_{02} – power sources of thermal exposure, W.

Each of the equations of the above system (1) – (5) is the heating equation for certain elements of the measuring unit of an atomic force microscope, the computational models of which are

shown in Fig. 2:

- heating equation of a cut cone element (probe): Fig. 2 – a, (1);
- heating equation of a plate of small thickness (sample): Fig. 2 – b, (2);
- heating equation of a plate of small thickness (cantilever): Fig. 2 – c, (3);
- heating equation of the end surface of a cylinder (piezoscanner): Fig. 2 – d, (4);
- heating equation of spherical element segment (probe tip): Fig. 2 – e, (5).

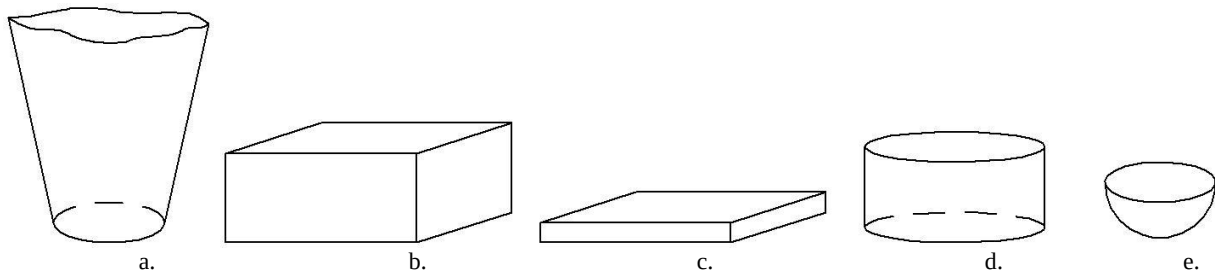


Fig.2. Calculation models of the main elements of the measuring unit of an atomic force microscope: a – of a cut cone element (probe); b – of a small thickness plate (sample); c – of a small thickness plate (cantilever); d – of the end surface of the cylinder (piezoscanner); e – of a spherical element segment (probe tip).

The coordination of the results of solving this system was carried out according to the heat balance scheme, in which the total thermal contribution was evenly divided between all the elements that took part in the heat exchange (the thermal and physical properties of the materials (heat capacity, thermal conductivity) of which these elements were made were not taken into account).

To confirm the adequacy of the considered models of heat transfer, an equivalent thermal scheme of the AFM was compiled and researched [7, 8].

The method of equivalent thermal circuits is most prevalent because of the simplicity and sufficient accuracy of the calculation. The disadvantage of the method is that it does not provide a complete picture of the temperature field, but only some average values of temperature for individual elements of the instrument or machine.

This method is based on the use of thermal resistances, which

are connected to a heating network that simulates the actual paths of heat flow in the device, and suggests an analogy of heat flow with electric current, based on the same form of the basic law of thermal conductivity (Fourier's law): $\Delta P = \Delta \theta / R_T$ and electric current (Ohm's law): $I = \Delta U / R_E$, where $\Delta \theta$ – temperature drop; R_T – thermal resistance of this gap in the heat flow path; ΔU – potential difference at the ends of the conductor; R_E – electrical resistance.

The system of equations for this scheme in the mode that is installed is given below. In this system of equations m – number of nodes equivalent thermal scheme; θ_B – temperature outside the element in question; $\Lambda_{ki} = 1/R_{ki}$ – thermal conductivity of the corresponding section of the scheme; P_i – heat losses in the i -th node.

$$\begin{cases} P_1 = \left(\sum_{k=1}^m \Lambda_{k1} \right) \cdot (\theta_1 - \theta_a) - \sum_{k=1}^m (\Lambda_{k1} \cdot (\theta_k - \theta_a)) \\ P_2 = \left(\sum_{k=1}^m \Lambda_{k2} \right) \cdot (\theta_2 - \theta_a) - \sum_{k=1}^m (\Lambda_{k2} \cdot (\theta_k - \theta_a)) \\ \dots \\ P_m = \left(\sum_{k=1}^m \Lambda_{km} \right) \cdot (\theta_m - \theta_a) - \sum_{k=1}^m (\Lambda_{km} \cdot (\theta_k - \theta_a)) \end{cases}$$

To calculate the non-stationary mode, the same thermal scheme is used, but each node is connected through a capacitor. In this case, the electrical capacitance is equivalent to the heat capacity of the body. The system of equations for the non-stationary mode is as follows:

$$\begin{cases} C_1 \cdot \frac{d\theta_1}{dt} = \left(-\sum_{k=1}^m \Lambda_{k1} \right) \cdot (\theta_1 - \theta_a) + \sum_{k=1}^m (\Lambda_{k1} \cdot (\theta_k - \theta_a)) + P_1 \\ C_2 \cdot \frac{d\theta_2}{dt} = \left(-\sum_{k=1}^m \Lambda_{k2} \right) \cdot (\theta_2 - \theta_a) + \sum_{k=1}^m (\Lambda_{k2} \cdot (\theta_k - \theta_a)) + P_2 \\ \dots \\ C_m \cdot \frac{d\theta_m}{dt} = \left(-\sum_{k=1}^m \Lambda_{km} \right) \cdot (\theta_m - \theta_a) + \sum_{k=1}^m (\Lambda_{km} \cdot (\theta_k - \theta_a)) + P_m \end{cases}$$

where C_i – heat capacity of the corresponding scheme node.

The systems of differential equations given above, in fact, are

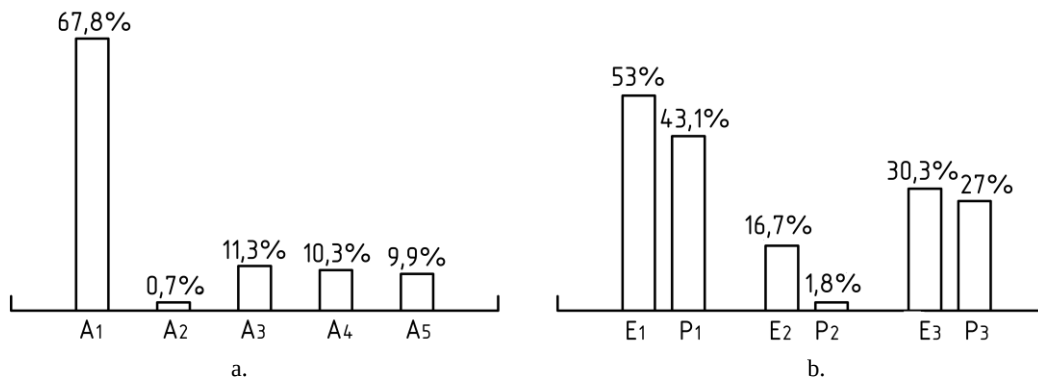


Fig.3. Heat emission diagrams (a) and energy heat input (b) in the zone of contact of the probe with the test surface. Heat dissipation: A_1 – on the mechanical friction of the probe on the test surface; A_2 – on the action of electromagnetic fields and electric charge; A_3 – on the bending of cantilever; A_4 – on the torsion of cantilever; A_5 – on the torsion of piezoscanner. Thermal energy contribution: E_1 – the energy that drives the piezoscanner; P_1 – useful work piezoscanner; E_2 – energy that is fed to the cantilever with the probe; P_2 – useful work of the sensing element; E_3 – laser positioner radiation energy; P_3 – laser energy that hits a photo detector.

As can be seen from the diagrams above, more heat generation in the contact zone arises due to friction of the probe on the test surface and is 67,8% (Fig. 3, a), at the same time, the maximum thermal contribution comes from the energy supplied to the piezoscanner (Fig. 3, b).

Thus, the use of the method of equivalent thermal circuits for evaluating mathematical models of the energy interaction of AFM probes with material surfaces, as well as further research on the distribution of thermal fields in the AFM control unit, differs from other numerous and analytical methods by sufficient accuracy and speed of calculations. Today, due to the lack of experimental methods for confirming the adequacy of mathematical models of thermal conductivity in the zone of interaction between the probe and the surface under study, this method is an alternative method for confirming the results obtained during the simulation. At the same time, the discrepancy between the results of mathematical

thermal models of heating individual elements of the measuring unit of an atomic force microscope. The main factors determining the accuracy of the calculation using this equation are:

- accuracy of setting sources of heat, i.e. losses;
- the accuracy of determining thermal conductivities, which in turn depend on:

a) thermal conductivity coefficients λ , which are prone to significant variation for technological reasons, under the influence of the appearance of air gaps, etc.;

b) heat transfer coefficients α , since empirical formulas and graphs available for their definition cannot take into account all factors and conditions affecting.

As a result of the calculations carried out, using both mathematical models using the inverse Fourier transform method and the equivalent thermal circuit method, the energy heat exchange was estimated and the main heat losses in the contact zone of the probe of the atomic force microscope with the surface were determined [9, 10] (Fig.3).

modeling and the results obtained with an equivalent thermal circuit does not exceed 5 – 8%.

3. Conclusion

The modeling of the energy interaction of AFM probes with the surfaces of materials at the preparatory, final stages and the scanning stage of the investigated surface was carried out taking into account the energy components of the processes occurring in the AFM control unit by minimizing factors (heat dissipation due to friction of the probe on the surface Q_1) negatively affecting both the control results and the state of the probe and surface established modes of stable operation of the AFM.

Mathematical modeling of the heating process of individual elements of the AFM measuring unit, confirmed by solving a system of differential equations with an equivalent thermal circuit,

which made it possible to conclude that the main heat losses and energy impact in the zone of contact of the probe with the surface under study. Thus, it was found that the most heat release in the contact zone (about 68%) arises due to probe friction on the surface,

while the heat contribution (53%) comes from the energy supplied to the piezoscanner.

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PROPERTIES OF NANODIAMONDS OF INDUSTRIAL DETONATION SYNTHESIS

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Abstract: Detonation synthesis nanodiamonds have been widely used in modern science and technology. Such kind of nanodiamonds can be obtained either from the carbon explosive molecule (DND) or from a mixture of explosives with the addition of graphite or soot (DALAN). Possessing nanoscale and high surface energy, diamonds have a structural and dispersion-strengthening effect being in contact with any materials. In many ways, the applications of nanodiamonds are determined by their dispersion, reactivity, and aggregative state in various environments. The given scientific research investigated the properties of industrial DND and DALAN produced by JSC RPE "SIDAL", being one of the leading manufacturers in Russia. There were estimated the following properties: crystallites sizes; elemental composition; thermal stability; specific surface area; unit sizes.

Keywords: NANO DIAMONDS, DETONATION SYNTHESIS, REACTIVITY, CRYSTALLITES, DISPERSION, UNITS

1. Introduction

Artificial diamonds are considered to be strategic materials all over the world, as they play a vital role in the development of the industry. With the establishment of a new detonation method for the synthesis of diamonds, there appeared fundamentally new opportunities for the implementation of advanced technologies [1,2]. The synthesis is carried out by detonation of explosives in an explosion chamber, while nanocarbon and nanodiamonds are formed in condensed detonation products with a high mass yield. Detonation synthesis is a fundamentally new and productive type of basic technology for producing nanostates and nanomaterials. Nanodiamonds (ND) of detonation synthesis is a unique material that combines the properties of diamonds and the advantages of nanostructures. Industrial development of the given method made it possible to actually reach large-volume production and consumption of ND in a number of industries [3,4]. ND is currently applied as anti-friction additives to motor, industrial oils and greases; in pastes and suspensions for super finishing material polishing; in wear-resistant electrochemical and chemical metal-diamond coatings; as dispersion-strengthening additives in composite materials based on polymers, metals, alloys and rubbers; as effective sorbents, catalyst carriers, biomarkers, transporter of medicinal substances, etc [5].

Science and technology are prone to use detonation synthesis, obtained both from carbon explosive molecules (DND) and from a mixture of explosives with the addition of graphite or soot (DALAN). In the latter case, they are larger.

The applications of ND are mainly determined by the dispersion of crystallites and particles, the reactivity and the aggregative state of ND powders in various environments. The given scientific research investigated the properties of industrial DND and DALAN produced by JSC RPE "SIDAL", being one of the leading manufacturers in Russia.

2. Preconditions and Means for Resolving the Problem

Detonation synthesis of DND and DALAN was carried out in industrial explosion chambers with a volume of 4 m³ in a buffer cooling environment out of their own detonation products. In order to obtain DND there were used cast charges from TNT – RDX alloys in a ratio of 40/60 by weight, respectively; as for DALAN there were used pressed charges of hexogen and graphite in a ratio of 80/20 by weight, respectively. The mass of the explosive charge was 0.5-2.0kg. The following DND and DALAN properties were evaluated: elemental composition; crystallite sizes and specific surface; thermal stability; particle size distribution according to the unit sizes in powders and water environment. The elemental composition (C, N, H, O) was determined on an elemental analyzer FlashEATM 1112 manufactured by ThermoQuest at the

incineration temperature of 900°C. The crystal structure and crystallite sizes of the samples were determined by means of wide-angle X-ray dispersion on a Rigaku Ultima IV X-ray diffractometer. There was used Cu radiation at a shooting speed of 1 deg/min, at a step of 0.01 deg and scanning angles of 5-75 deg. Processing of the obtained diffraction patterns was carried out by using the PDXL software package, the phase composition was determined by using the ICDDPDF2 database.

The specific area of the surface (S_{BET}) was determined by the BET adsorption method in a stream of nitrogen at the "ASAP-2000" automatic microvolume vacuum unit, Micromeritics.

The reactivity of the samples was determined by heating up to 800°C in the atmospheric environment with a heating rate of 10°C per minute by the method of simultaneous thermal analysis of DTA / TGA on a Shimadzu-60 device. There were estimated the initial temperature of intense mass loss, the temperature of the maximum and the thermal effect of samples combustion.

The size distribution of particles was measured in an Olympus OMEC DC130 microscope. The data were analyzed using OLYMPUS Particle Image Processor (PIP 9.0) software. In aqueous suspensions with the usage of ultrasound and without it the distribution was determined by the method of a laser diffraction on Horiba LA-950 laser analyzer. The frequency of ultrasound was 20 kHz.

3. Results and Discussion

Due to the fact that NDs are formed in fractions of microseconds under strongly different conditions, they possess a number of properties specific to nanomaterials. As for nanoparticles, the surface in terms of their volume is significantly larger in comparison with large crystals, that is why there can be expected the physicochemical properties of the surface layers. According to IR spectroscopy and polarography, the surface of ND is saturated with hydrocarbons, functional groups, and carbon structures with which these groups are directly connected. They contain hydroxyl, carboxyl and carbonyl groups, as a result of which the carbon content in the products decreases [6]. The results of studies of DND and DALAN properties are shown in table 1.

According to elemental analysis, ND powders contain C, H, N, O with a basic substance content of 86 ... 95% and an ash content of not more than 0.3%. The carbon content in DND samples is noticeably lower than for DALAN, the content of hydrogen, nitrogen, and oxygen is higher. It should be noted that there is no nitrogen in DALAN and this may be due to different kinetic conditions for the nucleation and growth of ND crystallites in the detonation wave. The crystallite sizes (the region of coherent scattering of the lattice) for DALAN are 1.5 times larger than for DND, while S_{BET} differs dramatically. DND particles are single-crystal formations, since the crystallite sizes coincide with the sizes

of particles, calculated by S_{BET} . A slight difference in values can be explained by the presence of distortions and defects inside the grain. This increases the width of the diffraction lines and decreases the crystallite size. DALAN particles are polycrystalline formations collected at the stage of synthesis from 6.8-7.4 nm crystallites into 58-89 nm particles.

Table 1: Main Properties of DND and DALAN

TITLE	DND	DALAN
Crystallites size [nm]	4,7-5,2	6,8-7,4
Specific surface (S_{BET}) [m^2/g]	220-246	21-32
Particle size (calculation) [nm]	7,6-8,5	58-89
Elemental composition [%]:		
C	86-88	93-95
H	0,8-1,2	0,3-0,9
N	1,7-2,1	0,0
O	9,7-10,3	3,7-4,6
ash content	0,1-0,3	0,1-0,3
Density [g/cm^3]:		
bulk	0,21-0,31	0,78-0,85
pycnometric density	3,05-3,21	3,35-3,48
temperature oxidation [$^{\circ}C$]:		
initial	540-580	630-650
maximum	590-614	693-714
Heat of combustion [kJ/g]	13,7-26,7	13,0-17,1
Unit diameter in powder [mkm]:		
mass average	2,5-3,3	2,6-3,2
median	2,2-5,6	2,5-3,2
Unit diameter in suspension [mkm]:		
average	35,2-63,4	14,1-21,2
median	21,3-48,3	9,1-11,3
Unit diameter in suspension after ultra sound processing [mkm]:		
average	2,3-4,7	3,4-6,1
median	1,8-5,6	3,0-6,1

The presence of a large number of uncompensated bonds on the surface of the particles leads to the appearance of "stored energy" and an increase in reactivity. The oxidation of DND and DALAN in air begins at 540-630 $^{\circ}C$, and occurs intensively at 590-714 $^{\circ}C$. For catalytic diamonds of static synthesis, the temperature of the oxidation onset is 670-850 $^{\circ}C$. Judging by the temperatures of the onset and maximum oxidation, the reactivity for DND is higher than for DALAN; in general, the heat of combustion is also higher. It is known that the formation enthalpy for crystalline diamond is 158 KJ/kg, for ND is in the range of 2560-2950 KJ/kg and may be higher according to calculations [3,6,7]. In our case, the heat of combustion for DND and DALAN reaches 26 and 17 KJ/g, respectively.

It should be noted that for DND and DALAN in the region of 300 $^{\circ}C$, a significant thermal effect is almost always observed on the DTA curve, which is comparable to a number of samples with the heat of combustion. Moreover, no mass loss is observed on the TGA curve (fig. 1,2). This behavior can be explained by the presence of excess surface energy, the destruction and oxidation of the surface layer.

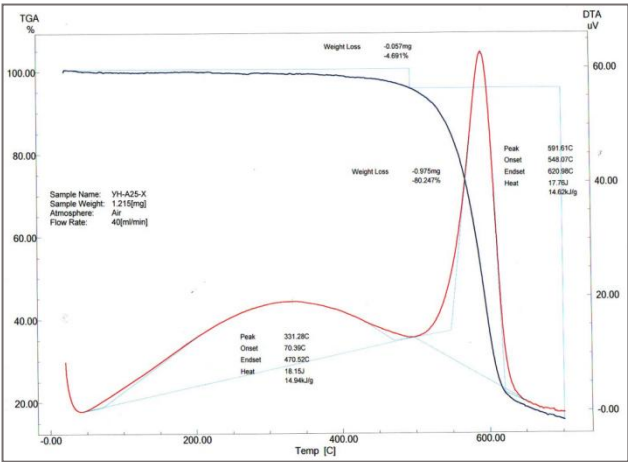


Fig.1 DTA and TGA curves for DND

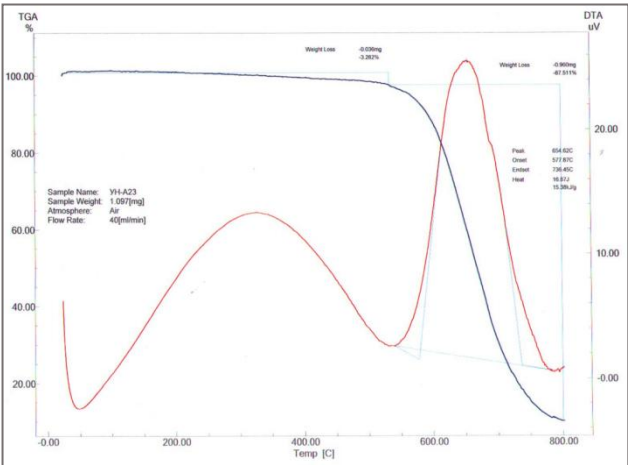


Fig.2 DTA and TGA Curves for DALAN

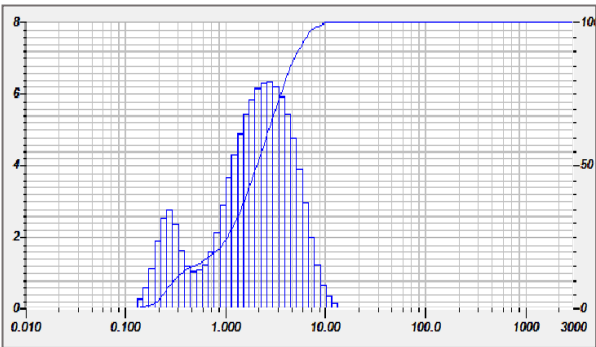


Fig.3 DND particle size distribution

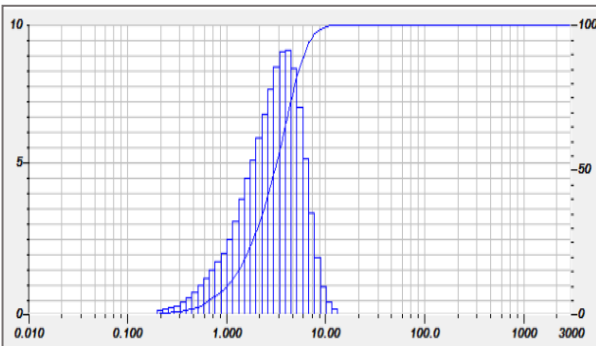


Fig.4 DALAN particle size distribution

Unsaturated surface bonds and surface energies lead to ND aggregation, forming a consistent hierarchical system of primary, secondary, and further units. The friability of units increases as they become larger while the density decreases.

The charging of DND and DALAN powders measured on a helium auto Picnometer is significantly lower than the X-ray density. In DND and DALAN powders, previously ground in an agate mortar, the unit sizes are approximately equal. In suspensions, without exposure to ultrasound, there are formed larger units. On the differential curves there observed two- and for DNDs also a three-modal size distribution of units. With ultrasonic processing, the dimensions of the units reduce and after 120 seconds they practically do not change. The unit sizes for DALAN and DND become comparable, however, for DND, the two-modal distribution of particles is sometimes preserved on the differential curve (fig. 3,4).

4. Conclusion

Studies have shown that the main difference between DND and DALAN is the crystal structure and particle size. DALAN has a polycrystalline structure with a particle size of 58-89nm, DND is a single-crystal structure with a particle size of 7.1-8.5nm. This explains other differences: carbon content; reactivity; energy saturation and propensity to form the units. The elemental and phase composition of nanomaterials is reproducible and stable. Crystallite sizes and granularity, energy intensity within the detonation production method can be controlled. As industrial nanomaterials, DND and DALAN are promising for use in materials science and nanoindustry.

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PECULIARITIES OF THE INTERACTION OF A HIGHLY CARBONIZED FERROCHROME WITH IRON DURING SINTERING OF 65% Fe-35% FH800 COMPOSITE

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Abstract: The influence of temperature of sintering on structure formation, phase composition, microhardness of components of powder composite 65 % wt. Fe – 35 % wt. FH800 were investigated. It has been established that the increase in the temperature of sintering from 1050 °C to 1250 °C leads to some increase in volumetric shrinkage, density and decrease in porosity of samples of material which was made from coarse-grained source powders of industrial production components. It was found that the sintering of green compacts in the range of 1000-1300 °C causes significant changes in the chemical and phase composition of the carbide component of the composite, which are described by a series of phase transformations: $M_7C_3 \rightarrow M_3C$ (1000-1150 °C) $\rightarrow M_7C_3$ (1200 °C) $\rightarrow M_3C$ (1250-1300 °C)

Keywords: powder materials, composite, sintering, FH800, microhardness.

Powder metal matrix materials of the class of carbide steels «iron-high carbon ferrochrome FH800» are promising wear-corrosion-resistant composites that can be successfully used for the manufacture of products operated under conditions of simultaneous action of friction forces, abrasive wear and corrosion-active media [1].

The complex of properties of such materials is determined not only by the composition and the ratio of the initial components, but also to a large extent by the additional formation at the sintering and interaction of the solid and metal components of the new phases. Obviously, such interaction inevitably leads to a redistribution of elements of the charge components between the solid and metal phases of the composite. Depending on the sintering temperature, the iron as a result of the interaction with the carbide component of the ferrochromium can form double chromosome carbides of different chemical compositions. Since the main solid component of the high-carbon ferrochrome FH800 is a complex carbide of (Cr, Fe)₇C₃ type so such an interaction reduces to its dissolution in the iron matrix of the composite. Information on the mechanism of dissolution (Cr, Fe)₇C₃ in iron and iron carbon alloys is very limited, and about sintering is practically absent. It was established [2,3] that with the increase in the temperature of the iron-carbon (2.9-3.3% C) melt, the dissolution rate of medium carbon ferrochrome (65% Cr, 3% C) increases from 0.41 at 1570 K to 7.152 kg/m²*s at 1720 K. The solubility parameter at temperatures close to solidus of ferrochrome (1570 K) approaches the magnitude of the diffusion coefficient of chromium in an iron-carbon melt, indicating that the diffusion mechanism is close to that of the dissolution.

The authors of the works [4,5] investigated the possibility of application for the creation of a powder-bearing material on the basis of iron carbonate ferrochromium FH650. It is established that the mechanism of dissolution of ferrochrome is similar to the mechanism of dissolution of orthorhombic chromium Cr₃C₂ carbide, but the dissolution process begins at slightly lower temperatures (~ 1000 °C) and in the interval 1000-1100 °C proceeds faster than Cr₃C₂.

It was found out that for the hot stamping method of wear-resistant structural material with carbide inclusions in an iron matrix it is expedient to use a powder of ferrochrome which contains both shallow fractions (less than 50 microns) and fractions of a size of about 200 microns [4].

The purpose of the work is to study the influence of the sintering temperature on the formation of the structure, phase composition and microhardness of the carbide and metal components of the powder composite 65% by weight. Fe - 35% by weight. FH800.

Objects and research methods

As the starting components for the production of samples of composite 65 wt. % Fe - 35 wt.% FH800 was used iron powder (manufactured by the State Enterprise "Powder Metallurgy Plant" (Brovary, Ukraine)), PCG 3.260.28 and a powder of high carbon (8% C) ferrochromium FH800 which was obtained by grinding

lump ferroalloys to a particle size of 100-200 μm. The powder mixtures were prepared in a laboratory drum mixer (2 h) and wet milling in a ball mill for 70 h. Experimental samples were pressed on a hydraulic press at a pressure of 800 MPa. Sintering was carried out in a vacuum electric furnace at a dilution of $1.33 \cdot 10^{-2}$ Pa in the temperature range 1000-13000 °C with a step of 500 °C at an isothermal expiration of 30 minutes. Density and porosity of samples were determined by the calculation and hydrostatic method in accordance with ISO 27.38.2009. The microstructure and phase composition of sintered specimens were investigated in an optical (Olympus LX-70) scanning (TESCAN VEGA) electron microscope, an X-ray diffraction analysis on a DRON-3 diffractometer. Local microscopic analysis was performed on the MS-46 microprobe. The microhardness of structural components was determined on the device PMT-3 for a load of 0.49 N. The mechanical properties were investigated using the INST RON 8802 machine.

Results and discussion

Investigation of the influence of the temperature of sintering on volumetric shrinkage, porosity, density and microhardness of the structural components of the composite 65%Fe - 35% FH800 (by wt.) showed (table 1) that the increase in the sintering temperature from 1000 to 1300 °C leads to some increase in shrinkage, density and decrease in porosity of samples. It should be noted that the sintering at a temperature of 1000 °C instead of shrinkage led to an increase in the volume of samples (negative shrinkage). This may be due to the Frenkel effect of the I genus, which manifests itself in the difference in the coefficients of heterodiffusion of chromium and iron in the composite. Also, an increase in the volume of samples results in a decrease in the diffusion mobility of γ-Fe atoms compared to the mobility of α-Fe atoms at temperatures slightly higher than α → γ (910 °C).

Table 1 – Influence of sintering temperature on physical and technological properties of samples of composite 65% Fe - 35% FH800

T, °C	Material Properties				
	ΔV/V, %	Porosity, %	γ, g/cm ³	H, μ, GPa	
				Matrix	Carbide
20	-	21,50	5,86	1,19	13,47
1000	+11	21,15	5,89	1,37	11,6
1050	0	20,74	5,92	1,39	10,43
1100	0	21,01	5,90	2,06	7,34
1150	1,25	20,60	5,93	1,24	12,26
1200	1,30	20,45	5,94	5,67	9,88
1250	5,10	17,80	6,14	4,98	10,39
1300	4,8	19,50	5,98	4,97	12,90

As can be seen from Table 1, sintering (even at temperatures of 1250 and 1300 °C), does not provide for obtaining specimens of material with minimum porosity and maximum density. This is due to the coarse-grained powders of the initial components of the mixture. It was discovered that the increase in the temperature of sintering up to 13000 °C leads to the partial melting and distortion

of the shape of the samples, therefore, to obtain virtually unpolluted material, it is necessary to use more dispersed iron powders and high carbon ferrochromium. The effectiveness of this approach has been demonstrated by us earlier in the works [6,7].

The study of microstructure of 65% Fe - 35% FH800 composites shows that small particles of high-carbon ferrochrome, already in the formation stage, are located on the boundaries between particles of iron powder, forming a kind of discrete shell around them (Fig.1).

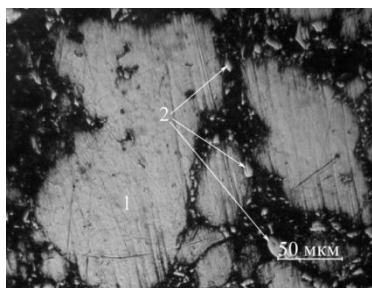


Fig.1 The fragment of the microstructure and the local X-ray microanalysis points are indicated by the arrows) pressing of the 65% Fe-35% FH800 composite

The X-ray microanalysis of the pressing components showed that, as expected, the middle of particles of iron powder contains (% at.): 99.48 Fe, 0.44 Cr, 0.07 C (t.1), and the basis of small particles of FH800 is chromium-iron carbide (57.28 Cr, 12.62 Fe and 30.09 C), corresponding to the formula M_7C_3 . Sintering of green compacts, except that affecting the volume shrinkage, porosity, density and microhardness leads to significant changes in the chemical and phase composition of the carbide and, to a lesser extent, the metal composite component (Table 2).

Thus, sintering at 1000°C leads to the conversion of the output carbide M_7C_3 into a cementite carbide type M_3C , which is stored to a temperature of sintering 1150°C. With the subsequent increase in the sintering temperature to 1200°C, the M_3C carbide is initially converted into an intermediate carbide M_7C_3 , after which the latter at a sintering temperature of 1250, 1300 °C is transformed into a double carbide of M_3C_2 type with a maximum carbon content and a minimum content of chromium and iron.

