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**INTERDISCIPLINARY PROBLEMS  
IN ADDITIVE TECHNOLOGIES**

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# INVESTIGATION OF THE EFFECT OF LASER RADIATION PARAMETERS ON THE STRUCTURE AND MECHANICAL PROPERTIES OF PARTS OBTAINED BY THE SLM TECHNOLOGY FROM A TITANIUM ALLOY

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Today 3D-printing is a fast developing technology that produces three-dimensional objects directly from digital models through additive process, typically by precipitation and "cured in place" successive layers of polymers, ceramics or metals. Unlike traditional production processes associated with subtraction, for example, a cutting process or shaping techniques, such as stamping, bending and molding, AT connects the material layers to create a final product. Originally, this technology was conceived as a method for producing prototypes, but currently additive manufacturing has improved to the extent that they are increasingly used to obtain the finished product. Therefore there are high demands on the quality of the synthesized material to details grown [1].

Selective laser melting technology (SLM) is a fast growing technology that allows to produce metal parts in a short time. Interest in the technology is growing from year to year. At the moment, there are several well known works devoted to the study of physical and mechanical properties and structure of the samples obtained by the SLM. For example, in works [2-3] studies on the impact of the mode selective laser melting refractory metal powder on the structure of the material are presented.

Despite the fact that the SLM technology has advanced considerably in recent years, its use is still limited due to the appearance of defects in synthesized parts, for example, such as hollows and cracks [4]. Also there are questions to the mechanical characteristics of the parts made by the SLM technology [5]. As is well known, the mechanical characteristics of the material obtained by the technologies of selective laser melting differ from the mechanical characteristics of the same material obtained by the traditional technologies.

The quality of the material obtained by the SLM technology is influenced by a large number of parameters, such as the laser emission power, scanning speed, diameter of the laser spot, the thickness of a sintered layer, structure, the properties of the material used, and many others [2]. The study of the influence of the SLM technology on the mechanism of structure formation and mechanical characteristics of the synthesized material is, therefore, of considerable interest.

Titanium alloys are widely used for manufacture of parts in aerospace, medical, automobile, chemical and other fields of industry [6, 7]. The main advantages of titanium alloys in comparison with structural steels are high unit strength in relation to material density, good rust resistance and high mechanical characteristics at influence of high temperatures.

VT-6 alloy (analogue ASTM Grade 5 titanium, Ti-6Al-4V) relates to titanium deformable  $\alpha+\beta$  to alloys. High durability such alloys is achieved through use of a heat treatment. Ultimate strength of material about 885 MPas at rather high plasticity  $\delta=10 \dots 13\%$ . With an increase ultimate strength of material up to 1100 MPas leads to receiving enough low values of plasticity, an order  $\delta=4 \dots 6\%$ . It is the main reason for the fact that BT6 alloy is in most cases used in not strengthened state.

VT6 alloy besides the  $\beta$ -stabilizer contains aluminum which is mainly dissolved in a  $\alpha$ -phase and strengthens it.

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# NUCLEATION MODELLING FOR SYNTHESIS OF THE NEW ADVANCED MATERIALS

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The new material design assumes to make the mixture of some components, which should be transferred into the metastable state and activated to start the chemical reactions or/and multiple phase transformations within the sophisticated technological procedures. Practically all chemical and physical treatments are presenting the energetic barriers overcoming, where new phase embryos are generating within the complicate sequence of the single steps of the new material design. Theory of nucleation is used generally to get a theoretical description and optimizing of these steps.

The first nucleation experiment is associated with measurements of liquid and crystals super cooling done by Fahrenheit (Ostwald, 1896). The nucleation of bubbles in gas saturated solutions was observed and the concept of critical embryos of a new phase was introduced in nucleation science during the second half of 19th century (Volmer, 1939). The quality of vapor/liquid nucleation rate results has improved substantially beginning in 1970s because of the development of new measurement systems. For example, the first prototype of the Flow Diffusion Chamber for vapor nucleation rate measurements was developed by Anisimov et al. (1978, 1982). Currently, the most significant problem in nucleation is the correspondence between experimental data and theoretical predictions of nucleation rate values. As a rule, theoretical and experimental data on nucleation rate are not in good agreement over a range of temperatures and/or pressures. It appears that there may be problems in both the experiments & theory and deficiencies can be identified in all versions of nucleation theories and practically all of the reported experimental results.

Current theories correspond to various modifications of Classical Nucleation Theory that was completed in the 1940s (Frenkel, 1975). The theoretical results look quite reasonable for sufficiently low vapor nucleation rates where the droplet approximation is applicable (Anisimov, 2003). However, these approaches have problems at the nanometer scale when the critical embryos contain of the order of 200 or less molecules (atoms). It appears that this quantity of molecules is near the threshold for the droplet critical embryo approximation, at least for organic vapors. Some researchers (Baydakov, 1995; Protsenko et al., 2006) have expressed optimism that nucleation theory and experiment are in agreement for the case of bubble generation from the superheated liquids.

At the present time, vapor-gas nucleation theory can produce values that deviate from the experimental results by up to several orders of magnitude (Fladerer and Strey, 2006; Brus et al., 2005). However, nucleation experiments using different devises also show significant inconsistencies in the measured rates (for example, see Brus et al., 2005). Both problems produce difficulties in establishing one or more standard vapor/liquid nucleation systems that could be used to test vapor-gas nucleation rate measurement systems. The problem of the nucleation rate standard is more complex than simply using the n-pentanol-helium system as was suggested by the International Workshop on Nucleation in the Czech Republic, Prague in 1995 as a candidate for a nucleation standard. The n-pentanol-helium system has unfortunately not produced sufficiently consistent data to date. The advantages and current problems of the vapor-gas nucleation experiments are discussed below and a view of the future studies is presented based on the assessment of vapor-gas/liquid nucleation experimental results.

In this review, results and problems related to aerosol generation experiments and nucleation theory development are discussed. Adiabatic expansion and gas-jet techniques, diffusion chambers, turbulent mixing apparatus were considered. It can be concluded that the development of accurate experimental techniques for vapor-gas nucleation research are still in progress. Measurable nucleation rates for the available experimental techniques span up to 19 orders of magnitude ranging from  $10^{-2} \text{ cm}^{-3} \text{ s}^{-1}$  up to  $10^{17} \text{ cm}^{-3} \text{ s}^{-1}$  and nucleation temperatures from cryogenic around 30 K to near 1300 K. Pressures in vapor-gas systems have been achieved within the interval from 30 kPa to 10 MPa. The detection of the critical vapor supersaturation for a given nucleation rate tends to be replaced by nucleation rate determinations that can vary by several orders of magnitude.

Important semiempirical information is obtained when the critical embryo parameters are derived from experimental nucleation rate data. These relationships were called "nucleation theorems." Kashchiev (2006) recently provided a theoretical review devoted to nucleation theorems. More easily

used equations to interpret experimental results have been given in several articles (Anisimov and Cherevko, 1985; Anisimov et al., 1987; Anisimov and Taylakov, 1989). Because of the inconsistencies in the experimental data, these relationship and the results derived from them should be considered as preliminary. It can be concluded now that the differences between theory and experimental data are too large to permit reconciliation of the theoretical predictions and experimental nucleation rates. Nevertheless some of the results and ideas reviewed above provide a reasonable basis for further development of the experimental vapor-gas nucleation research techniques.

It can be hoped that the uncontrolled parameter(s) will be identified in the near future and permit consistent nucleation rate data to be derived from different research methods. The introduction of one or several nucleation standard(s) is a major current problem. Success in the nucleation standard development and its introduction in nucleation research practice is a key issue for current nucleation experiments. The deeper understanding of the carrier-gas effects may clarify the nature of the different experimental set data inconsistencies. High pressure measurement systems need to be designed to permit the study of multi-channel nucleation at near critical and spinodal conditions where the nucleation rate surface topology needs to be clarified. While developing new experimental systems, the existing techniques can be used to study nucleation to explore the effects of the carrier gas and multiphase phenomena that need to be further explored. It is reasonable to attract the attention of researchers and practitioners to the new idea of semiempirical design of the nucleation rate surfaces (Anisimov, 2017). That idea can (and will) be used for data base on the nucleation rate surfaces design.

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# CALCULATION OF THE EFFECTIVE THERMAL PROPERTIES OF COMPOSITES BASED ON TITANIUM

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Recently, the scientific interest in Titanium-based composites has increased. Today titanium and its alloys are used in the aerospace industry, architecture, chemical processing, medicine, power generation, marine and offshore, sports and leisure, and transportation. This wide application is explained by its unique properties such as high strength, low density, good high temperature properties, biocompatibility and favorable corrosion resistance [1]. At the same time, the main disadvantages of this material, that has presented problems at some stages in its fabrication and in certain technological uses are lack of wear resistance and a susceptibility to galling [2]. For purpose of enhancing the surface properties of titanium alloys, different methods was employed to fabricate ceramics or intermetallics reinforced metal matrix composite coatings on the substrate. For example, TiC composite coatings offer significantly increased wear resistance and chemical stability [3]. TiB and TiC reinforcements can increase the oxidation resistance of titanium-matrix composites [4]. Dispersion hardening by particles of various compounds, for example, Ti<sub>3</sub>Al and Ti<sub>5</sub>Si<sub>3</sub> compounds, slightly improves the heat-resistant properties of titanium alloys. There are many studies on the mechanical properties of these composites [4]. However, these modifications can also affect the thermal properties of the resulting composite, which are also important for operation in extreme conditions.

To calculate overall thermal properties of the composites we will model them as a metal matrix containing spherical particles. The typical microstructure of metal matrix composite reinforced by particles can be represented as isotropic one. Properties required for calculations are presented in the Table 1 [1,5].

Table 1. Properties

Properties	Ti	TiC	Ti <sub>5</sub> Si <sub>3</sub>	TiB <sub>2</sub>
CTE, K <sup>-1</sup>	8,9-10,4 · 10 <sup>-6</sup>	7,4 · 10 <sup>-6</sup>	7,3 · 10 <sup>-6</sup>	6,4-7 · 10 <sup>-6</sup>
Bulk moduli, GPa	103,7	272	110	226
Young's modulus, GPa	112	460	156	529
Poisson's ration	0,32	0,19	-	0,11
TC, W/(m·K)	15,5-20	34-39	45,9	60-120

In this work we evaluated the effective properties of composites based on titanium, reinforced by TiC, Ti<sub>5</sub>Si<sub>3</sub> and TiB<sub>2</sub> inclusions. The effective properties are calculated using Maxwell's homogenization scheme in terms of contribution tensor of the inhomogeneity [6]. We analyzed change of effective thermal conductivity and effective coefficient of thermal expansion of composites with an increase of volume fraction of inclusions for two methods of calculation: the Maxwell method and mixture method. The results are compared with the experimental data.

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## THE VIBRATIONAL SPECTROSCOPY OF BIOCOMPATIBLE POLYMERS

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Biodegradable polymers are currently used in drug delivery and tissue engineering, polycaprolactone (PCL) being one of the most widely used materials. PCL acts as a bioresorbable scaffold having the ability to stimulate tissue growth. Its surface and bulk structure affects the cell adhesion and the scaffold's lifetime. The surface coating by titanium nitride is applied to increase cell adhesion to the samples, and in the future — implants. The deposition of thin titanium nitride coatings on the fibrous scaffolds' surface enhances cell adhesion [1]. At the same time, the process of coating the polymer scaffolds may modify its crystalline structure. Here, we aim at investigating the structural modifications in polycaprolactone coated with titanium nitride using Raman spectroscopy.

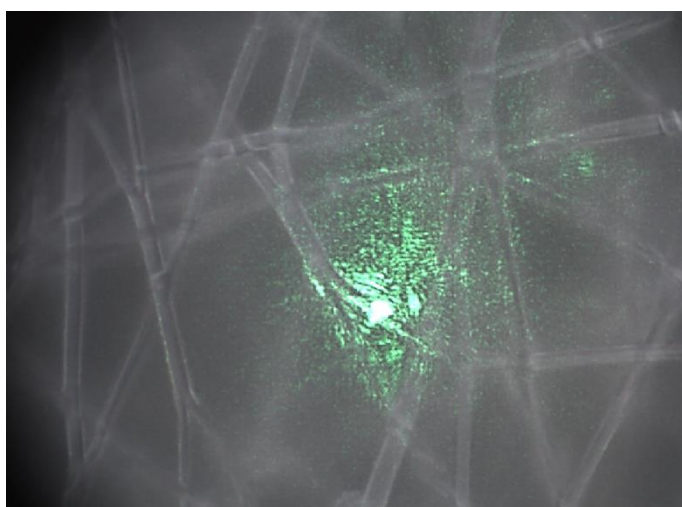


Figure 1. The fiber structure of the sample with green laser

The work was completed according to specifications: on one side of samples produced by the method of electrospinning from polycaprolactone (PCL) with a molecular weight of 80 000 had been deposited thin coating of TiN (titanium nitride) by magnetron sputtering [2]. One of the first goals is to evaluate whether it is possible to see differences that occur by changing the degree of PCL crystallinity depending on coating time from 0 to 240 seconds. Increasing the deposition time increases the amount of titanium nitride on the surface.

The method of Raman spectroscopy was selected for several reasons: the need for samples of small size, simplicity of sample preparation, access to a large amount of information, nondestructive and noncontact analysis. Raman spectroscopy is an optical spectroscopy based on the ability of the studied systems (molecules) to inelastically scatter monochromatic light. The sample is irradiated with a laser of 532 nm wavelength, and the Raman spectra is acquired using a Centaur U HR spectrometer; other lasers with wavelengths 405 nm (violet) and 785 nm (near-infrared) were used in a Renishaw Raman spectrometer. For each sample, at least two points were measured. Previous work showed the dependence of the width of the Raman peaks on the crystallinity of PCL.[2] We analyzed the Raman peaks at 2905, 2852, 1720, 1280, 1110  $\text{cm}^{-1}$ , but only 2 peaks have information about crystallinity: 1110  $\text{cm}^{-1}$  and 1280  $\text{cm}^{-1}$ . The spectra were filtered to remove cosmic rays, the background was subtracted, then all the spectra were normalized and compared. The dependence of the full width at half-maximum from the samples with different coating times of titanium nitride was investigated. The results shown in Figure 2 demonstrate us that samples with longer coating

time up to 120 s measured with 532 nm laser source have a smaller full width at half-maximum (FWHM), as seen in figure 2, consequently crystallinity of this samples is greater. At the coating time of 240 s the FWHM increases again.

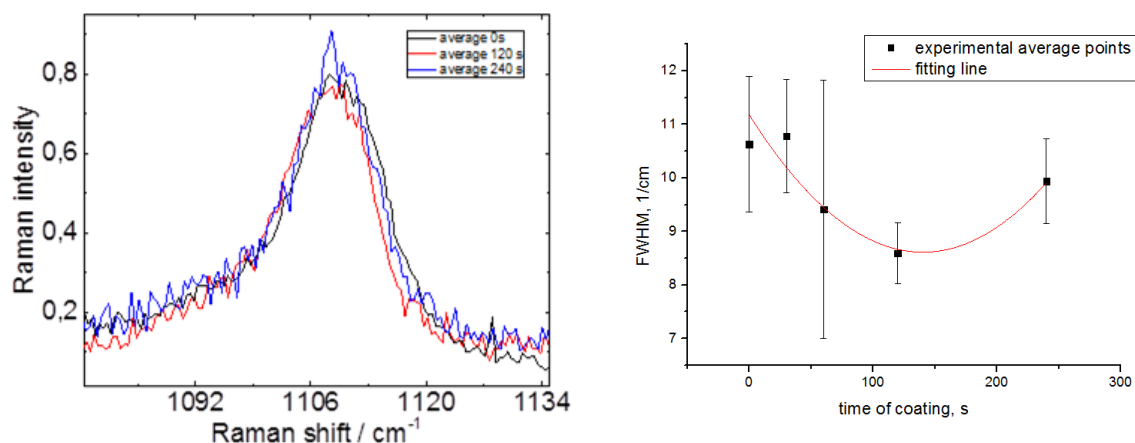


Figure 2. Left: Averaged Raman spectra in the region of the Raman peak  $\sim 1110 \text{ cm}^{-1}$ . Right: The dependence of the FWHM of the peak at  $1100 \text{ cm}^{-1}$  on the coating time using green laser with wavelength 532 nm

Previous studies showed that longer coating times result in improved crystallinity of the scaffolds due to the preferential destruction of the amorphous regions of the polymer in the process of titanium deposition. Also, the heating of the polymer material during the treatment promotes the crystallization process. The increase of crystallinity of PCL fibers is known to lead to the increase in their Young's modulus and mechanical strength [3].

A more thorough evaluation and the comparison of the results with the crystallinity obtained by other characterization methods (such as differential scanning calorimetry (DSC), X-ray diffraction (XRD) and direct measurements of mechanical properties by uniaxial stretching) could establish Raman spectroscopy as a good alternative method for non-destructive testing of crystallinity and mechanical properties of PCL electrospun scaffolds. That is why further work will aim at correlating crystallinity and mechanical properties of PCL electrospun scaffolds obtained by different methods.

**Acknowledgements.** The authors thank I.N. Lapin from Tomsk State University, Russia, for performing Raman measurements with 785 and 405 nm excitation.

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# MATHEMATICAL MODEL OF HIGH-TEMPERATURE SYNTHESIS OF THE Ni<sub>3</sub>Al INTERMETALLIC COMPOUND IN THE MODE OF THE POWDER PRESSING THERMAL EXPLOSION OF THE STECHIOMETRIC COMPOSITION INITIAL ELEMENTS

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The intermetallic compound Ni<sub>3</sub>Al ( $\gamma'$ -phase, ordered solid solution) is the main strengthening phase of modern nickel superalloys, the behavior of which under load largely determines the service life of alloys as a whole. The efficiency effect increase of the use of the intermetallic compound Ni<sub>3</sub>Al as the main component of high-temperature alloys is limited by its high tendency to brittle failure. Polycrystalline Ni<sub>3</sub>Al breaks along the grain boundaries, at both low and high temperatures. Therefore, one of the topical research areas in the field of developing new and improving existing intermetallic high-temperature alloys is to investigate the possibilities of increasing the strength and ductility of the Ni<sub>3</sub>Al compound. Increasing the strength and, accordingly, the plasticity of the intermetallic compound is possible by improving existing or applying new technological processes for its production. The high-temperature synthesis method of intermetallide in a powder mixture of initial elements of a given composition in the regime of a powder billet thermal explosion is the one of the latter. The thermophysical conditions of the synthesis bulk reaction of an intermetallic compound in a powder mixture of pure elements provide the synchronism of the phase transformations course in the entire volume of the powder perform. A unique feature of the thermal explosion process is the ability to consolidate individual structural fragments of the synthesis reaction products when the homogeneous structural-phase state reaches the thermoreacting system by applying an external pressure and forming a homogeneous structural phase state in the final product.