Thus, the thermally activated process of carbide transformations in the system of 65% Fe-35% FH800 occurs by diffusion of chromium and carbon atoms from the M_7C_3 carbide in iron and the counter flow of iron atoms from the matrix in the FH800.

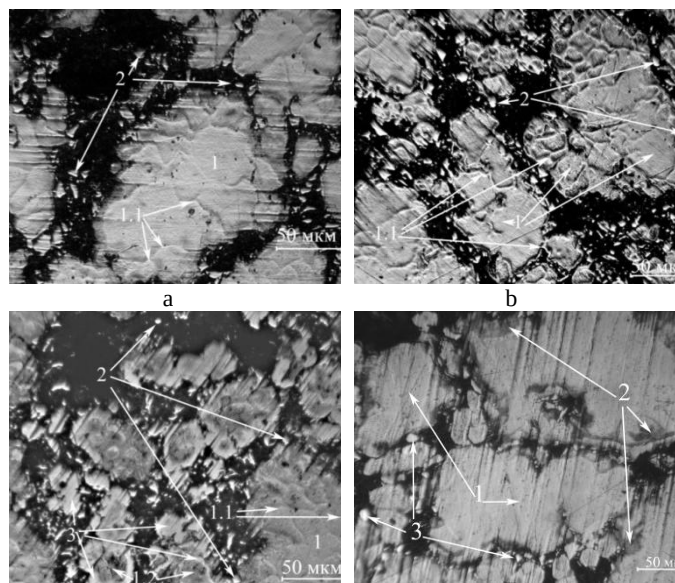
Summing up the above we can imagine the mechanism of carbide transformations of the studied system in the form of a chain $M_7C_3 \rightarrow M_3C$ (1000-1150 °C) $\rightarrow M_7C_3$ (1200 °C) $\rightarrow M_3C_2$ (1250-1300°C). The chemical and phase composition of the metal component with an increase in the temperature of sintering in the range of 1000-1300 °C practically does not change. Only a slight increase in Cr and C content (up to 0.87-0.94% to 2.11%, respectively) can be noted after sintering at 1250-1300 °C.

A detailed analysis of the microstructures and the chemical composition of the sintered samples of the composite shows that at the temperatures of sintering of 1000-1050 °C in separate large particles of iron the boundaries of grains on which diffusion concentration is concentrated (% at.) to 3.5% of chromium and about 1.14% carbon (Fig. 2 b, c). The increase in the temperature of sintering to 1100-1150°C causes the appearance of paraffin particles of the iron powder of chromium austenite edges (Fig. 2 g, d), which contains (% at.) to 1.11 Cr and about 2.27 C. The structure also stores a carbide phase in the form of M_3C (56.15 Cr -18.77 Fe-25.09 C). Burning at 1200-1250 °C leads to the appearance in the core particles of iron carbon, which amounts to 1.23(% at.) at 1200°C and increases to 2.11% at 1250°C. It should also be noted that light, smooth outer zones of iron particles contain (10,1-11,63% at.) Chromium and about 0,15-0,55(% at.) carbon at 1200°C, the M_3C carbide is transformed into a carbide of the type M_7C_3 (Fig. 2

is, g). In the middle of individual particles of iron powder at 1250°C, the amount of carbon reaches 8.38% at. And transitional eutectic zones based on iron are formed containing 3.28(% at.) Cr and 12.79 (% at.) C (t.5). The carbide phase of M_7C_3 undergoes transformation to M_3C_2 . Samples of the material which were sintered at 1300 °C consist mainly of two phases: metal, which in the form of light zones is formed in the form of particles of an iron powder and is an alloy of the type of chromium steel X15 (84,1% of Fe Fe - 15,11% of mass Cr – 0, 78% by weight C) and carbide, which is isolated on the boundaries of iron grains in the form of a complex carbide M_3C_2 . There is also the formation of separate eutectic zones based on iron containing (% at.) - 4.19 Cr and 4.98 C (Fig. 2).

Table 2 - Influence of sintering temperature on chemical and phase composition of metal and carbide components of composite 65% Fe - 35% FH800

T, °C	Content of elements in phases, % at., (% wt.)							
	Metal			Carbide				
	Fe	Cr	C	Cr	Fe	C	C _d /C _s	carbide type
20	99,4 (99,5)	0,45 (0,41)	0,07 (0,01 5)	57,2 (79,6)	12,6 (17,4)	30,5 (8,9)	0,4 3	M_7C_3
1000	99,0 (99,3 2)	0,66 (0,61)	0,27 (0,05)	58,3 9 (70,8 3)	17,1 (22,3)	24,4 (6,8)	0,3 2	M_3C
1050	99,1 (99,4)	0,59 (0,55)	0,22 (0,04 7)	56,6 0 (68,1 0)	19,5 (25,2)	23,8 (6,6)	0,3 1	M_3C
1100	98,9 (99,3)	0,64 (6,6)	0,36 (0,07)	56,0 7 (68,2 7)	18,8 (24,6)	25,0 (7,0)	0,3 3	M_3C
1150	99,1 (99,2)	0,78 (0,72)	0,06 (0,01)	56,1 (68,3)	18,7 (24,5)	25,1 (7,0)	0,3 3	M_3C
1200	99,2 (99,2)	0,55 (0,51)	1,24 (0,27)	53,0 9 (67,6 8)	17,2 1 (23,5 7)	29,6 (8,7)	0,4 2	M_7C_3
1250	96,9 (98,6)	0,94 (6,89)	2,11 (0,46)	44,6 8 (63,4 4)	15,3 (23,4)	39,9 (13,1)	0,6 6	M_3C_2
1300	98,7 (99,0)	0,87 (0,82)	0,39 (0,08)	45,0 6 (64,2 4)	14,7 (22,5)	40,2 (13,2)	0,6 6	M_3C_2



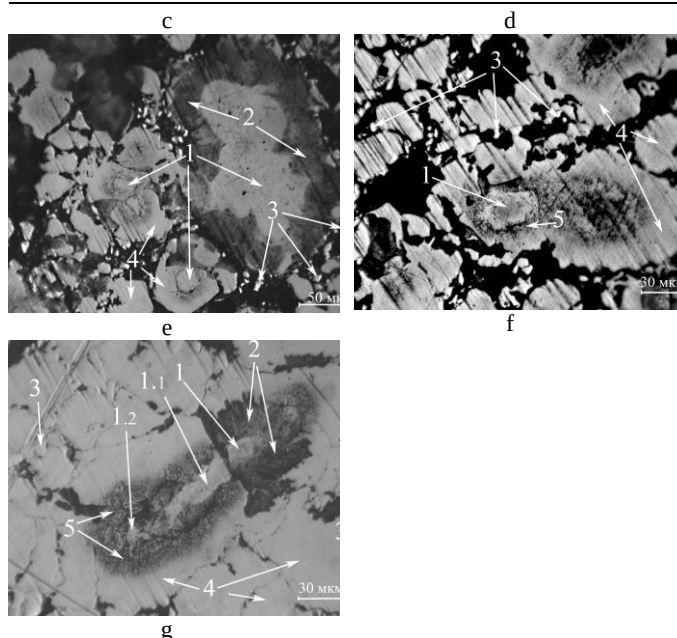


Fig.2 Fragments of microstructures and places of the local X-ray microanalysis points of 65% Fe composite - 35% of FH800, sintered at temperatures 1000-1300°C

Since it is known that mechanoactivation of powder mixtures leads to more intensive interaction of components, the effect of wet grinding in a ball mill for 70 hours on structural formation, the composition of the metal and carbide phases and the mechanical properties of 65% Fe - 35% FH800 composite were investigated. It was found that sintering of a fine-grained mixture at 1250 °C for 30 min. provides the formation of a practically unpolluted structure of samples of the material (Fig. 3) with high physical and mechanical properties (Table 3).

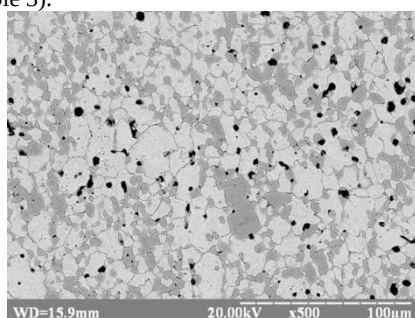


Fig. 3 Microstructure of composite of finely divided mixture 65% Fe - 35% FH800 sintered at 1250°C and carbon for 30 minutes

As can be seen from Fig. 3, the microstructure of the composite is heterogeneous and consists of 3 phases, light gray, dark gray and black.

Microrentgenospectral analysis showed that the light-absorbing phase contains, (% at.) 59.83 Cr - 25.10 C - 15.06 Fe and is a chrome-plated carbide of type M_3C . Dark soil consists of 81.22 (% at.) Fe - 18 (% at.) Cr - 0.77 (% at.) C, which, in terms of (% wt.) corresponds to chromium steel 17X17, and black represents pores.

The X-ray and phase analysis of the 1250°C sintered thin-layer composite composite showed that the X-ray spectrum fixes the most intense line belonging to a complex carbide of type M_7C_3 with a hexagonal lattice. Also observed in a small number of chromium carbide lines Cr_7C_3 and Cr_3C_2 , and austenitic phase γ -Fe with an elemental cell parameter of 0.3607 nm. thus, mechanical activation of a powder mixture of 65% Fe - 35% of FH800 and sintering at 1250 °C (30 min.) Ensures the production of chromium steel 17X17- (Cr, Fe) $_3$ C composite close to the equivalent composition. The study of properties confirmed that the use of

finely divided powders is provided by the rare-phase sintering at 1250°C to obtain practically non-porous composite specimens with high mechanical characteristics (Table 3).

Table 3 - Properties of specimens of composites of finely divided mixtures of 65% Fe - 35% of FH800 sintered at 1250 °C (τ = 30 min.)

$\Delta V/V$, %	f, %	γ , g/cm ³	HRA	δ , MPa	Microhardness, GPa	
					Matrix	Carbide
35.9	1.5	7.44	74.5	1760-1900	5.32	12.5

Conclusions

1. The influence of temperature of sintering on structure formation, phase composition, microhardness of components of powder composite 65 % wt. Fe - 35 % wt. FH800 were investigated. It has been established that the increase in the temperature of sintering from 1050 °C to 1250 °C leads to some increase in volumetric shrinkage, density and decrease in porosity of samples of material which was made from coarse-grained source powders of industrial production components.

2. It was found that the sintering of green compacts in the range of 1000-1300 °C causes significant changes in the chemical and phase composition of the carbide component of the composite, which are described by a series of phase transformations: $M_7C_3 \rightarrow M_3C$ (1000-1150 °C) $\rightarrow M_7C_3$ (1200 °C) $\rightarrow M_3C$ (1250-1300 °C)

3. The mechanical activation of the initial powdered charge by wet milling in a ball mill provides the method of liquid phase sintering at 1250°C of practically unpolluted composite type of chromium steel 17X17 - (Cr, Fe) $_3$ C equivalent (close range 50:50) composition with a density of 7.44 g/cm³ with a hardness of 74.5 HRA and a bending strength of 1760-1900 MPa.

4. The use of a fine powder (<50 microns) of high-carbon ferrochrome FH800 as a powder mixture can be used to obtain a chromium-plated steel of type 17X17 and a chromium-molybdenum carbide of the type M_3C through the sintering of liquid fossils at 1250°C.

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COMPUTER MODELING OF INFLUENCE OF PREVIOUS DEFORMATION DEGREE AND STRAIN RATE ON CARBONITRIDES PRECIPITATION KINETICS IN LOW-CARBON MICRO-ALLOYED STEEL

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Abstract: Using computer modelling with originally developed semi empirical physical grounded models a study was carried out to investigate influence of previous hot deformation on carbonitrides formation from austenite in low-carbon micro-alloyed steel. Studied in the article is an influence of degree and rate of the deformation on processes of nucleation and growth of Nb and Ti carbonitride particles. The model helps to predict not only process of changing in number and average size of the particles but also to estimate their final size distribution. One of additional peculiarities of the developed model is its ability to predict composition of the carbonitrides formed in certain conditions. The model takes into account process of recrystallization and returning, which affect carbonitrides precipitation and are influenced by it. Acceleration was shown of both nucleation and growth rates of the particles due to increasing of deformation degree and strain rate. Another result is that previous deformation significantly affects size distribution function of the particles precipitated with rather lesser effect on their average size. Kinetic curves and final size distribution plots are given.

KEYWORDS: STEEL, CARBONITRIDES, MODELLING, DEFORMATION

1. Introduction

The objective of the research is to demonstrate the developed model ability in prediction of processes of carbonitrides precipitation and show an influence of deformation on the particles size distribution.

Precipitation from solid solution (austenite) of carbonitride phases occurs in steels micro alloyed with Ti, Nb, V and Zr. Stimulating for the precipitation is previous deformation, which leads to increase in inner stress and dislocation density of the material. But owing to high temperature of the processes there have a place phenomena of returning and recrystallization [1].

A mathematical model and sufficient computational program was developed to describe kinetics of carbonitrides precipitation from austenite after deformation. The model takes into account returning and recrystallization their influence on carbonitrides nucleation and vice versa. Detailed description of the model can be found in our previous work [2], some information is also in our book [3] (in chapter 1). Here we do not consider on the essence of the model and its realization. It is a semiempirical but physical based model. Finite differences method was used for realization of the processes calculation.

Successful enough preempts were made before to describe theoretically kinetics of recrystallization and carbonitrides precipitation in deformed steel [4-6]. Even a program was developed [4], but it is not an open source. So program code realization for the model implementation was made originally by us. A remarkable feature of our model, qualitatively distinguishing it from the above one is including of thermodynamic calculation of the carbonitrides composition. For instance in the work [4] they consider carbonitrides as stoichiometric composition, which is a rather rough approximation and does not correspond to the composition of real inclusions. As a bases of thermodynamic module for carbonitrides composition calculation was taken our previously developed program. It was completely rewritten using Python language (original was in Delphi), program code optimization were made and precision increased (the nonideality of carbonitride as a mutual solid solution of the corresponding carbide and nitride is better taken into account). Another feature of current investigation is consideration on size distribution of the carbonitrides precipitated. In particular the influence of deformation degree and strain rate on the particles size distribution was studied using the model developed. In other papers known to us, the average indicators were modeled and investigated, but not the distribution functions. There are many others lesser particularities our model consisting in a more accurate consideration of many quantities and their dependencies but their description will lead to detail that make it difficult to understand.

2.1. Studied material

Chemical composition of the studied steel in this work is given in table 1.

Table 1

Chemical composition of the studied steel								
Fe	C	Mn	Si	Mo	Ti	V	Nb	N
base	0.10	2.00	0.25	0.15	0.015	0.02	0.045	0.006

The given composition is quit similar to one from our work [2] but amounts of Mn and Mo are higher and concentration of carbon is lesser. It's seen that the steel is low-carbon one and has rather high amount of Mn. It is micro alloyed with strong carbide and nitride forming elements, which are Ti, Nb and V. It is a typical strip steel.

2.2. Calculation and its results

The process modelling was carried out for four regimes of deformations that differ by deformation degree and strain rate. Values of the deformation degree and strain rate are given below in table 2.

Table 2

Regimes of deformation		
No.	Deformation degree	Strain rate, sec ⁻¹
1	0.10	0.5
2	0.10	3.5
3	0.30	0.5
4	0.30	3.5

Deformation temperature, which also was considered as the temperature of precipitation was 950 °C for all the regimes modelled.

Computation shows that only carbonitrides of Ti and Nb can form at the temperature. Carbides and nitrides of V have significantly lesser temperature of precipitation, so they do not occur under the considered conditions. According to thermodynamic calculations compositions of the carbonitrides formed are as follows: $\text{TiC}_{0.58}\text{N}_{0.42}$ and $\text{NbC}_{0.76}\text{N}_{0.24}$.

Primary studied were processes of the particles nucleation and growth. Figure 1 shows increasing of number of the particles by time flowing for Ti and Nb carbonitrides depending on the deformation regime.

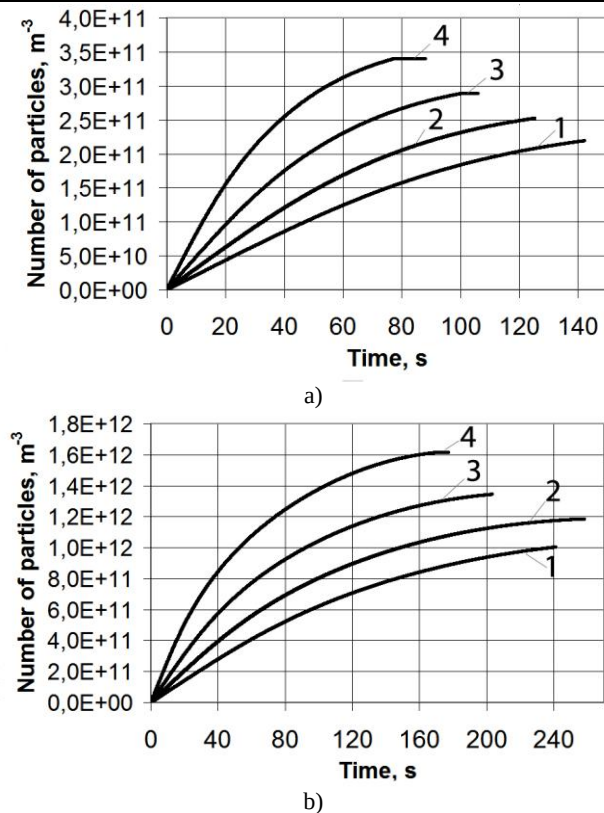


Figure 1 – Changing by time of number Ti(CN) – (a) and Nb(CN) – (b) particles depending on deformation regime. Numbers near curves correspond to the regimes listed in table 1

Increasing in number of particles occurs owing to nucleation process. When nucleation stops, the increasing stops too. According to the theory accepted in the model, the sites of nucleation of particles are the intersection sites of dislocations. So increasing of dislocation density during deformation leads to more places of nucleation. Besides increasing of precipitated particles number intensification of deformation also leads to acceleration of nucleation – the process goes faster. Significantly more particles of Nb(CN) precipitate than particles of Ti(CN), which is mostly owing to higher concentration of Nb compeering with Ti in initial chemical composition of the steel. Increasing both of deformation degree and strain rate has significant and similar influence. This distinguishes current situation from that considered in paper [7], where the influence of the strain rate on the kinetics of phase transformations was significantly less than the effect on the deformation degree on it.

In addition to the nucleation process, this model also describes the growth of carbonitride particles. Figure 2 demonstrates changing of the particles average size by the time flowing.

On figure 1 (a) – for Ti(CN) a strange effect is seen. In cases of higher deformation degree, at a certain time the average size of the particles begins to decrease. It is important to note that it's about average size only. Size of each particle grows. This effect is because of new particles nucleation during all the period. The larger a particle is the slower it grows. Intensive deformation makes better conditions for the emergence of a larger number of particles. Hence a situation is possible when originated at an early stage particles are already grown enough to slower their following growth but new ones are keeping appear. This is an explanation of the observed effect of decreasing of average size when all the particles grow. The same effect but significantly lesser is seen for Nb(CN) also that is shown on figure 2 (b).

Another interesting result is that despite the average size growth acceleration intensification of deformation does not affect significantly its final value.

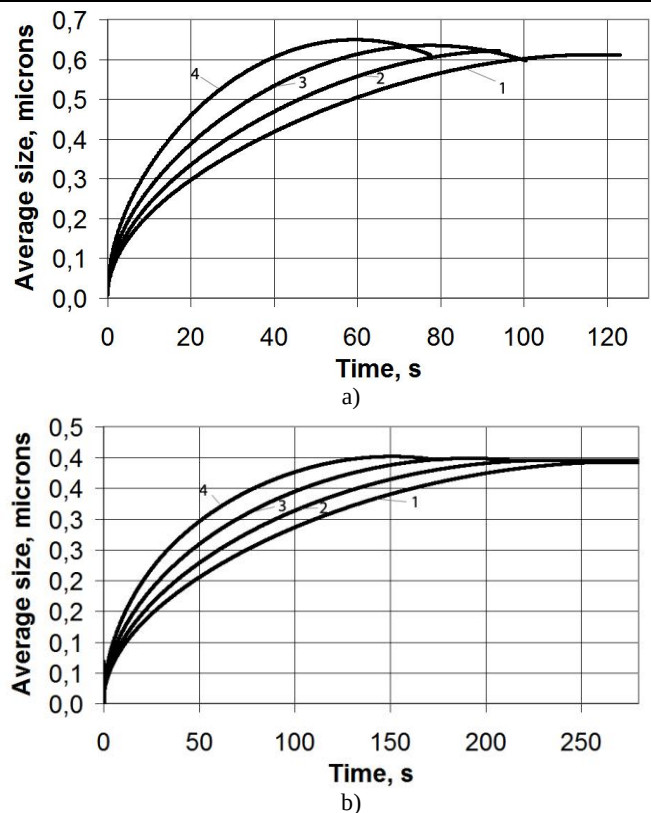


Figure 2 – Changing by time of average size of Ti(CN) – (a) and Nb(CN) – (b) particles depending on deformation regime. Numbers near curves correspond to the regimes listed in table 1

Below in table 3 are given predicted values of final average sizes of the particles obtained at each of the considered deformation regimes.

Table 3

Final average sizes of particles (in nm)

Regime No.	Ti(CN)	Nb(CN)
1	626	401
2	614	397
3	607	391
4	594	382

In spite of a little difference in an average size the particles number differs significantly. This effect has the following explanation. Deformation affects more the particle size distribution function than their average size. Plots of the final size distribution density functions predicted using the model are shown on figure 3.

The distribution function is lognormal for all of the cases. More significant influence of deformation regime on size distribution function is observed for Nb(CN) particles. With a lower intensity and degree of deformation, the particle size distribution function looks more smeared. That is, both smaller and larger particles will equally be present in the metal. An increase in the degree and rate of deformation leads to a narrowing of the distribution function and a smaller scatter of medium size. A large role in the narrowing of the distribution function plays an increasing of deformation degree than strain rate, which effect is additional. Deformation effect on distribution function for Ti(CN) particles is lesser but it is also observed. It is noteworthy that in terms of size distribution change for Ti(CN) in this case the effects of strain rate increasing with lower deformation degree is almost to deformation degree magnification but with lesser strain rate.

Thus, the model shows that an increase in the intensity of deformation leads not only to an increase in the number of carbonitride particles, but also narrows the range of variation of their sizes.

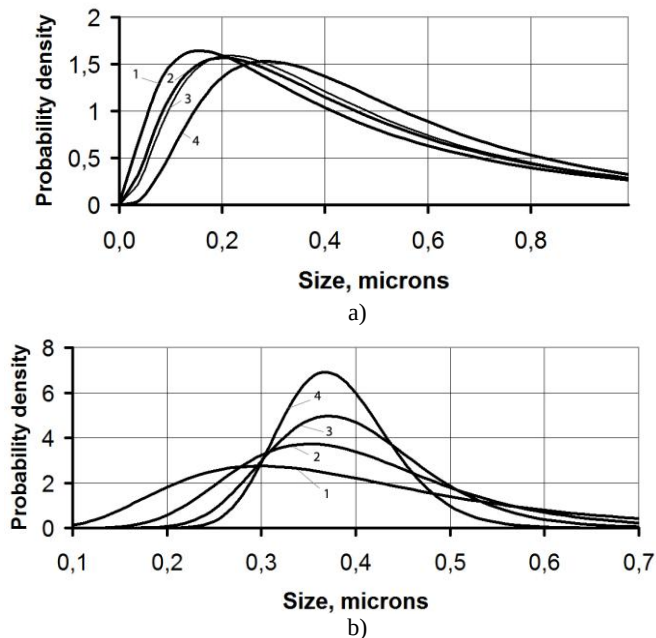


Figure 2 – Final size distribution function of Ti(CN) – (a) and Nb(CN) – (b) particles depending on deformation regime. Numbers near curves correspond to the regimes listed in table 1

The normal growth of particles at a given temperature stops when the system reaches equilibrium between the carbonitrides and solid solution. That is, simply put, when all that could have precipitated was already precipitated. After that the process of Ostwald ripening begins. It is when particles with smaller size become even smaller and then disappear and larger particles grow taking on material from smaller ones. Ostwald ripening stage of process is also included in the developed model but is out of consideration in current article. It is only to be said that this process is much slower than the nucleation and growth considered above.

3. Conclusions

Using a developed computer model a study was carried out to estimate influence of deformation on kinetics of Ti and Nb carbonitrides precipitation from austenite in low-carbon micro alloyed steel. An influence of the degree and rate of deformation on the number of carbonitride particles, the rate of their nucleation at a temperature of 950 °C was studied.

Composition of Ti and Nb carbonitrides was predicted for the invalidated conditions, it is $\text{TiC}_{0.58}\text{N}_{0.42}$ and $\text{NbC}_{0.76}\text{N}_{0.24}$. Information of chemical composition of carbonitrides is useful to conduct more precise kinetics computation.

It was shown that in spite of small difference in average particles size their amount differs significantly depending on deformation regime. Found here was that degree and rate of deformation affects significantly an obtained size distribution function of the particles precipitated. Especially increasing of both deformation degree and strain rate leads to the dispersion of the distribution becomes smaller.

The obtained results present in the paper are useful for developing of controlled rolling regimes for low-carbon steels micro alloyed with strong carbide and nitride forming elements.

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FEATURES OF STRUCTURE, PHASE COMPOSITION AND PROPERTIES OF HOT-FORGED HIGH-ENTROPY ALLOYS OF Ti-Cr-Fe-Ni-C SYSTEM

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Abstract. Powder high-entropy alloys (HEA) of TiCrFeNiC equiatomic composition were synthesized by hot forging (HF). The phase composition and parameters of the fine structure of the alloys are determined. It is shown that at all annealing temperatures of the alloys their phase composition does not change significantly and consists of two solid solutions of substitution - FCC and BCC and two carbide phases - TiC and Cr₃C₂. The mechanical properties of the alloys are at a rather high level - so the maximum strength of the alloy was 2243 MPa and the hardness is more than 62 HRC, which can be explained by the effect of high entropy and in situ synthesis of carbides in the manufacture of alloys.

Keywords: powder high-entropy alloy, phase composition, microstructure, hot forging, hardness, compressive strength

1. Introduction

A characteristic feature of high-entropy alloys is the content in their composition of at least 5 basic elements in the equiatomic ratio. The presence of a large number of heterogeneous but equal number of atoms with different individual properties imposes its specificity on the formation of solid solutions in these alloys [3]. The high entropy of mixing elements in the alloys causes the Gibbs free energy to be minimized and leads to the predominant formation of solid solutions with BCC, FCC or FCC + BCC structure. Reduced free energy of the alloy provides the stability of the solid solution during further heat treatment [1, 2]. Increased characteristics at high temperatures are ensured by the strong distortion of the crystal lattice due to differences in the atomic radii of the substitution elements [3, 4].

The purpose of the study is to study the phase composition, microstructure, structural imperfections of the crystal lattice and properties of high-entropy TiCrFeNiC powder alloys in the process of their synthesis.

2. Experimental materials and methods

The microstructure of the alloys was studied on an XJL-17 optical microscope as well as on a JEOL Superprobe 733 scanning electron microscope. Investigations of phase composition and structural component defects were performed by X-rays methods. X-rays of the samples were carried out on a DRON-3 diffractometer in filtered cobalt radiation by a stepwise scanning method in the range of angles $20 \div 130^\circ$. The scanning step was 0.05 deg, the angular velocity of the goniometer was $\frac{1}{4}$ deg/min. Quantitative micro-X-ray spectral analysis was performed on a CAMECA MS-46 x-ray microprobe at 20 kV, 12 nA probe mode and 3 μ m probe diameter.

Fe, Cr, Ti, Ni and C (graphite) powders with a purity of 99.5 - 99.9% were used as starting elements for obtaining equiatomic alloys of the Ti-Cr-Fe-Ni-C system. The original powders were dosed on an electronic scales. The mechanical activation of the powder mixture was carried out in a planetary mill at a ratio of the mass of the mixture to the mass of grinding bodies 1: 10. The speed of rotation of the drums of the mill was about 800 rpm, the grinding time - 60 minutes. In order to prevent oxidation and segregation of the powder particles, this process was carried out in ethyl alcohol. The drums were periodically cooled in water.

From the resulting mixture in a steel matrix pressed cylindrical blanks with a diameter of 40 mm at a pressure of 700 MPa. The consolidation of the powder blanks by hot forging was carried out on an forging press FB1732 at a temperature of 1100 °C in argon atmosphere. The annealing of the alloys after forging was carried out in an induction furnace in argon at temperatures of 1200, 1250 and 1300 °C. Annealing time is 2 hours. The density and porosity of the alloys were determined by hydrostatic weighing. The hardness was measured on a TK-14-250 hardness meter according to GOST 9013-75, and the micromechanical characteristics were measured on PMT-3. The strength characteristics were determined by the compression method according to GOST 25.503-97.

3. Results of the experiments and their discussion

Analysis of the X-ray spectrum of the mixture of TiCrFeNiC composition after grinding in a planetary mill for 60 min showed (Fig. 1) that it consists of the reflexes of the individual components that are part of the alloy being developed. Therefore, chemical interaction between the components does not occur, but the nature of the profile of the reflexes of the components of the alloy changes dramatically. Significant expansion of the reflexes indicates the distortion of the crystal lattice of elements, overlapping of x-ray reflections of elements having close angles of reflection, eliminates the possibility of profile selection for calculating the parameters of the fine structure. Defects in the crystal structure, intensify the sintering processes and greatly affect the peculiarities of the formation of the phase composition.

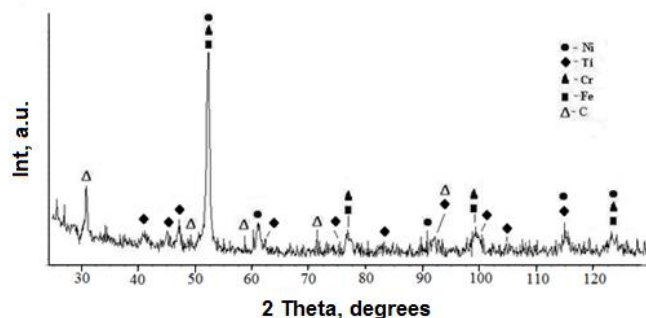


Fig. 1. Diffraction pattern of equiatomic TiCrFeNiC powder mixture after grinding for 60 min in a planetary mill

The X-ray spectrum of the TiCrFeNiC alloy synthesized by grinding the original charge for 60 min and subsequent hot forging at 1100 °C is mainly represented by the reflexes characterizing the FCC structure (Fig. 2 a). Significantly expanded lines of titanium carbide are clearly recorded on the X-ray of the alloy. The crystal lattice parameter of titanium carbide is 0,43267 nm, which corresponds to the ratio $C / Me = 0,82$. Many of the weak reflexes characteristic of this type of lattice are represented by Cr_3C_2 chromium carbide. Intermittent Cr_2Ti and Fe_2Ti interfaces with cubic and hexagonal structure are present in a small amount, respectively.

The use of hot forging after grinding showed a high defect of the solid solution based on the phase with the structure of the FCC: $\beta_{111} = 7,37$ mrad, $CSR = 24$ nm; $\beta_{311} = 21,79$ mrad, $\rho = 9,49 \cdot 10^{12} \text{ cm}^{-2}$, micro-distortion = $21,11 \cdot 10^{-2}$. In the course of hot deformation, at the same time as the introduction into the alloy of dislocations, their transformation into orderly groups always occurs. Hot stamping of porous blanks can be considered as one of the cycles of thermomechanical treatment, in the process of which a developed substructure is created. This developed substructure is the primary factor that determines all other structural causes of thermomechanical strengthening of the alloy and can contribute to the formation of solid solutions structures [5, 6].

On the X-ray spectrum of the alloy, no phase with the BCC structure is detected, but using the mathematical approach to decompose the maximum (111) of the FCC phase, the presence of two phases was found simultaneously (Fig. 1b). This can be explained by the significant slander of the BCC phase, which is associated with the blurring of the lines of this phase, which makes it impossible to distinguish from the low intensity line background.

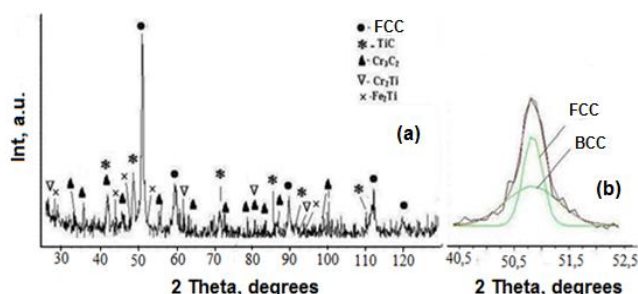


Fig. 2. Diffraction pattern of equiatomic TiCrFeNiC alloy after 60 min grinding and hot forging at 1100 °C (a) and the results of the decomposition of the FCC reflex (111) (b)

The performed calculations of the parameters of the fine structure according to the maxima of the component phases indicate

the significant imperfection of the crystal lattice of the structural components of the alloys.

At an annealing temperature of 1300 °C, the HEA is formed on the basis of a phase with the FCC structure. The FCC lattice parameter of 0,3597 nm is the largest compared to similar parameters in all technological modes. However, increasing the lattice parameter did not lead to an increase in hardness, which decreased to 49,1 HRC (Table 1). A similar feature of WEC alloys was observed by the authors of [4], explaining that one of the reasons may be the redistribution of the elements of substitution in the alloy with FCC structure. The defect in the HEA based on the FCC structure is characterized by the following values $\beta_{111} = 6,8$ mrad, $CSR = 25,8$ nm, $\beta_{311} = 17,76$ mrad, $\rho = 5,24 \cdot 10^{12} \text{ cm}^{-2}$, micro-distortion = $17,39 \cdot 10^{-2}$.

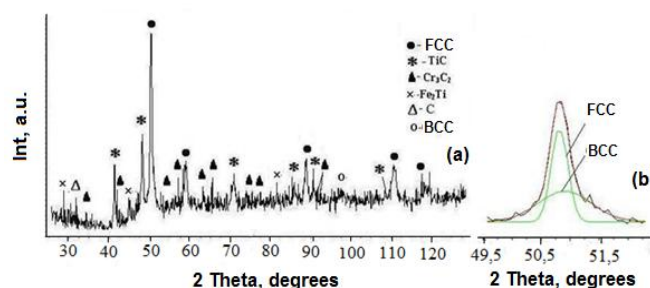


Fig. 3. Diffraction pattern of equiatomic TiCrFeNiC alloy after grinding, hot forging and annealing at 1300 °C (a) and the results of the decomposition of the FCC reflex (111) (b)

Structural formation during hot forging of porous powder blanks occurs under conditions of intense plastic deformation and accelerated cooling from contact with a relatively cold press tool, which provides a manifestation of the effect of thermomechanical treatment. The temperature and duration of heating before forging accelerate the flow of interparticle splicing processes when compacting the blanks and forming a finished product from it. In the case of HF, there is a partial annihilation of the dislocations acquired during the grinding process and the simultaneous introduction of dislocations. The heating temperature under forging ensures the alignment of the structure defect in the sample volume, the processes of structure formation are activated [7].

According to metallographic and micro-X-ray spectral analyzes, two solid-solution phases of substitution with FCC and BCC lattices and two carbide phases are present after annealing at all temperatures at TiCrFeNiC alloys (Figs. 4, 5). There is a layered structure, the layers of which are directed perpendicular to the direction of stamping, which is characteristic of hot-forged alloys. It is only after annealing at 1300 °C (Fig. 4, d) that a regular matrix structure is formed, in which a gray and light gray matrix and dark inclusions are present. The light area is a solid solution of iron with chromium in which there are gray inclusions of chromium carbide Cr_3C_2 . The dark gray area formed by the lattice of nickel contains the inclusion of titanium carbide TiC and a small fraction of iron and chromium (Fig. 5).

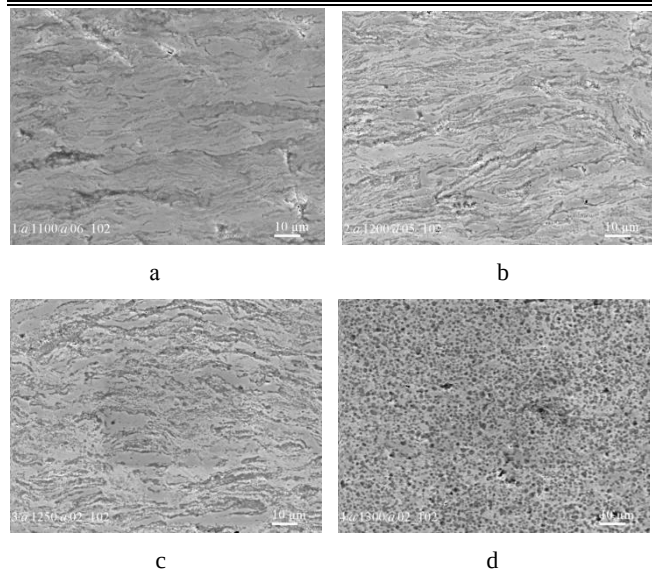


Fig. 4. SEM image of the microstructure of equiatomic TiCrFeNiC alloys after HF (a), after HF and annealing at 1200 °C (b), after HF and annealing at 1250 °C (c), after HF and annealing at 1300 °C (d)

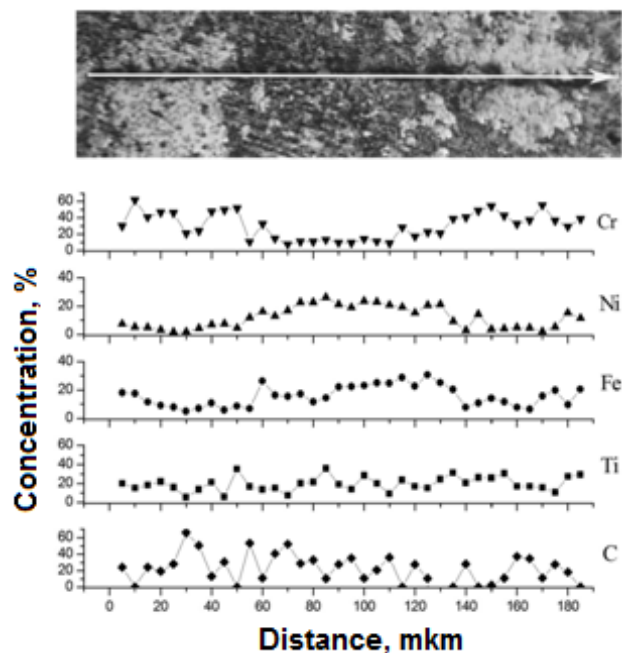


Fig. 5. The distribution of elements in the TiCrFeNiC alloy phases after annealing at 1300 °C

Therefore, a solid solution of substitution on the basis of the FCC structure is formed on the basis of the Nickel lattice. In the smaller volume of the bright region, the formation of a solid solution of substitution occurs when the mutual dissolution of chromium and iron with the formation of BCC structure on the basis of the chromium lattice. The data of the micro-X-ray spectral analysis confirm the results of the X-ray structural study on the formation of a high-entropy solid solution of substitution with the BCC structure.

In the table. 1 shows the density, porosity and hardness of the synthesized alloys. The theoretical density of the alloy is 6,65 g/cm³. The use of hot forging has yielded almost non-porous alloys, which is known to significantly affect their properties.

Table 1

Density, porosity and hardness of synthesized high-entropy TiCrFeNiC alloys

№	T _{annealing} , °C	Density, g/cm ³	Porosity, %	Hardness, HRC
1	without annealing	6,55	1,5	62,4
2	1200	6,51	2,1	58,2
3	1250	6,50	2,2	55,5
4	1300	6,48	2,5	49,1

Samples that were annealed have slightly higher porosity than non-annealed ones. This can be explained by the Frenkel II kind of effect due to the difference in the heterodiffusion coefficients of the alloy elements. The hardness of the samples decreases naturally with increasing annealing temperature, although this decrease is negligible due to the high entropy effect.

The results of tests for uniaxial compression are presented in table. 2. The high values of the mechanical characteristics of the alloys can be related to the effect of high entropy of element mixing in the alloys, as well as to the synthesis of carbides in situ during the process operations. The nature of the destruction of the samples is fragile, as evidenced by the low values of relative deformations. In the first three specimens, there is actually a microplastic deformation that does not exceed 0,7 %, and only in the last specimen does the plasticity increase significantly compared to other specimens. In general, the mechanical properties are clearly correlated with the annealing temperature of the alloys.

Table 2

Mechanical properties of synthesized high-entropy TiCrFeNiC alloys

№	T _{annealing} , °C	σ _{pr} , MPa	σ _{0,2} , MPa	σ _r , MPa	ε, %
1	without annealing	1381	1978	2243	0,41
2	1200	1081	1567	1971	0,65
3	1250	1055	1507	1663	0,69
4	1300	890	1266	1551	1,64

4. Conclusions

1. The synthesized high-entropy alloys of the TiCrFeNiC system are formed into solid solutions with FCC and BCC structures. The preferred phase is a solid solution based on the structure of the FCC.

2. Quantitative characteristics of the imperfections of the crystal lattice of HEAs indicate a significant distortion of the lattice of a solid solution with the BCC structure.

3. Along with the phases of the FCC and BCC structures, high-entropy chemical compounds are formed: TiC and Cr₃C₂ carbides, Fe₂Ti, Ni₃Ti, Cr₂Ti intermetallides, which play a large role in the solid solution formation on the basis of the FCC structure.

4. The high values of the mechanical characteristics of the alloys can be related to the effect of high entropy of element mixing in the alloys, as well as to the synthesis of carbides in situ during the process operations.

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MODELING OF THE METAL POWDER PRESSING PROCESS WITH A POROGEN

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Abstract: A model for calculating the metal powder pressing process with a porogen is proposed. An approximate method and algorithm have been developed that make it possible to take into account the presence of a porogen in the calculation of the pressing process and reflect all types of bonds at the contact boundaries of metal powder particles. Based on the constructed model, an approximate method and algorithm for calculating the properties of powder filter material (PFM) produced by pressing granular metal powders using a porogen have been developed. The advantage of the proposed mathematical model is the possibility of its development to describe the sintering process, as well as the possibility of its use for calculating the modified elementary cells used by researchers in modeling the structure of PFM with a porogen.

KEYWORDS: POWDER FILTER MATERIALS (PFMs), CALCULATION MODEL, METAL POWDER PRESSING, POROGEN

1. Introduction

Taking into account the influence of the use of porogens on expanding the range of PFM properties, modeling of a porous body with the presence of porogens is of interest. An analysis of the given models of the PFM structure shows that the model that most fully describing the structural features of the metal frame of the material and at the same time enabling to determine filtering properties is an elementary cell in which the properties of each element of the PFM volume are set by the elementary cell parameters in the form of a parallelepiped isolated from 8 powder particles. According to [1, 2], the presence of a porogen in this model is taken into account by shearing 1/4 of one of the faces in the direction of the corresponding axis of the nonorthogonal coordinate system (Figure 1).

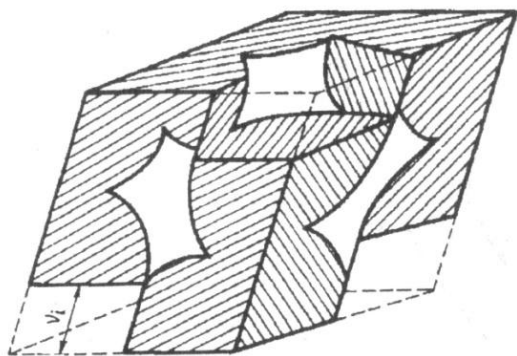


Figure 1 Scheme of powder particle displacement;
 v_i – Shearing area

The authors of Ref. [3] used the idealized (geometrically ordered) PFM structural model to define the relationship between various structural characteristics and determine the role of a porogen in the formation of a porous structure. It is assumed in a first approximation that the porogen particles, and therefore the cavities formed after their decomposition, have a spherical shape. These cavities in the finished material are uniformly distributed and form a simple cubic lattice with a step a . The gaps between the spherical cavities (the walls of these cavities) are formed by sintered metal powder and have a corresponding porosity, in the formation of which a porogen is not involved (the case is considered when the particle size of a porogen is much larger than the particles of the metal powder).

However, the first of given models does not take into account the particle size of a porogen, the second one – the particle size of the metal powder.

The purpose of this paper is to develop a mathematical model for calculating the PFM properties produced by pressing metal powders with a porogen.

2. Results and discussion

Let's consider the process of molding metal powders with porogen particles using mathematical modeling methods. This, in essence, means determining the properties and characteristics of the phenomenon or process by solving with a computer a system of certain equations – a mathematical model [4].

The main stages of the numerical solution of tasks in continuum mechanics are: 1) a choice of a model of a physical process; 2) a choice of a mathematical model or mathematical formulation of a task; 3) a choice and development of a numerical method for solving the corresponding mathematical problem; 4) development of a method for implementing an algorithm on a computer and conducting serial calculations, accumulation of experience, evaluation of the effectiveness and applicability limits of the algorithm.

In this paper, following the above stages, a mathematical model is developed to calculate the PFM properties obtained by pressing granules formed by particles of a porogen and metal powders.

Let's consider a cell as an elementary volume characterized by a cubic volume-centered packing of particles, with a porogen particle located in the center of the cube (Figure 2).

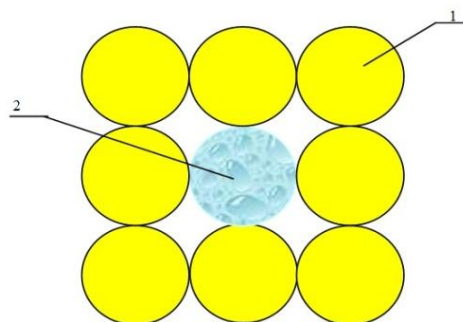


Figure 2 Elementary cell cross section
1 – metal powder, 2 – porogen

It was assumed that each particle is homogeneous and consists of a substance, which, as a separate compact body, obeys all the laws of classical mechanics. The mechanism of crumbling (pressing) of four particles under a given load was considered and a numerical model for calculating the resulting cavity (pore) was proposed. It should be noted that in the general case, the particles can be from different materials.

We show that the choice of a model with a porogen presented in Figure 2 expands the possibilities of obtaining a porous structure and makes it possible to predict the quality of PFM.

Based on the laws of continuum mechanics [5, 6] using static equations (the equilibrium equation of the selected medium element), geometric equations that determine deformations by the displacements of its points and the physical group of equations that take into account the mechanical properties of the material and determine the relationship between stresses and deformations of the element (Hooke's law), a mathematical model was proposed and an approximate algorithm for numerical calculation was described, which makes it possible to calculate pores and their sizes. The geometry of the pore was determined by a combination of some points M_k , called boundary with coordinates (r_i^k, z_j^k) , according to which it is easy to restore the closed curve that defines the pore.

When deriving the equations, Hooke's linear law [5] was used in the form:

$$T = \lambda_m D E^1 + 2\mu_m \varepsilon, \quad (1)$$

or in another form

$$T = 2\mu_m \left(\frac{\nu_m}{1-2\nu_m} D E^1 + \varepsilon \right), \quad (2)$$

where T – stress tensor, ε – deformation tensor, E^1 – unit matrix,

$D = \nabla \cdot \vec{v} = \text{div } \vec{v} = \frac{\partial u}{\partial r} + \frac{u}{r} + \frac{1}{r} \frac{\partial v}{\partial \varphi} + \frac{\partial w}{\partial z}$ – divergence of the

displacement vector, the index m indicates the properties of either metal ($m=1$) or the properties of a porogen ($m=2$), λ_m, μ_m – constant elastic moduli or Lamé coefficients for m – material, which are determined through the Young's modulus E_m and

Poisson's coefficient ν_m using formulas

$$\mu_m = \frac{E_m}{2(1+\nu_m)}, \quad \lambda_m = 2\mu_m \frac{\nu_m}{1-2\nu_m}.$$

Thus, for the displacement vector $\vec{u}$ arising in the m particle under the applied load, we have a system of nonlinear partial differential equations:

$$\frac{1}{1-2\nu_m} \text{grad } \text{div } \vec{u} + \nabla^2 \vec{u} + \frac{\rho_m}{\mu_m} \vec{Q} = 0, \quad (3)$$

where $\vec{Q}$ – volume or mass forces. In our case, the volume force is its own weight.

Taking into account cylindrical symmetry (when considering the task) and the assumption that the unknown quantities are independent of the angle φ , in the projections on the corresponding axes for the components of the displacement vector, we have system of equations

$$\begin{cases} \frac{1}{1-2\nu_m} \frac{\partial}{\partial r} \left(\frac{1}{r} \frac{\partial}{\partial r} (ru) + \frac{\partial w}{\partial z} \right) + \nabla^2 u = 0 \\ \frac{1}{1-2\nu_m} \frac{\partial}{\partial z} \left(\frac{1}{r} \frac{\partial}{\partial r} (ru) + \frac{\partial w}{\partial z} \right) + \nabla^2 w - \frac{\rho_m}{\mu_m} g = 0 \end{cases}, \quad (4)$$

where $\nabla^2 u = \frac{1}{r} \frac{\partial}{\partial r} \left(r \frac{\partial u}{\partial r} \right) + \frac{\partial^2 u}{\partial z^2}$,

$$\nabla^2 w = \frac{1}{r} \frac{\partial}{\partial r} \left(r \frac{\partial w}{\partial r} \right) + \frac{\partial^2 w}{\partial z^2},$$

u, v – projections of the displacement vector on the corresponding r and z axes.

Since the obtained system of equations contains partial derivatives, to uniquely determine the unknowns to the system of equations that determine the behavior of a linearly elastic body at points of its volume, it is necessary to add conditions on the surface S bounding it, which include particle surfaces, points of application of loads, points of contact with the solid surface (the base of the mold), and the points of contact or contact of the particles.

The boundary conditions are determined by the task or external surface forces or displacements of surface points, depending on the specific task. The points of the free surface are those points of the boundary of the particle contour that border gas (air) and the distribution of surface forces F is specified in them and the boundary condition is the equation of equilibrium on the surface

$$\vec{n} \cdot T|_S = \vec{F}, \quad (5)$$

where T – stress tensor at surface points; $\vec{n}$ – unit vector of the external normal to the surface of the body.

Guide cosines of the outer normal $n_r = \cos(r, \vec{n})$, $n_z = \cos(z, \vec{n})$ to the curve $z = z(r)$, determining the boundary of the surface are found by the formulas:

$$n_r = \frac{|z'|}{\sqrt{1+(z')^2}}, \quad (6)$$

$$n_z = -\frac{z'}{\sqrt{1+(z')^2}|z'|}. \quad (7)$$

Since the free surface S of the particle at the initial moment is known and in the dimensionless form on the plane (r, z) has the form

$$r^2 + (z-1)^2 = 1, \quad (8)$$

then according to formulas (2.24) and (2.25) it is easy to determine the direction cosines of the normal at the points of the boundary M^k

$$n_{r,M^k} = r^k, \quad (9)$$

$$n_{z,M^k} = \pm \sqrt{1-r^k}. \quad (10)$$

Task of 5 types of boundary conditions [7] leads to a closed system of differential equations, from the solution of which the stress state realized in the body and the displacement vector of the medium points realized in the body are determined. We formulate preliminary boundary conditions on the initial geometry of the body. Thus, the system of partial differential equations was finally obtained for the displacement vector $\vec{u}(u, w)$.

To numerically solve the system of equations (4) - (10) taking into account the boundary conditions [7], we will use the finite difference method. The solution is not found analytically, but on some selected set of points, called a difference mesh. The solution region is divided by coordinate lines with uniform nodes $M_{ij} = M_{(i,j)} = (r_i, z_j)$, $z_j = (j-1)h_z$ into cells (volumes), as a result of which we obtain a constant Eulerian mesh.

In the cells of the region, we specify the sign of filling the cell with substance according to the principle of the volume function, i.e. for a cell not fully filled with the substance, a scalar function F is introduced, the value of which is equal to the fractional volume of the cell filled with the substance. It is assumed that $F = 1$ when the cell is completely filled with the substance, and $F = 0$ in the case of

an empty cell. Cells with values $0 < F < 1$ contain a free surface. Using this feature, a decision algorithm is built taking into account the relevant conditions.

The system of difference equations for the numerical solution obtained by the integro-differential method [6] (by integrating the system of equations over the elementary volume or over the cell of the difference mesh) taking into account the dimensionlessness has the following form

$$\begin{cases} \frac{1}{\bar{r}}(r_{-0.5}u_{\bar{r}})_r + u_{zz} + \frac{1}{1-2\nu_m} \left(\frac{1}{\bar{r}}(r_{-0.5}u)_{\bar{r}} + w_z \right)_r = 0, \\ \frac{1}{\bar{r}}(r_{-0.5}w_{\bar{r}})_r + w_{zz} + \frac{1}{1-2\nu_m} \left(\frac{1}{\bar{r}}(r_{-0.5}u)_{\bar{r}} + w_z \right)_r - \frac{\rho_m}{\mu_m} gr_0 = 0, \end{cases} \quad (11)$$

for points $M_{ij} = M_{(i,j)} \in$ particle from m material.

The system is solved by the iterative method of establishing using the method of variable directions by iteration.

$$\begin{cases} \frac{u^{s+1} - u^s}{\tau_s} + \frac{1}{\bar{r}}(r_{-0.5}^{s+1}u_{\bar{r}})_r + u_{zz}^{s+1} + \frac{1}{1-2\nu_m} \left(\frac{1}{\bar{r}}(r_{-0.5}^{s+1}u)_{\bar{r}} + w_z^{s+1} \right)_r = 0, \\ \frac{w^{s+1} - w^s}{\tau_s} + \frac{1}{\bar{r}}(r_{-0.5}^{s+1}w_{\bar{r}})_r + w_{zz}^{s+1} + \frac{1}{1-2\nu_m} \left(\frac{1}{\bar{r}}(r_{-0.5}^{s+1}u)_{\bar{r}} + w_z^{s+1} \right)_r - \frac{\rho_m}{\mu_m} gr_0 = 0, \end{cases} \quad (12)$$

where τ_s - iterative parameter.

The method is based on explicitly finding the type of intersection of the boundary of the particle material with the coordinate lines of a constant Eulerian mesh.

We will focus on some stages of the system solution.

Stage 1: Solving an iterative system with respect to unknown displacements, we find a set of values $u = u_{ij}$, $w = w_{ij}$, which represent the values of the displacement vector in the nodes of the chosen mesh $u_{ij} = u(M_{ij}) = u(r_i, z_j)$,

$$w_{ij} = w(M_{ij}) = w(r_i, z_j).$$

Stage 2: Knowing the displacements found, it is easy to determine the coordinates of the displaced points $M^* = (r_i^*, z_j^*)$ by the formulas:

$$r_i^* = (r_i + u_{ij})r_0, \quad (13)$$

$$z_j^* = (z_j + w_{ij})r_0, \quad (14)$$

which determine the position of the deformable body. Finding new values $M^{*k} = (r_i^{*k}, z_j^{*k})$ of the boundary points $M^k = (r_i^k, z_j^k)$, we find a new surface of the deformable particle..

Stage 3: Next, we redistribute the cells along a given constant Eulerian mesh, taking into account the new position of the deformed particles. We form, based on the new coordinates of the points, a function indicating the filling of the volume in the cell, and introduce new markers, and etc.

When constructing a solution algorithm, to describe the behavior of a substance in an incomplete cell under the assumption of a linear nature of the substance change in the cell, 2 markers were introduced in incomplete cells located in the boundary cells at the points of intersection of the substance with the Eulerian mesh coordinate lines. Such a number of markers were enough to determine the normals and radii of curve at points on the free surface. The numbering (in ascending order) of the particle markers M^k in fractional cells is made so that the area filled with the substance is on the left.

The calculation algorithm considers 12 types of fractional cells, which differ from each other in the type of intersection of the free surface of the cell particles of the mesh region. Their configuration is shown in Ref. [7].

With the introduction of new coordinates of markers and signs of filling the cells, the algorithm is then repeated, but on a modified region. On its boundary conditions and the coupling conditions of various substances are determined according to the type of points.

The stress state at each point of the body (from the chosen system of points – the difference mesh) is determined by the stress tensor. We determine the components of the stress tensor through the found displacements using the following difference formulas:

$$\begin{cases} \sigma_{r,ij} = 2\mu_m \left(\frac{\nu_m}{1-2\nu_m} D_{ij} + (u_{ij} - u_{i-1,j}) / h_r \right), \\ \sigma_{\varphi,ij} = 2\mu_m \left(\frac{\nu}{1-2\nu} D_{ij} + \frac{u_{ij}}{r} \right), \quad \sigma_{z,ij} = 2\mu_m \left(\frac{\nu_m}{1-2\nu_m} D_{ij} + (w_{ij} - w_{i,j-1}) / h_z \right), \\ \sigma_{rz,ij} = \mu_m ((u_{ij} - u_{i-1,j}) / h_z + (w_{ij} - w_{i,j-1}) / h_r), \quad \sigma_{r\varphi,ij} = 0, \quad \sigma_{z\varphi,ij} = 0, \\ D_{ij} = (u_{ij} - u_{i-1,j}) / h_r + \frac{u_i}{r_i} + (w_{ij} - w_{i,j-1}) / h_z. \end{cases} \quad (15)$$

Stresses in the medium can be represented as consisting of stresses associated with uniform hydrostatic pressure (all three normal stress components are equal), plus stresses characterizing the resistance of the material to shear deformations. Therefore, if we decompose each stress $\sigma_r, \sigma_\varphi, \sigma_z$ into the hydrostatic component P and the deviator component S , then we will have the following ratios

$$\sigma_r = -P + S_r, \quad (16)$$

$$\sigma_\varphi = -P + S_\varphi, \quad (17)$$

$$\sigma_z = -P + S_z. \quad (18)$$

where P – arithmetic average of three stresses.

$$P = (\sigma_r + \sigma_\varphi + \sigma_z) / 3. \quad (19)$$

We will use Mises yield criterion to describe the yield stress. If the principal stresses are known, then the yield criterion is written as follows:

$$(\sigma_r - \sigma_\varphi)^2 + (\sigma_\varphi - \sigma_z)^2 + (\sigma_r - \sigma_z)^2 = 2(Y_0^m)^2, \quad (20)$$

where Y_0^m – yield stress for simple stretching for a particle of m material. We continue the calculations until the stress values on the left side become less $2(Y_0^m)^2$.

The solution of a nonlinear system of differential equations allows one to determine displacements that describe the pore space of the simulated cell and make it possible to determine the specific surface, pore size, and porosity with subsequent calculation of permeability according to known techniques [2].

The advantage of the developed mathematical model is the possibility of its development to describe the sintering process.

Schematically, the option of pore formation as a result of pressing and sintering is shown in Figure 3.

Moreover, the developed mathematical model can be used to calculate modified elementary cells. One of the options for such a modification is presented below in Figure 4, which schematically shows a granule formed by a metal powder and a porogen.

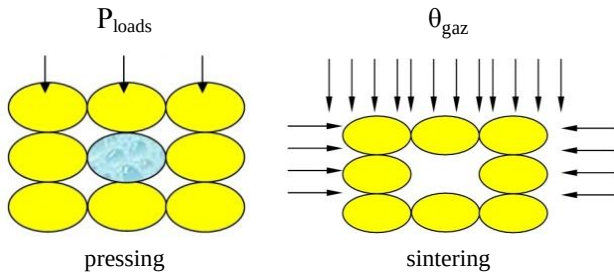


Figure 3 Scheme of pore formation during pressing and sintering:

P_{loads} – sample load, θ – sintering temperature

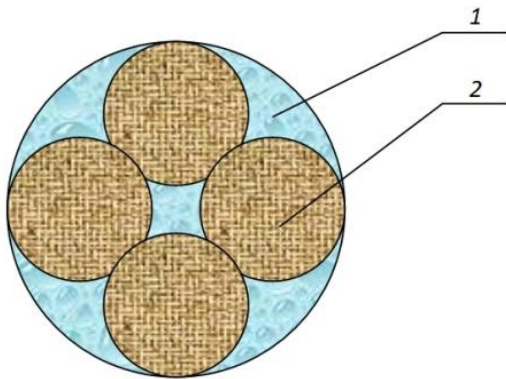


Figure 4 Granule scheme (homogeneous metal particles in porogen media):

1 – a porogen, 2 – metal

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3. Conclusion. PFM model is proposed, which enables to take into account the presence of a porogen when calculating the pressing process and reflects all types of bonds at the contact boundaries of metal powder particles. Based on the constructed model, an approximate method and algorithm for calculating the properties of PFM obtained by pressing granular metal powders using a porogen were developed.