To initiate a bulk exothermic reaction (thermal explosion) in a powder blank, various methods of heating the preform are used. The general principle of heating the preform before autoignition is to minimize the temperature gradients in the volume of the preform under controlled heating conditions. To the fullest extent, the latter is achieved by heating the compact from the original powder mixture in a steel mold-reactor with high-frequency currents. The heating of the mold-reactor walls under controlled conditions to heat the powder compact before self-ignition, followed by a power seal of the high-temperature synthesis product.

The mathematical model of high-temperature synthesis of intermetallic in the thermal explosion regime of a powder compact is proposed. The model takes into account all stages of heating the mixture in a steel cylindrical mold.

The model consists of two parts. It's electromagnetic and thermal.

From the solution of the electromagnetic problem, the dependence of the heater temperature on the strength of the magnetic field was obtained. With a body with this temperature, the heat transfer of the reagent is carried out by thermal radiation, for which the following part of the problem is needed. Using this dependence, we solve the thermal problem of initiating the reaction in a powder mixture in a cylindrical mold. The problem includes the equation of thermal conductivity for a mixture of powders and a steel cylindrical mold, takes into account the heating due to thermal radiation from the inductor and the chemical reaction of formation of the final product of Ni<sub>3</sub>Al.

The problem was solved numerically. The model makes it possible to study not only temperature changes in space and time, but also to investigate in dynamics the influence of the heating conditions and the properties of the powder mixture on the thermal explosion modes in this



technological process. The model allows for taking into account the dependence of thermophysical properties on temperature and composition.

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## PERSONALIZED APPROACH TO OSTEOSUBSTITUTION WITH CERAMIC IMPLANTS

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Personalized approach to medical care – one of the strategies of scientific and technological development of the Russian Federation objectives. Such approach becomes particularly important in the provision of constructive medical care for patients with bone defects as a result of cancer, genetic disorders or complex injuries. In these cases, required an exclusively individual approach, not only to the osteoprosthesis tactic, but also to the design of osteoimplants.

Reconstruction of bone tissue emerging defects is a complex clinical task. Patients who have received medical assistance, have to overcome social and psychological barriers associated with disease. Mostly this applies to patients who underwent prosthetics. Great importance in this case have the aesthetic side of prosthesis, especially in the face area.

The solution to the Existing problem solution can be find in the osteoimplant high-precision formation according to the 3D model of the planned for resection bone site. Class of oxide ceramics [1, 2] is the most preferable as a material of an implant, in case of the lack of polymers and metals risks of causing an acute inflammatory reaction due to their low biocompatibility and integrating capacity with bone tissue. The identity of chemical bonding type and the mechanical parameters of ceramics to natural bone tissue prevents the potential possibility of disrupting bone physiological functionality and structural and phase changes in the bone tissue [3].

This work is the result of the consolidated scientific work of materials scientists, biologists, physicians and engineers and consists in developing a technological approach to the bone defects reconstruction in the skull visceral region by personalized implants based on porous ceramic osteosubstitution material. The technology consists in individual 3D-modeling of endoprosthesis based on a patient computer tomogram, with subsequent constructed model reproduction in ceramic material by methods of additive production.

At present time, the first microsurgical reconstruction of the face left middle zone with the ceramic implant has been carried out, figure 1. Using of this technology is not limited by the skull visceral region and can be extrapolated to other skeleton areas [4].

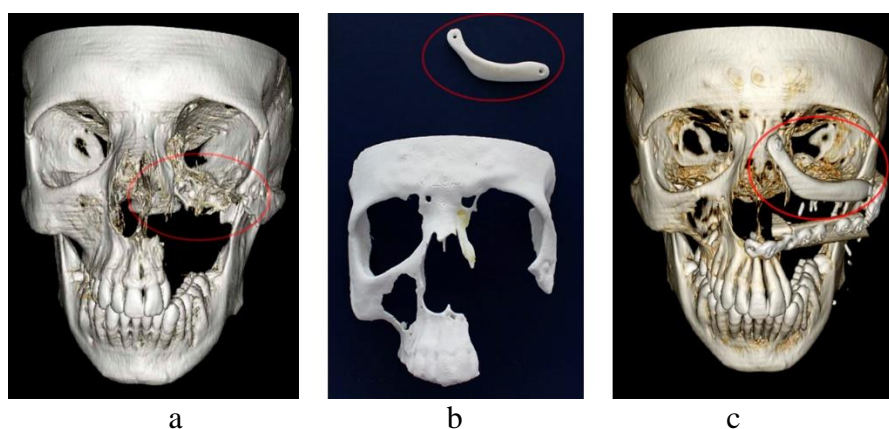


Figure 1. a) Computer tomogram with a marked area of prosthetics; b) Ceramic osteoimplant and stereolithographic model of the skull made of plastic, obtained by the additive prototyping techniques; c) Postoperative computer tomography with a marked implanted ceramic endoprosthesis.

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## MECHANICAL TREATMENT OF COMBINE (ZrB<sub>2</sub>-SiC)-POWDERS FOR ADDITIVE MANUFACTURING OF HARD CERAMIC COMPOSITES

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The refractory compounds are the basic components of materials used in high-temperature engineering, such as thermal protection of space vehicles, electronics, etc. Among materials with high melting temperature, a special attention is paid to ZrB<sub>2</sub>-based composites but due to a very high melting point the manufacturing of products based on these materials are difficult because a treatment of materials are almost impossible due to a high hardness. The new technique called additive manufacturing may be used for making samples with finely shapes and sizes after sintering process however it is needs to prepare such powders before forming and sintering.

It is known, that addition of SiC to ZrB<sub>2</sub> increases the density of sintered materials due to smaller melting temperature of SiC. The ZrB<sub>2</sub>-SiC composites are usually obtained under pressing at temperatures higher than 2000 °C [2], and to decrease the sintering temperature, the powders undergo mechanical treatment in high-energy ball-milling. In this case, subsequent sintering will be activated due to increased number of defects, acceleration of diffusion processes etc., so sintering can be carried out during SPS-process. Unfortunately, data on the influence of mechanical treatment on the properties of powders and the process of subsequent hot pressing are poorly investigated. So, the aim of this paper is to study the influence of mechanical treatment of ZrB<sub>2</sub>-SiC powders on their properties and properties of ceramic composites sintered by hot pressing.

The research was carried out using powder mixtures of ZrB<sub>2</sub> (d<sub>50</sub> = 2.5 μm) and SiC (d<sub>50</sub> = 4.2 μm) with SiC content of 10, 15 and 20 vol.%. The powders were mechanically treated in a planetary mill with acceleration of approximately 30g with time up to 20 minutes. Hot pressing of ceramic composites was carried out at the temperature of 1800 °C and pressure of 50 MPa with isothermal sintering for 30 minutes. X-ray with CuKα radiation was used to study structure, phase content and coherently diffracting domains (CDD). Scanning electron microscope Tescan VEGA-3SBH was used to determine the structure and average grain size.

It have been shown that increasing of treatment time are accompanied by increase of relative density, in addition, the morphology of particles has appreciably changed, from separate particles in the beginning state up to the formation of agglomerates in the end of treatment. The X-ray phase analysis of mixtures has shown that during the treatment there were no changes; addition of SiC to the mixture leads to the occurrence of its peaks. With increased treatment time, we have found a broadening of peaks due to increasing number of lattice defects and decrease of CDD or grain size from 46 down to 37 nm.

After sintering phase content did not change and the increase of treatment time in the planetary mill before sintering have no effect on CDD of sintered materials. This means that all defects are annealed during sintering process.

Addition of SiC leads to essentially increased sample density: its value goes up to 99% of a theoretical one for a powder with 20% of SiC, as compared to ZrB<sub>2</sub> going not higher than 76%.

It have been shown that relative density change of sintered materials are well described by a simple function like  $Y = A \cdot x^n$ , where parameter  $n$  characterizes the speed of density change and according results addition of SiC to the mixture leads to up to four-fold decrease of  $n$ -value. This is due to that all defects accumulated during mechanical treatment are annealed during sintering and there are no changes of CDD values in sintered ceramics.

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# MODELING OF COMPOSITE SYNTHESIS DURING SHS IN THE TI+C SYSTEM

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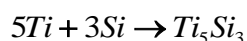
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One of the methods of composite manufacturing based on titanium with inclusions of carbides borides and silicides is self-propagating high-temperature synthesis (SHS) or combustion synthesis. However, this process is nonequilibrium process and it is not possible to predict the composition of the synthesis product because of the presence of a wide range of homogeneity on the phase diagrams of some systems. Therefore, the aim of this work is to develop a model and theoretical study of the synthesis of the *Ti-TiC* composite in the combustion regime.

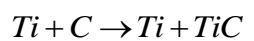
It is known that, an igniter is usually used for difficult flammable non-gas systems to initiate the reaction, which is brought into contact with the ignition system. Ignition of the reaction mixture in this case is carried out by a combustion wave going along the igniter to the place of contact of the materials.

A mathematical model of the process of initiation of the reaction in a powder mixture of a metal (*Ti*) and graphite (*C*) is considered. The sample is a cylinder of radius *r* consisting of two layers of powder fillings (figure 1). We assume that layer *l* (igniter) is a stoichiometric mixture of *Ti* and *Si* powders and the thickness of the layer is *l*. The second layer of thickness *L* (reaction mixture) is a mixture of *Ti* and carbon *C* (carbon black) powders.

We assume that titanium in the second mixture is presented in excess, so that it is not completely consumed in the reaction. The fraction of titanium *Ti* that is not consumed in the reaction is  $\eta_e$ . We consider the chemical transformations in the system are described by the total reactions "reagent-reaction product" for the first layer



and second layer



As a result, we must obtain a composite of the form titanium-titanium carbide.

In the energy equation, we take into account the heat losses to the environment due to convection (if the synthesis is carried out in an inert gas atmosphere) and due to thermal radiation. Excess titanium consumes heat and it role an inert component. This is formally taken into account through the heat capacity. We consider the kinetic equation for conversation level corresponding to the reaction with strong retardation of layer reaction product. The melting of the component with the lowest melting point (*Ti*) was taken into account by changing the effective heat capacity and density in the vicinity of the melting temperature.

Since the structure of the powder system is changing and is unknown at any time, we use the rule of the mixture to calculate the effective composite properties. The effective coefficient of thermal conductivity of the mixture was calculated similarly.

As calculations showed, an increase in excess titanium  $\eta_e$  leads to a decrease in the temperature in the second reaction layer and a slowing of the propagation velocity of the reaction front, which qualitatively corresponds to the experiment. A stationary combustion wave in the igniter is not formed. The formation of a wave occurs almost instantaneously in the reaction mixture. The termination of the reaction after initiation is realized with the inhibition parameter of  $m = 25$  and  $\eta_e = 85\%$ .

The work is supported by Russian Science Foundation (RSF), grant number 17-19-01425.

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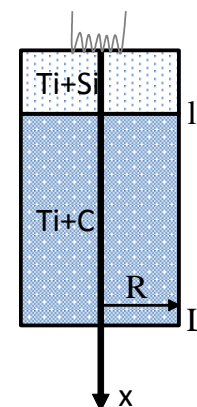


Figure 1. Scheme of process

# NANOPOWDERS PRODUCTION OF AL BY ELECTRICAL EXPLOSION OF WIRES

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Metal powders nowadays are in usage in additive technologies, they play a role of “raw materials” in 3D printing.

Main ways of metal powders obtaining are: plasma dispersion, gas atomization, centrifugal dispersion, etc., each method has its own advantages and disadvantages.

Electrical explosion of wires (EEW) allows to obtain sphere shaped particles which find their application in 3D printing.

The main advantage of this method is simplicity and universality of the equipment, low energy consumption and possibility of receiving product's structure and size regulation.

In current work it is shown that it is possible to change the particle sizes of aluminum wires obtained by the electric explosion method by adjusting the energy level introduced into the conductor during an explosion, and also changing of structure of the gas environment in which EEW is carried out.

It is established that the powders received in the EEW modes with the low level of the energy injected into the wire –  $0.4e_s$  ( $e_s$  – specific energy of sublimation of aluminum  $32.9 \text{ J/mm}^3$ ) in the environment of argon consist at least of three fractions.

The average size of the first fraction - large fraction is 30 microns (figure 1, a), the entrance is about 55%, the second fraction consists of particles from 2 to 6 microns. The average size of the third one is nanometers, 40 nm.

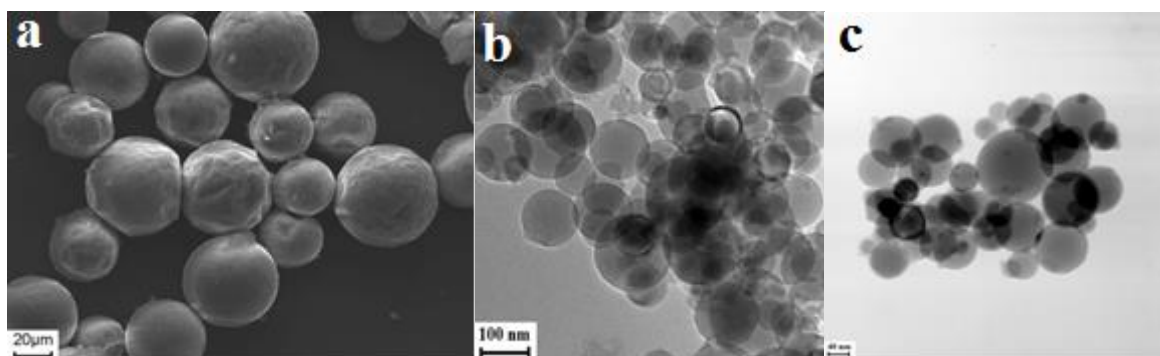


Figure 1. Photos of Al of the powders received at various levels of energy.

a) Ar -  $0.4e_s$ ; b) Ar -  $1.9e_s$ ; c) He -  $1.9e_s$ .

Increasing of energy up to  $1.9e_s$ , at EEW in argon leads to "disappearance" of micron fraction of particles and reduction of the average size of particles of nanometer fraction up to 30 – 300 nanometers with a maximum at 110 nanometers (figure 1, b).

EEW in the environment of helium at energy of  $1.9e_s$  allows to receive powders with the largest specific surface area – up to  $20 \text{ m}^2/\text{g}$ , with the narrow histogram of particles size distribution and with the average size about 80 nanometers (figure 1, c).

With reduction of the size of particles also the content of metal aluminum in them decreases. It is caused by increase in their reactionary activity. Particles with an average size of 30 microns at 99% consist also metal. Particles with an average size of 400 nanometers contain – 93% of Al, and in powders with a size of particles about 100 nanometers the content of metal is about 87%.

This study was supported by the Russian Foundation for Basic Research (grant RFBR-mol-a 16-38-00051)

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# ELECTRON MULTIBEAM ADDITIVE PROCESS FOR HIGH PRODUCTION MANUFACTURING LARGE METALLIC COMPONENTS

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Fast development of additive manufacturing is a global challenge in modern technology development, which determines provisions for developing and using new advanced, high-production and competing processes. This work has been focused on developing an electron multibeam additive directed energy wire deposition process and commercial high-production robotic equipment for manufacturing large metallic components.

This process involves a layer-by-layer deposition of metal by electron beam melting of wire and obtaining then an near-net-shape component. The advantage of this process is its high deposition rate up to 12 kg/h which is unachievable with other additive processes. Also it allows making large up to 5000 mm size fully dense and structurally homogeneous components from both refractory and heat-resistant alloys. Extra feature of this process is a feasibility of simultaneous deposition of dissimilar metals and thus forming a composite structure inside a vacuum chamber. Therefore, it excludes any oxidizing of the component [1-3].

Samples will be manufactured from hot-strength alloy ZhS6U, stainless steel 12Kh18N10T and titanium alloy VT-6. The process should meet the requirements as shown in Table.

Table 1. Equipment characteristics

Building rate	5 - 12 kg/h
Wire feed	automatic
Number of wire feed devices	1 to 3
Number of electron guns	1 to 3
Residue pressure in a chamber	$\leq 1 \times 10^{-4}$ mm mercury column
Positioning accuracy	$\leq 0,1$ mm
Max current	100 mA
Max voltage	30 kV
Focal distance	200 mm

The project implementation allows filling a commercial niche both in home and abroad markets of equipment and materials needed for high-production additive manufacturing of large complex shape components. Achieving such a goal will provide our technological leadership in high-production electron beam additive manufacturing.

The work was financially supported by the Russian Federation via the Ministry of Education and Science of the Russian Federation (Agreement No. 14.610.21.0013, project identifier RFMEFI61017X0013).

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# THE MICROSTRUCTURE OF HETEROGENEOUS MATERIALS BASED ON NI AND B4C POWDERS USING A COLD SPRAY AND STRATIFIED SELECTIVE LASER MELTING TECHNOLOGIES

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The work is dedicated to the creation of new ceramic-composite materials based on boron carbide, nickel and using a laser treatment in order to obtain three dimensional objects henceforth. The perspective way of obtaining which has been suggested by the authors combined two methods: cold spray technology and subsequent laser post-treatment [1,2]. At this stage, the authors focused on the interaction of the laser with the substance, regardless of the multilayer object development. The investigated material of this work was the metal–ceramic mixture based on boron carbide, which has high physical and mechanical characteristics, such as hardness, elastic modulus, and chemical resistance. The titanium alloy VT-20 was used as substrate. The nickel powder as a binder and different types of boron carbide were used. The ceramic content varied from 0 to 90% by mass in initial mixture. The cold spray coating thickness was ranged from 250 to 50  $\mu\text{m}$ . Cold spray coatings could be characterized as heterogeneous cermet coatings (Fig. 1a) with low porosity and ceramic content at range 0 to 27% by mass. After laser melting (Fig 1b) is shown compaction of deformed nickel particles, reduction of pores in the volume, smoothing of the coating surface, disappearance of the boundaries between the coating particles.