The advantage of the proposed mathematical model is the possibility of its development to describe the sintering process, as well as the possibility of its use for calculating the modified elementary cells used by researchers in modeling the structure of PFM with a porogen.

ANALYSIS OF PLASTICITY OF QUENCHED TOOL STEEL DURING STRESS RELAXATION AT ELEVATED TEMPERATURE

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Abstract: Phase transformations in metals have a major influence on the material behaviour in several common engineering applications. Steels exhibiting enhanced response to transformation-induced plasticity (e. g. high strength TRIP-steels for automotive production) are examples of the important role martensite formation can play. An externally stressed specimen in the process of a phase transformation may show a significant nonlinear behaviour, which is known as transformation plasticity. Even under an externally applied load stress with the corresponding equivalent stress being small in relation to the “normal” yield stress of the material, plastic deformation occurs.

An aim of a research was to determine relaxation and transformation plasticity properties of alloyed tool steel while is tempered at elevated temperatures and for different tempering duration.

Keywords: TRANSFORMATION PLASTICITY, TOOL STEEL, TEMPERING, STRESS RELAXATION.

1. Introduction

Quenched steel is in a metastable state. When martensite is tempered at higher temperatures and in some cases, retained austenite as well, carbon is precipitated in carbides form. After cooling, secondary martensitic transformation is possible [1]. Transformation plasticity effect can also be observed during these processes [2-5]. When first heated, the tempered steel is relatively ductile; after reheating at the same temperature the ductility is negligible.

Changes in the properties of quenched steel due to time and temperature have long been observed. According to Geler [6], by maintaining 9XC quenched but not tempered steel at 20°C temperature, its strength initially decreases and then increases. Since the carbon concentration and martensite workability do not change under these conditions, this phenomenon can be explained as a result of stress relaxation processes.

These issues are particularly relevant in the manufacture of tools, as most of them must be straightened to eliminate quenching distortions. Straightening can be performed in various ways: at cold state, after hot washing and often after tempering. Although the tools that are tempered are less brittle than the ones that have just been quenched, they are harder to smooth because quenched and low-tempered steels are very elastic and spring-like. Great force must be used to bend the bent tool to the opposite side. If smoothed, the geometrical parameters of such tool are not stable. Over time, and especially after heating during operation, the straightened tool or part will be distorted again.

The most effective method of the straightening of curved quenched parts is to make them straight during the transformations while tempering. The parts are bent and pressed inside the device for the fixed deflection and then tempered. During the transformations of tempering, the steel becomes very plastic for a short time, the elastic strains partly relax and the part of the elastic deflection changes to the plastic [7]. The form of the straightened parts is not geometrically stable and changes in time, especially when the parts are heated. This phenomenon, called self-deformation was also researched.

The article presents investigation of relaxation properties during tempering of low alloyed tool steel XBF (GOST standard). This steel is used for cutting tools with increased resistance to quenching distortions, thread gages, matrix and punches for cold forming.

2. Methodology

Investigation of relaxation properties was performed using low alloyed tool steel. Its chemical composition is presented in Table 1 and critical points of the steel grade are listed in Table 2.

The specimens of rectangular cross section with parameters of 6×8×100 mm were oil quenched from 840°C temperature (Fig. 1). Then, cooled specimens were bent elastically and fixed inside the special device with the deflection of 0.5; 1.0 or 1.5 mm (the value of y_e) and tempered at the temperature of 200-400°C for 10 min till 4 h (Fig. 2). After tempering at this fixed position, the specimens were withdrawn out of the device, the plastic deflection y_p of the specimens was measured. Then the coefficient k of the elastic stress relaxation was calculated:

$$k = \frac{y_p}{y_e} \quad (1)$$

The self-deformation of the elastically bent specimen was observed during the tempering at the same temperature as they were bent for 1 hour. Then the change of the plastic deflection Δy_p was measured in accuracy of 0.01 mm and the relative self-deformation $\Delta \varepsilon$ was calculated:

$$\Delta \varepsilon = \frac{\Delta y_p}{y_p} \quad (2)$$

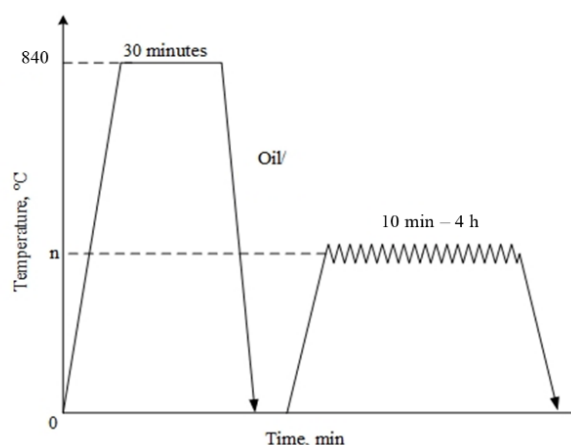


Fig. 1 The circle of heat treatment of steel during investigation of relaxation properties

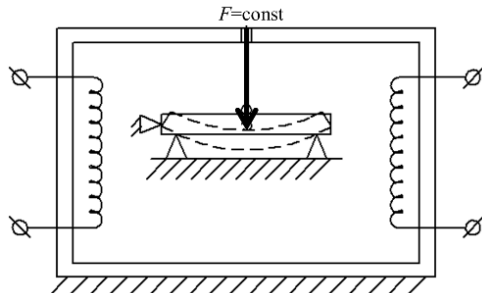


Fig. 2 The schema of bent and fixed specimen during tempering in the furnace

Table 1: Chemical composition of investigated low alloyed tool steel (% wt) [8]

Grade (GOST)	Elements, %					Equivalent of alloy grade
XBΓ	C	Si	Mn	Ni	S	107WCR5 EN
	0.9-1.05	0.1-0.4	0.8-1.1	max 0.4	max 0.03	
	P	Cr	W	Cu	Fe	
	max 0.03	0.9-1.2	1.2-1.6	max 0.3	Bal.	

Table 2: Temperature of critical points for steel XBΓ [8]

Critical point	A_{c1}	A_{cm}	A_{r1}	M_s
Temperature, °C	750	940	710	210

The microstructure of steel used for experiments was examined by optical microscope optical microscope Nikon equipped with video camera Nikon DS-2 16 MP and objectives Nikon TU Plan Fluor 10×/0.30 and Nikon TU Plan Fluor 100×/0.90.

The temperature of specimen during tempering was measured by welded chromel-alumel thermocouple of 0.3 mm wire diameter.

3. Results and Discussion

The microstructure of quenched steel XBΓ consists of martensite, retained austenite and carbides (Fig. 3). Amount of retained austenite can vary from 18 % to 20 % [9].

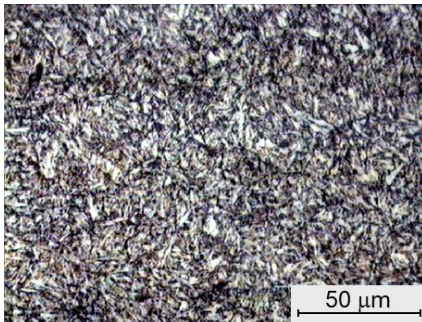


Fig. 3 Microstructure of oil quenched steel XBΓ: dark martensite and white retained austenite

The influence of tempering duration at 250°C temperature on the remained plastic deflection of specimens is shown in the Table 3.

Table 3: Values of elastic and plastic deflections of steel specimens before and after tempering at 250°C when initial fixed elastic deflection is 1 mm

Tempering duration, min	Fixed elastic deflection y_e , mm	Remained plastic deflection after tempering y_p , mm	Coefficient of stress relaxation, k
10	1.05	0.03	0.03
30	1.03	0.14	0.14
60	1.02	0.51	0.49
120	1.02	0.59	0.59
240	1.05	0.77	0.73

In the same way, the results were obtained at tempering temperatures 200, 225, 275, 300 and 400 °C and at fixed initial

elastic deflections of 0.5 mm and 1.5 mm. The graphic expression of the results is presented in Fig. 4.

Transformation plasticity of quenched steel, which causes the relaxation of elastic strains, depends on the microstructural metastability and progression of tempering transformations. Therefore, the maximum effect of plasticity (in our way – relaxation of elastic strains) was then, when the specimens were quenched from the temperature, which can be characterized by martensite with enough of carbon and not much of retained austenite, and then tempered at sufficiently high temperature. On the contrary, when the specimens were bent at low temperature, especially those, which were already tempered, the plastic properties were obtained minimal.

Investigating influence of the magnitude of retained plastic deflection after tempering of stressed specimens, it was determined, that the biggest effect of the progress of relaxation is then, when the duration of tempering is 1 hour (Fig. 5).

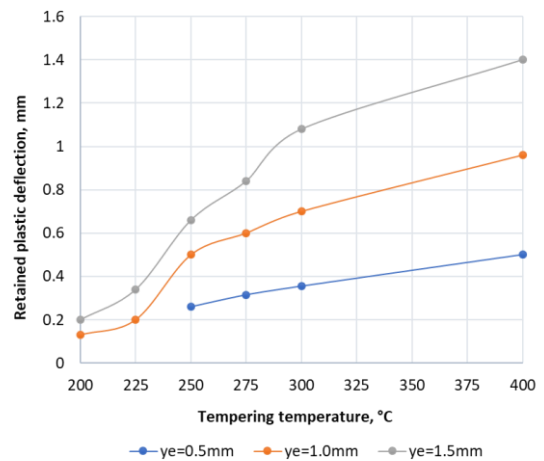


Fig. 4 Influence of tempering temperature on retained plastic deflection of elastically bent specimen when tempering duration is 60 min

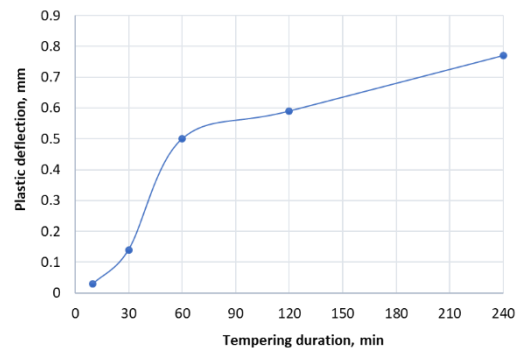


Fig. 5 Influence of tempering duration on relaxation properties of steel XBΓ specimens

The second (stabilizing) tempering of already tempered in stressed state specimens was performed for the reason to clarify, if the microstructure of tempered specimens was stable. The change of the plastic deflection of steel specimens after stabilizing tempering is shown in Fig. 6. After second tempering at stabilizing 170 °C temperature, the certain part of plastic deflection eliminated due to metastability of microstructure. The lower tempering temperature was set, the higher percentage of eliminated plastic deflection occurred.

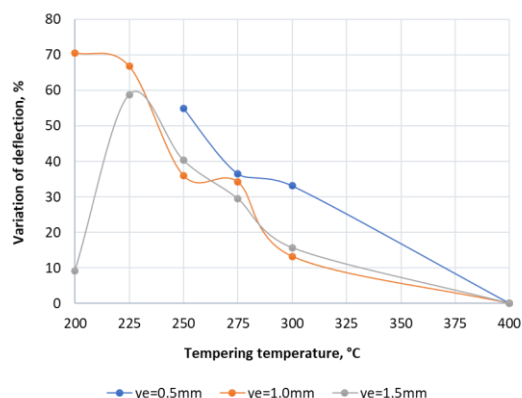


Fig. 6 Magnitude of percentage of plastic deflection elimination after the second tempering (stabilizing) at 170 °C temperature

The biggest variation of plastic deflection is then, when tempering temperature is low (less 300°C). This shows that at the lower tempering temperature stresses in steel microstructure have the biggest influence on tempering transformations, e.g. tension stresses stop mostly the transformation of martensite. That is why martensite has heterogeneous structure after tempering and, therefore, increased self-deformations. The continuance of tempering also makes the same influence: after the short-term tempering the self-deformation is rather big – up to 40-75 %.

4. Conclusions

Following the investigation of the relaxation properties of steel XBF during tempering, the following conclusions were formulated:

1. The quenched steel becomes significantly plastic during tempering; therefore, the part of elastic stresses relaxes – the part of elastic deflection becomes plastic. Relaxation properties of steel increase with the increasing of tempering temperature and heating duration.

2. For the straightening the tools must be tempered at the temperature 170°C for 1 hour, bent in the device (evaluate relaxation properties), heated in the furnace at the certain temperature for 1 hour, then cooled in the air and tempered at the same temperature for 1 hour. The initial fixed elastic deflection of tool in the device is calculated considering the relaxation properties of steel at the temperature of tempering and self-deformation during the stabilizing tempering.

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THE CONNECTION BETWEEN COEFFICIENT CLUSTERISATION WITH TEMPERATURE

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Abstract: A statistical model is proposed to analyze the relationship between the clustering coefficient (CC) and the temperature and atom-atom interaction potential. CC is defined as the ratio of the number of atoms in clusters to the total number of atoms in a system. The proposed technique is based on the statistical concept of entropy. The clustering coefficient is considered for the equilibrium state.

KEYWORDS: CLUSTERED LIQUID, CLUSTERING, FREE ENERGY, INTERATOMIC POTENTIAL, ENTROPY.

1. Introduction

In various liquids there are regions with a large number of atoms with degree of ordering more than the average for the whole volume of the liquid. Such regions are called as clusters, and the fluids in which they emerge are called as clustered. At a temperature close to the evaporation one temperature, the concentration of clusters is minimal, and at crystallization their concentration has maximal value. If the number of atoms in the system is N and the number of atoms in the clusters is C , the ratio $q = C/N$ is a clustering coefficient (CC). Each cluster is a dynamic system with a lifetime τ . But the total number of atoms C remains unchanged if $T = \text{const}$. The value q at a fixed temperature depends on the properties of the liquid, which are individual for each liquid substance. It means that cluster have possibility to appear and to disappear. But the dependence $q = q(T)$ has common form.

This fact may be explained on the basis of methods of physical kinetics.

The aim of the research is to create a mathematical model based on the minimization of free energy at the equilibrium state of the cluster system.

The object of the investigation is the Helmholtz equation, which includes the potential of atom-atom interaction and entropy of the system.

The object of the research is the relationship between CC, temperature and potential of atom-atom interaction.

There are following research tasks:

1. To constructed a model for calculating the entropy of a cluster system at a fixed free energy when $T = \text{const}$;
2. To look for a connection between the CC and the internal energy for isothermal conditions at equilibrium;
3. To show that the CC in liquids monotonically decreases if temperature changes from the melting point of the crystal (T_{cr}) to the point of evaporation of the liquid (T_{ev}).

2. Degree of atomic-molecular ordering

Crystalline objects are atomic-molecular systems with regular arrangement of homologous points in any direction. This means that if one selects any point in the crystal and draw a straight line through it, then on this straight line, at a distance from the initial point equal to a , there's a point absolutely identical in structural and chemical relation to the starting point. For an ideal crystal, the distance from the initial point to n -th point (a_n) is described by the condition:

$$a_n / n \equiv a \quad (1)$$

In real crystals, the distance from the starting point to the n -th one equals to:

$$a_n / a = n + \Delta \quad (2)$$

This is explained by the fact that the distances between neighboring atoms fluctuate. As repulsive forces are significantly stronger than attractive forces at the same change $a_n - a_{n-1}$, then Δ in formula (2) is always positive. If the Δ exceeds any fixed value, for example $a/2$, the value of n determines the degree of ordering. For perfect crystals $n \Rightarrow \infty$, for gases $n=1$.

The degree of ordering is a subjective characteristic to some extent, since Δ can take other values. For example, if Δ_1 is accepted as equal to $a/2$ or $\Delta_2 = \frac{a}{4}$, that the degree of ordering in the first case "will appear" higher.

However if the same limit value is taken for a number of samples with the same structure and composition but different concentrations of defects, then regardless of its magnitude a hierarchical series is constructed from the sample with the highest degree of ordering to the sample with a lower value q or vice versa.

If the substance in the crystalline state is described by the $Fm3m$ group, which corresponds to a three-layer closed sphere package, for an ideal crystal, the radius of the n -th coordination sphere is equal to:

$$R_n = \sqrt{n} \cdot d, \quad (3)$$

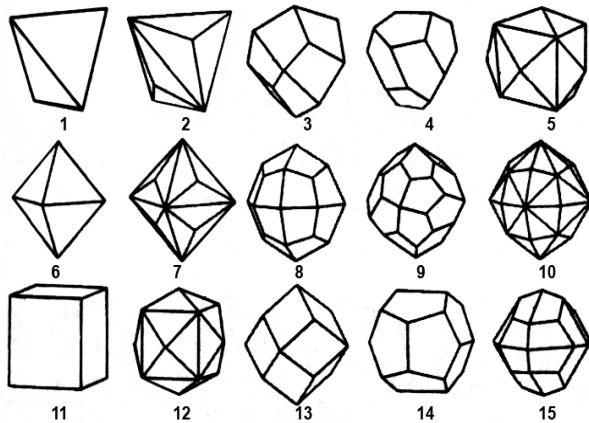
where d is the diameter of the atom.

That is, a monoatomic substance is considered, in which, within the framework of the model of the densest sphere packing, the degree of ordering determines the defect of the crystal due to various factors.

The example of these objects is metallic crystals [1]. In this case, the criterion of the degree of ordering is the difference of the radius of the n -th coordination sphere and its number for metals of the corresponding point $Fm3m$ group or other groups. In particular for metals with $Fm3m$, $Im3m$, $6/mmm$ point groups. These three groups describe the structures of almost all metals, ice and other substances.

In liquid environment, the average atom-atom distance is almost equal to the value for crystalline objects. But the degree of atomic ordering in liquids is much lower than in crystals. So for liquids the notion of middle and even closest order applies. At the same time, groups of atoms can interact with each other to create a three-dimensional configuration similar to a nanocrystalline particle.

As the temperature decreases, the stability of this system increases. At the point of crystallization, each system of this kind which is called a cluster, becomes the center of crystallization. That is why metals create polycrystalline objects during crystallization. Let's consider the correct forms of crystals of the $Fm\bar{3}m$ group.



1. Tetrahedron
2. Trigontrioctahedron
3. Tetragontritetrahedron
4. Pentagontritetrahedron
5. Hexatetrahedron
6. Octahedron
7. Trigontrioctahedron
8. Tetragontritetrahedron
9. Pentagontritetrahedron
10. Hexaoctahedron
11. Hexahedron
12. Tetrahexahedron
13. Rhombododecahedron
14. Dodecahedron
15. Didodecahedron

Fig. 1. - The cubic crystals polyhedral right forms

Figure 1 shows the names of possible polyhedra.

These habitus are preserved for the nanocrystals. As the particle size increases, the polyhedral form takes the shape of a sphere, as the $Fm\bar{3}m$ group in the limiting transition from a polyhedron to a sphere becomes a group of infinite order $\infty : \infty \cdot \infty$.

In general case, clusters are the objects with the following characteristics. The first, the degree of atomic ordering is greater than the average volume of the cluster fluid; secondly, they are a dynamic system, which even in thermostatic conditions disappear and occur in other parts of the system.

The total number of atoms in the cluster state remains unchanged.



Fig. 2. – Macroscopic model of the atomic cluster

An example of a cluster system is shown in figure 3, which demonstrates that clusters with structural characteristics different from non-clustered matrix are visible in the electron-microscopic image of the pure metal.

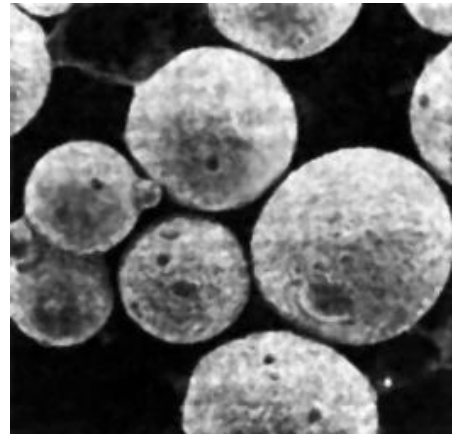


Fig. 3. – Electron microscopic image of copper particles, magnification $5.2 \cdot 10^{-7} \text{ m}$ [2].

3. The Helmholtz equation for cluster liquids

To determine the relationship of the clustering coefficient for liquids, the Helmholtz equation was used which is applicable for solids, liquids and gases, especially for cases with high pressure.

This equation has the form:

$$F = U - TS, \quad (4)$$

where F is the free energy of Helmholtz or the isochoric-isothermal thermodynamic potential determined by the difference between the total energy and the parameter configuration, depending on the number of States in thermodynamic equilibrium.

The total energy U is the sum of the kinetic energy of the motion of atoms (ions, molecules) and potential energy of atom-atom interaction. The kinetic energy of one atom modeled by a sphere with three degrees of freedom is:

$$K = 3kT, \quad (5)$$

where k is Boltzmann's constant.

For polyatomic molecules

$$K = f kT, \quad (6)$$

where f is a factor depending on the number of degrees of freedom of an atom (ion, molecule).

The potential energy of the pair atom-atom interaction (P) in the general case can be represented as:

$$P = \alpha \exp \frac{E}{kT}, \quad (7)$$

When α , E are energetical parameters which depends on properties of matter and start temperature point.

If is consider the following temperature range:

$$T_{cr} < T < T_{ev}, \quad (8)$$

where T_{cr} , T_{ev} – crystallization and evaporation temperatures respectively.

For specific situations, the form of the potential may be different [3]. The potentials of Lennard – Jones, Morse, of Sutherland, Yukawa, models of hard spheres, quantum wells with different parameters [4, 5]. All these potentials can be considered as constants for fixed interatomic distances. If the temperature increases, the atom-atom distance increases. This means that the parameter of atom-atom interaction decreases with increasing temperature. That is, this degree of approximation is applicable to the analyzed temperature range.

Entropy $S = k \ln \Omega$, where Ω is the number of the system states. In this case entropy as thermodynamic function has statistical sense. General number of atoms (ions, molecules) equal N in the system. From N atoms C atoms are displaced in clusters. That is,

$$C/N = q, \quad (9)$$

it is a clustering coefficient with $0 \leq q \leq 1$.

Atoms in a cluster interact with each other.

The total potential energy for all clusters is equal to

$$U_C = \frac{C}{2} \alpha \exp \frac{E}{kT}, \quad (10)$$

For atoms outside the cluster system, the gas model is applicable, when atoms interact only in a collision.

This model is used not only for liquids but also for solids [6]. On its basis it is proved that

$$C_V(T)_{T > \theta_D} = 3R, \quad (11)$$

where θ_D – Debye temperature.

This conclusion is in good agreement with the experiment, which indicates that classical concepts are applied legitimately.

The total energy in the Helmholtz equation (4) for the system under consideration is

$$U(T, C) = (N - C) f k T + \frac{C}{2} \alpha \exp \frac{E}{kT}, \quad (12)$$

The scheme of change of the clustering coefficient for any cluster systems is shown in figure 4.

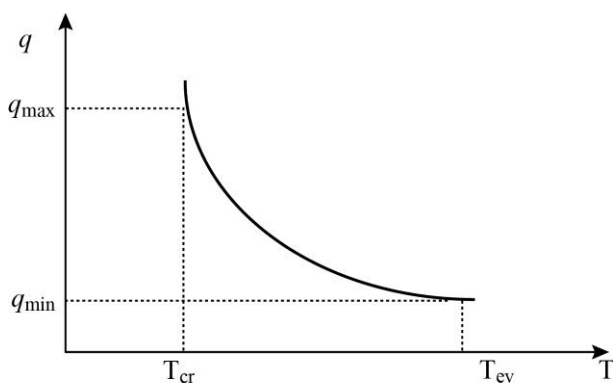


Fig. 4 – The scheme relation between coefficient of clusterization (q) and the temperature (T) [2].

From this scheme clearly visible, that $q_{\max}$ meets the requirements T_{cr} , and $q_{\min} - T_{ev}$.

The numerical values of these parameters depend on the properties of the substance and the methods of temperature change. For example, the sudden cooling of the melt (method of

spinningovye or punching through the capillary) can be fixed clustering for intermediate temperatures of the melt.

4. The entropy of a cluster system

The cluster system can be considered as a two-phase substance with phase concentration $(N-C)/N$, C/N .

In the framework of the classical kinetic model atoms are distinguishable, that is, the number of States in the system (Ω) is determined by the condition:

$$\Omega = \Pi_{(N-C)} \cdot \Pi_C = (N - C)! C!$$

$$S = k \ln \Omega = k \cdot \ln(N - C)! + \ln C!, \quad (13)$$

When $\Pi_{(N-C)}$ is according to Stirling's formula and $(X \gg 1)$

$$\ln X! = X \ln X - 1 = X \ln X, \quad (14)$$

and considering that N , $N-C$, C immeasurably more than one, we get:

$$S = k \ln \Omega = k((N - C) \ln(N - C) + C \ln C) = k[N(1 - q) \ln N(1 - q) + qN \ln qN] \quad (15)$$

According to formulas (13 – 15) the Helmholtz equation (4) will take the form:

$$F(T, q) = N(1 - q) f \cdot kT + q \frac{N}{2} \alpha \exp \frac{E}{kT} - kT \{ N(1 - q) \ln N(1 - q) + q \cdot N \ln qN \} \quad (16)$$

Free energy at a constant temperature depends only on the degree of clustering determined by the value of the clustering coefficient q . When the system is in the equilibrium state the following ratio must be true

$$\frac{dF}{dq} = 0. \text{ Then}$$

$$\frac{1 - q}{q} = \left(0,5 \exp \frac{E_0}{kT} - f k T \right). \text{ Then}$$

$$F(q) = (1 - q) f k T + \frac{q}{2} \cdot \alpha \exp \frac{E}{kT} + (1 - q) \ln N(1 - q) + q \cdot \ln Nq \quad (17)$$

At a constant temperature, the equilibrium state of the system is determined by condition:

$$\frac{dF}{dq} = 0. (T = \text{const}).$$

That is:

$$\left(-f k T + \frac{1}{2} \alpha \exp \frac{E}{kT} + k T \ln \frac{q}{(1 - q)} \right) = 0, \quad (18)$$

From here

$$\ln \frac{q}{1-q} = \frac{\alpha}{2kT} \cdot \exp \frac{E}{kT} - f, \quad (19)$$

In thermostatic conditions, the clustering coefficient of the system depends on only the potential of atom-atom interaction.

In the quasi-stationary transition to higher temperatures, the right side of the equation (19) decreases, hence the clustering coefficient also decreases. Using the model the size of clusters and their shape cannot be determined. Because it needs property of matter.

However, given the dynamic processes of cluster formation, we can understand why, for example, it is very difficult to obtain single crystals during solidification of cluster fluids.

Each cluster at a lower temperature is characterized by a longer lifespan and can become the embryo of the crystal.

At sharp cooling of metal melts, when relaxation in the cluster is not achieved, the metal solidifies.

Since the cluster does not necessarily reflect the symmetry of the crystal state, structures can occur even with non-crystallographic symmetry.

This may explain the example of quasi-crystalline objects with pentagonal symmetry.

5. Conclusion

The model of calculation of entropy of cluster system for conditions of thermostatic equilibrium has been constructed.

The relationship between the clustering coefficient and the internal energy for isothermal conditions at equilibrium has been analyzed.

The focus of the study has been the dependence of energy freedoms on temperature, the parameters of the atom-atom interaction potential and the ratio of the number of atoms in clusters to the total number of atoms in the system, also known as the clustering coefficient.

It is found that the clustering coefficient in liquids monotonically decreases with temperature change from the melting point of the crystal to the point of evaporation of the liquid.

The proposed model for determining the dependence of the clustering coefficient (q) in cluster fluids, based on the method of physical kinetics, explains the dependence of the clustering coefficient (q) on temperature T , and is applicable to all types of atom-atom interaction potentials.

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ULTRAFINE-GRAINED MAGNESIUM ALLOYS WITH ENHANCED STRENGTH AND IN-SERVICE PROPERTIES AFTER DIFFERENT DEFORMATION METHODS

УЛЬТРАМЕЛКОЗЕРНИСТЫЕ МАГНИЕВЫЕ СПЛАВЫ С ПОВЫШЕННЫМИ ПРОЧНОСТНЫМИ И ЭКСПЛУАТАЦИОННЫМИ СВОЙСТВАМИ ПОСЛЕ РАЗЛИЧНЫХ МЕТОДОВ ДЕФОРМАЦИИ

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Abstract: Recently, the possibility of imparting an ultrafine-grained (UFG) structure to various metals and alloys by severe plastic deformation (SPD) techniques, such as high-pressure torsion and equal channel angular pressing, was demonstrated quite convincingly. However, the SPD methods do not always successfully fit into the technological chain of industrial manufacturing. In this regard, another group of processes based on severe shear deformation are of interest. In this talk, we will show that radial-shear rolling and rotary swaging, which involve severe shear and are amenable to industry-scale manufacturing, lead to the formation of a UFG structure of magnesium-based alloys. This results in an increase of their strength and in-service properties, including the fatigue limit, the corrosion resistance, and biocompatibility. For comparison, the effect of multiaxial deformation – another SPD process that is viable at industry manufacturing scale – on the microstructure and the characteristics of the processed magnesium alloys will be considered.

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KEYWORDS: MAGNESIUM ALLOYS; SEVERE PLASTIC DEFORMATION; ULTRAFINE GRAINED STRUCTURE; TEXTURE; MECHANICAL PROPERTIES; IN-SERVICE PROPERTIES

SYNTHESIS AND DESIGN OF HYPERBOLOID GEAR DRIVES: METHODOLOGY AND ASPECTS

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Abstract: *The high current requirements for the characteristics determining the accuracy, reliability and efficient manufacturing of modern machines, apparatus and devices applicable in various fields of industry and transport in a big extend dictate the applied scientific methodology to the technological synthesis, design and production of gears. In the processes of synthesis and design of the different types of gear transmissions, the designer faces an extremely complex set of questions to solve, which together define the desired optimal construction. In this case, an optimal design means a gear transmission that is capable of delivering preliminary defined kinematic and / or strength requirements at minimum design, production and operation costs (including the repair costs during their exploitation).*

This work presents a review of the methodology, developed by the authors, oriented towards the synthesis and design of spatial gear mechanisms (hyperboloid gear drives), realizing rotations transformation between crossed shafts. The main principles, on which the construction of adequate software programs are based for the synthesis of treated transmissions, are presented.