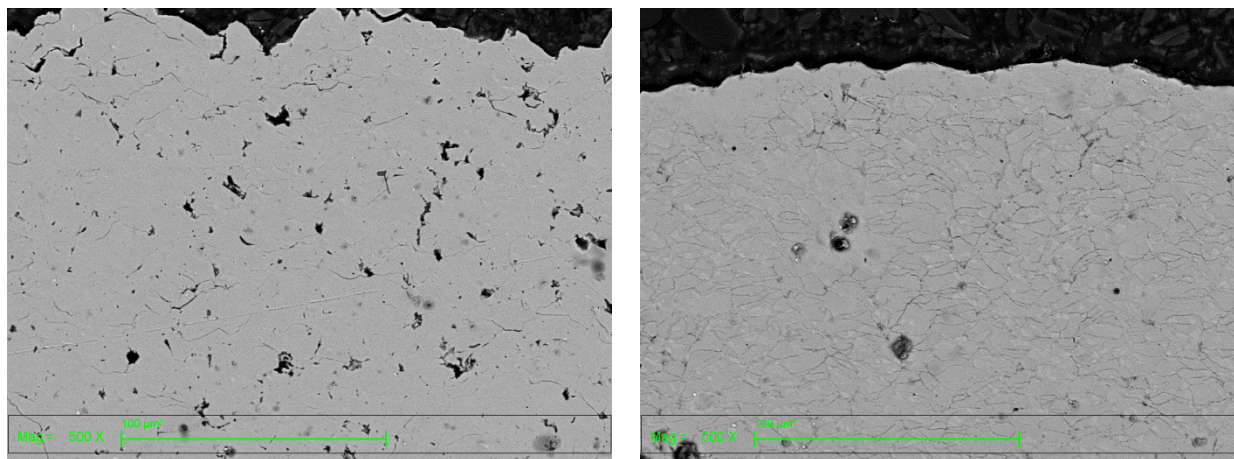


Figure 1. SEM-image cold spray coating pure nickel a) before and b) after laser treatment

Thin ceramic layers were obtained by the combined method and cross-sections microstructure of different seams were studied. It was shown that at low ceramic concentrations melted coating contains bubbles with ceramic particles. At ceramic concentrations 90% in initial mixture a continuous cold spray coating layer transforms to seams under laser radiation. There are some chemical reactions in the seam cavity (Fig.2 a,b). The authors made an assumption about the chemical transformation of boron carbide to whiskers of titanium carbide as at work [3]

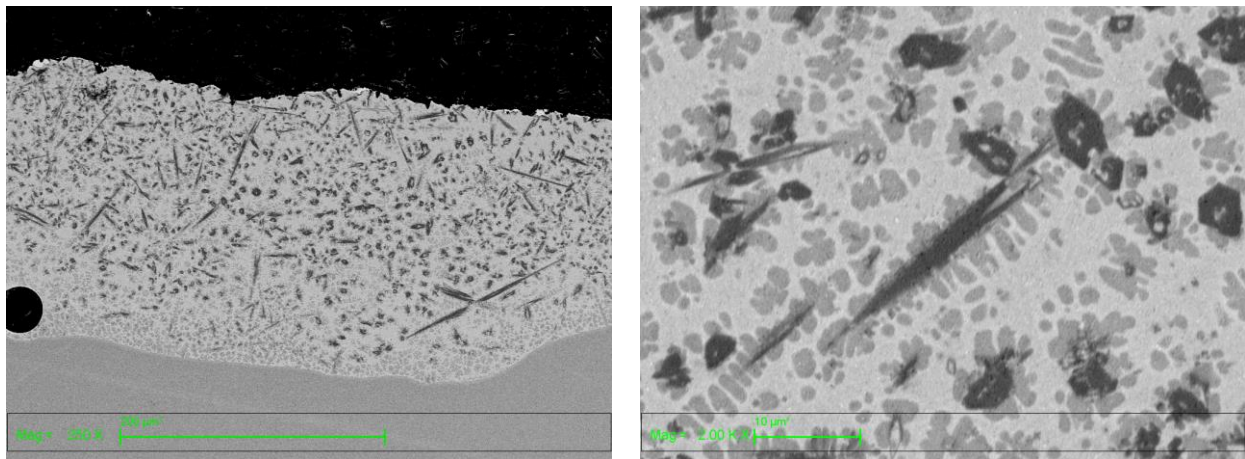


Figure 2. SEM-image the seam cavity cermet coating with different magnification:  
a) 250X, b) 2000X

**Acknowledgements.** This work was supported by a grant of the Russian Scientific Foundation "The physical basis for the creation of heterogeneous materials using additive technologies" №16-19-10300.

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# ANALYSIS OF THE THERMAL PROCESSING INFLUENCE ON THE MICRO-STRUCTURE OF A METAL-CERAMIC 3D PART CREATED BY THE ADDITIVE TECHNOLOGY METHOD

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Today, the time of production of wear- and corrosion-resistant coatings is a topical task. The coatings with high physical and mathematical characteristics are necessary in many areas of industry (high-strength cutting tools, elements of protection for parts acting in aggressive media, etc.). To solve these problems, metal-matrix composite coatings based on mixed metal powders and ceramics of different chemical content are used [1,2].

The paper deals with the influence of the thermal processing on the micro-structure of a 3D composition created by the additive technologies method. A multi-layer coating is created by the CO<sub>2</sub>-laser; it consists of the WC and Ni powders mixture. The thickness of the grown composition is about 5 mm. Then the samples are put in a furnace for two hours, the temperature regimes 700°C, 800°C, and 900°C. The thermal processing regimes were chosen on the base of [3]. Analysis of the microstructure is carried out on an electronic scanning microscope Zeiss EVO MA 15. The change in the clad micro-structure after the thermal processing is discovered (Fig. 1). The micro-hardness of the clad coating is measured.

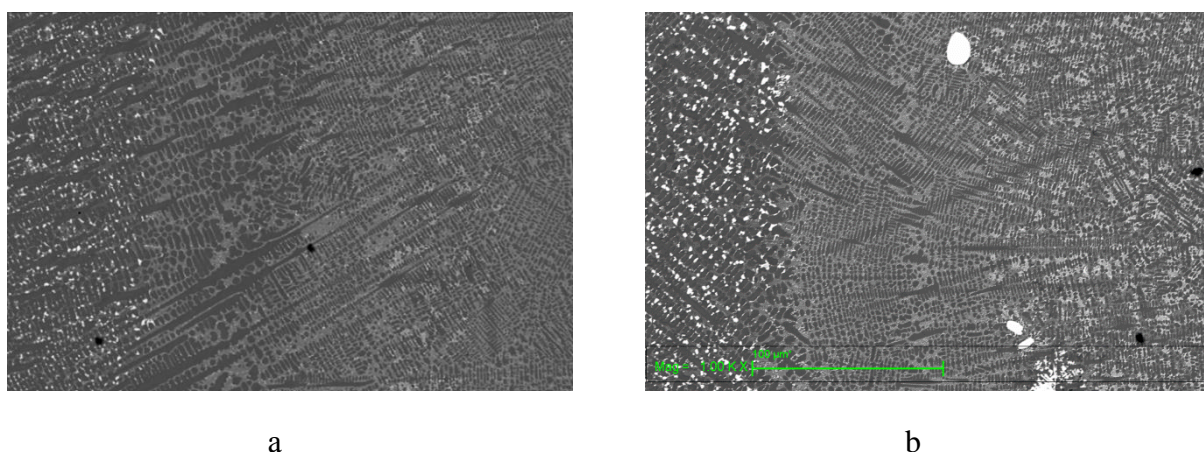


Figure 1. Photos made by the electronic scanning microscope  
a) – the sample without post-processing, b) – the sample after 800°C thermal processing

**Acknowledgements.** This work was supported by a grant of the Russian Scientific Foundation "The physical basis for the creation of heterogeneous materials using additive technologies" №16-19-10300.

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# THE IMPACT OF THE STRATEGY OF LASER TREATMENT ON STRUCTURE FORMATION OF STEEL IN A STATE OF PRE-TRANSFORMATION

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**Introduction.** Study of the relationship between the temperature distribution and the formation of regions with different structure in the heat affected zone (HAZ) is an urgent task, since this could make it possible henceforward to carry out optimization of laser cutting technology (LCT), including in terms of preventing a significant decrease in the mechanical characteristics of the processed material in the HAZ. This work is devoted to the study of the temperature distribution near the cutting zone after LCT by example of steel St3 sheets and its comparison with the location of various structural domains found in the HAZ.

**Materials and methods.** The object of investigation was sheets of steel St3 (A53 AISI), with a thickness of 6 and 10 mm. LCT was carried out using a unit equipped with an EIP1119 optical head produced by STC IRE-Polus [1,2]. The parameters of laser cutting were varied within the following ranges: radiation power  $W = 1200\text{--}1900$  W, cutting speed  $V = 700\text{--}1600$  mm/min, gas pressure  $P = 0.015\text{--}0.05$  MPa, focal length  $F = 295\text{--}305$  mm. The investigation of the HAZ microstructure was performed using an Observer Dim light microscope at  $\times 200$  and  $\times 500$  magnification. For making microsections  $10 \times 25$  mm in size, the samples were cut out near the surface of the cut after LC performed in different modes. The etching of the samples was performed via immersion in a 4% solution of nitric acid in alcohol.

**Results and discussions.** A calculation is performed for the temperature distribution and studies are conducted for the structure of the HAZ in sheet steel St3 with a thickness of 6 and 10 mm after LCT. It is shown that, with increasing LCT speed, the temperature in any section of the HAZ is reduced. Increasing laser power in the studied range has almost no effect on the temperature distribution in the HAZ. The temperature fields appearing in the course of LCT are compared with the location of the HAZ regions differing in structure. The area with ferrite structure under heating stays in a state of pre-transformation before a eutectoid transformation; the cause of its formation could be an enhanced diffusion mobility of interstitial atoms near the critical point  $Ac_1$ .

**Conclusions.** The calculation of the temperature distribution in the heat-affected zone during the laser cutting of steel St3 sheets for different modes has allowed us to compare the temperature field with the arrangement of different structural domains and to reveal that the area with the most defective structure in the course of heating was in a pre-transformation state before the eutectoid transformation. (2) The estimate of the carbon diffusion coefficient in the heat-affected zone of steel sheets indicates an accelerated diffusion of carbon in the course of laser cutting and confirms the possibility of decarburization of the alloy areas the temperature of which corresponded to a temperature range inherent in the state before transformation in critical point  $Ac_1$ .

**Acknowledgments.** The work was performed within the framework of the Federal program «Research and development on priority directions of development of scientific-technological complex of Russia for 2014-2020» on the theme « Development of prototype of IPO based on high-performance computing to evaluate mechanical characteristics of products manufactured using additive technologies (by means of selective laser sintering) based on the strategy of making» (the unique identifier of the project RFMEFI57717X0271).

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# MODELING OF SPECIAL CONDITIONS OF DIFFICULTLY DEFORMABLE HETERO-PHASE METAL SYSTEMS

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When developing engineering software for predictive modeling of the state of a product in the process of its creation by the method of selective laser sintering, one of the questions is the development of a methodology for modeling hard-to-deform heterophase metal systems. The methodology of complex investigation of processes of increased deformation capacity (superplasticity) by methods of mathematical modeling, phase and structural analysis of hard-deformed heterophase metal systems (on the example of their typical representative - multicomponent high alloy steel of the iron-tungsten-molybdenum-chromium-vanadium-carbon system of the traditional redistribution of steel - grade P6M5).

With the use of optimal experiment planning and mathematical modeling, adequate mathematical models of the processes of plastic forming of steel for various stress state schemes have been constructed. On the basis of developed multifactor mathematical models of isothermal deformation of P6M5 steel, temperature-velocity intervals of increased plasticity and superplasticity (SP), kinetics and staging of their manifestation are determined.

Systematic data on the temperature-velocity parameters of SP in the interval of temperatures of phase transitions 750...850°C and strain rates 0.0001 ... 0.042s<sup>-1</sup> are obtained. Temperature-velocity maps of lines of equal values of the investigated criteria ( $\sigma$ ,  $\delta$ ,  $m$ ,  $Q$ ), increased plasticity and superplasticity of P6M5 steel are constructed. The superplasticity temperature range for steel draft was 800...820°C, and the strain rate is from 0.002 to 0.005s<sup>-1</sup>. When stretching, the temperature intervals of increased ductility ( $\delta > 90\%$ ) and superplasticity ( $\delta > 120\%$ ) are detected: 760...770°C and 825...835°C. Superplasticity develops intensively at a temperature of 830°C and a deformation resistance of 80 MPa.

It is established that superplastic deformation increases the dispersion of the carbide phase and the chemical homogeneity of the P6M5 steel. In the case of draft and tension in the region of increased plasticity, the dispersion of the steel structure also increases in comparison with the initial state. Formation of blanks from steel R6M5 with increased performance characteristics during draft is ensured by SP due to increased dispersion and structural homogeneity. Degree of deformation of steel P6M5 with increased ductility exceeds 90%, and at SP 120%.

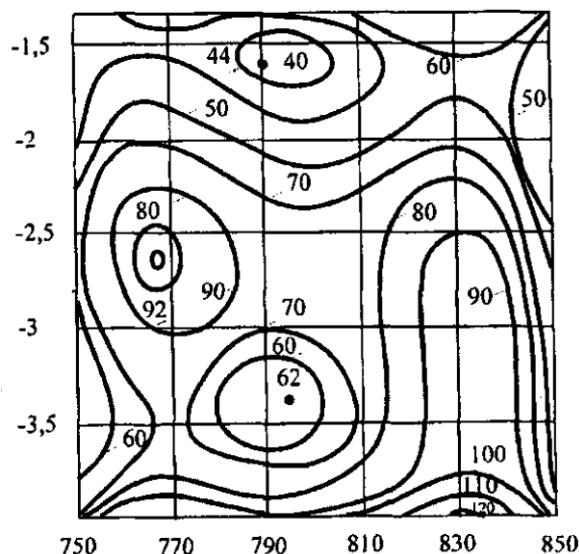


Figure 1. Lines of equal values of the relative elongation  $\delta$  when tensile steel P6M5

A method has been developed for producing blanks of a metal cutting tool made of P6M5 steel under conditions of increased ductility and superplasticity, which allows to increase of coefficient of metal utilization to 0.8...0.9, reduce energy consumption and increase durability tool in 1,8...2,2 times. This allows it to be attributed to low-waste, resource-saving technologies of rational nature management. It is established that a protective coating based on liquid glass in SP conditions allows increasing the service life of the steel deformation capacity by 15...20%.

Areas of increased ductility ( $\delta > 90\%$ ) at 770°C and superplasticity ( $\delta > 120\%$ ) at a temperature of 830°C are found in which it is advisable to conduct resource-saving treatment of P6M5 steel at high degrees of deformation (Fig.1).

Analytical models of the effective activation energy of the process of plastic deformation under tension in a temperature-velocity field are obtained. It is established that a correlation exists between the deformation resistance  $\sigma$ , the relative elongation  $\delta$  and the effective activation energy of the plastic deformation  $Q$ : a decrease in  $\sigma$  corresponds to an increase in  $\delta$  and a decrease in  $Q$  in the temperature-velocity field under study.

Modeling of special states of metal systems (states of pre-transformations, increased deformation capacity, superplasticity) can be used to develop low-waste and resource-saving technologies for processing materials under various conditions and conditions [1-5].

The work was performed by the L.N. Tolstoy Tula State Pedagogical University with financial support from the Ministry of Education and Science of the Russian Federation (Project 14.577.21.0271, project ID RFMEF157717X0271).

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## APPLICATION OF AUTOMATED TECHNOLOGICAL COMPLEXES IN ADDITIVE PRODUCTION

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Many world manufacturers of products consider technologies and equipment of additive production (AP) as a means of obtaining the final product. AP provides an opportunity to implement innovative projects that in the past were considered impossible or unprofitable. Modern equipment and AP technologies are built on the layer-by-layer synthesis of products, but differ from each other by the materials used, the methods of creating and joining the layers among themselves. These differences determine not only the physical and mechanical properties of the material and the accuracy of the resulting shape of the product of the specified model, but also affect the manufacturing time, the need for finishing and the total cost of the product.

Among a wide variety of additive technologies, one of the most promising and least expensive directions is the layered formation of a model of sheet material (Laminated Object Manufacturing - LOM).

Scheme layered formation of products from sheet material is shown in the figure. It is based on the algorithm for breaking the computer model into layers. Cutting sheet material is carried out using modern automated systems.

The initial information for the product can be a solid model (when designing a new product), prepared in a graphic editor - Creo, KOMPAS 3D, SolidWorks, Unigraphics, etc. or a digital model obtained with the help of 3D scanners and coordinate measuring machines (when copying a standard).

At the next stage of production, the computer model of the product is rationally divided into layers of different thicknesses, according to a certain algorithm, which takes into account the possibility of cutting the edges at an angle.

After sorting layers by thickness, the contour of each layer is formed on an automated cutting complex. To program the contour to be processed, it is necessary to use the CAM (Computer Aided Manufacturing) module (Hypermill; Unigraphics; Turbonest, etc.), designed for the preparation of control programs for CNC-controlled machines. Writing the program code depends on the CNC system (Sinumerik; Fanuc; Heidenhain) used in the automated system. Programming is carried out using G-code.

The expanded sheet material (product layers) enters the subsequent layer-by-layer assembly, which can be performed in various ways, indicated on the diagram (figure 1) depending on the intended purpose of the product. If necessary, the product assembled for this technology can be further processed.

The final production cycle is the control of the mechanical and geometric parameters of the finished product.

The use of modern technological complexes with the automated control system makes it possible to prepare and cut edges along a curved path at an angle, which significantly reduces the number of product layers due to an increase in their thickness and reduces the need for subsequent processing of the resulting product.

To improve the quality and reduce the production time of non-separable products using LOM technology, it is advisable to cut and assemble the product using concentrated energy flows, which makes it possible to perform these operations on the same equipment while reducing interoperational time and transportation costs. All this affects the decrease in the final cost of the product.



Technological approbation on the basis of JSC "NPO Center" of the National Academy of Sciences of Belarus has shown that the use of modern technological complexes with the automated control system provides a transition to the application of additive technology of sheet cutting and assembly of products.

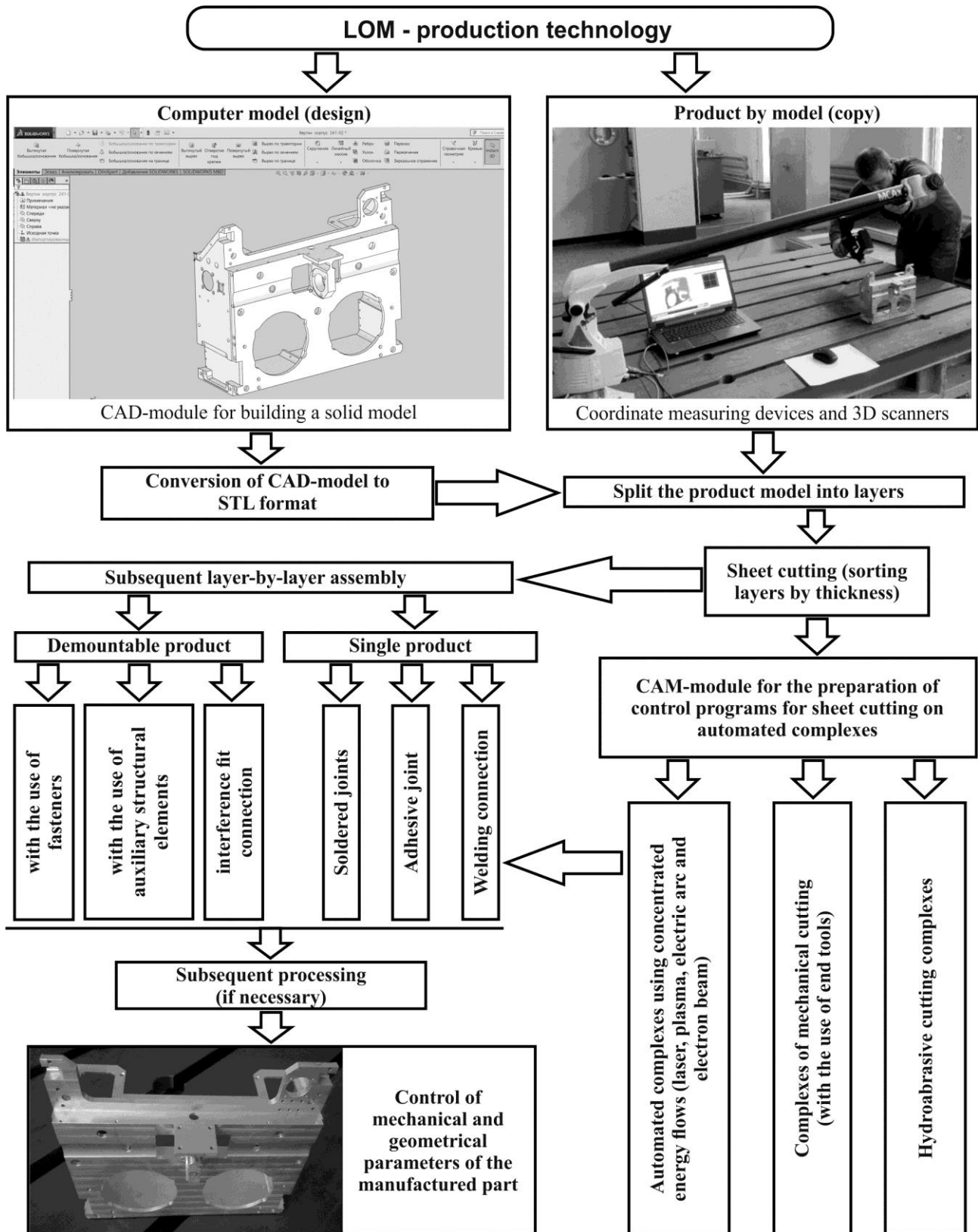


Figure 1. Scheme of additive production by the method of layered product formation

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# MODELING 2D MATERIAL HETEROSTRUCTURES FOR OPTOELECTRONIC APPLICATIONS

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Scientific progress does not stand still, with it the requirements for the rapid operation of modern technologies are growing. Current materials' characteristics are not satisfying the growing standards for future development. Recently, materials such as graphene, MoS<sub>2</sub>, and graphene oxide, which represent a group of 2D materials, have become more prominent to scientists. These materials have proven themselves as highly effective for wide applications in the fields of electronics and optoelectronics not only due to their remarkable electrical and optical qualities, but also because of their mechanical features such as high strength. The use of these materials opens up a completely new format for the vision of the current world of electronic devices. A good example of 2D materials is gallium selenide (GaSe). However, there remains a large set of physical and chemical properties that can be simultaneously exploited in this kind of materials for high-performance applications. This contribution provides computational and experimental results on the enhanced photonic properties of GaSe for applications in optoelectronics.

The objective of this work is to determine the light trapping capabilities of 2D semiconductor layers. We aim at providing a framework for engineering the physical dimensions of 2D layers to maximize the quantum efficiency and performance of optoelectronic devices. Thin layers of GaSe are investigated by the finite element method programming environment, which allows us to study the influence of light on the material using electromagnetic wave frequency domain physics. The Figure 1 illustrates the model of studying material – GaSe as a layer of 140 nm thickness placed on a highly ordered pyrolytic graphite (HOPG) substrate, illuminated by plane wave with  $\lambda=600$  nm. The choice of the direction of the propagation vector is conditioned by the heterogeneous layered structure of GaSe. We perform a multiparametric sweep investigation as a function of GaSe thickness and incident wavelength that allows to maximize the light trapping efficiency of GaSe.

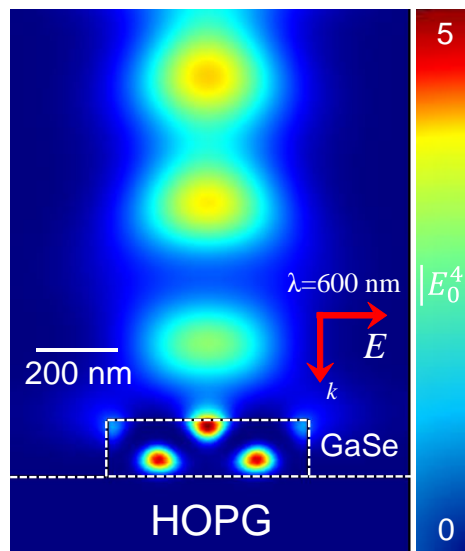


Figure 1: Finite element method simulation result of a GaSe layer on HOPG illuminated by a plane wave of 600 nm wavelength. The electric field enhancement reaches values of about 500% increase with respect to the magnitude of the incident excitation light.

We compare the simulation results with the experimental observations of electric field enhancement obtained by hyperspectral Raman spectroscopy imaging. As a Raman active probe, a 2 nm ultrathin layer of cobalt phthalocyanine (CoPc) was deposited on the GaSe/HOPG sample under ultra high vacuum conditions using organic molecular beam epitaxy. The hotspots, regions with highest electric field enhancement visible in Figure 1 are located at the top and bottom of the GaSe layer enhancing both the substrate and the CoPc layer on top of it. The computational result is in agreement with the experimental observations shown in Figure 2, where the Rayleigh and Raman signal from the substrate and CoPc are enhanced. This is exactly the effect that we hypothesized. Our research results show that the light trapping capabilities of these layers used in optoelectronic devices could be largely increased.

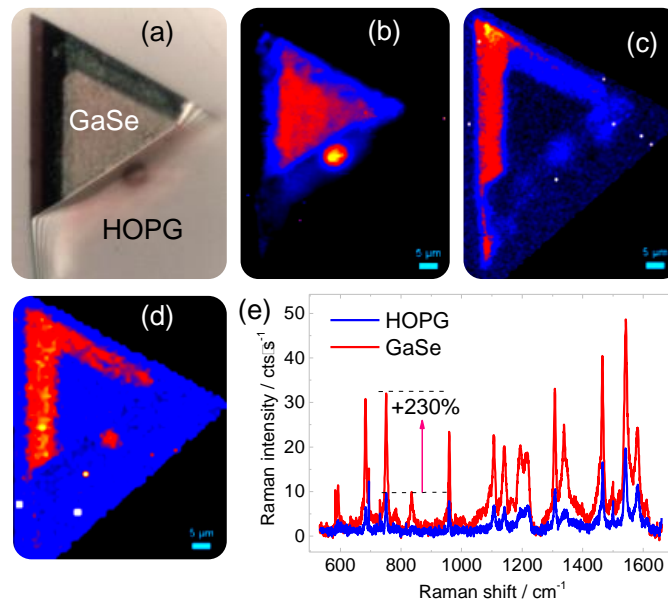


Figure 2: a) Optical microscopy image of a GaSe flake on HOPG. b) Rayleigh scattering image, c) CoPc Raman intensity for the mode around  $1435 \text{ cm}^{-1}$ , d) Raman signal from the G mode of HOPG, e) Raman spectra comparison for regions with and without GaSe.

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# THE DEVELOPMENT OF ADDITIVE TECHNOLOGIES FOR PRODUCTION OF CERAMIC COMPONENTS AND PARTS OF A NEW TYPE BY MEANS OF SELECTIVE LASER MELTING

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Increasing demands for range and intensity of maneuvering hypersonic vehicles at the moment is hampered by the complex dependent factors: weight, heat resistance and mechanical strength of structural materials at elevated temperatures. Existing to date technology of hot pressing and conventional sequential molding followed by sintering of ceramic materials, can not provide the required characteristics of the structural parts for hypersonic vehicles. This is especially true of hollow ceramic parts of cellular structure. Technology of production of ceramic units and parts by selective laser fusion, which refers to the additive technology will enable the engineering industry to solve complex materials science problems and to create a programmable structural material of cellular structure. Therefore, the formation of products on the basis of ceramic materials is developing in the direction of use of the additive technologies.

The significance of the problem being solved with the use of additive technologies from the perspective of overcoming technical, technological, resource, environmental limitations relevant to the areas of development of the country is formed by the set of advantages of additive manufacturing technologies over traditional machining of workpieces. So, for example:

- a significant savings when production is started. The data required to start the production, can be stored digitally, and reproduced without material cost;
- ability to make amendments at any stage by a simple adjustment of the CAD file;
- customization production line: additive technologies allow to make party, in which every object (item) may be different from the previous one.
- the effectiveness of additive technologies is the increase in utilization of material, reduced weight and lower cost parts through the optimization of the technological cycle.
- ecological purity of production, since the space of additive machines are generally closed and includes in its design the filtration system, all the gases passing through the chamber is filtered from particles of powder. Unused powder during the process is sent back to the head of the production cycle.

It should be noted that the analysis of modern trends of development of the relevant area of science and technology shows a rapidly growing segment of additive manufacturing refers to production technology of ceramic components and parts by means of selective fusing.

Thus, the proposed project is fully consistent with modern trends in the field of additive technologies and the development of the aerospace industry, and contributes to the solution of questions of import substitution in the domestic market and has export potential.

The project will develop new materials micro and nano structured type, which ensures the creation of goods by means of additive technologies for operation in extreme conditions. The materials will be used in new high-tech industries and provide substantial (more than 30 %) to increase the thermal stability of the products.

In addition, the project will be developed production technologies of ceramic components and parts of hypersonic vehicles by means of selective laser melting behaviour and methods of diagnosis of processes and obtained products. The same will determine the best modes of fusing ceramic materials depending on particle size distribution.

In the course of the project it is expected to obtain at least two results, capable of legal protection is itself modified powdered ceramic material and a useful additive model to streamline the installation.

The object of this project is the technology of production of ceramic units and parts by selective laser fusion with the use of innovative methods of diagnostics of processes and obtained products. This technology relates to new processes layer-by-layer combining materials to create the object and, according to foreign classification ASTM F2792-12a, in the course of the project will create a technology called "Powder bed fusion" - the fusion of material into a pre-formed layer.

Work is performed under support of Federal target program.

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# STRUCTURE FORMATION DURING SINTERING OF THE SYNTHESIZED POWDER MATERIALS BASED ON TITANIUM CARBIDE

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Wide applying of additive technology is inhibited by a narrow range of consumables used. Most often the powder mixture is applied as consumables according to functional features of equipment used. Generally the final material composition of the formed part is determined by the mixture of trade powders. It causes the difficulties related to the structure control; it's homogenously, the degree of faultiness.

One solution to this problem is to use consumable powder materials with a pre-formed multicomponent structure, which under the conditions of the additive process can be transformed into a controlled type to ensure the functional properties of the resulting part. Secondary structure formation studies of compacted samples from synthesized multicomponent powder materials were carried out to assess the possible degree of structure transformation of these powders under conditions of vacuum sintering.

The composition based on titanium carbide with titanium binder (TiC-50 vol% Ti) was selected as the test material. The powders were obtained by the SHS method in the regime of layerwise combustion with subsequent grinding and screening of the fraction used 40-60  $\mu\text{m}$ . Grinding of synthesized cakes was carried out both in the standard mode and with the use of additional mechanical activation. Sintering was carried out in a vacuum at a temperature of 1200-1350  $^{\circ}\text{C}$  with an exposure time of 180 minutes. The residual porosity, compaction, grain size and phase composition before and after sintering were evaluated as the main parameters of the structure change of the synthesized powder material.

The carried out researches have shown, that carbide grains undergo significant changes during sintering: a partial coalescence or the carbides growth with a decrease in the thickness of the interparticle interlayer of the titanium binder occurs. At the same time the pore volume content significantly reduced. The intensity of compaction increases when mechanical activation of the synthesized powder material is additionally used.

The results obtained give grounds to assume that the studied synthesized powder materials (TiC-50 vol% Ti) can be successfully used in surfacing processes with low temperatures due to a significant degree of their sinterability which can be increased by the additional mechanical activation.

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# NUMERICAL MODELING OF AIR COOLING OF CONTAINERS FOR DESUBLIMATION OF FLUORINE HYDROGEN

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A condensation-evaporation plant is an element of production for the separation of uranium isotopes. The main components of the resulting mixtures are UF<sub>6</sub>, hydrogen fluoride (HF), air components and other non-condensable impurities. The method used to separate the gas mixture consists in the movement of the gas mixture through a series of special thermostatted containers, the cooling regime of which differs in temperature. In this case, desublimation of UF<sub>6</sub> and HF occurs in different containers. To cool the containers, liquid nitrogen is used to condense HF. It was shown in [1] that the temperature of the cold air of the BXM-0.56/0.6-H air cooler can reach 130-140 K. To simulate the possibility of using air cooling of containers for HF desublimation, modeling of several containers cooling was carried out (Fig. 1).

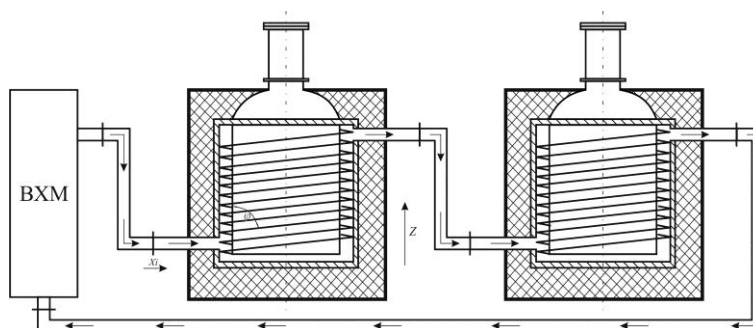


Figure 1. The scheme of two containers cooling.

A physico-mathematical model of the flow of cold air in a branched network of pipelines and heat exchange of several containers during the condensation of the product is developed. Hydrogen fluoride is considered as the condensation product. The dependence of the saturated vapor pressure on temperature for HF is taken from [2]. The intensity of heat release in connection with the condensation of HF is taken from [3]. An algorithm and a computer program have been developed that realize the calculation of the air flow parameters in a branched pipeline network for transporting cold air to the containers. Numerical simulation of the processes of containers - air heat exchange taking into account the air flow characteristics of BXM-0.56/0.6-H, heat exchange with the environment and technological flows into the tank is carried out. The analysis results of heat transfer calculation during container cooling have shown that the power of one BXM-0.56/0.6-H is enough for thermostating six containers.

The work was carried out with the financial support of the RFBR grant No. 16-48-700732 p\_a.

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# SINTERING OF COMPOSITE POWDER ON THE SUBSTRATE, CONTROLLED BY ELECTRONIC BEAM

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In additive technology the creation of the product using electron beam energy is possible in many ways. In one case the electron beam runs along the previously poured powder layer. In the second case, the powder is poured into the molten bath, initially formed by the electron beam in the substrate or in the previously deposited layer. Synthesis of the composite from the mixture of titanium and carbide powders under these conditions is practically impossible to realize due to the large difference in the melting temperatures of the components, their different molar masses, and the problems of the solid particle wetting by the melt. Therefore, we assume that the powder used for layer-by-layer product growth is a Ti-TiC composite, which precludes named problems. However, it is necessary to make choice of processing conditions which ensures uniformity of sintering and a predicted shrinkage value.

During small item growing, the process is essentially nonstationary; therefore, it is required to set the mode of beam movement and energy distribution explicitly.