Keywords: HYPERBOLOID GEAR DRIVES, SPIROID AND HELICON GEARS, SYNTHESIS, DESIGN, MATHEMATICAL MODEL, COMPUTER PROGRAMS

1. Introduction

The high current requirements to the accuracy and reliability characteristics of the machines, apparatus and devices applicable in industry and transport in a big extend dictate the applied scientific methodology to the technological synthesis, design and manufacture of gear drives. In the processes of synthesis and design of the various types of gear transmissions, the designer faces an extremely complex set of questions to solve, which together define the desired optimal construction. In this case, an optimal design means a gear transmission that is capable of delivering the preliminary defined kinematic and / or strength requirements at minimum design, production and operation costs (including the repair costs during their exploitation. By its essence, the systems of requirements for different quality characteristics of the gear drives, ensuring its optimality, are [1]:

- **geometric ones**, which control the kinematic exactness, smoothness of the work process, the character of the contact (the placement of the contact point, an orientation of the contact lines in the contact points) in the relation of the gear drives' loading capacity and etc.;
- **dynamics ones**, that influences the noise and vibrations in the gear systems, the conditions for occurring the resonance phenomena and etc.;
- **strength ones**, determining durability and reliability of gear drives, including the transfer of nominal power in the process of rotations transformation with avoidance of "scratching" (scoring), "pitting" and etc. on the active tooth surfaces of the synthesized mechanism;
- **economic ones**, defining the production costs (e.g., per unit of power), energy loss in motions transformation (coefficient of efficiency) and etc.

Considering the wide variety of requirements to the quality characteristics of the gear drives, in connection with the actual existing technological, manufacturing and economical price limitations, it is obvious that the solution of a real task of creating an optimal gear design is realized with some approximation with respect to the theoretical variant. The realization of this only possible approach to creating real gear drives requires it to be complex one, which should be foreseen in the processes of synthesis and design of the gear systems.

2. Aspects of Computer Design

The wide variety of gear mechanisms used in industry and transport, as well as the permanent pursuit of researchers to create new and improved gears drives on one hand and on the other- the different and changing approaches to the mathematical modeling for

synthesis and design, make it difficult to realize universal CAD systems. In connection to all said above, it specially should be noted the note the extremely dynamic development of modern technical computing tools and software programs. This often requires a reassessment not only of the way in which computer programs are organized, but also leads to informal changes of applied mathematical models [2]. In order to realize these researches into the field of theory gearing, as well as to provide an adequate scientific support for this type of production, the computer design has evolved, forming three types of software:

First type. The programs, included here, are designed to study the mutual influence between various kinematic, structural, technological and exploitation parameters of the studied gear drives. Essentially, this type of software is not subordinate to a particular strategy associated with the construction of CAD systems. The elaborated mathematical models, algorithms and computer programs are designed to determine the influence of one or other real-life existing parameters on the qualitative characteristics of the concrete gearings. However, the programs created in this case can be used as software modules, which are elements of system of criteria for the quality control of the synthesized gear drives.

Second type. This group includes computer programs organized on the basis of algorithms, contained in standardization documents [3], company methodologies [4] or handbooks (guides) [5-8]. Here, it should be included, the program products, elaborated on the basis of algorithms for geometric and strength calculation of the traditional types of gear drives: cylindrical involute with external and internal mating gears, cylindrical worm gears, bevel gears with straight teeth and so on. It should be noted, in particular, that the algorithms used in the prevailing number of cases do not ensure the optimization in the synthesis and design of gear transmissions. For example, the known algorithms for the synthesis of cylindrical involute, conic and worm gears [6] are able to provide only the technological and instrumental requirements for their manufacture, in terms of standardized determined modules (ptiches) and in terms of the occurrence of undercutting on the tooth surfaces under instrumental gearing or interference - in work gearing. Secondly, this category of software products it can also be included those, through which the strength characteristics of the already geometrically and technologically synthesized gear drives [3] are examined. As such, these computer programs can be treated as tools for analysis of the gear mechanisms.

Third type. Included in this category of computer program products are those based on mathematical models, developed on the basis of the specially oriented scientific studies. For example, for Bulgaria, these are the computer programs that covers the synthesis and design of Spiroid and Helicon gear sets (Spiroid and Helicon

are registered trade mark of Illinois Tool Works, Chicago, Illinois) [1, 9, 11], and with conic and hypoid gear mechanism – type Gleason [5, 12, 13]; and others. For the contemporary gear transmissions, including even classical gear mechanisms, treated in terms of current engineering requirements, the elaboration of new mathematical approaches to their geometric, technological and strength synthesis is required. The process of optimization synthesis, in this case, is realized by applying the **method of direct search**. This method provides an opportunity to reduce the number of calculated gear pairs, which compose the synthesized gear mechanism. It will be reminded, that the essence of this method is as follows:

- input parameters are defined, as well as those that will not be changed throughout the whole synthesis process;
- the variables parameters are determined as well the way of their variation, respectively;
- the process of varying the defined variable input parameters compared to their initially given value continues, until the preliminary defined optimization criteria are fulfilled;
- from the calculated pairs of conjugated gear sets, a final variant is chosen for which, there is the best fulfillment of the additional conditions (restrictions) introduced in the mathematical model.

In other words, the process of optimization synthesis and design of the third type of software is based on adequate iterative procedures, by which the desired solution is found by variation of certain parameters.

3. Computer Programs: Principles and Methodology

The computer programs designed for the synthesis and design of linear contacting hyperboloid gear mechanisms belong to the third type software. Considering, the known methodological limitations [14], the following sequence is kept when constructing this type software, for elaboration of the system for the computer design of hyperboloid gears with linear contacting tooth surfaces.

3.1. Basic Principles and Approaches of the Mathematical Modeling for Synthesis and Design

When profiling the kinematically conjugated surfaces, by means of which the rotations transformation between crossed axes is realized, the basic observed principles are the principles of T. Olivier [1]. Here, it will be summarized only that part of them, which is directly related to the construction of the concrete computer programs.

When generating the tooth surfaces Σ_1 and Σ_2 , firmly connected with the bodies B_1 and B_2 , through the generating (instrumental) surface Σ_J , firmly connected with the body B_J , the mentioned bodies B_1 , B_2 and B_J realize rotations motion with different vectors of rotation. In this case, relative motions are characterized by velocity ratios $i_{12} = \omega_1/\omega_2$, $i_{ij} = \omega_i/\omega_j$ ($i = 1, 2$). If the surfaces Σ_2 (or Σ_1) are respectively a one-parameter envelope of the instrumental surface Σ_J , when the conditions $\Sigma_J \equiv \Sigma_1$ ($\Sigma_J \equiv \Sigma_2$, respectively), $B_J \equiv B_1$ ($B_J \equiv \Sigma_2$, respectively) and $i_{12} = i_{j2}$ ($i_{12} = i_{j1}$, respectively) it is said that the tooth surfaces are generated in accordance with the second principle of T. Olivier. In this case, Σ_1 and Σ_2 have always a linear contact.

In [1, 14-17], are formulated two approaches (applied by the authors) to the elaboration of mathematical models for the synthesis: mathematical modeling, which defines and optimizes the geometric, technological and exploitation characteristics of the design gear drives in a small vicinity of the *pitch contact point* and

a mathematical modeling related to the ensuring of the quality characteristics in the whole *region of mesh*.

In connection with all said, it is obvious that the methodological difference between the two approaches for the synthesis of spatial gear drives requires to define in advance the adaptability of the future designed hyperboloid gear drive to one of the two mentioned approaches. The determination of the adaptability of the planned procedure for creating of an adequate mechano-mathematical model is a complex creative process, requiring the knowledge of both the theoretical content of the approaches to the synthesis and the specific technological and exploitation requirements characterizing the created transmissions.

3.2. Principles of Organizing the Design Process

The following principles are obvious and have been commented by various authors [1, 2, 9, 12-15]. Here, the focus will be put on those which are determining for the construction of the computer programs for synthesis of gears type Spiroid and Helicon.

Determination of the groups of independent and variable input parameters. To the group of independent input parameters a set of standardization modules should be included, that determines the technological capabilities of the hobbing machines; coefficients that define the tooth geometry as a function of the modules; coefficients of frictions between the different pairs of materials applicable for producing of the toothing of the conjugated gear pairs; coefficients, linear and angular values associated with the design of the instrumental equipment and etc.

To the input data parameters, among which the variable ones are chosen, as a rule are included those which define the overall geometry of the calculated gear system. Here belongs the parameters determining the dimensions of the gear structure: the offset, distances from the offset to the planes in which the pitch circles lie; the angles defining the orientation of the above said planes relative to the pitch normal and etc. The variable input data include also those, from which the geometry of the conjugated active tooth surfaces depends: the independent coordinates of the tooth surfaces; their helical parameters; parameters which determine face width of the teeth, etc.

Introducing the main analytical relations, which are based on the chosen approach to the mechano-mathematical modelling.

Here are included solutions of the fundamental tasks of the synthesis upon a pitch contact point and upon a mesh region with the application of the adequate geometric interpretations of the basic equation of meshing, namely [1, 14-17]: the task for the synthesis of pitch circles; the task for the defining the geometry of the active tooth surfaces by their linear and angular characteristics in the pitch contact point; the task for defining the singularity in the pitch contact point (without describing the analytical type of the tooth surfaces); the task for analytically defining of the entire mesh region; formulations of relations, which are used to determine the optimal dimensions and placement of the region of mesh on the active surface and etc.

This principle of organizing the computer design includes also the introduction of geometric and kinematic relations, intended for the reduction of the input parameter sets.

Constructing the complex process for synthesis and design of hyperboloid gear drives. This is realized by defining the separate sub-stages of the synthesis and design in their sequence and interconnection. This principle, applied in the design of each computer program, is directly dependent on the type of functioning, in the elaborated algorithm, complex of criteria. Those criteria determine the preliminary defined quality characteristics of the gear mechanism in dependence of the accepted approach to the mathematical modelling. A distinctive characteristic of the accepted principle for construction of the complex process for synthesis and design is the chosen approach for the estimation of the calculated option of gear drive.

4. Programs for Synthesis of Spiroid and Helicon Gear Drives

The shown considerations for designing of software programs, applicable to the synthesis of hyperboloid mechanisms are also realized in connection with constructing three types' software products for the design of Spiroid gears, which functional relations are shown in Fig. 1 [1]. Each one of the illustrated three main directions, has its own importance. It means that the user can restrict himself to use the results of only one program; to analyze and interpret these results and then after an adequate assessment, to go through the entire process shown in the figure.

Further below, each of the directions, that includes the whole process of synthesis and design of Spiroid gears, will be considered.

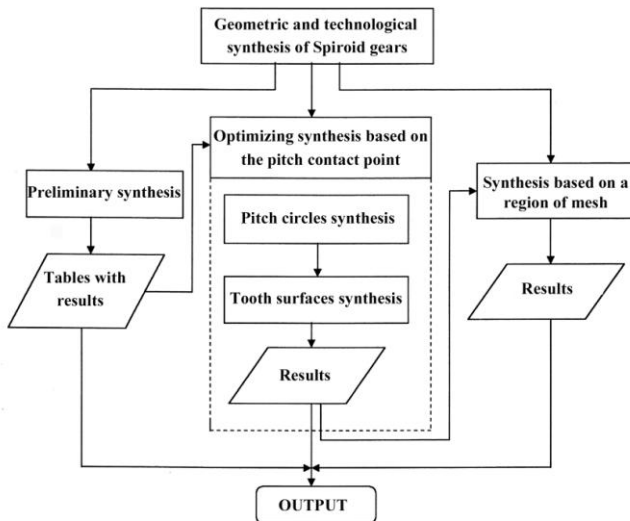


Fig. 1 General block-scheme of the approaches for the synthesis of Spiroid gears

4.1. A Software Program for a Preliminary Synthesis

The aim of the preliminary synthesis is to be calculated the main geometric parameters of the special case of Spiroid gear, when the angle, at which the rotations axes are crossed, is 90° , and pinion is of cylindrical form. With other words, the preliminary synthesis of the Spiroid gears is essentially oriented to the geometric dimensioning (without an optimization) of hyperboloid drive of type Helicon [11, 18].

The mathematical model upon a pitch contact point is in the basis of the program for a preliminary synthesis. It is assumed that this point is placed on the outside cylinder of the Helicon pinion [19].

The input data of the computer program are: gear (velocity) ratio u ; offset a_w ; Z_1 - number of teeth of the pinion depending on the degree of accuracy and the conjugation type of the tooth surfaces; a standard pinion tooth deflection (thinning) - Δb ; set of standard axle modules; coefficients and etc. In the commented program, the possibility of defining the following geometric-technological requirements is taken into consideration when choosing the outer diameter d_{a2} of the gear:

- in the case of the double bearing of the pinion, when one support is located in the field of the offset (in this case the synthesized gear drive is characterized by smaller sliding velocities [18]);
- to ensure the possibility of generating the gear sets, over a wide range of external diameter d_{a2} variations, and with the use of hobbing machines.

The program works for velocity (gear) ratios of up to 100 and offset not exceeding 250 mm. The only optimization ensured by this

program is related to the selection of a standard axle module of the Helicon pinion (the calculated axle module has to belong to the set of standard input modules).

The data for u , Z_1 , a_w , Δb and one of the mentioned geometric-technological conditions is input by the designer at the start of each computational procedure.

Thus, only by inputting the above mentioned input data by the designer, the program calculates and chooses the values of the standard axle module, outer and inner diameters of the pinion and the crown (gear), the width of the toothing of the gear and the length of the generated part of the pinion, the minimum mounting distance of the pinion and etc. The program enables a technological check of the values of the big profile angle of the teeth (corresponding to low-side driving) as well as for obtaining variations of the gear drive for the minimum and maximum possible values of the number of the teeth of the gear (crown).

The organization of the program for a preliminary synthesis is illustrated on Fig. 2 [1].

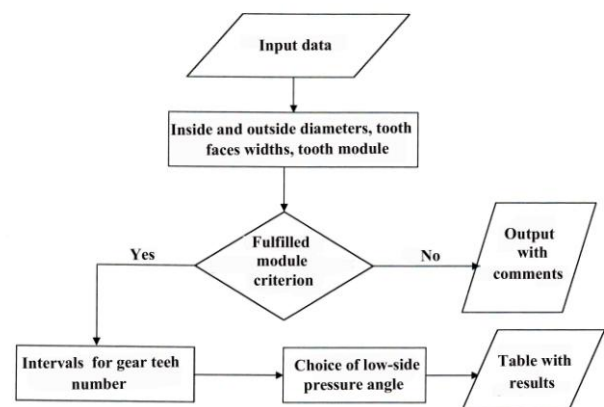


Fig. 2. Scheme of preliminary synthesis program

4.2 Program for Optimization Geometric and Technological Synthesis of Spiroid Gears upon a Pitch Contact Point

This program includes solving of the following tasks [1]:

- Synthesis of geometric pitch circles.
- Synthesis of the active tooth surfaces of the Spiroid pinion and of the cutting tool for generation of the Spiroid crown (Spiroid hob);
- Verification if that the accepted quality criteria of the gear drive are fulfilled.

From the formulation of the defined tasks, it can be seen that the algorithm of this program corresponds to the approach to mathematical modeling for synthesis upon a pitch contact point [1, 14-17]. In this sense, when designing the Spiroid gears, it is of particular importance to select the location of the pitch contact point in the fixed space. The placement of the pitch contact point (as a common point of the pitch circles and conjugated active tooth surfaces) affects on one hand on the common geometry of the designed gear system (overall dimensions of the gear pair) and on the other- on the geometry and proportions of the gears' teeth, as well as on the gears' quality (through the geometric, kinematic and strength characteristics of the conjugated gear pair).

For the purposes of this study, here will be briefly outlined some of the information contained in [1, 14-17], dealing with the geometric nature of the externally contacting geometric pitch circles.

The pair of the pitch circles ($H_1^c : H_2^c$) from the mentioned type are circles H_i^c ($i = 1, 2$), having only one contact point P . Their centers are places on the axes of rotations $i-i$ ($i = 1, 2$) of the movable links, and the corresponding circles are perpendicular to those axes. Mutual placement of the crossed axes is uniquely determined by the angle $\delta = \text{constant}$ between the skewed axes and by the length of their axis – the offset (center distance) $a_w = \text{constant}$. The position of the geometric circles H_i^c ($i = 1, 2$) in respect with the rotations axes $i-i$ ($i = 1, 2$) and with their offset line is defined by four parameters for each circle. The condition for the two circles to have a common contact point is expressed by five independent scalar equations. Hence, the mutual position of two circles is not defined in a unique way. It is a function of 5 independent parameters. The synthesis of geometric pitch circles is preceded by the decision, which five of the eight parameters (for both circles) to be chosen for independent ones and how to choose the intervals for their variation.

For this specific case, these five independent parameters are chosen as: an angle δ between the axes of rotations $i-i$ ($i = 1, 2$); the offset a_w ; the angle δ_1 , which is concluded between H_1^c and the pitch normal $m-m$ (half of the angle at the top of the pitch cone H_1^s of the Spiroid pinion); radius r_1 of H_1^c and the distance a_1 between the offset line of the gear drive and the plane in which H_1^c lies.

The ambiguity of the solution enables the possibility these free parameters to be discreetly changed within certain limits and among many pairs of geometric pitch circles to search for those ones, which parameters ensure that the preliminary defined requirements for the quality of the synthesized gear drives in the vicinity of the pitch contact point are satisfied. The criteria used in the program will be discussed further below.

The program allows choosing the type of conic linear helicoid, applied as active tooth surfaces of the Spiroid pinion: convolute, Archimedean or involute ones. The calculation of the necessary and sufficient geometric and technological parameters for the design of the Spiroid gear pair and cutting instruments are realized for the required type of conic helicoid.

The indicators, which serve to control the quality of the gearing, are essential for the design process. As it has already been mentioned, the dependence on the solution of the task for the synthesis of the pitch circles from concrete free parameters, should be searched among the optimal geometric, kinematic and technological quality characteristics in the vicinity of the pitch contact point. They will be briefly explained.

Basic technological criterion. This is the main criterion related to the technology of elaboration of Spiroid gears. It is related to the reduction of the nomenclature of cutting tools by ensuring the conditions for the design of Spiroid hobs with standard modules (hob parameters are functions of this module). This causes the requirement that the calculated in the pitch contact point module to coincide (with a given exactness) with any of the modules contained in the input set of standard modules.

Criterion controlling the singularity in the pitch contact point. The constructed criterion is analytically described in [1]. The ensuring of the implementation of this criterion helps to reduce the ordinary nodes from the mesh region of the synthesized gear drive. Hence, it leads to the improvement of its loading capacity, of the efficiency and of the durability. It will be reminded, that the elimination of the singularity of first order, by this criterion, is guaranteed in vicinity of the pitch contact point. The optimization,

when using this criterion, is realized by the verification of the analytical dependencies introduced for each of the selected combination of the five independent variables.

Criterion for controlling the transmission angle of the normal force (pressure angle). This criterion provides optimization of gear drive in terms of the transmission of the normal forces from the pinion to the big gear (crown), when the gear mechanism is operated under the conditions of the rotations transformation at low-side driving.

Criterion for controlling the value of the Spiroid pinion spiral angle. By this criterion is carried out the control of the value of the angle in the pitch contact point. Its values have to belong to definite intervals in accordance to the purpose of the designed gear drive.

Here, it should specially be noted, as it is shown in [6], that the choice of the appropriate values for the pressure angle and the Spiroid pinion spiral angle of the longitudinal line of the active tooth surface $\Sigma_1^{(1)}$ in the pitch contact point substantially affect the efficiency of the gear drives. Therefore, by appropriately selecting of the geometric characteristics of the tooth surfaces of the Spiroid pinion, an indirect control of the gear drives' efficiency is achieved. It also should be mentioned, that from the calculated equivalent variants of the synthesized gear mechanisms, from a geometric and technological view point, the program allows to select that one which has the highest value of the coefficient of efficiency for the computational (pitch) contact point.

Criterion related to the durability of the gear drive. It controls the magnitude of the sliding speed at the computational contact point, depending on the chosen material for the toothing of the Spiroid gear - different types of bronze.

Criterion controlling the hydrodynamic conditions of meshing. This optimization aims the accomplishment of the following conditions: the synthesized gear drive has to obtain a maximum as a value of the summed circumferential velocity $|\bar{V}_\Sigma|$ in the pitch contact point and a minimum value of the angle Ω , which $\bar{V}_\Sigma$ concludes with the normal to the contact line in the pitch contact point.

Technological criteria for hobbing. These criteria are related to the choice of the minimum value of the axial (normal) profile angle of the Spiroid hob, in order to provide optimal conditions for hobbing, both in terms of cutting the metal and in relation to the strength characteristics of the cutting elements of the hob and etc.

In number of design cases, some of the initially defined independent parameters could be fixed due to the specific requirements (or example because of requirements for the maximum sizes of the gear drives and the mutual position of the shafts of the gears), which results in reduction of the number of independent variables without limitation to search and find an optimal geometry of the tooth surfaces.

Input parameters of the software program: number of the Spiroid pinion' threads and number of Spiroid gears' spiral teeth; offset; standard pressure angle; type of the conical helicoids (type of the tooth surfaces of the Spiroid pinion); type of the bearing of the gear shafts (on two bearing supports or on a console); frequency of revolution and etc. Keys parameters will take values of 1 or 0 depending on whether a given criterion will be taken in consideration for the synthesis or not. For each of the free parameters, the minimum and maximum values as well as the pitches of variation, are chosen. The independent variation cycles in the computer program are equal to the number of the free parameters.

On Fig. 3 the main block-scheme of the commented above program can be seen. The table of the results consists of: the basic geometric parameters of the Spiroid pinion and of the Spiroid gear, the design parameters of the Spiroid hob, geometric and constructive parameters of the three-link spatial gear mechanism,

parameters related to the quality of meshing at the pitch contact point, such as coefficient of the efficiency of the gear drive, forces acting in the pitch contact point (pole of meshing); loading of the bearing supports, etc.

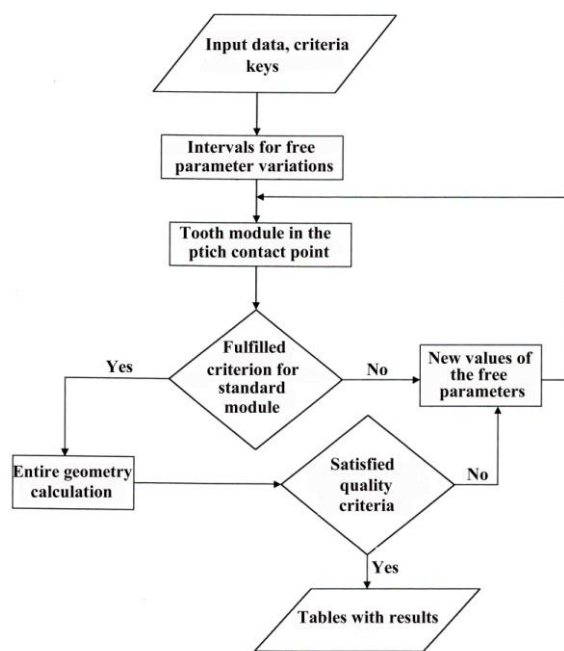


Fig. 3 Program scheme for an optimization synthesis of a Spiroid gear pair

It should be mentioned that, when the bearing supports are calculated, the distances between them and the pitch circles in real dimensions mm or dimensionless - as the ratio of the distances to the diameter of the pitch circle of the Spiroid gear are given. In the program, all forces, that act in the pitch contact point and at the bearing supports are dimensionless in relation to the peripheral force, acting on the Spiroid gear. If in the input of the program, the torque on the gear shaft or a torque on the pinion shaft is given, then this peripheral force has a concrete value and all forces and loads are calculated in $[N]$. Analogically, the sliding velocity is calculated as the ratio with the magnitude of the Spiroid pinion' angular velocity. If in the input, the numbers of pinion' revolutions per minute are given, then the sliding velocity is obtained in $[m/s]$.

4.3. A Computer Program for Synthesis upon a Mesh Region

This program for synthesis of Spiroid gear, based on the commented in [1] mathematical model, represents a logical final stage of the organized complex process of the synthesis for this class high-reduction spatial gear mechanisms. This approach is applied in the following two cases:

When synthesizing a gear drive for which elaboration it has to be used the existing instrumental equipment (in particular an existing Spiroid (Helicon) hob).

In this case, in the designing of the gear drive, it is not possible to define an optimally positioned pitch contact point. In a number of cases, the existing instrumental equipment is suitable for generating the gear mechanism, for which it is not possible to define a pitch contact point in the mesh region. In this case, the constructively defined region of mesh is tested for the singularity of first and second order. In case, when the constructively defined gear drive contains an unacceptable amount of undercutting points and ordinary nodes, a change of the location of the mesh region in the fixed space is used.

When types of Spiroid (Helicon) reductor are synthesized. The type reducers represents a family of products, which are elaborated on the basis of the same: corpus details; gear blanks;

purchased groups of assembled details and parts. A rational and applied approach by the authors of this research is as follows:

At the beginning a basic model is designed, the synthesis of which is realized by using the computer program for synthesis upon a pitch contact point. The rest of the elements of the family of the created type are designed, by using adequate analytical dependencies [1], that ensure design of gear drives, so the ordinary nodes (contact points) are completely eliminated from their mesh region. Thus, the selected types of gear set, together with the base gear drives, are tested for the presence of singularity of second order in the whole region of mesh.

Through the created software program for the synthesis upon a mesh region, the entire region of mesh is checked in order to determine the type and amount of singularity. For the discrete variation of the meshing parameter φ_1 (an angle of rotation of the Spiroid pinion), the contact lines on the active tooth surfaces $\Sigma_1^{(j)}$ of the pinion are defined as a function of one of their curvilinear coordinates $u^{(j)}$, $g^{(j)}$. The parameter φ_1 varies in the interval $k\pi$, as the real number k depends on the geometric-kinematic characteristics of the synthesized gear drive, as well as from the goals of the study. The curvilinear coordinate $g^{(j)}$ varies in an interval, which depends on the length of the generated part of the pinion - L_1 and its axial module m , i.e. $g^{(j)} \in [-L_1/m, +L_1/m]$. The interval is divided by the number of points chosen by the designer. For each of these values, it is firstly checked whether the curvilinear coordinates belong to the mesh region, and then checking if that these curvilinear coordinates satisfy the analytical dependencies that indicate the presence of ordinary nodes and undercutting points. The finding is noted with the relevant text in the software program.

The input data in the program are [1]: number of teeth of the Spiroid pinion Z_1 , number of teeth of the Spiroid crown Z_2 , offset a_w , a distance from the offset line to the origin of the coordinate system $S_p(O_p, x_p, y_p, z_p)$, in which the equations of conic linear helicoids, profile angle, the length of the generated part of the Spiroid pinion, the module of the tooth surfaces are written. These data can be obtained from the synthesis of the base model upon a pitch contact point. In this case after the check with the program for synthesis upon a mesh region, the base gear drive is synthesized upon the so-called **combined approach**. The input of the program can be set from the constructive considerations, depending on the available instrumental and technological equipment. In this case, the synthesis upon mesh region is transformed into evaluating of the constructively chosen parameters.

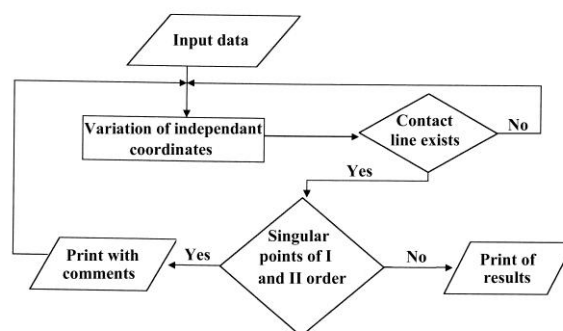


Fig. 4. Computer program for synthesis upon mesh region

The computer program is organized upon following Fig. 4 [1]. In the appendices to the publication [14], exemplary calculation

results are presented from the execution of the computer programs for synthesis of Spiroid gear sets.

5. Conclusion

The current work presents an adequate review of the created methodology and the approaches to the synthesis and design of hyperboloid gear mechanisms with face mated gears. It is an actualized and added presentation of the illustrated in [20] strategy for synthesis of gear drives with crossed axes.

The main features of its content are as follows:

- A classification of the types of specialized software applicable in the analysis, synthesis and calculation of gear drives is realized.
- The principles, applied by the authors, for organization of the process of the synthesis and design of hyperboloid gear mechanisms, are briefly defined (described).
- The essence of the elaborated computer programs for preliminary synthesis, optimization synthesis upon a pitch contact point and upon a region of mesh, applicable in the design of Spiroid and Helicon gears, are summarized.

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PROCEDURE FOR DETERMINATION OF THE CALIBRATION CURVE OF THE MEASUREMENT DEVICE USING THE METHOD OF LINEAR REGRESSION

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Abstract: The result of each measurement consists big or small measurement uncertainty. Many sources of uncertainty, especially in industry measurements, are not well studied and require further researchment. Therefore, it is important to have good knowledge about measurement uncertainty in order to minimize as much as possible.

The results of performed analyse and the study of the opportunities for application of the method of linear regression for determination the calibration curve of the measurement device are presented in this paper. The researchment refers to the assessment of the measurement uncertainty type A and B, as well as the combined and extended uncertainty of the measurement result.

By the fulfillment of the assumption for realization of an engineering experiment, with possession of mathematical, technical and practical experience of the operator and the computer supported analytical process, the calibration curve of the measurement device is determined, as well as the extended uncertainty of the measurement result. In this way, a stable measurement process will be achieved, a consistent measurement result, with an increased level of confidence.

KEY WORDS: measurement uncertainty, linear regression, calibration curve.

1. Introduction

As a consequence of the action of numerous factors which cannot be controlled, the conditions for experiment realization are constantly changed. Therefore, an uncertainty exists at the result of each measurement which may occur in any segment of the measurement process, as well as in the links between segments.

The result of the engineering experiment that is subject to analysis, contains measurement uncertainty which is the summary - consists of two components type A and B. Their relationship depends on the used measurement device as well as the conditions in which the experiment is realized.

Measurement process testing is usually realized by applying of models. Generally, the model is used to assess the original system or to make conclusions about its behaviour. We use a deterministic model for the analysis in our case where the seria of measurements can be conceptualized as a measurement process, where the true value of the measurement size x_t is an input of the measurement process and the process produces the measured value x_m , as its output (Fig. 1).