For a thermally thin plate and thin surface layer of the substrate we neglect the temperature distribution over the depth. We suppose that the shrinkage and the kinetic parameters in the porosity variation law depend from hydrodynamics. The thermal part of the problem includes a two-dimensional heat equation. The thermal conductivity equation takes into account the heat loss from the surface according to the Stefan-Boltzmann law and the effective heat loss into the substrate.

The porosity variation leads to change in the thermophysical properties and the shrinkage of the powder layer. The specific heat and thermal conductivity of the poured particles are calculated by the rule of the mixture, depending on the fraction of titanium carbide. The thermal conductivity of the powder layer depends on the porosity. The thickness of the powder layer varies according to the kinetic law.

Figure 1 shows the change in the thickness of the stacking over time layer for different scanning modes.

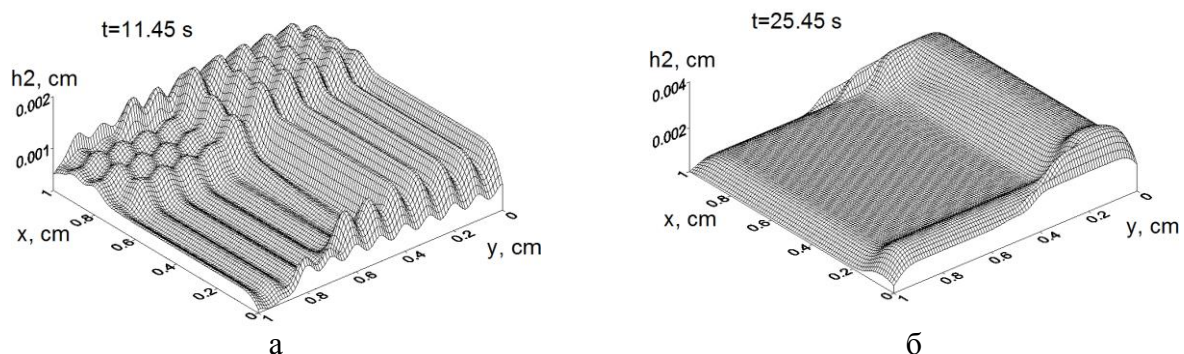


Figure 1. Dynamics of changing the thickness of the stacking layer in the process of electron beam fusion for different scanning steps a)  $h_{0x} = h_{0y} = 100$ ; b)  $h_{0x} = h_{0y} = 50$

Mathematical modeling allows studying nonequilibrium phenomena accompanying the synthesis of composite powders and the layer-by-layer formation of products as well as the laws of the formation of properties at the synthesis and fusion regimes changing.

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## CONSIDERATION OF NICKEL ADDITIVES AS A METHOD OF CONTROL OF SHS

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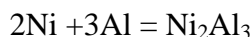
The traditional way of HLW isolation from the biosphere is their immobilization in phosphate and borosilicate glasses of different composition. However, glass does not completely satisfy the requirements to the matrices for the preservation of long-lived radionuclides for a long period of time [1].

There is about 560 million cubic meters of radioactive waste accumulated within the territory of Russian Federation; moreover, 50 % of it is located in temporary storages before further reprocessing. Besides that, 5 million cubic meters of radioactive waste is originated every year.

Along with the improvement of glasses material necessary for long-term HLW disposal in the geological environment, various types of ceramics based on the unique rock-forming minerals with high chemical, thermal and radiation stability are being developed. The basic idea of this method - the inclusion of chemically bound active atoms of radionuclides and toxic waste components in the form of a solid solution into the crystal lattice of the minerals' matrix similar to the stable natural minerals, where such radionuclides exist for thousands of years with the levels of radioactivity or toxicity hundreds of times smaller than if they were in free or mechanically blocked state. Nowadays over 20 compounds differing in capacitance depending on the elements mentioned, chemical and radioactive strength were proposed. These compounds include zircon, zirconolite, perovskite, yttrium-aluminum garnet (IAG), britholite, monazite, pyrochlore.

To prepare the mixture we used industrially manufactured PA-4 aluminum powders and neodymium oxide with a chemical purity grade TU 48 – 4 – 186 – 72.

The initial mixture was prepared for the exothermic reactions:



By heating the initial sample to a temperature of about 500-600 K (depending on the preparation of the initial mixture of components), the combustion wave was initiated at the ends' edges that spread over the sample volume. In this process the temperature increased rapidly, then stabilized, and burning continued almost isothermally under the temperature of 1000-1250 K. After the passage of the combustion wave along the sample volume, it cooled down to ambient temperature of the medium.

Table 1 presents comparative data of x-ray phase analysis of samples at different degrees of dilution of the initial charge.

Experiments have shown that the combustion temperature depends on the nickel additive amount in the initial mixture of reactants. Thus, when the content of additional components in a sample is about 25% wt. there is a significant increase in temperature (up to 2700 K) of the process; in case of 30% wt. – thermochemical degradation of the sample during synthesis for any stoichiometry considered due to significant heat release of the reactions in the mixture.

Thus, by increasing the content of additives amounts up to 25% wt. and compressing pressure of 30 MPa, the content of the modified aluminum-based perovskite based in the samples reaches almost 50 wt.%, and the amount of unreacted neodymium oxide is less than 5% wt.

Table 1. The effect of the degree of dilution of the initial charge on the phase formation.

Образец	NiAl	Ni <sub>2</sub> Al <sub>3</sub>	NdAl <sub>2</sub>	NdAlO <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>
NiAl+20%Nd <sub>2</sub> O <sub>3</sub>	56,6%	-	26,4%	17%	-
NiAl+30%Nd <sub>2</sub> O <sub>3</sub>	-	35,97%	13,27%	6,49%	44,27%
NiAl+40%Nd <sub>2</sub> O <sub>3</sub>	20.51 %	-	12,16 %	8.36 %	58.97 %
NiAl+60%Nd <sub>2</sub> O <sub>3</sub>	-	30.15%	12,16 %	8.22 %	49.57 %
NiAl+60%Nd <sub>2</sub> O <sub>3</sub>	-	18.09%	7.93%	8.19%	65.79%
NiAl+70%Nd <sub>2</sub> O <sub>3</sub>	-	16.67%	7.73%	10.73%	64.87%

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# ON POSSIBILITY OF APPLYING PARTS PRODUCED BY ADDITIVE TECHNOLOGY IN HIGH PRESSURE GATE VALVES

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Selective laser melting (SLM) is a promising additive technology capable of reducing costs and resources. It makes it possible to produce metal powder parts with high mechanical and operational qualities which are not typical for prototypes after machining. For instance, this can be said about stainless austenitic steels that are used for production of gate shutters and valves designed for conducting water-gas-oil mixtures and aggressive media which are likely to cause corrosion.

The purpose of this research is to look into the properties and to complete a comparative analysis of service life of parts in the gate unit of a wedge gate valve (Fig. 1); the valve has a port area of  $D_n$  15mm, the pressure of the medium is 2.5 MPa, the parts were made by machining AISI 321 rolled steel and welding CN-12M (%: C 0.08; Si 4.00; Mn 3.67; Ni 6.97; Cr 15.46; Mo 5.74; Nb 1.08; S 0.010; P 0.029) material onto the sealing surface and by applying the selective laser melting technology making use of EOS PH-1 steel powder (%: Cr 14.0-15.5; Ni 3.5-5.5; Cu 2.5-4.5; Mn $\leq$ 1; Si $\leq$ 1; Mo $\leq$ 0.5; Nb 0.15-0.45; C $\leq$ 0.07).

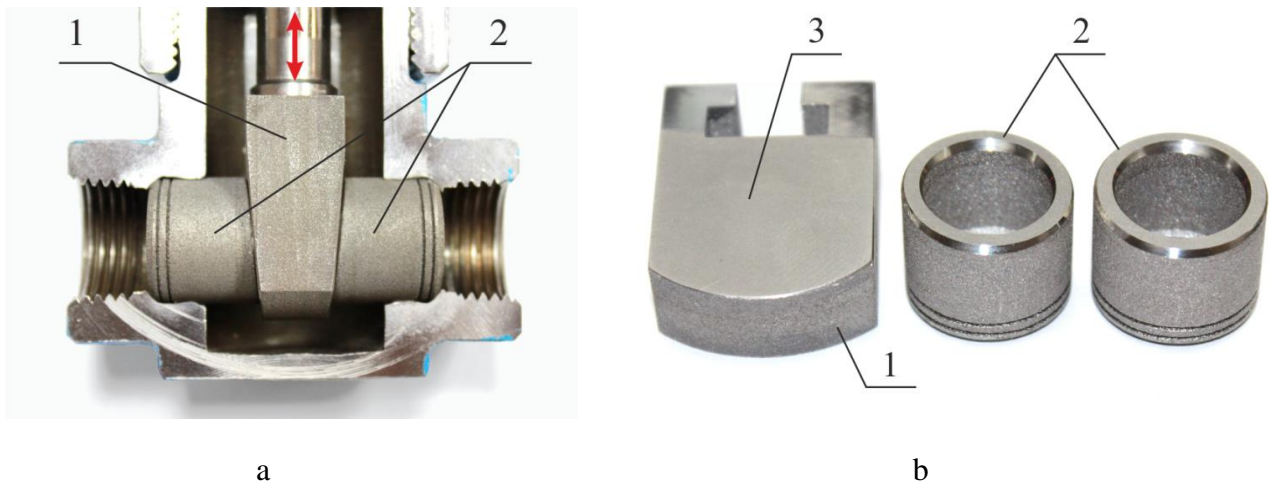


Figure 1. Gate unit a) and parts of the gate unit b): 1 – wedge; 2 – seats; 3 – sealing surface located at 5° to the axis

The parts ‘wedge’ and ‘seat’ of the gate units fabricated from rolled steel were machined on MIKRON VCE600 Pro и Takisawa E810 CNC machines. The welded surface was ground on a 3B722 machine to obtain  $Ra=0.4 \mu\text{m}$  roughness. Selective laser melting was done on an EOSINT M280 unit, the parts were made by layer-by-layer horizontal adding, the thickness of the layer being 30  $\mu\text{m}$ . The structure of the melted material is presented in Fig. 2.

After the addition had been completed and the pad had been separated from the part, fine milling of the sealing surfaces was done on a MIKRON VCE600 machine with a KORLOY RM4PCM4050HR-M end mill with LNMX 15 1008 and PNR 5300 plates made from PC 5300 alloy. The feed modes were as follows: the spindle ran at  $n=800 \text{ rev/min}$ ; the cutting rate was 125 m/min; the feed was  $f_z=0.03125 \text{ mm/tooth}$ ; the depth the removed layer was  $a_p=0.1 \text{ mm}$ .

Milling modes were selected so that no overheating could occur; the cutting was dry. A special stand was used for lapping of sealing surfaces on the wedges and seats with ASMG NOMG

10/7 TU2-037-506-85diamond paste to provide for required nonflatness not over 0.02 and roughness  $Ra=0.6 \mu\text{m}$ .

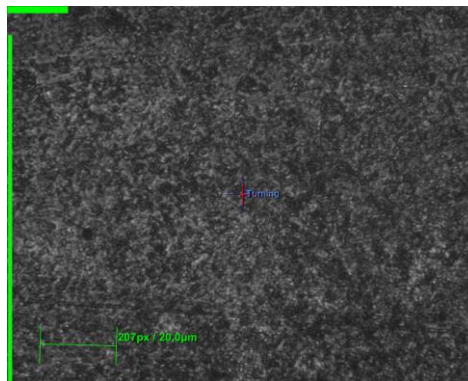


Figure 2. Structure of the material of the wedge produced by laser melting from PH-1 powder.

Research methods. Roughness of the sealing surfaces on the wedge was measured with a Wyko NT1100 optical 3D-profilometer, microhardness – with an EcoHard XM1270C microhardness tester; a PIM-DV-1 instrument was used to measure hardness and mechanical properties. To find out the service life in cycles the valves were tested on a special test stand until they began to leak. Roughness, mechanical properties of the sealing surfaces and service lives of the valves are illustrated in table 1. The values that describe mechanical properties of the wedge fabricated by selective laser melting are practically the same as those cited in the paper [1].

Table 1. Roughness and mechanical properties of wedge sealing surfaces, service life of the valve

Technology of producing wedges	Roughness $Ra, \mu\text{m}$	Micro-hardness $HV_{0.05}$	Hardness HB	Yield strength YS, MPa	Tensile strength TS, MPa	Cycles number
Machining and welding	$0.4 \pm 0.05$	$488 \pm 23$	$364 \pm 22$	$1034 \pm 36$	$1252 \pm 40$	3609
Selective laser melting	$0,53 \pm 0.05$	$394 \pm 13$	$327 \pm 25$	$923 \pm 83$	$1131 \pm 88$	3450

Tests showed that application of components produced by the SLM technology in gate units of valves meets customers' requirements on service life – 2000 cycles of closing and opening. Yet, the cost effectiveness of these components remains high, reaching 20% of the total cost of the valve, while the cost effectiveness of the components produced by conventional technologies does not exceed 11%.

Further fine-tuning of the modes employed for thermal production of the wedge is a promising trend for future research into the properties of valve components produced with applying the SLM technology.

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# MESOSCOPIC SIMULATION OF SOLIDIFICATION PROCESS WITH MELT CONVECTION

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Dendritic solidification is a phenomenon with immense technical and theoretical importance. Its technical importance clearly stems from the fact that the overwhelming majority of all metallic workpieces have once in their lifetime undergone solidification with a dendritic structure. The grain size, shape and the solute distribution affects significantly the mechanical properties of the materials produced.

Under normal growth conditions, i.e., if we do not have a microgravity setup, the inhomogenous distribution of temperature and solute concentration in the solidification sample will inevitably lead to thermal convection. Convective flows can substantially influence the growth process and the features of the resulting pattern.

The phase-field method is widely used in simulations of dendritic solidification (e.g. Refs. [1-2]). Its main advantage is the absence of the necessity of tracking the interface, together with the possibility of keeping good accuracy at moderate computational cost. The lattice Boltzmann (LB) method is presently a well-established tool to simulate fluid flows, especially flows in complex geometries [3].

We describe the combined mesoscopic scheme for the simulation of solidification with presence of melt flow. We consider the case of the solute-driven process, but the thermal-driven case can be considered as well [4]. The scheme is based on the multiphase-field method [1] for calculating the solidification. The interphase boundary is considered as a thin transition layer in which all the material parameters change gradually. The dynamics of the phase-field variable which represents the local concentration of the solid phase is governed by the minimization of the free-energy functional. The concentration of solute is obtained from the diffusion equation, with the local redistribution of solute between solid and liquid phases according to the linear phase diagram.

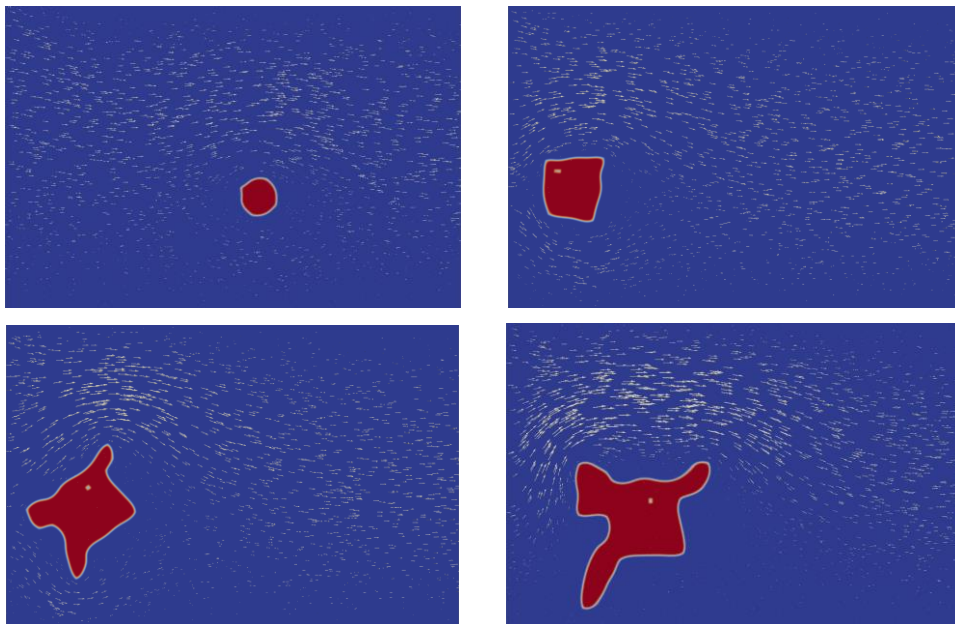


Figure 1. Moving grain in a channel flow. Time is (left to right, top to bottom)  
 $t = 0.5 \text{ s}, 1 \text{ s}, 1.5 \text{ s}.$



The lattice Boltzmann method [3] is used to simulate the fluid flows. The fluid is presented as a set of pseudoparticles which stream to neighbor lattice nodes and undergo local collisions. Friction forces due to the relative velocity of solid and liquid phases are taken into account by the Exact Difference Method [4]. Buoyancy force due to the thermal expansion and the inhomogeneous distribution of the solute concentration can also be included. Motion and rotation of growing solid grains is described by the equation of motion of a rigid body.

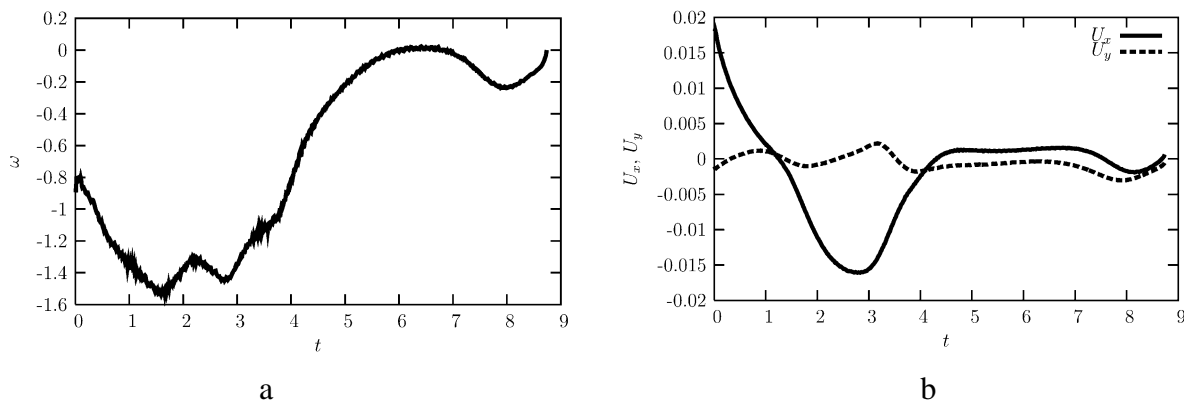


Figure 2. Moving grain in a channel flow  
a) time dependence of the grain velocity and b) angular velocity

We simulated the growth and the rotation of a dendrite in a parabolic flow, the grain growth and rotation in a shear flow, and the grain evolution and motion in a parabolic flow under the action of gravity. In first two cases, the growing dendrite rotates due to the interaction with the flow. Results for the moving grain are shown in Fig. 1. The direction of flow is from left to right, the gravitational force is directed to the left. Time dependence of the center of mass velocity and the angular velocity is shown in Fig. 2. When the size of the grain increases, the drag force also increases, and the grain stops and then starts to move with the flow. The angular velocity also first increases, then decreases and changes sign. In all the cases, the upwind branches grow faster since the flow brings fresh material into its vicinity.

In conclusion, the mesoscopic approach based on the combination of the phase-field and lattice Boltzmann methods can be successfully applied to simulation of solidification processes with melt convection in a wide range of problems.

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# EXTRUDABLE COMPOSITES BASED ON POLYETHERETHERKETONE FOR ADDITIVE TECHNOLOGIES

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Polyetheretherketone (PEEK) is an engineering structural plastic that is characterized by high values of thermal resistance, strength, and melt flow index (MFI) which ensure its wide application in aerospace engineering and medicine. Also, additive manufacturing technologies might be attracted for the fabrication of complex shape items, in particular, for medical applications [1]. However, the unfilled PEEK possesses not high enough wear resistance as well as is characterized by high friction coefficient (0.3-0.4) which limits its use in friction units for mechanical engineering and orthopedics.

In order to use the fillers as a solid lubricant as well, PEEK-based composites filled with carbon nanotubes (nanofibers), carbon microfibers and polytetrafluoroethylene particles were fabricated. Their mechanical properties and tribotechnical behavior under dry sliding friction were studied. A comparative analysis of the effectiveness of loading carbon nano- and microfillers, as well as polytetrafluoroethylene micron size particles in formation of tribotechnical characteristics of polymer composites based on the extrudable thermoplastic matrix was carried out.

PEEK powder (F450 by Victrex), Taunit carbon nanofibers (outer  $\varnothing = 60$  nm) and Tuball carbon nanotubes ( $\varnothing = 10$  nm), short carbon microfibers ( $\varnothing = 10$   $\mu\text{m}$ , length = 80  $\mu\text{m}$ , aspect ratio  $\sim 10$ ), polytetrafluoroethylene particles ( $\varnothing = 14$   $\mu\text{m}$ ) were used as fillers. Composites on the basis of PEEK were fabricated by hot compression molding at a pressure of 14 MPa and the temperature of 400°C with subsequent cooling rate of 5 C/min.

The wear resistance of composites under study at dry sliding friction was determined by the "pin-on-disk" scheme at the load of 30 N and a ring sliding velocity 0.3 m/s (according to ASTM G77). Examination of the composite's structure was carried out with the use of scanning electron microscopy, differential scanning calorimetry and IR spectroscopy.

It is shown that loading of small amount of the nanotubes/nanofibers (less than 1 wt. %) makes it possible to twice increase wear resistance of the PEEK-based composites. The loading of microfillers (short carbon fibers, polytetrafluoroethylene) in the content of up to 20-30 wt. % gives rise to increasing wear resistance of the PEEK-based composites three-fold while maintaining high enough melt flow index. A comparative analysis of nano- and microcomposites based on PEEK with extrudable composites based on ultrahigh-molecular matrix UHMWPE fabricated with the help of compatibilizers [2] was carried out. It is shown that the PEEK-based composites while having the high flowability are promising for additive manufacturing technologies at fabricating complex shape items for mechanical engineering and medical applications.

**Acknowledgments.** The work was financially supported by the Russian Federation via the Ministry of Education and Science of the Russian Federation (Agreement No. 14.604.21.0154, project identifier RFMEFI60417X0154).

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# PROSPECTS OF ESTABLISHING A TRABECULAR TANTAL IMPLANT BY THE ADDITIVE METHOD

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**Introduction.** Today, porous orthopedic implants reveal new horizons in skeletal reconstructive surgery, in the general replacement of the hip joint, recently called the "operation of the century" [1]. Until now, general arthroplasty of the hip joint has become a routine operation, when the treatment of hip osteoarthritis and the number of surgical interventions has been increasing since the 1970s. The most common causes for revision are mechanical weakening, infection and instability / dislocation [2]. Most of these failures are due to the wear of polyethylene and periprosthetic osteolysis and, more recently, metallosis [1,3]. These defects of bone tissue need to be restored and filled during the revision operations with new structures on which new elements of the prosthesis are attached. These new structures can be autografts, polymethylmethacrylate (bone cement), or allografts. Currently, transplants are the best solution for creating a mechanical stable reconstruction that can withstand postoperative mechanical loading, in cases of large bone defects.

**Purpose and subject of research.** The purpose of this work is to determine the relevance of the development of technology for the production of trabecular tantalum implants by the additive method.

The first porous implants had only porous coatings of cobalt-chromium, but were soon replaced by the surgical type 5 Ti-6Al-4V, which is still the most widely used material for porous biomaterials [1]. An alternative with a large potential is tantalum (Ta). Ta is a solid, plastic, highly chemically resistant material with good biocompatibility to human bone. It has been used successfully as a biomaterial since the 1940s in clinical medicine, but because of its high cost and complexity in processing, the use of Ta as a biomaterial has been limited. Despite its promising biological properties, Ta is not considered to be a suitable material for large implants because of its high density and high price. Recent work devoted to research depending germination bone implant (trabecular implants are based on a porous alloy Ti-Nb-Ta, obtained by powder metallurgy techniques) showed a strong dependence on the alloy porosity and pore dimensions themselves. The best biocompatibility was shown by Ti-Nb-Ta based alloys with pore sizes from 200 to 400  $\mu\text{m}$ , which correspond to the pore sizes of animals and human bones. The porosity of the alloy should be at least 70% with a plateau voltage of 53 MPa and an elastic modulus of 3.4 GPa

**Results and discussion.** The research team made an attempt to assess the competitive advantages of the proposed technology in comparison with the main market implants produced in the classical way.

A study of the endoprosthesis market showed that among the metals that are used for implantation, the proportion of titanium and its alloys that are best for implantology, in terms of biocompatibility and specific strength, is about 5%. This is due to the low processability of titanium, and, consequently, the high cost of implants, which are mainly manufactured by machining. In addition, the final finish of the bone-contacting surfaces is quite difficult.

The unique quality of tantalum is its high biological compatibility, i.e. The ability to get accustomed to the body without causing irritation to surrounding tissues. This property is based on the widespread use of tantalum in medicine, mainly in reconstructive surgery.

Tantalum has an advantage over titanium, stainless steel and other metals, which are used for the production of implants. Tantalum implants are more porous than titanium, which promotes bone growth and elasticity of the prosthesis itself.

However, only 5% of tantalum produced in the world is spent on medical needs, about 20% is consumed by the chemical industry. The bulk of tantalum - over 45% - goes to metallurgy. Leaders in the field of tantalum implants are the American company ZIMMER.

Tantalum production in East Kazakhstan is the only one in the CIS and one of the largest in the world, equipped with powerful modern equipment capable of processing any tantalum-niobium-containing raw materials into ingots, chips, powders, rolled products.

The use of tantalum in medicine will allow us to master a new segment, increasing the Kazakhstan content in the full cycle of production. The market of tantalum implants remains undeveloped due to various factors considered above. We are able to become the first in the market of modern implants to have a raw material base, a growing demand for endoprosthetics, and the use of new technologies in the production of implants. The combination of these factors will allow Kazakhstan to become a market leader with a fundamentally new approach to the production of medical products.

**Conclusion.** Unlike titanium, tantalum has a higher biocompatibility. Tantalum is a unique material - it has a high biological compatibility with the tissues of a living organism, without causing rejection, which is why it is widely used in medicine: reconstructive surgery, orthopedics. Tantalum plates cover the damaged skull, tantalum with alloys of other metals used to make endoprostheses.

Similar properties - biological compatibility - are alloys of tantalum with niobium. The lower density of alloys compared with tantalum makes them more promising in medicine, including implantology. The human body perceives tantalum not as a foreign body, but as its own bone tissue.

Increasing the share of medical use of tantalum will allow solving a wide range of tasks in traumatology, orthopedics and surgery. Diversified use of tantalum properties makes this metal a promising raw material in the future medicine. However, in the modern world there is no commercial application of tantalum implants obtained by the additive method, having scientific developments are at the stage of preclinical research.

The use of tantalum as a material in prototyping will allow:

- improve the efficiency of medical care, reduce the time of surgery and reduce risks to the patient;
- use tantalum raw materials in a completed production cycle;
- increase the share of Kazakhstan content in endoprosthetics.

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# STUDY OF THE INFLUENCE OF INITIAL PARAMETERS OF THE CHARGE ON PHASE FORMATION NICELLANEOUS MATRIX OBTAINED IN THE TECHNOLOGICAL MODE OF BURNING

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There is about 560 million cubic meters of radioactive waste accumulated within the territory of Russian Federation; moreover, 50 % of it is located in temporary storages before further reprocessing. Besides that, 5 million cubic meters of radioactive waste is originated every year. [1]

SHS-based materials synthesis included the following steps:

- mixing powders of initial reagents according to the stoichiometric calculation for the corresponding reaction equations;
- drying the initial mixture of reactants in a technical vacuum;
- mixing in a cubic mixer; - compressing the initial reactants mixture into cylindrical pellets of different diameter and height with variations in material density values in samples obtained by changing the pressure;
- running the SHS process in a laboratory facility and obtaining functional materials samples [2].

To obtain the result, which is the most appropriate to the characteristics, the samples with various contents of Ni (15 ... 45 wt.%) and compaction pressure (20 ... 30 MPa) were prepared.

Phase formation of the samples, synthesized through variation of nickel percentage and compaction pressure, is listed in the table 1.

Table 1. Phase formation of the samples according to nickel percentage variation

№	Phase percentage, %					P, MPa	Nickel additive percentage, %
	Ni <sub>2</sub> Al <sub>3</sub>	NiAl <sub>3</sub>	Nd <sub>2</sub> O <sub>3</sub>	NdAlO <sub>3</sub>	Al		
1	14,4	15,6	32,4	7,9	29,7	20	10
2	16,8	22,4	25,8	8,4	26,6	25	
3	25,3	23,7	17,9	10,3	22,8	30	
4	18,4	20,4	25,1	10,3	25,8	15	15
5	18,2	19,3	26,7	11,2	24,6	20	
6	18,3	21,3	24	12,3	24,1	25	
7	17,2	21,1	24,9	14,5	22,3	30	
8	19,0	30,9	20,1	18,4	11,6	15	20
9	21,1	25,3	26,5	19,9	7,2	20	
10	22,0	24,7	30,1	22,5	0,7	25	
11	19,7	20,6	33,8	25,9		30	
12	35,9	30,6	2,3	31,2		15	25
13	35,7	23,6	1,8	38,9		20	
14	35,8	18,7	1,2	44,3		25	

It is established that the samples obtained by compression of the mixture with the addition of Ni samples at the compaction pressure increased from 15 to 30 MPa experience the phase shift towards the final product formation due to the higher energy yield.

The maximum amount of aluminum perovskite is observed when the initial mixture compaction pressure equal to 30 MPa and 25% wt. nickel.

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# SLM TECHNOLOGY PROBLEMS FOR METAL-MATRIX COMPOSITE FABRICATION

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Metal-matrix composites (MMC) possess an unique combination of hardness, fracture toughness and wear resistance. Therefore MMC commonly used for wear resistant coatings. MMC structure consists of disperse hard inclusions uniformly arranged in a metal matrix. Titanium carbide is the most often used as a disperse strengthening phase in MMC. The reasons are extremely high hardness among the metal carbides and equal axes, rounded shape of the inclusions. Titanium, cobalt, iron and nickel alloys are used as the metal matrix in the titanium carbide base MMC. The most commercial interest for wear resistant coatings are TiC-Ti metal-matrix composites. TiC inclusions in Ti or in Ti alloy matrix multiple reduce wear as compared with titanium or alloys. There are a lot of publications on laser induced deposition of titanium and titanium carbide powder mixtures on the titanium base. The results of the investigations are a good background for additive technology application to TiC-Ti composite materials. To get a high strength together with high wear resistance it is enough "to print" TiC - Ti surface layer on volume fraction of the part printed of high strength titanium alloy. As a rule TiC and Ti use as a powder admixtures in additive technologies. Powder additives either put on base surface in a thin layer shape for following fusion or are poured directly into molten pool. In the most cases primary TiC particles dissolve in the liquid melt and precipitate at the cooling from Ti - C liquid solution in dendrite or equal axes crystals form.

A size and morphology of the carbide inclusions depend strongly on the molten bath cooling rate. When TiC - Ti powder mixes are poured into the molten pool a segregation of TiC and Ti powders can occur owing to difference in density and dispersity of the carbide and titanium powders. To avoid the powder components segregation they use composite powders instead of particular powder mixtures. There are some different methods to fabricate TiC-Ti composite powders. One of them is sputtering of TiC-Ti suspension in any organic liquid. Drying of the suspension drops in a hot gas flow results in solid granules formation. The granules (composite powders) consist of TiC and Ti particles uniformly distributed over granule volume. A mechanical treatment of TiC-Ti powder mixtures in planetary grinding mills is used in another method of composite powder fabrication.

Self-propagating high temperature synthesis (SHS) is the most high-productive and energy-saving technology for "TiC-Me" composites production. SHS in titanium and carbon powder mixtures results in TiC +Ti composite production when there is a titanium excess in the reactive mixture. After crashing and following screening of the porous SHS cake granules consisting of TiC particles in Ti matrix can be obtained. The granules can be used in selective laser melting (SLM) additive technology to get hard and wear resistant coating on the surface of titanium alloy part.

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# DEVELOPMENT OF MODELS, ALGORITHMS AND SOFTWARE FOR EVALUATING THE EFFECTIVE CHARACTERISTICS OF MATERIALS OBTAINED BY USING ADDITIVE TECHNOLOGIES USING MULTISCALE PHYSICAL MODELING

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One of the vectors of the development of our country's economy is the transition to the so-called "digital economy". In the message of the President of the Russian Federation to the Federal Assembly on December 1, 2016, "It is necessary to focus on areas where the powerful technological potential of the future is accumulating, and these are digital, other so-called end-to-end technologies that today determine the appearance of all spheres of life" [1]. One of the elements of the digital economy is 3D printing, additive technologies, or layer-by-layer synthesis technologies, one of the most dynamically developing directions of digital production in our country and abroad. These technologies combine one circumstance: the construction of a part occurs by adding material (from English add - "add") in contrast to traditional technologies, where the creation of a detail occurs by removing "excess" material.

In turn, this raises the problem of developing a mathematical, algorithmic and software apparatus for modeling the characteristics of newly created materials. This problem in all industrially developed countries of the world is solved by replacing the real object with its mathematical model, reproducing the basic functions of the original and the like in given relevant aspects.

At present, there is no single generally accepted approach directly linking the features of the microscopic structure of inhomogeneous composites produced using additive technologies with their macroscopic properties, which limits the functionality of the systems of engineering analysis (SAE) (universal strength computational packages). In this regard, the development of new physical models, computational methods and algorithms that would allow to connect the results of modeling at the microlevel with macroscopic properties, is especially urgent [2].

For these reasons, it seems relevant in the framework of the proposed PSI to develop methodologies for approach to computer modeling combined by a common ideology of multiscale modeling and parallel computing technologies, the exchange and processing of input and output data

In the Tula State Pedagogical University. L.N. Tolstoy together with the FKP "Aleksinsky Chemical Plant" are carrying out research within the framework of the Federal Program "Research and Development in Priority Areas for the Development of the Scientific and Technological Complex of Russia for 2014-2020" on the theme "Development of models, algorithms and a prototype of a software module for assessing the effective characteristics of materials produced using additive technologies using multiscale physical modeling "[2].

The overall goal of the work is a significant acceleration and optimization of the process of developing multicomponent materials obtained by using additive technologies with given physical and mechanical properties due to multiscale physical modeling and increasing the efficiency of using modern high-performance computing systems for computer simulation and design of new materials.

Other research objectives are:

– development of scientific and technical software in the field of software for evaluating the effective characteristics of materials obtained by using additive technologies using multiscale physical modeling;

– acceleration and optimization of the process of development of multicomponent materials obtained by using additive technologies with specified deformation-strength properties due to increasing the accuracy of prediction of structure and properties;

– increasing the efficiency of using modern high-performance computing systems for computer modeling and design of new materials and structural elements from them.

Carrying out a multilevel modeling of the processes of deformation and destruction of materials obtained with the use of additive technologies, suggests a study, during which should be:

1. Models have been developed to evaluate the effective characteristics of materials obtained by using additive technologies using multiscale physical modeling.

2. Algorithms have been developed for carrying out scalable computations in multilevel modeling problems of effective characteristics of materials obtained by using additive technologies.

3. Prototypes of software modules are created that implement the developed algorithms for evaluating the effective characteristics of materials obtained by using additive technologies.

4. Experimental verification of the results of theoretical studies, first of all, using methods of numerical simulation of the effective characteristics of materials obtained with the use of additive technologies, and confirmed the possibility of carrying out multilevel calculations combining micro and meso levels.

5. The mechanisms of deformation and destruction of materials obtained using additive technologies using multiscale physical modeling are analyzed, and the efficiency of the proposed modeling techniques is analyzed from the point of view of their use for the development of new materials obtained with the use of additive technologies.