Mathematic model of the measurement process [1] will be:

$$x_m = x_t + f(x_t) + R \quad \text{or} \quad x_m - x_t = f(x_t) + R \quad (1)$$

where:

x_m – measured value of the measurement size,

x_t – true value of the measurement size,

$f(x_t)$ – measurement uncertainty function – calibration curve (measurement uncertainty type B),

R – residuals (measurement uncertainty type A).

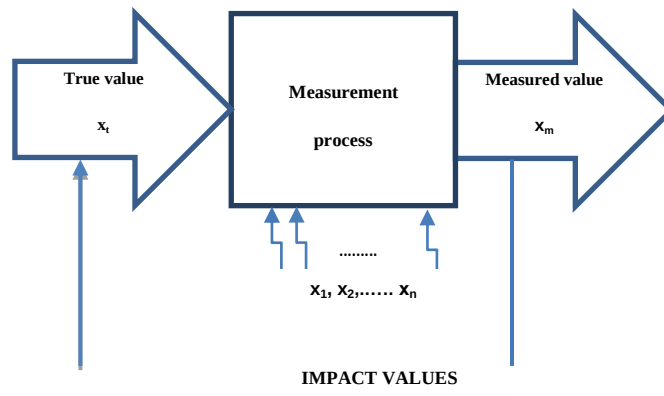


Fig. 1 Schematic view of the model for estimation of the measurement uncertainty [2]

According to the probability for occurrence, the total measurement uncertainty is divided into two components: [2]

Measurement uncertainty type A – $U(a)$: Factors which contribution to the measurement uncertainty can be assessed using the statistical distribution of measurement results. The measurement uncertainty type A is displayed as scattering of the measurement result. In the series of measurements when measuring the same value of the measurement size, different measurement results are always obtained. In order to determine the average and the standard deviation of each point of the measuring area, several measurements are realized.

Measurement uncertainty type B – U(b): Factors whose contribution is assessed in other ways, by other means and in relation to other laboratories (with plans for taking a representative sample, by experience e.c.t.).

Function of the measurement uncertainty (measurement uncertainty type B) according to changes dependent on the measurement size, is divided into three components: additive, multiplicative and nonlinear errors [3]. Moreover, additive component of the total uncertainty does not depend on the measurement size. Examples of sources of additive error: measuring system displacement from the zero position, drift e.c.t. Multiplicative component is changed in proportion to the measurement size. Sources of a multiplicative component are factors which have influence to the measurement devices parameters. Nonlinear component of measurement uncertainty includes members of the second or higher exponent of the measurement size.

In addition to the basic components of the measurement uncertainty, it is also defined [2]:

- Combined measurement uncertainty U_C

$$U_C = \sqrt{U_A^2 + U_B^2} \quad (2)$$

- Extended measurement uncertainty U :

$$U = k \cdot U_C \quad (3)$$

Where k is an extension factor. The value of the factor is chosen depending on the confidence level of the measurement result.

Table 1: Extension factor (according addition of GUM) [4]

Confidence level P%	68,27	90	95	95,45	99	99,73
Extension factor k	1	1,645	1,960	2	2,576	3

Calibration [5] is a set of procedures that establishes a relationship between the values of the size indicated by the measurement device or measurement system, or the values representing a materialized measure or reference material with appropriate values of the achieved etalon, under certain conditions. According to [6], the calibration of the measurement device can be expressed by an uncertainty statement, shown as calibration function, calibration curve or calibration table.

The aim of this paper is to present in a systematic way the procedure for experimental statistical identification of the calibration curve of the measurement device and its display as a polynomial function using the method of linear regression.

2. Preparation, planning and realization of the engineer experiment

Calibration curve of the measurement device $f(x_i)$ and the standard deviation of the measurement uncertainty type A, σ^2 , will be determined through an analytical process, by realization of a certain number of measurements n , through the measurement area of the device, in a pre-specified measurement conditions. Series of true values x_i are in fact values of referent measuring sizes (etalons). On that way is created a set of data with n values of referent (x_i) and the calibration points - averages of the measured (x_m) values.

Several measurements are realized in order to estimate the impact of measurement uncertainty type A in each calibration curve. Average of a series of measurements is considered as representative of the behavior of the measurement device in the calibration point, i.e. by fulfilled conditions for normality of the measured values and homogeneity of the variations can be considered that the calibration point - the average of the measurements contains only the measurement uncertainty type B. Only one measurement in each calibration point is usually realized in the automatic regulation process.

If the calibration curve $f(x_i)$ contains p parameters, the number of points (pairs) of calibration base should be at least $n = p + 1$, in order to calculate the required elements in the analysis. That is the minimum required number of measurements (pairs of values). More number of measurements is better in a statistical point of view, but this is also a question of time, effort and resources used in the creation of the calibration database.

While determining the mathematical form of the calibration curve $f(x_i)$ (determination of polynomial function degree) should be taken into account that has a critical impact on the quality of the measurement uncertainty assessment. Shape of the calibration curve $f(x_i)$ depends on the nature of the measurement system and the specific reasons for uncertainty. Higher order parametric function better reflects the relation in the set of values (reference and measured). This can be seen by the correlation coefficient, which is higher, but also is more practical to not work with more complex function than is necessary. For optimal realization of such an experiment, the function of the calibration curve $f(x_i)$ should always be some compromise between theory and practice.

Processing of the measurement results and determination of the calibration curve for the experimental measurement equipment, is done through realization of specific iterative procedure:

1) As a first step in the process of determination the calibration points from the set of measured values, harsh errors are identified and eliminated. Harsh errors are clearly perceptible errors that significantly exceed the boundaries of the analyzed measurement process. After identification of harsh error using the Grabs method [3], this error is excluded from the measurement result.

2) In order the average of the measured values at each calibration point to be considered as relevant for further processing and performing valid conclusions, it is necessary to perform testing for meeting the requirements for normality of the measured results and homogeneity of variations. This is done by testing hypotheses using multiple tests:

- for testing of normality: χ - test, λ - test, Anderson – Darling, Ryan – Joiner, Kolmogorov – Smirnov test, by using of probability diagram or by p - value analyse e.c.t.
- for testing of homogeneity : z – test, Student t – test, Fisher F – test e.c.t.

3) If the conditions for normality of the measurement results and homogeneity of variation are fulfilled, the averages of the series of measurements are calculated. On that way, the calibration points are obtained, for which can be considered that contain only measurement uncertainty type B.

4) Shape of the calibration curve $f(x_i)$ will be determined by visual testing of the diagram, where on the y-axis will be calibration points - averages ($x_m - x_i$), and on the x-axis real (etalon) values x_i .

5) Regression analysis will be made with the assumed $f(x_i)$. The correlation coefficient will be an indicator of the quality of the selection of the calibration curve. Higher correlation coefficient means greater connectivity of measured values and reference values. In case [1] the correlation coefficient is less than 80%, the selected calibration curve $f(x_i)$ is not appropriate for the set of values and selection of new calibration curve and repetition of the whole procedure is needed. The function obtained on that way is good and the standard deviation of measurement uncertainty type A can be calculated.

3. Results

In order to illustrate the above procedure an experiment with a relatively small number of measurements is realized (in our example 11 calibration points with 5 measurements at each point – Table 2). Basically, this means determining the calibration curve of the measurement device through a small number of measurements, which results on optimization of the time spent, engaged resources and costs for experiment realization.

• Harsk errors identification and elimination

In accordance to Grabs criteria [3] for cutoff value 10% (process measurements) $G_{crit}=1,77$. Since all values of G_{max} and G_{min} are smaller than G_{crit} (maximal value $G=1,63$ in the fourth measurement point), the hypothesis of existence of a harsh error in the set of realized measurements is rejected.

- Checking the normality and homogeneity of variation in the calibration database,

Checking the normality of distribution of the obtained results for each calibration point will be made by testing the hypotheses using Kolmogorov-Smirnov test and by p - value analyse, for confidence level 90% (process measurements). This kind of checking the normal distribution of the measurement results is realized for each calibration point.

Although it relates to series with a relatively small number of measurements (five measurements at each point), all series of measurements show a high degree of adherence to the normal distribution (the smallest value $p = 0.142$ at measurement point 4, but still greater than the cutoff value 10%).

Kolmogorov-Smirnov test with allowable error of 1%, for confidence level 90% (process measurements), value is calculated by the following formula [7] :

$$TEST = \frac{1,22}{\sqrt{n}} \quad (4)$$

In our example, $n = 5$, and for the test value, the obtained value is 0.55. In our example, the largest (in absolute value) $D_{max}=0,3$ is achieved in the measurement point number 4, which is less than the value of TEST. Based on this, it can be concluded that the measurement results for the seventh calibration point do not deviate significantly from the normal distribution.

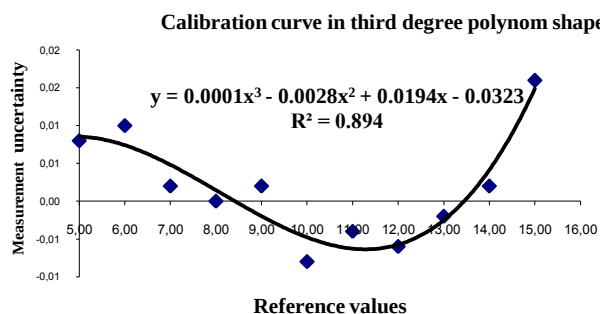
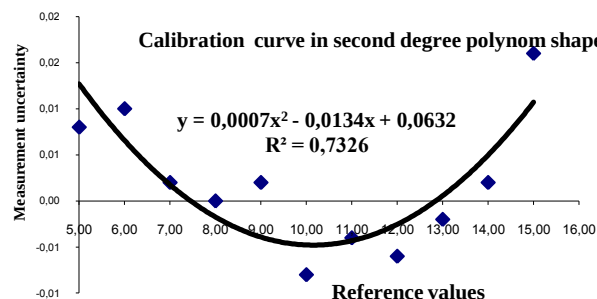
Homogeneity of the variations in the database will be tested by the application of Fishers F-test. The basic and alternative hypothesis is are:

H_0 : Two samples belong to the same basic set of values.

H_A : Two samples do not belong to the same basic set of values.

We calculate F-test indicator. The subject of analysis in our case will be calibration points number 6 and 11 (the point with the smallest and biggest standard deviation). The first sample (item 6) $n = 5$, $\bar{X}_6=9,992$ $S_6^2=0,00007056$. The second sample (item 11) $n = 5$, $\bar{X}_{11}=15,016$ $S_{11}^2=0,00027889$. The value of F is 3.95. Value F_{crit} ([8] p. 563) for $q = 0,05$ $F_{crit}=6,33$ while for $q=0,01$ $F_{crit}=15,98$. Because in both cases, $F < F_{crit}$, we can confirm the reliability of the base hypothesis.

- Selection of the calibration curve function of the measurement device



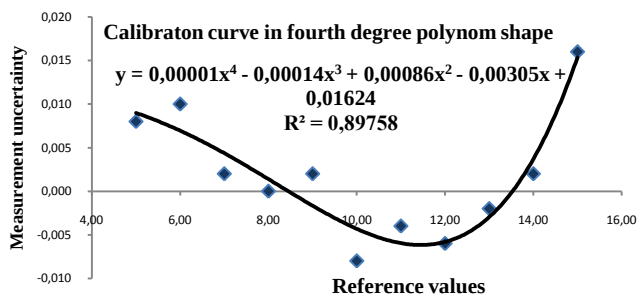


Fig. 2 Calibration curves in second, third and fourth degree polynomial shape

Due to the significantly bigger correlation (Fig. 2) coefficient at the fourth degree polynomial (which associates to bigger connectivity of the measurement uncertainty, i.e. the measured values with reference values), the fourth degree polynomial shape is adopted as a calibration curve for the tested measurement device. It can be noticed that increasing the degree of polynomial function does not significantly increase the correlation coefficient and further increasing the degree of polynomial function would be irrational.

- *Checking the normality of the residuals*

Checking the normality of distribution of the residuals for the calibration curve will be made by testing the hypotheses, using the Kolmogorov-Smirnov test and by p - value analyse (Fig. 3), for confidence level 90% (process measurements). The value of the test indicator, according to (4) is 0.367.

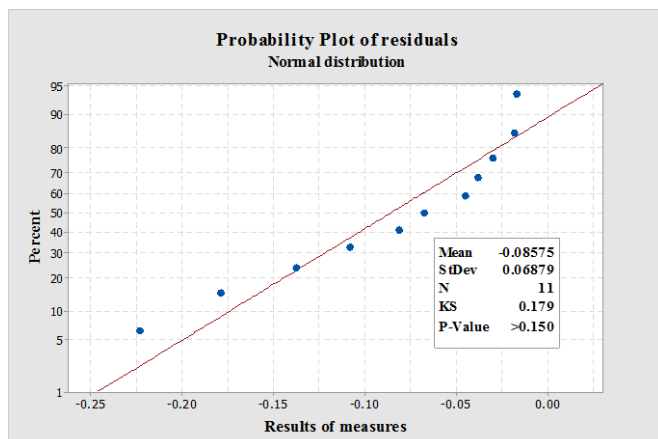


Fig. 3 Analysis of the residuals normality by using a probability diagram

$D_{\max} = 0,179$ which is less than the test value and $p > 0,150$, which means that the basic hypothesis of normal distribution of the residuals can be accepted.

- *Calculation of the standard deviation of the residuals*

Using a calibration curve and the calculated standard deviation of the set of residuals in relation with the calibration curve function of the tested measurement device (in our example $\sigma = 0,0687$), the estimated value of the calibration point and the confidence interval

around the estimated value can be determined. For example, the measured value $x_m = 14,80$. By replacing this value in the following function $x_m - x_t = 0,00012x_t^3 - 0,00283x_t^2 + 0,01939x_t - 0,03230$, and solving the function it is obtained $x_t = 13,10$, and for the confidence interval $\pm 1,645\sigma$ ($P=90\%$ probability of production measurements distribution) it is obtained 0,113. That means that the estimated value of the calibration point will be located in the interval $x_t = 14,63 \pm 0,113$ $\kappa = 1,645$, $P = 90\%$.

4. Conclusion

The calibration curve of the tested measurement device with controlled costs for the work of the laboratory is determined by optimal realization of such an experiment. The implementation of this method allows accrual determination of the estimated value and the confidence interval, correction of the measurement results, as well as control the accuracy of the measurement device during its exploitation and determination of the moment for starting an action for improvement the metrological characteristics of measurement device. Due to its simplicity the displayed method is especially efficient and practice in its application in conditions different from the laboratory conditions.

The determination of the calibration curve of the measurement device, above all, must be in function of customer satisfaction for the services of the measurement device, especially the customer requirements regarding the reliability and confidence in the results of the realized measurement.

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TABLE 2: *Measured values at measurements realized in whole measuring area of the measurement device*

	REF.	FIRST	SECOND	THIRD	FOURTH	FIFTH		MEASURE	STANDARD
NB.	VALUE	MEASURE	MEASURE	MEASURE	MEASURE	MEASURE	AVERAGE	ERROR	DEVIATION
1	5,00	5,01	5,00	5,01	5,00	5,02	5,008	0,008	0,0084
2	6,00	6,00	6,02	6,01	6,00	6,02	6,010	0,010	0,01
3	7,00	6,99	7,00	7,01	7,01	7,00	7,002	0,002	0,0084
4	8,00	8,01	8,00	8,01	8,00	7,98	8,000	0,000	0,0123
5	9,00	9,00	8,98	9,00	9,02	9,01	9,002	0,002	0,0148
6	10,00	9,99	10,00	9,98	10,00	9,99	9,992	-0,008	0,0084
7	11,00	10,98	11,00	10,98	11,00	11,02	10,996	-0,004	0,0167
8	12,00	11,98	12,01	11,98	12,00	12,00	11,994	-0,006	0,0134
9	13,00	12,98	13,00	13,00	13,00	12,99	12,998	-0,002	0,0148
10	14,00	14,00	14,01	13,98	14,00	14,02	14,002	0,002	0,0148
11	15,00	15,00	15,02	15,02	15,00	15,04	15,016	0,016	0,0167

SIMULATION TESTS OF THE BRAKING PROCESS OF AN AGRICULTURAL TRACTOR

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Abstract: The agricultural industry is growing very quickly. Large areas of fields make tractors work at higher speeds, with which they also move on public roads. Except acceleration, the agricultural tractor must also be stopped. There are emergency situations on the roads that require sudden braking, and therefore a quick stop of the vehicle. Drum brakes are used in most farm tractors. They contain a friction pair (shoes and drum) which as a result of cooperation changes the kinetic energy of the motion into thermal energy. Unfortunately, this creates a huge amount of this energy. This can cause overheating of the friction lining, which can lead to permanent damage. This paper presents the results of simulation tests of the heating process during single emergency braking. It has been shown that in some situations the temperature can reach a higher value than that which is safe for friction material components.

Keywords: BRAKES, AGRICULTURAL TRACTOR, FRICTION, HEATING PROCESS, COOLING PROCESS

1. Introduction

Agricultural tractors have been a must for every farm for many years. They are used mostly in agricultural works, but not only. The specificity of agricultural production means that the acreage of processed fields is increasing. Therefore, the number of kilometers covered increases naturally. Farmers, in addition to cultivation and harvesting, must deliver their crops to collection points. These are usually several or even over a dozen kilometers away. Tractors are also used to overcome them. In combination with trailers, they are a sufficient means of transport [1].

The specificity of today's times which results in a dizzying pace of life means that no one can afford to waste time. This situation is being met by producers of agricultural equipment who are offering bigger, stronger and faster tractors. Constructions sold today easily reach speeds of 50 km/h together with a loaded trailer (or even several) [2].

However, accelerating such a large mass (reaching several dozen tons) is not all the problems. The real challenge is quick and efficient braking. On public roads there are often emergency situations in which a sudden loss of speed is required [3, 4]. In the event of a breakdown, there is a real danger to the health or even life of road users. It is therefore important that the components of the braking system work quickly and reliably throughout the entire service life [5].

Drum-type friction brakes are used in most agricultural tractors [6]. The braking process consists in pressing the linings against the drum rotating together with the wheel. The resulting friction effectively changes the kinetic energy of a moving tractor into thermal energy. It is then returned to the environment due to conduction, convection and radiation. However, it is not always possible to dissipate heat energy quickly enough. In the case of a sudden braking, it happens that the temperature of the friction elements reaches up to several hundred degrees. For some materials, this means a loss of stability and the formation of so-called fade phenomena [7, 8]. It involves sublimation of the matrix of the friction material, which creates a gas cushion between the friction couple. This is associated with a loss of braking capability. **This work is aimed at checking whether there is a risk of fading during emergency braking in modern, fast agricultural tractors.**

2. Mathematical model of braking process

The force of the braking process may be described as a negative derivative of the vehicle's kinetic energy [8]:

$$P = -\frac{d}{dt} \left(\frac{mv^2}{2} \right) \quad (1)$$

or

$$P = -mR^2 \omega(t) \alpha \quad (2)$$

where: m - vehicle's mass, v - speed of the vehicle, R - dynamic radius of the road wheel, ω - angular velocity of the wheel, t - time, α - angular deceleration. Assuming that the braking deceleration is constant, we may say that:

$$\omega(t) = \omega_0 + \alpha t \quad (3)$$

A simple stability analysis of the vehicle made demonstrated that approximately 60% of the total braking force comes from the front wheels (roughly 15% per each of the four brake shoes on the front axis) [1]. It has been assumed that each of the brake pads generates identical braking force. The relation between the braking force of one wheel and the complete vehicle is as follows:

$$F_b = \frac{100\% \cdot F_{fb}}{2 \cdot 15\%} \approx 3.33 \cdot F_{fb} \quad (4)$$

where: F_{fb} - braking force generated by one front wheel, F_b - total braking force of the vehicle. On that basis, the braking force of the vehicle may be described as [9]:

$$P = -3.33 \iint f_f \cdot dA \cdot v_d \quad (5)$$

where: f_f - frictional force per surface unit, $v_d = \omega(t) \cdot r$ - brake drum linear speed at radius r , A - disc and shoe contact surface area (in our case $A = 0.01 \text{ m}^2$). The braking force is also expressed as the relation [9]:

$$P = f_f(t) \cdot \omega(t) \iint r_m \cdot dA \quad (6)$$

where: r_m - distance from the shoe's centre to the disc rotation axis (in our case $r_m = r$). Comparison of the two equations makes it possible to determine the f_f coefficient:

$$f_f = -\frac{mR^2 \alpha}{3.33 r_m A} \quad (7)$$

Assuming that the vehicle's deceleration occurs only through the action of the brake disc and pad, the heat flux may be designated using the equation [10]:

$$q(r, t) = -f_f \cdot v_d(r, t) \quad (8)$$

or by substituting:

$$q(r, t) = -\frac{mR^2 \alpha}{3.33 r_m A} r (\omega_0 + \alpha t) \quad (9)$$

Amontons - Coulomb friction law [11] makes it possible to determine the contact pressure, which in the analyzed case is as follows:

$$p = \frac{P}{\mu \cdot v} \quad (10)$$

where: μ - coefficient of friction between the disc and pad. Also, the study takes into account the heat exchange occurring between the disc and pad, expressed as the following relation [12]:

$$\rho \cdot C_p \frac{\partial T}{\partial t} + \nabla \cdot (-k \cdot \nabla T) = Q - \rho \cdot C_p \cdot u \cdot \nabla T \quad (11)$$

where: k - thermal conductivity, C_p - thermal capacity, u - heat flux rate, Q - heating power per density unit, ρ - density, T - temperature. During the action of the brakes, the following amount of heat is released through convection and radiation [9]:

$$q_d = -h(T - T_r) - \varepsilon \sigma (T^4 - T_r^4) \quad (12)$$

where: h - convection coefficient, $T - T_r$ - temperature different between the friction material and ambient temperature, ε - emissivity of material, σ - Stefan-Boltzmann constant. The relation between the convention coefficient and the speed of the vehicle is described as follows [9]:

$$h = \frac{0.037k}{l} \left(\frac{\rho \cdot l \cdot v}{\mu} \right)^{0.8} \cdot \left(\frac{C_p \cdot \mu}{k} \right)^{0.33} \quad (13)$$

where: l - disc diameter, μ - viscosity.

3. Object and methodology of research

The object of research was CAD models of the braking system parts (Fig. 1) from a popular agricultural tractor in Europe (linings and drum). The most important technical specifications of the tractor are summarized in table 1.

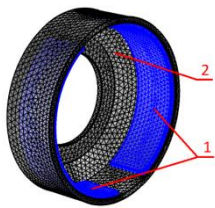


Fig. 1 CAD model used in FEM testing together with the mesh: 1- brake linings, 2- drum.

Table 1: Mechanical properties of selected powder materials

Description	Symbol	Value
Wheelbase	c	2925mm
Rear axle width	b	2602mm
Center of gravity distance from the front axle	c1	1345mm
Center of gravity distance from the rear center of the left tire	b1	1301mm
Height of the center of gravity	h	910mm
Total mass	m	16000kg
Maximum speed	$V_{\max}$	50km/h
Maximum slope angle	ϕ	20°

The developed model has been simplified relative to the real parts of the tractor. The simplification concerned geometry (shapes and fragments that did not have a significant impact on the heating process were removed) and it was aimed at avoiding unnecessary compaction of the mesh. Ultimately, the grid consisted of nearly 6,000 elements, which gave over 27,000 degrees of freedom. Comsol Multiphysics 4.4 software was used for simulation tests. The necessary material data, i.e. density, thermal conductivity and thermal capacity of the friction pair were determined experimentally. A summary of the above data is shown in Table 2. In addition, the friction coefficient of friction, which is 0.36, was determined.

Table 2: Technical data of friction materials.

Shoe lining	Thermal conductivity	140 [W/(m*k)]
	Density	2600 [kg/m ³]

Drum	Heat capacity at constant pressure	1080 [J/(kg*K)]
	Thermal conductivity	49 [W/(m*k)]
	Density	7850 [kg/m ³]
	Heat capacity at constant pressure	484 [J/(kg*K)]

During the tests it was assumed that: braking takes place without slip, the tire adhesion coefficient to the ground is 1.0 (this leads to a traffic delay of 9.81m / s²), the ambient air temperature is 25°C, the invariability of the friction coefficient of the linings against the drum (in fact it changes with temperature), contact pressure that is constant and even for both linings, uniformity of the material and contact with the entire surface, no influence of external factors (e.g. unevenness of the road, air resistance, operation of the vehicle suspension). The initial speed of the vehicle was set at 50km / h.

4. Results and discussion

In the research, the first 10 seconds counted from the start of was analyzed. The study step was 0.05s. The temperature was measured at the central contact point at a depth of 0.2 mm both in the drum and in the lining. Temperature profiles are shown in figure 2 (lining temperature) and figure 3 (drum temperature).

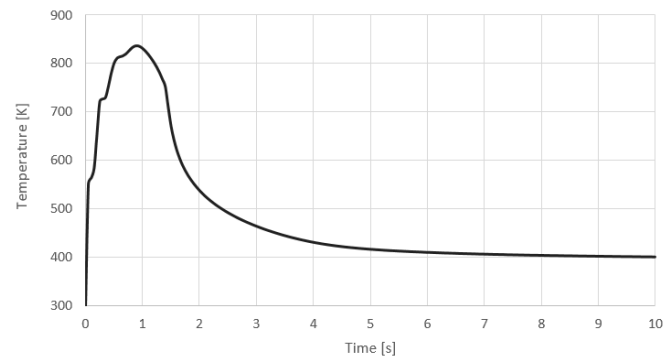


Fig. 2 Graph of temperature of agricultural tractor brake lining during emergency braking.

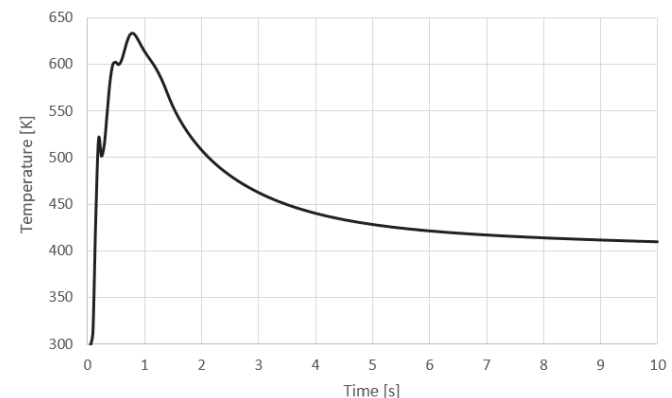


Fig. 3 Graph of temperature of agricultural tractor brake drum during emergency braking.

Analyzing the graphs above, it can be seen that there is a very rapid temperature increase, especially in the initial braking phase. The friction material of the brake linings heats up more, reaching 835.9K in just 0.9s. The drum reaches its maximum temperature (633.15K) 0.1s earlier, 0.8s from the start of braking. The characteristic sharp lines result from the transfer of thermal energy from one material to another. The probable cause is seen in the different properties of both materials, especially the different heat capacity and thermal conductivity. The above conclusion, as well as the correctness of performed simulations, are confirmed by two

successive charts: thermal energy produced in the braking process (fig. 4) and given to the nearest environment (fig. 5). Both lines have a "smooth" course, so it can be assumed that the simulation went correctly.

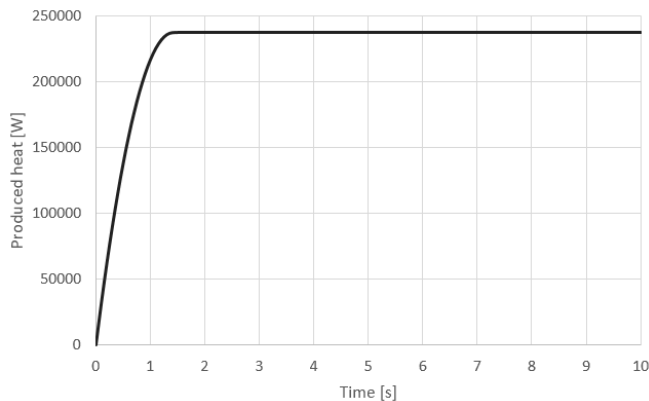


Fig. 4 Heat produced in the process of braking the agricultural tractor.

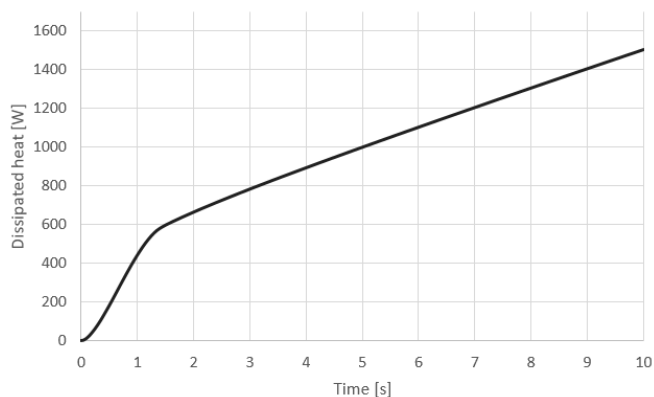


Fig. 5 Heat dissipated as a result of convection and radiation in the braking process of an agricultural tractor and immediately after it.

Analyzing the obtained data, it can be stated that 237.28kW was produced in one of the front wheels during braking, while just over 1.5kW was lost within 10 seconds of starting braking due to convection and radiation. This means that the cooling process is much slower than heating and the brakes remain "hot" for a long time after stopping. In the event that there would be a need for emergency braking in the near future, the risk of fade would increase significantly.

5. Summary and conclusions

This paper presents a mathematical model of the agricultural tractor braking process and simulation tests carried out on its basis. It was found that:

1. Emergency braking of an agricultural tractor with a total weight of 16000kg from a speed of 50km / h takes about 1.4s;
2. 237.28kW of thermal energy is generated as a result of friction generated in the brakes;
3. The brake lining heats up to 835.9K during 0.9s from the beginning of braking; after this time the temperature drops because the vehicle speed is already low, and the heating process is slower than cooling;
4. The drum heats up to 633.15K in 0.8s, followed by cooling for the same reason as in the linings;
5. Due to the fact that the friction material is a composite material whose matrix is usually a resin with a maximum allowable operating temperature of 500-800K [13, 14] there is a real risk of fade.

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РАЗВИТИЕ И ТЕНДЕНЦИИ ПРИ МЕБЕЛИТЕ С АКУСТИЧНИ ХАРАКТЕРИСТИКИ.