Thus, the object of research are material bodies created from materials obtained with the use of additive technologies (MIAT) with various mechanical and physical properties.

At the micro level, the processes occurring throughout the composite body are described by differential equations with variable coefficients whose values depend on which of the volumes the point belongs to. Moreover, these coefficients undergo finite discontinuities when passing through the boundaries of volumes [3-5].

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## PHASE COMPOSITION AND STRUCTURE OF NICKEL-BASED ALLOY OBTAINED BY HIGH-SPEED DIRECT LASER DEPOSITION

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The structure and phase composition of products generated during additive manufacturing has a number of features that depends on the operating parameters of device, which affect the final mechanical properties of products. Thus, it is possible to manufacture products with a given mechanical property by varying the operating parameters. The structure and phase composition of nickel-based alloy obtained by the additive manufacturing of high-speed direct laser deposition is presented in the paper.

Lately one of the most promising methods of additive manufacturing of is the technology of high-speed direct laser deposition, since the use of technology is applied not only to creation of new parts of products with complex geometry, but also can be used for recovery operations of worn parts of products [1-3].

The main aim of the investigation was to study the influence of operating parameters on the structure and phase composition of nickel-based alloy obtained by high-speed direct laser deposition on the experimental device at Peter the Great St. Petersburg Polytechnic University. The laser power was varied from 450 to 1200 W during deposition of samples. Scanning velocity (45 m / sec) and powder feed rate (20 g / min), as well as the diameter of the laser spot, which are the main determining parameters of additive processing [4-6], remained unchanged. The choice of unchanged parameters and the coaxial geometry of nozzle was justified on the basis of previous experimental studies and mathematical modeling of complex geometry products [7-8].

After EDX analysis the powder was assigned to the Ni-Co-Cr system. The average size of spherical particles was 70 ... 90  $\mu\text{m}$ . The surface of the particles had a smooth surface with dendritic structure. The materials of such system belong to nickel-based superalloys. High-temperature strength of alloys is provided by refractory alloying elements, forming a solid solution with nickel, and also by forming precipitating phase.

Structure of longitudinal section (relative to the laser beam) of all samples investigated with a help of light microscopy and scanning electron microscopy had a dendritic morphology with separated layers from successive laser trajectory. Dendritic grains had an axes of the second order, which size depended on the used laser power. The height of the individual layer and the thickness of the grown wall of each sample also had a linear correlation with respect to the laser power. The cross section (relative to the laser beam) was typically equiaxed cellular structure the samples obtained at low laser power contained incompletely molten particles of the initial material with border pores. Areas with the growth of grains from the centers of the powder particles were revealed with increasing of laser power. Such areas indicate the occurrence of bulk crystallization [9]. The structure with dendritic grains oriented toward the direction of laser scanning was observed at power above 900 W.

Decoding of the results of X-ray diffraction analysis of the initial powder and deposited samples shown the presence of solid solution based on nickel in all cases. The EDX analysis of the longitudinal section, carried out perpendicular to dendritic grains for identify the concentration zones of individual chemical elements, also showed the chemical composition corresponding to the initial powder throughout the samples section. Precipitating phase, as well as carbide and boride inclusions, were not detected at this stage due to their small size [10-11].

It is planned to conduct studies using transmission electron microscopy for determining the complete phase composition of the samples obtained at different laser powers.



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# INFLUENCE OF TECHNOLOGICAL PARAMETERS OF SLM ON THE POROSITY OF ALLOY TI-NB

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Selective laser melting (SLM) allows making products with a complex geometry. The laser beam under control of a computer program, selectively affects the particles of the powder. Particles of the powder are heated, sintered, and then fused together. By changing process conditions SLM is possible to obtain products with a given structure and physical and mechanical properties. SLM composite powder in mass proportions of 55% Ti and 45% Nb (Ti-45Nb) is made on an experimental machine of layer by layer laser sintering «VARISKAF-100MVS». As a result of the experiments, it was obtained panel of samples. Samples were made with a side of 10 mm and a thickness of 1.5 mm. The change in the porosity was considered according to the technological conditions of SLM. The used processing parameters for forming 3D specimens were laser power, 100 W; scan speed, 70 mm/s; scan interval, 0.1 mm; temperature of powder, 300 °C. The influences of the thickness of a powder layer of 0.025-0.05 mm and various scanning strategies on the change in porosity are considered. The values of technological parameters of SLM are established at which the minimum porosity in the samples is reached (Fig. 1).

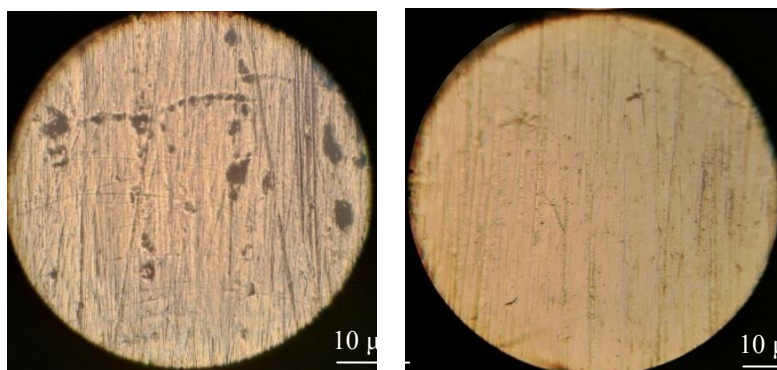


Figure 1. Changing the porosity in the samples

By changing the technological parameters of SLM, a minimum porosity value is obtained in samples of the Ti-Nb alloy. But the problem of the formation of cracks in the volume of samples is not solved.

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## INTEGRATION OF PLASMONIC NANOPARTICLES IN 3D PRINTING

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The new generation of sensors can be designed to work in conjunction with smartphones exploiting the detection and optical illumination capabilities of handheld devices. Plasmonic nanostructures have a significant potential to become the new element of the optical sensing devices. A recent study showed that with 3D printing it is possible to create new functional materials using additive assembly nanomaterials<sup>1</sup>. Depending on the deposition parameters, it was possible to obtain functional 3D-printed plasmonic structures with either graded or sharp interfaces. We propose to produce plasmonic/polymer structures by 3D printing based on filament precursor containing nanoparticles made of noble metals.

The technology for integration of plasmonic nanoparticles into 3D structures is the main objective of this study. It consists of several stages. First, we need to determine which polymer will be used. The polymer should be transparent and have a smooth surface in order to act as efficient optical elements. Given the thermal sensitivity of the plasmonic nanoparticles, we investigate the printing temperature of different polymers to minimize particle melting and aggregation that result in degradation of the plasmonic activity.

Two ways for introducing the nanoparticles into the 3D polymer ink will be investigated. The first method consists of mixing the nanoparticles with the dissolved polymer, and then making the mixture completely homogeneous using chemical methods. In this approach, polylactic acid (PLA) a biodegradable polymer with relatively low melting temperature is used. This material is advantageous for this study since it is widely employed in 3D printing and can be chemically functionalized with the nanoparticles. The finished mixture is melted in an extruder to obtain the filaments used in the 3D printing process.

The second method for introducing the nanoparticles in 3D printing involves the use of polyethylene terephthalate (PET). Because it is soluble only in chemically hazardous reagents, purely physical methods of mixing of nanoparticles and PET will be used. It will be mixed directly in the extruder at relatively high temperatures than those used for PLA. The main risk is that nanoparticles can fuse together during the polymer melting process.

The polymer filaments with nanoparticles will be optically analyzed, and the rationality of its future uses and the methods of parameter optimization will be discussed. Ultimately, we aim at determining the best method of preparation of the mixture of polymer nanoparticles, including the choice of polymer.

The sensor that will be created based on the plasmonic structure will be used as a portable device for chemical detection in different media such as blood, urine, and other liquids<sup>2</sup>. The devices will be developed based on numerical simulations and prototyping based on the finite element method.

Figure 1 presents a scheme of the sensor. Here, the camera acts as the optical detector, and the light from the display as the light source. The light falls directly on the structure, which contains the analyte flow, after it is refracted by the prism and focused on the front camera of the smartphone. The physical principle of detection is based on the phenomenon of Localized Surface Plasmon Resonance (LSPR). Due to the LSPR, the local electromagnetic field around the metallic nanoparticles will be enhanced dramatically, with the resonant wavelength being sensitive to the variation of the local dielectric environment. Noble metal nanoparticles, such as gold and silver, exhibit a strong optical extinction at visible and near-infrared (NIR) wavelengths and generate LSPR phenomenon which is sensitive to the surrounding medium. Accordingly, the camera will show the image in which the color of the absorbed wavelengths will be missing. Such selective decrease in intensity will be correlated with the presence of the analyte to be detected<sup>2</sup>.

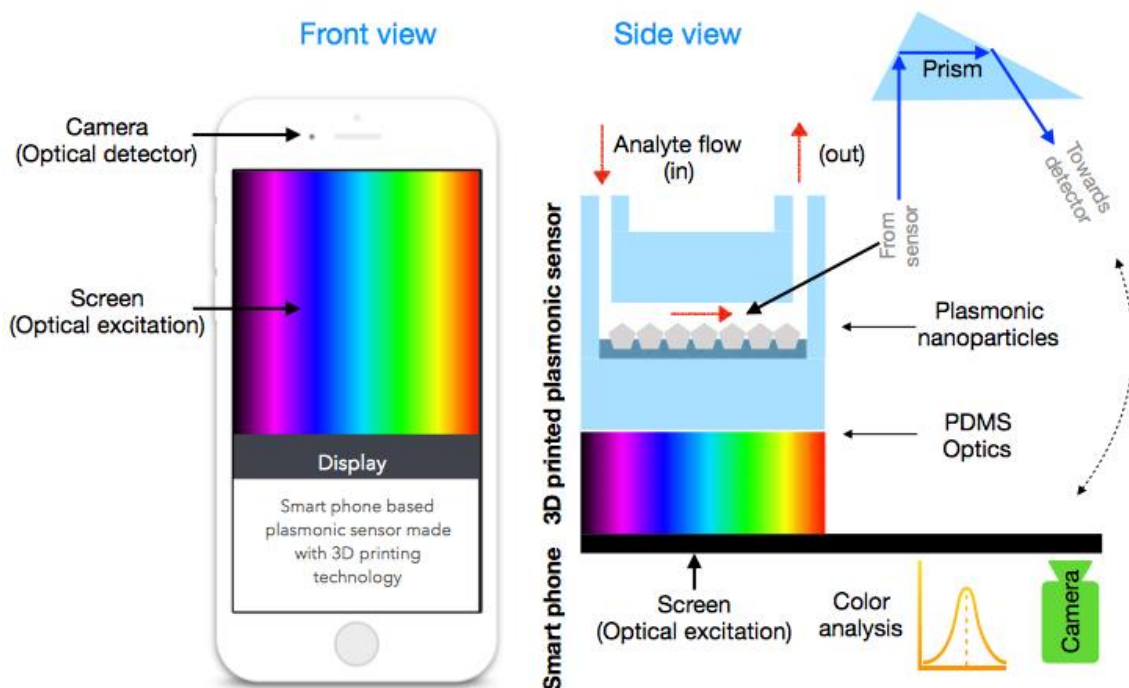


Figure 1. 3D-printed plasmon sensor based on a smartphone

We expect that our technology will make an innovative contribution for the inexpensive fabrication of portable optoelectronic devices that can compete with conventional sensor technologies. It will be a very simple way to implement biomedical chemical detection and for other applications where the only extra part required is a device that most people from both developed and under developed countries already have access to.

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## OBTAINING OF BIOINERT ALLOY WITH LOW YOUNG'S MODULUS AND PRODUCTS FOR MEDICAL APPLICATIONS BY SELECTIVE LASER MELTING

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Ti-Nb alloys are perspective materials for implants production. Due to their features alloys of Ti-(40-50) wt. % Nb have Young's modulus close to that of bone tissue. Selective laser melting (SLM) allows obtaining low-modulus Ti-Nb alloys and parts of them. SLM significantly reduces material consumption and allows controlling shape, size and porosity of produced items. Change of SLM parameters affects the size of structural elements and phase composition of resulting product. By using methods of mathematical simulation of physical processes it is possible to predict SLM-modes necessary for production of the item with certain properties.

By the molecular dynamic methods, the model of processes at laser beam influence on separate particles of metallic powders was created. The mathematical two-dimensional model of laser alloying of composite Ti-Nb powder layer on the Ti substrate was suggested. It was shown that melting kinetics has a great influence on the SLM process dynamics. The 3-D specimens of Ti-(40-50) wt. % Nb alloy were obtained in Yurga Institute of Technology (Russia) on "VARISKAF-100MVS" installation and in Tomsk Polytechnic University on "LUCH" installation. To produce the specimens composite powder of titanium and niobium was obtained by mechanical alloying of titanium and niobium powders mixture in AGO-2S ball mill (AltSTU, Barnaul, Russia) and was layered on titanium substrate. The microstructure of 3-D specimens was investigated by methods of optical metallography (OM) on AXIOVERT-200MAT microscope in bright-field and DIC (differential interference contrast) and on Zeiss Axio Observer microscope, transmission electron microscopy (TEM) on JEM-2100 microscope and scanning electron microscopy on SEM 515 Philips microscope. Change of elemental composition was evaluated by energy dispersive microanalysis. Evaluation of phase composition was carried out by the method of X-ray diffraction analysis (XRD) on DRON-7 (Burevestnik, Russia) in  $\text{CoK}_\alpha$ -radiation and on Shimadzu XRD-6000 (Shimadzu, Japan) in  $\text{CuK}_\alpha$ -radiation. Such physical mechanical properties as nanohardness and Young's modulus were investigated on NANO Hardness Tester NHT-S-AX-000X. The microhardness was measured on DURAMIN-5. The research was conducted in CCU "Nanotech", Institute of Strength Physics and Materials Science (ISPMS), SB RAS and TRCCU National Research Tomsk State University (Tomsk).

The results of investigation have shown that Ti-(40-50) wt. % Nb alloy obtained by SLM-method has a structure represented by two-phases. They are the main  $\beta$ -solid solution of titanium and niobium and  $\alpha$ '-martensite phase. The microstructure contains zones with fine and medium grains. The Young's modulus changes due to the change of elemental composition and SLM-mode in the range of 55-90 GPa.

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# POSSIBILITIES OF ADDITIVE TECHNOLOGIES FOR MANUFACTURING OF GRADIENT MATERIALS WITH RECORD HIGH MECHANICAL CHARACTERISTICS

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**Introduction.** The urgency of the problem of creating materials with record high mechanical characteristics is due, first of all, to the needs of the aerospace industry. No less responsible and socially significant is the use of heavy-duty structural materials in medicine, sports, safeguarding equipment, since their use significantly increases the reliability of the functioning of the respective products while significantly reducing their size and weight. In the above-mentioned high technology applications, the characteristics of specific strength and rigidity (form stability) are more critical than the material cost, because losses due to major accidents, injuries and lethal outcomes are incommensurable with the costs of manufacturing structural elements.

The aim of the work is to create materials with the highest possible strength and modulus of elasticity based on optimal modulation of structure and properties of interphase layer.

**Methods and results.** It seems that this goal can be achieved in the mainstream of two dominant trends in modern composite materials science – the use of computer design of materials [1] and application of the principle of additivity (additive technologies) on nanoscale level [2].

More specifically, the possibility of additive (layer-by-layer) formation of a nanosized interphase layer (IL) in the form of a gradient material that is optimal in terms of the load-carrying capacity of a composite as a “matrix-filler” system is considered. The optimal gradient of the structure and properties of the interphase layer for a particular composite modulating this way is proposed to be calculated by methods of computer-aided design of materials. As shown in figure 1, this approach is implemented in the classes of disperse-reinforced (a) and directionally reinforced (b) composites, both on the polymer and on the metal matrix.

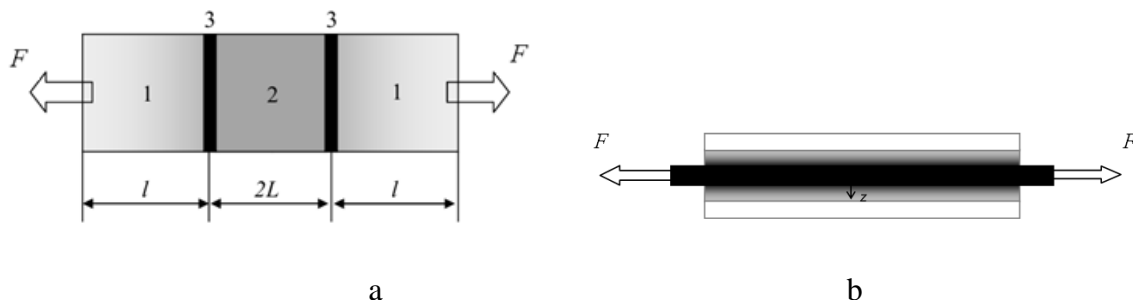


Figure 1. Interphase layer of composite with gradient of mechanical properties shown in grey half-tone (uniaxial tension): a) disperse reinforcing (1 – matrix, 2 – filler, 3 – interface layer), b) directional reinforcing.

We have to state that the possibilities of increasing the specific strength and modulus of elasticity of the named composites by choosing a filler in the form of reinforcing fibers and a matrix (binder), as well as reinforcement schemes, are mostly exhausted. The disadvantages of directionally reinforced composites are the low interlayer strength and the “quality” (expressed unevenness) of stress transferring in the “fiber-matrix” adhesion joint. In this connection, the applicants consider the possibilities of optimizing the structure and properties of the least studied component in the form of an interphase layer (IL) formed near the interface, whose role in transferring mechanical stresses between the filler and the matrix is well known.

But in common case, IL is formed spontaneously in the course of complex physical-chemical processes not related to the operational stress-strain state of the composite. Thus, the structure of the

IL, as a rule, does not ensure the uniformity of its stress state, i.e., is not optimal by the criterion of strength. It seems that by controlling the structure of the interphase layer and providing the desired gradient of its deformation properties, it is possible to achieve a significant increase in the efficiency of the transferring of stresses from the filler to the matrix and a radical change in the character of failure.