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Резюме: В докладът се представя развитието на мебели с допълнителна функционалност, свързана с акустични характеристики. В последните години все повече се обръща внимание на завишеният звуков фон в работни и обществени пространства. Тази този шум доказано води до понижена трудоспособност. От ЕС има множество обновявани всяка година предписания за предприемане на мерки за понижаването му. Един от начините са шумопоглъщаща обработка на помещенията. Естествената нужда от функционалност довежда до конструирането на мебели с допълнителни шумопоглъщащи свойства. Все по-популярни на пазара от мебелните фирми, се представят разнообразни решения, конструкция, материали и дизайнерски решения.

Ключови думи: ЗВУКОПОГЛЪЩАНЕ, ДИЗАЙН, АКУСТИКА, АКУСТИЧНИ МЕБЕЛИ, ЗВУКОВ ФОН, ШУМ

1. Въведение

В съвременното шумът е все по-голям фактор като източник на негативни реакции върху човешката физиология. Ако живеем в постоянна шумна среда, ще бъдем перманентно уморени и нетрудоспособни. Поради тази причина трябва да се обръща внимание на шумовия фон не само в работната среда, но и в останалата част от ежедневието. Шумът е нежелан или вредоносен звук. Всеки ден милиони служители в Европа са изложени на шум по време на работа и рисковете, които това може да доведе по данни 7% страдат от трудности със слуха, свързани с работата. Проблемът със слуха, причинен от шума, е една от най-често съобщаваните професионални заболявания в ЕС. Високите нива на шум затрудняват слушането и общуването на персонала, което може да повиши вероятността от злополуки. Стресът, свързан с работата (в който шумът може да бъде фактор), може да доведе до проблеми.

В офис пространствата фоновият шум също е със завишени нива. Работните места в офиса, в които се събира, обработва, съхранява и съобщава информация, могат да бъдат намалени в много области, например административни офиси, база данни, дизайнерски офиси и офиси за покупко-продажби.

Работата се подпомага от модерна комуникационна технология, превръщаща повечето работни места в работни станции. За разлика от фабриките, където избягването на увреждания на слуха е от първостепенно значение, в офисите намаляването на нивото на звуковото налягане не е единствен приоритет. Въпросът е да се разгледа структурата на шума (звука), ако искаме да намалим неблагоприятните ефекти от него върху способността ни да се концентрираме и стреса, който причинява. Освен в тези сфери на работа проблем с шума има и в заведенията за хранене, училища и детски градини, както и в дома. Често се налага човек да върши работа в къщи, за целта му трябва тишина и възможност за концентрация, но не всеки интериор е съобразен с това. Често се случва членовете на семейството забавлявайки се да създават шум, както и работата на телевизор или звукова уредба. Все повече дизайнери дори и в интериора на дома започват да се съобразяват с акустичната обработка на помещенията, което може до някаква степен да намали напрежението и стреса не само в работа, но и в къщи.

2. Съвременни тенденции в производството на мебели с акустични характеристики

Акустиката е актуална тема на последните мебелни изложения в Европа и у нас, като дизайнерите и марките предлагат продукти, които да намалят фоновия шум. На много мебелни изложения се популяризира производството на мебели с акустични характеристики. Те са насочени към изолация на човека, докато почива от околната среда и най-вече от шума. На тези изложения се разполагат продукти по-разнообразни от всякога, като обичайните звукопоглъщащи стенини панели се

присъединяват от тапицирани екрани на работния плот за работа с компютър, губещи кабинки за стена за телефонни разговори и дори преградни куполовидни форми, предназначени да висят точно над главите на работниците. Последните тенденции са за разработка на все повече възможности в мебелния и акустичния дизайн. Повече фирми предлагат и работни места за офиса, но и често се срещат мебели, проектирани за фойета и обществени места, които да помагат за спокойствието и релаксацията на човек. Те имат модерен и разнообразен дизайн. Освен столове и паравани към дивани се проектират подобни на беседки пространства за по-голям брой хора в различна от работната среда. "Намаляването на шума изглежда е основна загриженост в скандинавските интериори в наши дни, което едва ли е изненадващо, тъй като дървените подове и суровите повърхности, които са синоним на скандинавския дизайн, не поглъщат много добре звука", каза главният редактор на „Dezeen“ Маркус Файрс. Много от скандинавските и други техни европейски колеги споделят мнения за акустичните мебели.

Уолтър Бетенс от списание *Damn* и Аделин Сейдел от „Stylepark“ добавят, че дизайнерите пренебрегват прекалено много шума в интериора. Новите дизайнери не решават проблемите на акустиката, "казва Бетенс, редактор на списание „Damn.“ Те са твърде заети с текстури и цветове, но това е много важно нещо. Тя трябва да бъде част от образованието на дизайнер. " Той добави: "Наскоро отидох в нов ресторант на" дизайнер "в Милано и не можах да чуя какво говорят хората заради ужасната акустика.", "Дизайнерите мислят за светлината, формата и повърхността, но често не мислят за звук", каза Сейдел, главен редактор на „Stylepark“. "Те мислят само дали изглежда хубаво."

Карл Джемтелиус от шведската марка за акустични продукти „Zilenzio“ заявява, че търсенето на неговите продукти расте бързо, тъй като компаниите се опитват да намалят разходите, като набират персонал в спартански офиси с отворен план. Страничният ефект от това е, че производителността на работниците все повече се влияе от разсейването на шума. В допълнение, той заяви, че тенденцията за по-гъвкави офис площи, където работниците се насърчават да използват най-различни работни условия през деня, означава, че има увеличено търсене на тихи зони. "Това изисква различни офис среди. Все пак те трябва да бъдат приятни за работа. Ето защо акустиката е актуална." [13]

Шведската марка „Form2“, пусна пазара стена-стенд, който позволява на работниците да правят непрекъснато телефонни обаждания. (фиг. 1). Изработена е от основа от минерално дърво, пресирано с горната част, която е от сгъстена влакнеста вата и същото за малкия рафт. Този стенд запазва звука вътре и не позволява на хората от вън да чуят разговора. Във все повече офис пространства, както и в обществени места се налага употребата на този тип звукоизолиращи кабинки. С течение на времето те ще бъдат затвърдени като функционална

необходимост. Подобен род кабинки бяха изложени и в последното мебелно изложение в София.

Освен кабинките за разговори фирмите работят и за места за срещи и обсъждане на проекти или просто изолация на компания посредством специфично изградено за целта тяло



Фиг. 1

подобно на беседка. На фигура 2 са представени различни варианти на разработки на този тип обекти.



Фиг. 2

Северните дизайнери от няколко години са пионер в акустичния дизайн. За груповата работа, базирана в Стокхолм „Form Us With Love“, е проектирана Hood, извит филцов купол, който виси от тавана над големи маси. Компанията „Baugh“ стартира производството на цветни и шарени изолационни панели, изработени от дървена вата - комбинация от дървени стърготини, цимент и вода. „Baugh“ е замислен да внесе известен дизайнерски усет в често скучния свят на акустични продукти.

"Дизайнът не е важен фактор (в индустрията на акустиката), но това, което виждаме сега, е, че купувачи като архитекти или интериорни дизайнери всъщност искат дизайнерски стойности в материали, които обикновено са чисто функционални. "В момента никой наистина не отговаря на тези искания." Споделят от фирмата.[13]

„Baugh“ е фирма предлагаща продукти чиято основна гама, от различните цветове, се произвежда от дървените стърготини, които се обръщат при нагряване по време на производството.

Главните материали от, които се произвеждат акустичните мебели са дърво или производни на дървесината, вторични материали и различни видове тъкан от сорта на филца. Интерес будят трансформируемите мебели, които могат да променят части от своите детайли спрямо търсената функционалност. (фиг. 3)



Фиг. 3

Такива примери за дивани с столове се срещат все по-често, дори се предлагат във фирми за офис оборудване специфични шумоизолиращи офис столове. Те са на принципа на стандартни кресла около, които са монтирани около облегалката и подлакътниците филцови стени, които продължават по височина на стола и предпазват стоящия от страничния шум. Така удобно изчакващ за среща клиент ще стои спокойно без да бъде обезпокояван от околния шум. Освен доста стандартни като форма и дори леко тромави кресла, биват разработвани и по-иновативни като дизайн столове със същата функционалност, но доста по-нестандартна визия (фиг. 4) В случая към стандартен стол може просто да бъде добавен един подобен на яйцевиден абажур елемент, който е поставен на метална стойка. В случая спомагащ за шумоизолацията на стоящият под него човек.



Фиг. 4

Друг подобен по идея, но изпълнен по по-различен начин са въртящи столове отново с формата на яйца, които имат вариант със и без шумоизолираща приставка. Минусът на тези две

разработки е, че тази горна част не е мобилна и дефакто човек не може да я поставя или маха в зависимост от нуждите. Горната част е изградена от метални шини, които са уплътнени с порест материал и облечени с филц, като цвета повтаря този на долната част на стола, но двете части са с различен материал на дамската (фиг. 5).

На пазара на този тип мебели се появяват нови и нови идеи за работни места и други многофункционални идеи. Някога отделните шумоизолиращи прегради за бюра вече вървят заедно с места за работа в различни форми разцветки и вариации. (фиг.6)



Фиг.5



Фиг.6

Разделителните за стени със звукопоглъщащи свойства са особено гъвкаво решение за големи площи и могат да се използват поотделно или с няколко свързани блока. Те се състоят главно от масивна дървена или алуминиева рамка и

звукопоглъщащ мек акустичен пълнеж, биха могли да играят ролята на библиотека или етажерка. (фиг.7)

Най-новата тенденция е освобождаване от конвенционалните мебели, които имат акустични качества и навлизането на авангардни и иновативни идеи. Например единствената специфика на креслата на фигура 7 е материала, от които са изработени, в същото време са придружени от маса, която напомня повече на арт инсталация от колкото на нормална функционална мебел. В центърът ѝ са поставени дървени обли плоскости облечени във филц. Предстои да видим как ще се развие тази нова и доста иновативна ниша в мебелния дизайн.



Фиг. 7

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РЕАЛИЗАЦИЯ НА ДИЗАЙНЕРСКИ ПРОДУКТ С ВЪЗМОЖНОСТИТЕ НА ДИГИТАЛНОТО РИСУВАНЕ

REALIZATION OF A DESIGN PRODUCT WITH THE POSSIBILITIES OF DIGITAL PAINTING

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Abstract: The report presents the materialization of the design idea, starting from a sheet of paper to the digital screen image. The individual approach to moving from traditional creative media to computer graphics is exposed. The process tracks the steps of creating a sketch, through its software processing to a completed raster image. An author's product, which is part of the design concept of characters, subject of dissertation research, is realized using the tools and techniques in digital painting.

Keywords: TRADITIONAL DRAWING, RASTER GRAPHICS, SOFTWARE, ADOBE PHOTOSHOP, METHODOLOGY, DIGITAL DRAWING, DESIGN PRODUCT.

1. Увод

От първата пещерна рисунка до днес, човекът е превърнал дейността рисуване в изкуство. А именно- средство за влияние на емоционално и интелектуално ниво. През дългия си път на развитие изобразителното изкуство се проявява в разнообразие от средства и техники на рисуване, всяка от които маркира определен исторически период. Днес артистът има пълната свобода да създава изкуство във формата, която пожелае. Традиционните техники се превръщат в основа за създаването на нови. А съвременната дигитална медия предоставя среда за безкрайно и неограничено експериментиране, където традиционната изобразителна форма бива постигната при нови и различни условия. При дигиталното рисуване, с набор от едни и същи инструменти се постига пресъздаване на различни изобразителни техники, което го прави универсален и иновативен подход за развитие на изкуството.

Adobe Photoshop е програма за обработка на дигитални изображения, осигуряваща разнообразие от възможности за достигане на търсения от артиста резултат. Докладът представя стъпка по стъпка създаването на дигитално изображение посредством възможностите на Adobe Photoshop. Изображението е част от серия български митични създания, разработвани в специфична стилистика.

2. Методология

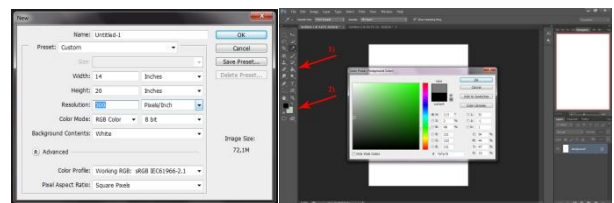
Усещането при традиционното рисуване върху хартия може да се пресъздаде в дигиталната медия посредством графичен таблет и писалка. Технологичният пазар предоставя широко разнообразие от графични таблети, с разнообразни възможности и размери. В настоящата разработка се използва тип Tablet Monitor с USB порт, при който се рисува директно върху плоска стъклена повърхност. Устройството е свързано с компютър, където протича работата в Adobe Photoshop.

2.1. Всичко започва с идеята върху лист хартия. Чрез така наречените груби скици се достига до избор на позиция и израз на персонажа. Изгражда се митичният образ на Змеицата от българския фолклор, където често срещано е описанието ѝ като мечка стръвница.

2.2. След изясняване на концепцията, започва същинската работа с графичната програма.

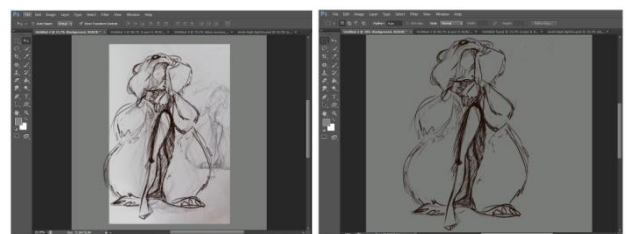
Първата стъпка е създаване на работно поле. Open Photoshop > File > New. Фиг.1 показва опциите за настройване на желаните параметри. Целта е да се създаде изображение с висока резолюция, което в същото време да не натоварва програмата. В този случай резолюция 300 пиксела осигурява адекватно качество на графиката. Работи се в RGB цветови

модел, освен ако продуктът не е предназначен за печат, когато е препоръчително цвятна настройка в SMYK модел. Създаденото работно поле е бяло. Цветовете върху бял или черен фон се възприемат като по-тъмни или ярки. За да се избегне тази илюзия, е препоръчително да се работи върху неутрален сив фон. За целта чрез Paint Bucket Tool (от менюто с инструменти 1) фиг.2) фонът се променя в желания цвят (2) фиг 2).



Фиг.1. Отваряне нов документ. Фиг.2. 1) Paint Bucket Tool; 2) Color.

2.3. Следващата стъпка е отваряне на сканираните скици в работното поле. Има няколко подхода за работа: ако скицата е прецизна, програмата позволява обработването ѝ до завършване на сканирания файл; в разглеждания случай се работи с т.н. груби скици, което изисква създаване на нова дигитална скица, базирана на сканираната. В Adobe Photoshop се работи със слоеве (Layers). Всеки слой има настройки за прозрачност и режим на работа. Фигура 3 показва сканираната скица, намираща се в слой 1. При увеличаване на прозрачността тя избледнява. Създава се нов слой, избира се четка и се рисува върху контурите, принадлежащи на долния. След като скицата е прерисувана, долният слой се изключва, само новият остава видим (фиг.4).

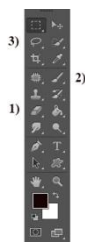


Фиг.3. Два слоя – дигитална рисунка и сканирана скица.

Фиг. 4. Един слой – дигитална рисунка.

2.4. Базисната скица може да се променя и подобрява с помощта от набора инструменти (фиг. 5). Променя се линията на движение на фигурата, за да се създаде по-динамичен характер. Както при традиционното рисуване, и тук също може да се изтрива с гума (1) фиг.5). За да се провери дали цялостният образ изглежда добре, изображението се обръща огледално: Image > Image Rotation > Flip Canvas Horizontal. Тъй като окото свиква с образа в процеса на работа, някои нередности остават незабелязани, но когато се обърне

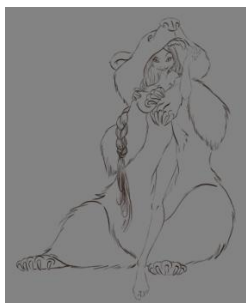
огледално, изображението се разглежда с нов поглед и местата, които се нуждаят от корекции стават видими.



Фиг. 5. Инструменти.

2.5. Следващият етап е създаване на фина скица. От менюто с инструменти се избира вид четка (Brush Tool – 2) (фиг.5). Четката може да се регулира спрямо търсения ефект, който да създаде. В случая се избира Erodible Point от базисния пакет, включен в програмата. Тази четка наподобява рисуване с графит.

Увеличава се прозрачността на грубата скица и се създава нов слой, който се преименува като “fine sketch”. Подобно предходните стъпки, с четката се прерисуват линиите от грубата скица, но този път с по-голяма прецизност, защото това ще са видимите контури в илюстрацията. На този етап се добавят детайли, които да обогатят рисунката.

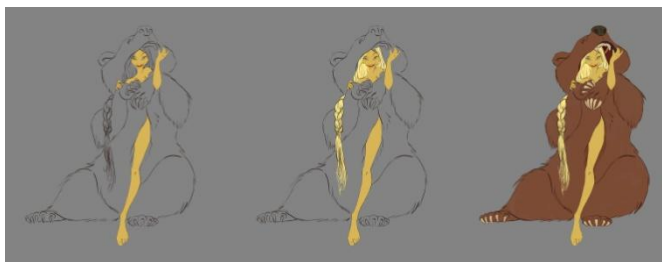


Фиг. 6. Фина скица.



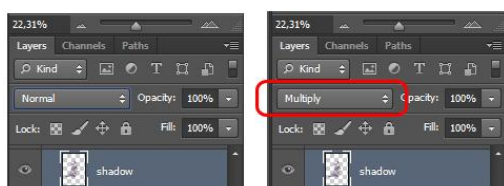
Фиг. 7. Фина скица- детайл.

2.6. След като фината скица е готова, всички долни слоеве се изтриват и се започва работа по добавянето на цвят. Създава се нов слой – „local color”, който да се намира под „fine sketch”. Избира се нова четка от селекцията видове четки, решава се базисният цвят и се пристъпва към оцветяването. Процесът е подобен на традиционно оцветяване. Внимателно се запълват отделните части на рисунката, като гумата винаги е „под ръка”.



Фиг. 8. Поетапно запълване на отделните елементи с цвят.

2.7. Следващият момент е добавяне на сянка и светлина. Създава се нов слой, над слоя „local color”, който се записва „shadow”. Настройва се режим на слоя - Multiply, като по този начин цветът на сянката ще се обедини с локалния цвят. Избира се цвят/нюанс на сянката, избира се и се настройва четка за работа.



Фиг. 9. Смяна на режим на слоя: от Normal в Multiply.

Посредством четката започва нанасянето на сянката, според избора на светлинен източник (фиг. 10). За придаване на повече драматизъм и фокус на изображението, в нов слой се добавя по-мощна сянка, отново в режим Multiply (фиг. 11). След което идва ред на маркиране на светлината. Създава се нов слой, наречен „highlights”, този път в режим Overlay (фиг. 12). Изборът на цвят във всеки един от разгледаните процеси не е окончателен. Винаги може да се промени нюанса със следните настройки: Image > Adjustments > Hue/Saturation (фиг. 13).



Фиг. 10. Сенки.

Фиг.11. Допълнителни сенки.

Фиг.12. Добавяне на светлини.



Фиг. 13. Промяна на цвят/нюанс с настройки на „Hue/Saturation”.

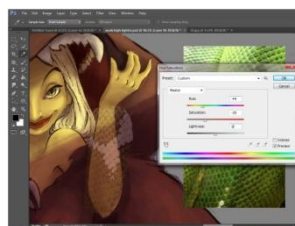
2.8. Ключов етап от изграждането на графичното изображение е добавянето на детайли. Героинята, криеща се под мечата кожа, е Змеица, чието тяло е покрито с люспи. За създаване на такава текстура по кожата се извършват следните действия: подбери се снимка на змийска кожа в близък план с високо качество; след което се добавя в работното поле: File > Place. Чрез Lasso Tool (3) (фиг. 5) се селектира част от снимката и се поставя на мястото, което трябва да има въпросната текстура; увеличава се прозрачността на изрязаната част (фиг. 14); изпълнява се: Image > Adjustments > Exposure, по този начин се придава контраст (фиг. 15); следва: Image > Adjustments > Hue/Saturation – регулиране на цвета (фиг.16), за да се доближи до цвета на кожата на Змеицата; Edit > Transform > Wrap – за да приеме формата на ръката (фиг.17).



Фиг.14. Наместване на селекцията и регулиране прозрачността.



Фиг. 15. Придаване на контраст.



Фиг. 16. Регулиране на цвят.

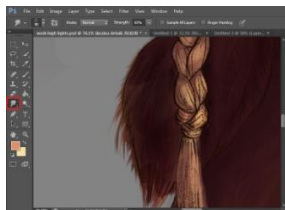


Фиг. 17. Промяна на форма.

За пресъздаване текстурата на мечата козина, се отваря ново работно поле: File > New; създава се нов слой; избира се Hard Round Brush, с която произволно се поставят точки по работното поле с черен цвят; следва: Edit > Define Brush Preset, с което се създава нова четка. Обратно към основния работен файл; следва работа с инструмента Smudge Tool (фиг. 18); от менюто с четки се избира новата, която се създаде в предходната стъпка (фиг. 19); работи се върху слоя с локален цвят, посредством Smudge Tool, четката деформира цвета в слоя извън очертанията, под формата на снопове козина (фиг.20).

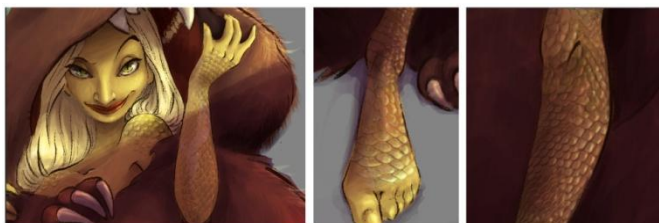


Фиг.18. Избор на Smudge Tool. Фиг. 19. Избор на четка.



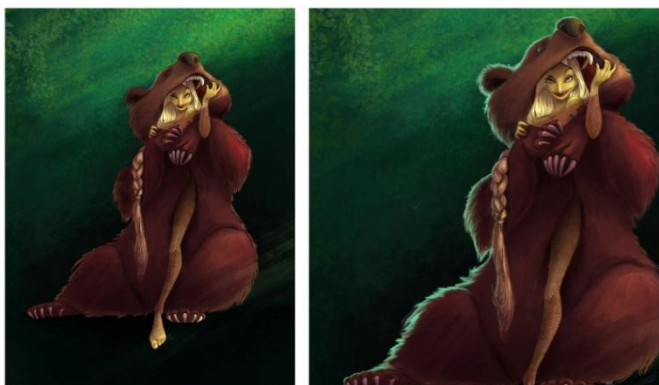
Фиг. 20. Създаване ефект на козина.

2.9. След създаване на текстурата, се пристъпва към добавяне на светлосенки и детайли по нея. Отваря се нов слой в режим Multiply, където се нанасят допълнителни сенки към текстурите. Аналогично се постъпва и с добавянето на светлини и отблясъци в нов следващ слой, който се намира в режим Overlay.



Фиг. 21. Детайли по текстура.

2.10. Идва ред на изграждане на фон. Чрез Paint Bucket Tool цвета на „Background“ слой се променя. Създава се нов слой, в който, с помощта на Brush Tool, извеждаме осветените и затъмнени части, спрямо светлосенките на персонажа (фиг.22).



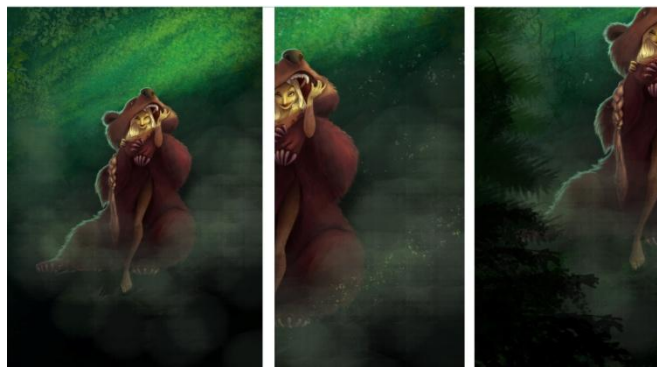
Фиг. 22. Създаване на фон.

Фиг. 23. Добавяне втори източник на светлина.

2.11. Създава се нов слой, в който ще се добави втори светлинен източник, отново чрез Brush Tool. Тази студена

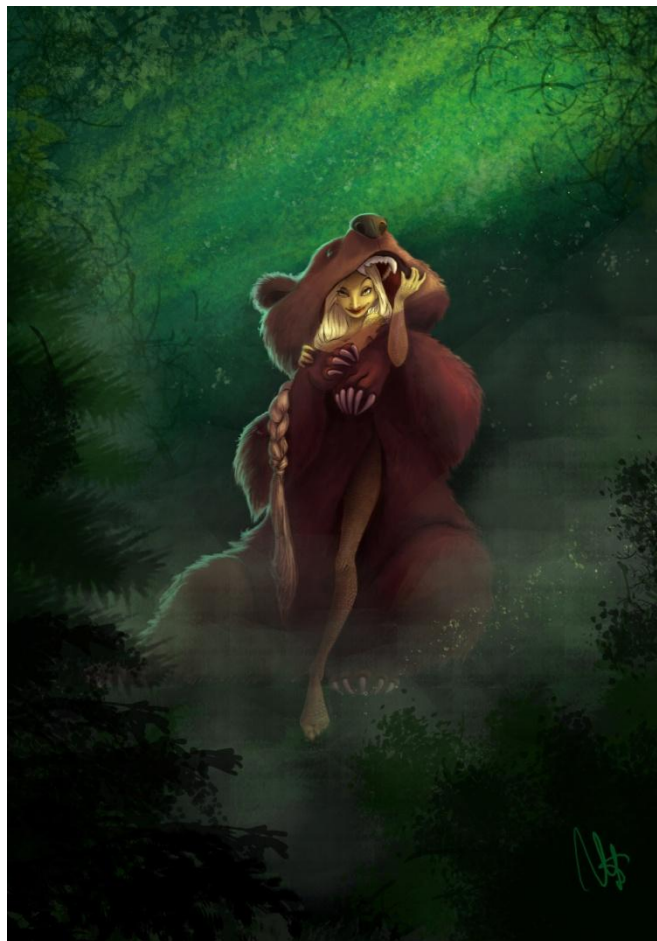
светлина осветява от задния план на композицията, което подчертава по-силно формите и силуета (фиг. 23).

2.12. Един от последните етапи по изграждането на илюстрацията е създаването на атмосфера/среда. Чрез Brush Tool, слой след слой се създават атмосферни (фиг. 24) и светлинни (фиг.25) ефекти, последвани от растителни елементи (фиг. 26), отново постигнати с Brush Tool.



Фиг. 24. Мъгла. Фиг. 25. Проблясъци. Фиг. 26. Растителност

2.13. Крайният резултат от графичното оформление е представен от фигура 27. През целия процес изображението се изгражда слой по слой. Всеки нов слой допринася за създаването на по-тежък файл. За да се улесни работа с готовото изображение на един следващ етап, е необходимо преобразуване, което ще направи файла значително по-лек. Един от начините е чрез опцията: Layer > Flatten Image. По този начин всички слоеве се обединяват в един. Препоръчително е преди всяко обединяване на слоевете, да се прави резервно копие, съдържащо отделните работни слоеве, ако се предвижда допълнителна работа по проекта.



Фиг. 27. Завършено дигитално изображение.

3. Резултати и дискусия

Крайният резултат е многоцветно графично дигитално изображение с резолюция, позволяваща печат или „web“ разпространение (фиг. 27). Програмата позволява запаметяване на дигиталната графика в богато разнообразие от видове файлове, което благоприятства широко приложение.

Процесът на работа в Adobe Photoshop е последователно структуриран, позволяващ корекции и промени във всеки един етап. Това значително спестява много време и усилия, което е едно от основните предимства пред традиционното рисуване.

Така представено, дигиталното рисуване създава усещане за един механичен процес, но в действителност са необходими същите артистични умения и действия, типични за традиционното изобразително изкуство.

Неограниченото експериментиране, възможността за надграждане и усъвършенстване са големите предимства при компютърната графика. Изображението, което се създава стъпка по стъпка може да се подобри и промени посредством ефектите и филтрите, с които разполага програмата.

Adobe Photoshop е достъпен за изучаване и универсален за прилагане продукт. Няма правилен или неправилен начин на работа. Всеки артист има индивидуален подход и свобода на изразяване.

4. Заключение

Развитието на компютърната графика предлага нови възможности пред съвременните артисти. Това не значи загърбване на традиционните методи и техники на рисуване, напротив, те също са част от този нов процес на създаване на изкуство. Програмата Adobe Photoshop дава поле за изява и приложение на разнообразните начини и похвати в изобразителното изкуство, за което са необходими само 3 технически средства – компютър, таблет и писалка.

Дигиталното рисуване улеснява и ускорява процеса на реализиране на идеята на дизайнера, предоставяйки многообразие от възможности за развитие.

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EXAMPLES OF SOFTWARE CONFIGURATION MANAGEMENT

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Abstract: When you build software the changes happens. Because it happens you need to manage it effectively; this is a set of activities designed to manage shanges by identifying the work products that are lakely to change, establishing relationships among them, defining the mechanism for managing work products, controlling the changes imposed ang repochanges made. In the report is made the distinction between software support and software configuration managemet ang give the examples of using.

Keywords: Software engineering, Software Configuration Management (SCM), Elements of SCM System, SCElements,SCM Layers SCM features

Introduction

Software configuration management (SCM), also called change management, is a set of activities designed to manage change by identifying the work products that are likely to change, establishing relationships among them, defining mechanisms for managing different versions of these work products, controlling the changes imposed, and auditing and reporting on the changes made.

SCM activities are developed to: identify change, control change, ensure that change is being properly implemented, and report changes to others who may have an interest.

Software configuration management is a set of tracking and control activities that are initiated when a software engineering project begins and terminates only when he software is taken out of operation (in opposite to Support that is a set of software engineering activities that occur after software has been delivered to the customer and put into operation).

Software Configuration Management

A primary goal of software engineering is to improve the ease with which changes can be accommodated and reduce the amount of effort expended when changes must be made.

The output of the software process is information that may be divided into three broad categories: computer programs (both source level and executable forms), work products that describe the computer programs (targeted at various stakeholders), and data or content (contained within the program or external to it). The items that comprise all information produced as part of the software process are collectively called a *software configuration*.

As software engineering work progresses, a hierarchy of *software configuration items* (SCIs) - a named element of information that can be as small as a single UML diagram or as large as the complete design document - is created. If each SCI simply led to other SCIs, little confusion would result. Unfortunately, another variable enters the process - *change*. Change may occur at any time, for any reason. In fact [1,2] states: "No matter where you are in the system life cycle, the system will change, and the desire to change it will persist throughout the full life cycle."

There are 4 sources of changes:

- New business or market conditions dictate changes in product requirements or business rules.
- New stakeholder needs demand modification of data produced by information systems, functionality delivered by products, or services delivered by a computer-based system.
- Reorganization or business growth/downsizing causes changes in project priorities or software engineering team structure.

- Budgetary or scheduling constraints cause a redefinition of the system or product.

Elements of Software Configuration Management System

Software configuration management is a set of activities that have been developed to manage change throughout the life cycle of computer software. SCM can be viewed as a software quality assurance activity that is applied throughout the software process. In the sections that follow.

A typical CM operational scenario involves a project manager who is in charge of a software group, a configuration manager who is in charge of the CM procedures and policies, the software engineers who are responsible for developing and maintaining the software product, and the customer who uses the product.

The goals of the configuration manager are to ensure that procedures and policies for creating, changing, and testing of code are followed, as well as to make information about the project accessible. To implement techniques for maintaining control over code changes, this manager introduces mechanisms for making official requests for changes, for evaluating them and for authorizing changes. The manager creates and disseminates task lists for the engineers and basically creates the project context. Also, the manager collects statistics about components in the software system, such as information determining which components in the system are problematic.

The White paper on software configuration management [3] identifies four important elements that should exist when a configuration management system is developed:

- *Component elements* - a set of tools coupled within a file management system (e.g., a database) that enables access to and management of each software configuration item.
- *Process elements* - a collection of actions and tasks that define an effective approach to change management (and related activities) for all constituencies involved in the management, engineering, and use of computer software.
- *Construction elements* - a set of tools that automate the construction of software by ensuring that the proper set of validated components (i.e. the correct version) have been assembled.
- *Human elements* - a set of tools and process features (encompassing other CM elements) used by the software team to implement effective SCM.

In the context of software engineering, a baseline is a milestone in the development of software. A baseline is marked by the delivery of one or more software configuration items that have been approved as a consequence of a technical review. For example, the elements of a design model have been documented and reviewed. Errors are found

and corrected. Once all parts of the model have been reviewed, corrected, and then approved, the design model becomes a baseline. Further changes to the program architecture (documented in the design model) can be made only after each has been evaluated and approved. Although baselines can be defined at any level of detail, the most common software baselines

The software configuration item are defined as information that is created as part of the software engineering process. In the extreme, a SCI could be considered to be a single section of a large specification or one test case in a large suite of tests. More realistically, an SCI is all or part of a work product (e.g., a document, an entire suite of test cases, or a named program component).

In addition to the SCIs that are derived from software work products, many software engineering organizations also place software tools under configuration control. That is, specific versions of editors, compilers, browsers, and other automated tools are "frozen" as part of the software configuration. Because these tools were used to produce documentation, source code, and data, they must be available when changes to the software configuration are to be made. Although problems are rare, it is possible that a new version of a tool (e.g. a compiler) might produce different results than the original version. For this reason, tools, like the software that they help to produce, can be baselined as part of a comprehensive configuration management process.

In reality, SCIs are organized to form configuration objects that may be cataloged in the project database with a single name. A *configuration object* has a name, attributes, and is "connected" to other objects by relationships. Referring to Figure 1, the configuration objects, **DesignSpecification**, **DataModel**, **ComponentN**, **SourceCode**, and **TestSpecification** are each defined separately. However, each of the objects is related to the others as shown by the arrows. A curved arrow indicates a compositional relation. That is, **DataModel** and **ComponentN** are part of the object **DesignSpecification**. A double-headed straight arrow indicates an interrelationship. If a change were made to the **SourceCode** object, the interrelationships enable you to determine what other objects (and SCIs) might be affected

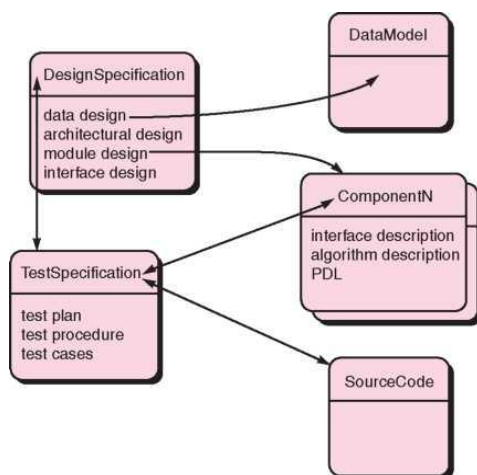


Fig. 1

Today, SCIs are maintained in a project database or repository. *Webster's Dictionary* defines the word *repository* as "any thing or person thought of as a center of accumulation or storage." During the early history of software engineering, the repository was indeed a person - the programmer who had to

remember the location of all information relevant to a software project, who had to recall information that was never written down and reconstruct information that had been lost. Sadly, using a person as "the center for accumulation and storage" (although it conforms to Webster's definition) does not work very well. Today, the repository is a "thing" - a database that acts as the center for both accumulation and storage of software engineering information. The role of the person (the software engineer) is to interact with the repository using tools that are integrated with it.

Software Configuration Management Features

To support SCM, the repository must have a tool set that provides support for the following features:

Versioning As a project progresses, many versions of individual work products will be created. The repository must be able to save all of these versions to enable effective management of product releases and to permit developers to go back to previous versions during testing and debugging.

The repository must be able to control a wide variety of object types, including text, graphics, bit maps, complex documents, and unique objects like screen and report definitions, object files, test data, and results. A mature repository tracks versions of objects with arbitrary levels of granularity; for example, a single data definition or a cluster of modules can be tracked.

Dependency tracking and change management The repository manages a wide variety of relationships among the data elements stored in it. These include relationships between enterprise entities and processes, among the parts of an application design, between design components and the enterprise information architecture, between design elements and deliverables, and so on. Some of these relationships are merely associations, and some are dependencies or mandatory relationships.

The ability to keep track of all of these relationships is crucial to the integrity of the information stored in the repository and to the generation of deliverables based on it, and it is one of the most important contributions of the repository concept to the improvement of the software process. For example, if a UML class diagram is modified, the repository can detect whether related classes, interface descriptions, and code components also require modification and can bring affected SCIs to the developer's attention.

Requirements tracing This special function depends on link management and provides the ability to track all the design and construction components and deliverables that result from a specific requirements specification (forward tracing). In addition, it provides the ability to identify which requirement generated any given work product (backward tracing).

Configuration management. A configuration management facility keeps track of a series of configurations representing specific project milestones or production releases.

Audit trails. An audit trail establishes additional information about when, why, and by whom changes are made. Information about the source of changes can be entered as attributes of specific objects in the repository. A repository trigger mechanism is helpful for prompting the developer or the tool that is being used to initiate entry of audit information (such as the reason for a change) whenever a design element is modified.

The software configuration management process defines a series of tasks that have four primary objectives: to identify

all items that collectively define the software configuration, to manage changes to one or more of these items, to facilitate the construction of different versions of an application, and to ensure that software quality is maintained as the configuration evolves over time.

A process that achieves these objectives need not be bureaucratic or ponderous, but it must be characterized in a manner that enables a software team to develop answers to a set of complex questions:

- How does a software team identify the discrete elements of a software configuration?
- How does an organization manage the many existing versions of a program (and its documentation) in a manner that will enable change to be accommodated efficiently?
- How does an organization control changes before and after software is released to a customer?
- Who has responsibility for approving and ranking requested changes?
- How can we ensure that changes have been made properly?
- What mechanism is used to apprise others of changes that are made?

These questions lead to the definition of five SCM tasks—identification, version control, change control, configuration auditing, and reporting—illustrated in Figure 2.

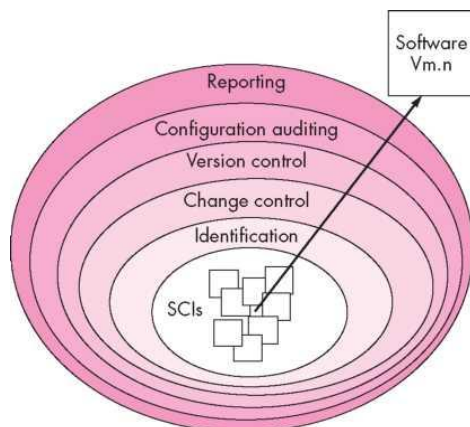


Figure 2

Referring to the figure, SCM tasks can be viewed as concentric layers. SCIs flow outward through these layers throughout their useful life, ultimately becoming part of the software configuration of one or more versions of an application or system. As an SCI moves through a layer, the actions implied by each SCM task may or may not be applicable. For example, when a new SCI is created, it must be identified. However, if no changes are requested for the SCI, the change control layer does not apply. The SCI is assigned to a specific version of the software (version control mechanisms come into play). A record of the SCI (its name, creation date, version designation, etc.) is maintained for configuration auditing purposes and reported to those with a need to know. In the sections that follow, we examine each of these SCM process layers in more detail.

Software Configuration Management Layers

To control and manage SCI is need to used object-orientated approach. Two types of objects can be identified [4]: basic objects and aggregate objects. A *basic object* is a unit of information that you create during analysis, design, code, or test. For example, a basic object might be a section of a

requirements specification, part of a design model, source code for a component, or a suite of test cases that are used to exercise the code. An *aggregate object* is a collection of basic objects and other aggregate objects. For example, a **DesignSpecification** is an aggregate object. Conceptually, it can be viewed as a named (identified) list of pointers that specify aggregate objects such as **ArchitecturalModel** and **DataModel**, and *basic objects* such as **ComponentN** and **UMLClassDiagramN** (Fig. 1).

Configuration object identification can also consider the relationships that exist between named objects.

For example:

Class diagram <part-of> requirements model;

Requirements model <part-of> requirements

in SCIs hierarchy:

DataModel <interrelated> DataFlowModel

DataModel <interrelated> TestCaseClassM

The identification scheme for software objects must recognize that objects evolve throughout the software process. Before an object is baselined, it may change many times, and even after a baseline has been established, changes may be quite frequent.

Version control combines procedures and tools to manage different versions of configuration objects that are created during the software process. A version control system implements or is directly integrated with four major capabilities: a project database (repository) that stores all relevant configuration objects, a *version management* capability that stores all versions of a configuration object (or enables any version to be constructed using differences from past versions), a *make facility* that enables you to collect all relevant configuration objects and construct a specific version of the software. In addition, version control and change control systems often implement an *issues tracking* (also called *bug tracking*) capability that enables the team to record and track the status of all outstanding issues associated with each configuration object.

A number of different automated approaches to version control have been proposed over the last few decades. The primary difference in approaches is the sophistication of the attributes that are used to construct specific versions and variants of a system and mechanics of construction's process.

For a large software project, uncontrolled change rapidly leads to chaos. For such projects, change control combines human procedures and automated tools to provide a mechanism for the control of change. The change control process is illustrated schematically in Figure 3. A *change request* is submitted and evaluated to assess technical merit, potential side effects, overall impact on other configuration objects and system functions, and the projected cost of the change. The results of the evaluation are presented as a *change report*, which is used by a *change control authority* (CCA) - a person or group that makes a final decision on the status and priority of the change. An *engineering change order* (ECO) is generated for each approved change. The ECO describes the change to be made, the constraints that must be respected, and the criteria for review and audit.

Prior to an SCI becoming a baseline, only *informal change control* need be applied. The developer of the configuration object (SCI) in question may make whatever changes are justified by project and technical requirements (as long as changes do not affect broader system requirements that lie

outside the developer's scope of work). Once the object has undergone technical review and has been approved, a baseline can be created. Once an SCI becomes a baseline, *project level change control* is implemented. Now, to make a change, the developer must gain approval from the project manager (if the change is "local") or from the CCA if the change affects other SCIs. In some cases, formal generation of change requests, change reports, and ECOs is dispensed with. However, assessment of each change is conducted and all changes are tracked and reviewed.

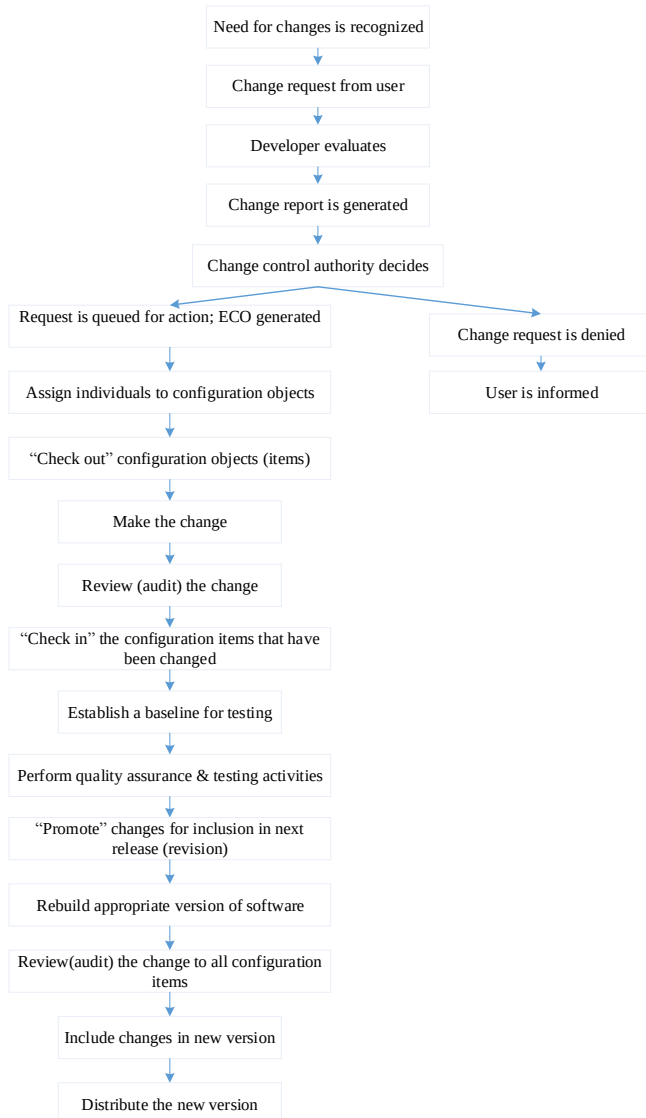


Figure 3

When the software product is released to customers, *formal change control* is instituted. The formal change control procedure has been outlined in Figure 3.

The change control authority plays an active role in the second and third layers of control. Depending on the size and character of a software project, the CCA may be composed of one person - the project manager - or a number of people (e.g., representatives from software, hardware, database engineering, support, marketing). The role of the CCA is to take a global view, that is, to assess the impact of change beyond the SCI in question. How will the change affect hardware? How will the change affect performance? How will the change modify customers' perception of the product? How will the change affect product quality and reliability? These and many other questions are addressed by the CCA.

A *software configuration audit* complements the technical review by assessing a configuration object for characteristics that are generally not considered during review. The audit asks and answers the following questions:

1. Has the change specified in the ECO been made? Have any additional modifications been incorporated?
2. Has a technical review been conducted to assess technical correctness?
3. Has the software process been followed and have software engineering standards been properly applied?
4. Has the change been "highlighted" in the SCI? Have the change date and change author been specified? Do the attributes of the configuration object reflect the change?
5. Have SCM procedures for noting the change, recording it, and reporting it been followed?
6. Have all related SCIs been properly updated?

Configuration status reporting (sometimes called *status accounting*) is an SCM task that answers the following questions: What happened? Who did it? When did it happen? What else will be affected?

The flow of information for configuration status reporting (CSR) is illustrated in Figure 3. Each time an SCI is assigned new or updated identification, a CSR entry is made. Each time a change is approved by the CCA (i.e., an ECO is issued), a CSR entry is made. Each time a configuration audit is conducted, the results are reported as part of the CSR task. Output from CSR may be placed in an online database or website, so that software developers or support staff can access change information by keyword category. In addition, a CSR report is generated on a regular basis and is intended to keep management and practitioners apprised of important changes.

Summary

SCM is an umbrella activity that is applied throughout the software process: identifies, controls, audits, and reports modifications that invariably occur while software is being developed and after it has been released to a customer. All work products created as part of software engineering become part of a software configuration. The configuration is organized in a manner that enables orderly control of change. The SCIs are produced as a result of some software engineering activity. In addition to documents, programs, and data, the development environment that is used to create software can also be placed under configuration control. All SCIs are stored within a repository that implements a set of mechanisms and data structures to ensure data integrity, provide integration support for other software tools, support information sharing among all members of the software team, and implement functions in support of version and change control. The evolution of a program can be tracked by examining the revision history of all configuration objects.

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ВЛИЯНИЕ НА МЕЖДУНАРОДНИЯ ТИПОГРАФСКИ СТИЛ ВЪРХУ УЕБ ДИЗАЙНА

IMPACT OF INTERNATIONAL TYPOGRAPHIC STYLE ON WEB DESIGN

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Abstract: This report reviews the main visual characteristics of the International Typographic Style. Its key principles and elements have been outlined, as well as its leading compositional specifics. Analyzed is its impact on the contemporary web design. Shown are examples of current web design used on web pages, with the application of Swiss- design characteristic details and compositional techniques.

Keywords: INTERNATIONAL TYPOGRAPHIC STYLE, SWISS DESIGN, GRIDS, SANS-SERIF FONTS, MINIMALISM, TYPOGRAPHY, WHITE SPACE, ORDER, GRAPHIC DESIGN, WEB DESIGN, WEB TYPOGRAPHY, LEGIBILITY.

1. Увод

В доклада са разгледани основните визуални характеристики на Международния типографски стил. Очертани са неговите ключови принципи и елементи. Разгледани са водещите композиционни особености на стила. Анализирано е въздействието му върху съвременното оформление на уеб дизайна. Направен е опит да се посочат примери от актуалния дизайн на уеб сайтове, които използват елементи и композиционни похвати характерни за Швейцарския дизайн.

2. Изложение

През 50-те на XX в. в Швейцария се появява нов графичен стил в дизайна, който ще се превърне във водещ стил в графичния дизайн по света през 70-те г. на XX в. Поради силната си зависимост от типографски елементи новият стил става известен като Международен типографски стил (International Typographic Style). Някои автори го наричат още и Швейцарски стил (Swiss Style), Швейцарски дизайн (Swiss Design), а други използват термина Швейцарска школа.

Стилет се развива основно в две дизайнерски училища в Швейцария, едното е в Цюрих под ръководството на Йозеф Мюлер-Брокман, а другото е в Базел и водещи фигури там са Армин Хофман и Емил Рудер. Всички те са учили при Ернст Келер (фиг. 1) в Цюрихското училище по дизайн преди Втората световна война и са били обучавани в принципите на Баухаус и на Новата типография на Ян Чихолд [1, 2, 3, 4].

Новият стил става водещ в графична идентичност на много швейцарски културни институции, които използват рекламни плакати за своите представления. Армин Хофман прави серия плакати за държавния театър в Базел (фиг. 2), а Йозеф Мюлер-Брокман е известен със своите плакати за операта в Цюрих и Tonhalle оркестър в Цюрих (фиг. 3).



Фиг. 2 Плакати на Армин Хофман за театъра в Базел



Фиг. 1 Плакати на Ернст Келер, считан за основоположник и пионер на Международния типографски стил



Фиг. 3 Плакат на Йозеф Мюлер-Брокман за Операта в Цюрих и книгата му „Rastersysteme für die visuelle Gestaltung“ – фундаментален труд за мрежовите системи за оформление в графичния дизайн

Характерно за произведенията на Мюлер-Брокман и Хофман е силното подчертаване на контрастите между различните елементи на дизайна и водещата роля на типографската йерархия, а също и употребата на черно-бели фотографии. В работите на Йозеф Мюлер-Брокман, Армин Хофман и Емил Рудер се виждат всички основни характеристики и принципи на Международния типографски стил. Водещи принципи стават рационалните композиции, редът, приоритетното значение на типографията – това води до обективно представяне на информацията, чрез функционален и минималистичен дизайн [5].

Други известни представители на Швейцарския дизайн са Тео Балмер, Херберт Матер, Макс Бил, Рихард Паул Лозе и др.

Ключовите принципи и основните изразни средства на Международния типографски стил са:

- Използване на математически изчертана мрежа, за да се предостави цялостно подредена и единна структура за оформление на текст и изображения;
- Употреба на безсерифни шрифтове (особено Helvetica и Univers);

През 1957 г. швейцарският дизайнер Адриан Фрутигер създава шрифтовото семейство Univers, което включва 21 начертания. Неговият шрифт става един от най-известните шрифтове на XX в. Шрифта е базиран на Akzidenz-Grotesk от 1898 г.

Друг много популярен шрифт от този период е Helvetica – той става една от иконите на Швейцарския дизайн. През 50-те и 60-те години на XX в. шрифтът става световен образец за функционална и ясна визуална комуникация. Балансираната му и неутрална форма предхожда експресивността на постмодерната типография. Helvetica е гротесков безсерифен шрифт – гредите му са с еднаква плътност, а рисункът стъпва на формите на Akzidenz-Grotesk на Бертхолд от 1898 г.

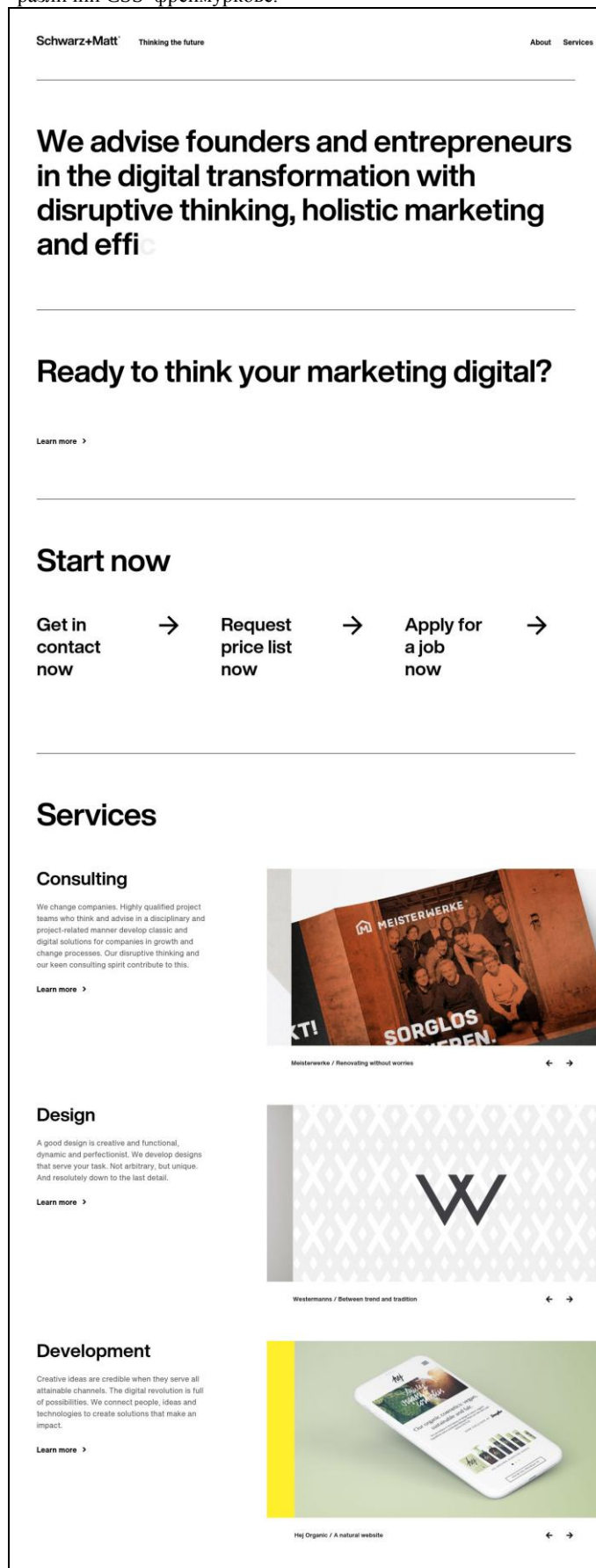
- Силно присъствие на типографската йерархия с използването на големи контрасти в кеглите на шрифта;
- Използване на ляво подравнен текст с нахсан десен край и асиметрично оформление на страниците;
- Широка употреба на фотографията (много често черно-бяла), на мястото на ръчно нарисувана илюстрация;
- Използване на много широки бели полета, които стават основен композиционен похват;
- Постигане на ритъм с използване на геометрични форми – квадрати, правоъгълници, кръгове и линии.

След като се разгледаха основните характеристики на Международния типографски стил в графичния дизайн ще се анализира и въздействието му върху уеб дизайна.

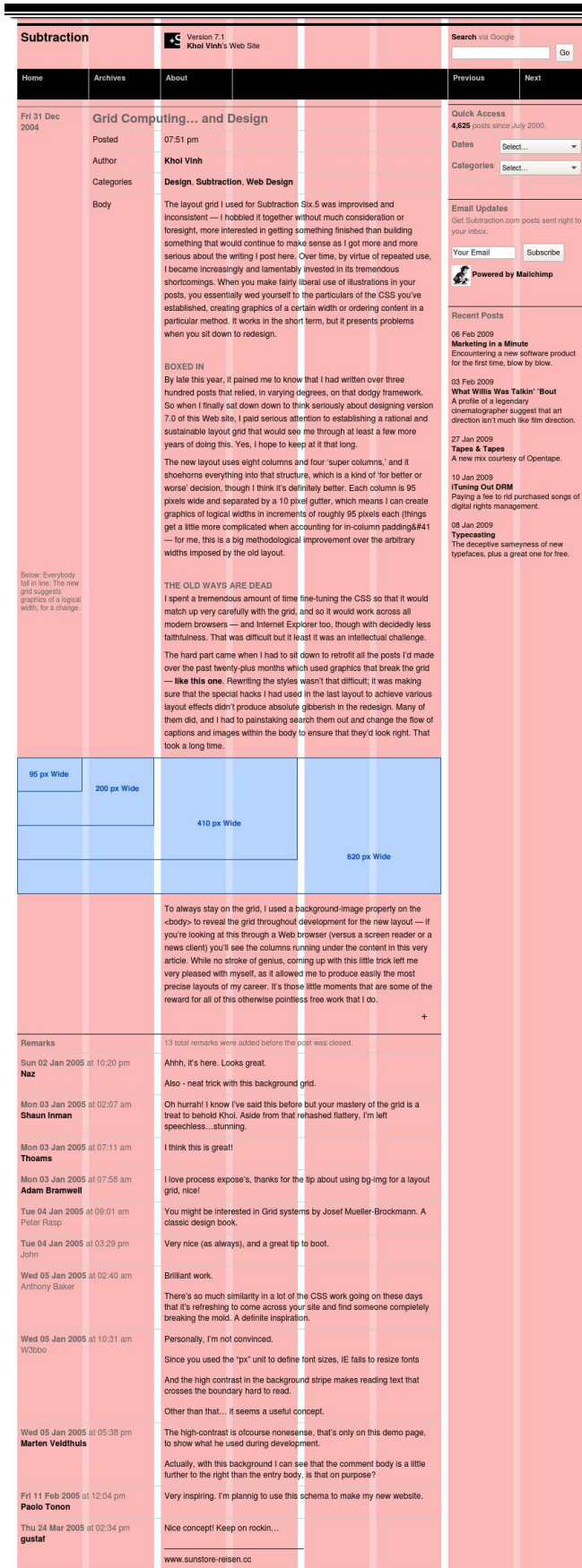
На фиг. 4 е показан пример за качествено оформление на уеб страница с използване на характерните за Швейцарския дизайн композиционни похвати. В уебсайта на германското студио за дизайн Schwarz+Matt е използван безсерифния шрифт Helvetica – постигната е много ясна визуална йерархия на основните структурни елементи на текста. Основният текст е ляво подравнен и е използвано много голямо бяло пространство, което дава достатъчно въздух на композицията и акцентира върху важните елементи на уеб страницата – текста и изображенията.

Един от първите уеб дизайнери, които се занимават с възможностите мрежите да се използват за оформление в уеб дизайнът е известният дизайнер Хой Вин. При редизайна на своя блог Subtraction.com през 2004 г. Вин прилага осем колонна мрежа за оформление на елементите в уебсайта (фиг. 5). Мрежата помага да се въведе ред и подравненост във взаимодействието между текст и изображения и така се постига единност и цялостност в композицията на отделните уеб страници в целия сайт. В книгата си излязла през 2011 г. *Ordering Disorder: Grid Principles for Web Design* [6] Вин анализира изчерпателно предимствата на оформлението на сайтове с мрежи и високата организация на съдържанието при този вид дизайн. Книгата е един от ранните опити да се систематизират основните теоретични и практични методи за употреба на мрежите при проектирането на уебсайтове.

В съвременния уеб дизайн употребата на мрежи за оформление става масова, благодарение на използването на различни CSS фреймворкове.



Фиг. 4 Уебсайта на Студио за дизайн Schwarz+Matt (Германия) използва минималистичен дизайн, базиран на Швейцарския дизайн <https://www.schwarz-matt.com/en>



Фиг. 5 Блога Subtraction.com на Хой Вин от 2004 г. Дизайнерът използва осем колонна мрежа за оформление на елементите в уебсайта

На фиг. 6 е представено портфолиото на американския уеб дизайнер Уилсън Майнър. Неговия дизайн е много минималистичен и отново е използван безсерифен шрифт за оформлението с достатъчно негативно пространство, като

основен композиционен способ. Типографското изграждане на уебсайта е с много отчетлива визуална йерархия. Кегела на шрифта в заглавието е голям и привлича погледа на потребителите на уеб сайта. Благодарение на акцентирането на заглавието – става пределно ясно с какво се занимава автора на портфолиото.



Фиг. 6 Портфолио на американския уеб дизайнер Уилсън Майнър-
<https://wilsonminer.com/>

3. Заключение

В заключение може да се стигне до извода, че стиловете в графичния дизайн оказват своето влияние върху уеб дизайна. В съвременния уеб дизайн все повече се усеща въздействието на Швейцарския дизайн и Модернизма. Характерни тенденции в дизайна на актуалните уебсайтове е използването на мрежи за оформление, употребата на безсерифни шрифтове с ясно изразена визуална йерархия и умелата употреба на много негативно пространство и бели полета. По този начин се постига ясно структуриран уеб дизайн с акцент насочен към информацията и водещо значение в цялостната композиция на уеб типографията.

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