Thus, optimal gradients of structure and properties, for example, porosity, are characteristic for rationally constructed in course of evolution biological materials – wood, solid (bone and dental) and soft tissues, where the strongest elements, even at the cellular level, are located in the zones of maximum stresses, and relatively low-modulus (damping) intermediate layers of lignin and elastin are localized in such a way as to "absorb" significant strains without destruction.

Expediently organized (modulated) continuous or discrete gradations of the composition of the interphase layer between unlike components (metals, ceramics, polymers) allow redistributing internal stresses and reducing the probability of local fracture and crack formation. Similarly, the level of local stresses that promote the growth of cracks along the interfaces can be regulated by the appropriate distribution along it of the gradients of the elastic and plastic properties of the interphase layer. The effectiveness of using the principles of gradient and deformation compatibility is proved in microelectronics, where thin intermediate layers of variable composition ensure the thermal strength of hetero- and epitaxial layered structures in semiconductor devices.

One of the means to achieve the required gradient of the properties of the interphase layer is the use of nanoparticles introduced into the glaze composition and contributing to the formation of the dendritic structure of the IL. In addition, the introduction of nanoparticles will allow for a more dense packing of the fibrous filler, which also contributes to the strength of the composite.

To synthesize a composite interphase layer reinforced with structurally-forming nanoparticles, solid-phase, liquid-phase (eg, sol-gel) and gas-phase additive technologies can be used. The advantage of solid-phase technologies, in particular, the "ball-mill" process, is the relatively low processing temperature, which avoids undesirable reactions and obtains a qualitative microstructure of dispersed-reinforced materials. However, when using long reinforcing fibers (hardeners with a high aspect ratio), it is difficult to obtain a uniform distribution of the nanoparticles. Only recently there have been reports of the possibility of solid-phase formation of a strong interphase layer as a result of intensive mechanical interaction.

It seems promising to form the aprotic nanocomposite coupling agents in the plasma of amine-like groups using an additional (eg, metallic) cathode that is a source of nanoparticles as well as ion implantation method [3].

**Conclusions.** The use of additive technologies opens up wide possibilities for increasing the strain-strength characteristics of dispersional and directional reinforced composites by forming an optimal gradient of the material properties of the interphase layers, which ensures the most efficient transfer of mechanical stresses between the rigid reinforcing filler and the elastic matrix.

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## ELECTRIC ARC SYNTHESIS OF TITANIUM CARBIDE PARTICLES

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The carbon arc is used to synthesize a wide range of nanomaterials. The discovery of such carbon nanostructures as fullerenes and carbon nanotubes in the reaction products (soot) brought the fame to the reactor based on the graphite arc. Another important application of the electric arc discharge is the synthesis of various metal-carbon composite materials. In this case, additions of various elements with the pressure of saturated vapors not lower than that of graphite are introduced into the material of electrodes (or one of the electrodes). The reaction products can be metallic nanoparticles in a carbon matrix, particles of metal oxides and carbides.

In this research, the materials formed in the gas phase were studied at joint electric arc spraying of titanium-graphite (Ti-C) and TiC-G rods. The Ti-C nanostructured material is synthesized by a DC plasma-arc setup. The reactor is a vacuum chamber, which is pumped out and then filled with helium to the working pressure. In the reactor, there are two electrodes, and the distance between them can vary. A DC arc discharge glows between them. The cathode is a graphite tablet with the diameter of 20 mm; the anode is a rod with the diameter of 8 mm and length of 70 mm. A water-cooled removable screen is mounted around the electrodes at the distance of 5 cm to collect the synthesis products. Under the conditions of the present experiments, the interelectrode distance is maintained to achieve the arc voltage of 20 V; the discharge current of 125 and 150 A was set. The anode is a graphite rod with an aperture along the axis, filled with silicon-graphite powder in a predetermined ratio. The rods were made of carbon with the density of 1.82 g/cm<sup>3</sup> and purity of 99.99. The powder of pure titanium and titanium carbide was used as a titanium additive. The molar fraction of metal in the sprayed electrode was 6.4% and 20%. The synthesized material was annealed in air at the temperatures of up to 950°C. The materials were analyzed by transmission electron microscopy (JEOL - 2010).

Due to the works performed, the experimental data on the synthesis of titanium-carbon composites under various discharge conditions were obtained. The shape, size and phase composition of titanium particles in the carbon matrix were investigated. It is shown that when the carbon matrix is removed, the nanoparticles are sintered and oxide particles larger than 100 nm are formed.

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# INVESTIGATION OF THE MOBILITY OF A CONCRETE MIXTURE AS A FUNDAMENTAL FACTOR IN THE FORMATION OF MIXTURES FOR 3D-PRINTING

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An important task for realizing the possibilities of 3D-printing is the selection of mixtures of composite building materials that meet the required performance characteristics of the erected building and meet the requirements of the equipment. Fine-grained concrete mixtures are used that have the necessary strength, rigidity, frost resistance, increased adhesive and cohesive properties, and hardening speed are mostly used for this purpose.

At present, there are no recommendations on the selection of mixtures of concrete with the necessary set of properties. When designing the mixtures of composite materials, it is also necessary to take into account the features of the 3D-printer (mixture delivery time, mixture pressure, contact surface materials).

Concrete mixture transported through hoses must have relatively high mobility to allow its mechanized feeding to the extrusion site and not to separate, while it must retain its shape after extrusion. Hardened concrete must have high strength and deformability, especially with bending loads and high adhesion to concrete.

Therefore, the actual task is to develop a complex additive for a concrete mix, which gives the concrete mix high thixotropic properties with simultaneous increase in strength of the formed concrete stone and its adhesion to concrete.

The aim of this work is to study with the help of Agocel S-2000 additive based on polyacrylamide the control of the basic properties of solutions (water retention, mobility, strength).

The samples, cubes with dimensions  $20 \times 20 \times 20$  mm stored in air-moist conditions, were prepared to study the properties of cement-sand mortars modified with polyacrylamide, after which tests were carried out for strength in accordance with the State Standard 10180-2012 at 3, 7, 28 days of hardening. To carry out the tests, cement-sand mortars were prepared, the ratio is binding: the ratio of sand was 1: 2. The rheological characteristics of freshly prepared solutions were also studied: mobility, water-retaining capacity (the State Standard 31356-2013).

The following were used in the work: portland cement class Cem I 42,5N, according to the State Standard 31108-2016. The sand test was carried out in accordance with the State Standard 8736-2014. The grain composition and the size modulus of the sand, the bulk density, the true density, the content of silty and clay particles, mixing water were determined in accordance with the State Standard 23732-2011 "Water for concretes and mortars. Technical conditions ».

A modifying additive Agocel S-2000 with a viscosity of 1,500mPa s (5 g/l, 20°C, Brookfield RVT 20 rpm) based on polyacrylamide was chosen for a study.

The mechanism of action of polyacrylamide is connected to the interaction with mixing water. Presumably, the formation of a coagulation structure in the mixture, providing thixotropic properties of the mixture, is due to the formation of a spatial phase grid. At the same time, due to the fact that the formation of the structure occurs due to weak physical forces, the structure of the mixture is easily destroyed when the mixture is subjected to mechanical loads, thus the viscosity of the mixture does not increase during transportation. Thus, the introduction of polyacrylamide into the mixture for 3D-printing reduces the mobility of the solution, preventing spreading after the product formation process.

To determine the rational "additive-cement" ratio, additives were mixed with cement in the amount of 0.002; 0.004; 0.006% of the weight of the dry components. The dry components were first mixed, after which the mixture was covered with water. The water-cement ratio was 0.53.

The use of polyacrylamide showed a high thickening power when small amounts of additive were added to the cement-sand mixture. However, when introduced into the formulations, the

water-holding capacity of the mixture is decreased. In the future, this property can lead to stratification of the mixture into fractions, thereby negatively affecting the strength of the finished product. But at the same time, the introduction of an additive makes it possible to increase the strength characteristics of concrete. Thus, it is expedient to use this additive as a part of multicomponent polyfunctional action additive when forming mixture compositions for 3D-printing.

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# SURFACE MODIFICATION OF ADDITIVE MANUFACTURED METAL SPECIMENS BY AN INTENSE ELECTRON BEAM

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On the example of VT6 titanium alloy it is shown that successive surface modification of additive manufactured metal specimens in vacuum at an argon pressure of  $3.5 \cdot 10^{-2}$  by ten pulses with  $200 \mu\text{s}$ ,  $45 \text{ J/cm}^2$  and then by three pulses with  $50 \mu\text{s}$ ,  $20 \text{ J/cm}^2$  provides a considerable decrease in their porosity and surface roughness (20 times for  $R_a$ ) while their surface microhardness, friction coefficient, and wear level remain almost unchanged. After electron beam irradiation, the ultimate tensile strength of the material increases on 12%, and its tensile strain on 10% (fig. 1). For specimens obtained by conventional metallurgy and irradiated in the same modes, no such effects are observed.

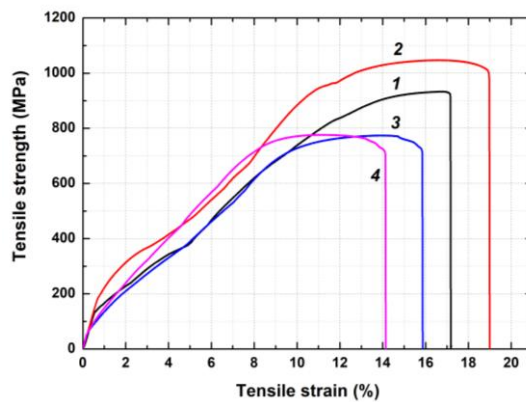


Figure 1. Strain hardening curves for VT6 titanium alloy under tension: 1 – additive manufacturing; 2 – additive manufacturing and irradiation; 3 – conventional metallurgy; 4 – conventional metallurgy and irradiation.

Our study shows that the difference in the mechanical properties of the materials is due to differences in their defect structure. After intense pulsed electron beam irradiation, the additive manufactured material reveals a polycrystalline structure with a grain size of  $15\text{--}60 \mu\text{m}$  and a cellular structure with a cell size of  $0.5\text{--}1.2 \mu\text{m}$  in the grain volume (fig. 2,a). The material obtained by conventional metallurgy assumes a polycrystalline structure with a grain size of  $50\text{--}800 \mu\text{m}$  and a rough lamellar structure in the grain volume (fig. 2,b).

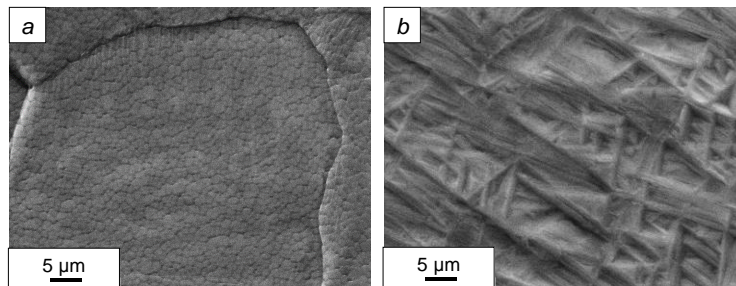


Figure 2. Intragrain structure of the surface layer of VT6 titanium alloy, formed as a result of irradiation with an intense pulsed electron beam of samples prepared a) by the method of additive technologies and b) methods of traditional metallurgy

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## NEW ACOUSTIC MATERIALS BY ADDITIVE TECHNOLOGY

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The main goal of this paper is to demonstrate the advantages of additive technology in the development and fabricating of new acoustic materials with unique properties, so called acoustic metamaterials (AMM). Several samples of AMM absorbers (AMMA) with high sound absorption are presented. The samples in the form of cellular thin-walled periodic structures are synthesized by photopolymer material using the additive PolyJet technology. One AMMA model represents the cellular thin-walled internal structure with cells of the resonant type coupled by tubes with open end faces of 1 cm diameter. The geometrical parameters of one cell were chosen from a condition of obtaining high sound absorption in the frequency range, 300-600 Hz (the sizes of one cell is 50x50x50 mm and walls of 1 mm thick). Another basic AMMA structure chosen is a labyrinthine type structure composed of a number of compactly "coiled-up" resonators with various parameters. It is given in Figure 1.

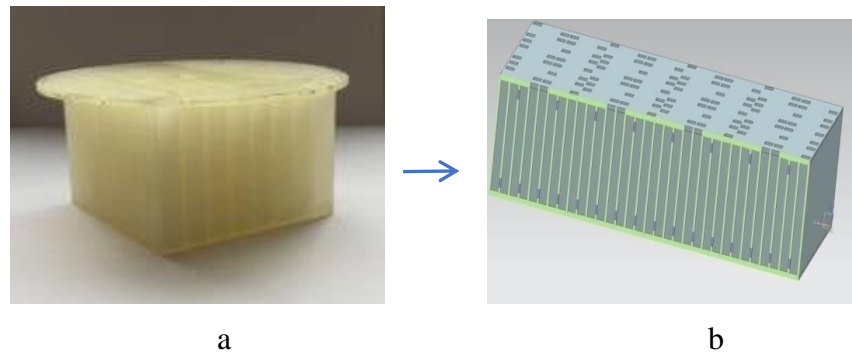


Figure 1. Labyrinthine cell-structure made by PolyJet technology:  
a) the sample general view and b) its 3D digital cross-section

An essential feature of AMMA sample design are thin walls and complex space geometry with the horizontal elements, which require to use of supporting materials in the process of manufacture that complicates it, and the subsequent finishing processing. The task was to produce the desired structure without loss of quality. Two forms of additive manufacturing technology, the stereolithography (SLA) and the PolyJet 3D printing, were considered and the technical data of available printers LS 250 and Objet Eden 350 were compared.

The choice was made in favor of the PolyJet 3D printing and Objet Eden 350 printer, which has sufficient build platform and fabrication accuracy to create the 3D object with required parameters (based on the authors experience). To avoid possible distortion of the given form of the AMMA sample (e.g., effect of warping walls) additional experimental study has been conducted. For this purpose, several samples of thin plates were constructed by Objet Eden 350 printer. The optimum arrangement of the AMMA sample on a build platform was defined for the printed object to have no distortion.

The peculiarities of the synthesized process are studied and optimal parameters are found to provide the required shape of the samples. The results of experimental study of sound absorption efficiency of the synthesized samples are also presented that demonstrate twice higher sound absorption efficiency comparable with commonly used absorbers.

The work is supported by the Russian Science Foundation (grant no. 15-19-00284).

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## PHASE FORMATION STUDY OF A MATRIX RECEIVED BY THE SHS METHOD

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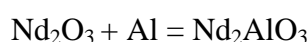
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There is about 560 million cubic meters of radioactive waste accumulated within the territory of Russian Federation; moreover, 50 % of it is located in temporary storages before further reprocessing. Besides that, 5 million cubic meters of radioactive waste is originated every year. [1].

Matrix-based materials are expected to help overcome the above-mentioned drawbacks. One of the most prospective matrixes for radioactive waste immobilization is perovskite ceramics. It is an analogue of natural stable minerals, capable of meeting structural behavior requirements for radioactive waste immobilization.

Initial charge material was prepared on the assumption of the following exothermic reaction:



Sample 1 with Al/Nd<sub>2</sub>O<sub>3</sub> proportion 1/1 and compaction pressure 25 MPa (Fig. 1) after SHS has the following phase composition: 69,1% Al, 19,6% Nd<sub>2</sub>O<sub>3</sub>, 11,3% NdAlO<sub>3</sub>.

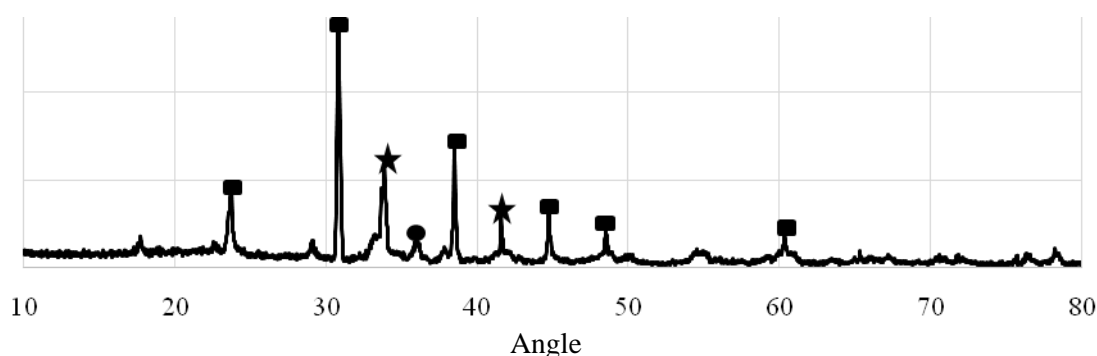


Fig. 1 – Sample 1 X-ray diagram,  
where ■ – NdAlO<sub>3</sub> – 11,3 %, ● – Nd<sub>2</sub>O<sub>3</sub> – 19,6%, ★ – Al – 69,1 %.

Analyzing the data obtained by XRD, it was established that the reaction temperature in the samples during the flow of SHS does not reach the required level for the formation of perovskite NdAlO<sub>3</sub>, at the periphery of the samples. It is possible to achieve an increase in the reaction temperature by adding Ni to the initial charge and carrying out the synthesis in a vacuum.

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# NUMERICAL MODELING OF TWO PHASE FLOW FOR DIRECT METAL DEPOSITION

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Laser cladding is an effective method of production of various coatings including functional-gradient, multi-layer ones, plus it is a flexible way to produce the details of complicated geometry.

One of the main scientific and technological problem of technology is increasing the coefficient of the use of powder. Other important problems are increasing spatial resolution of material deposition and reducing the roughness of the cladded bead, protecting the optical elements of the laser nozzle head from the damage of hot powder particles.

The structure of gas-powder flows formed by the laser-cladding nozzle and the accuracy of focusing the jet of powder into the region of the melt created by the laser beam is of decisive importance of the tasks mentioned above.

In this work, physical and mathematical models of powder transportation by gas during laser cladding are discussed. A comparison of numerical modeling results and experimental data is presented.

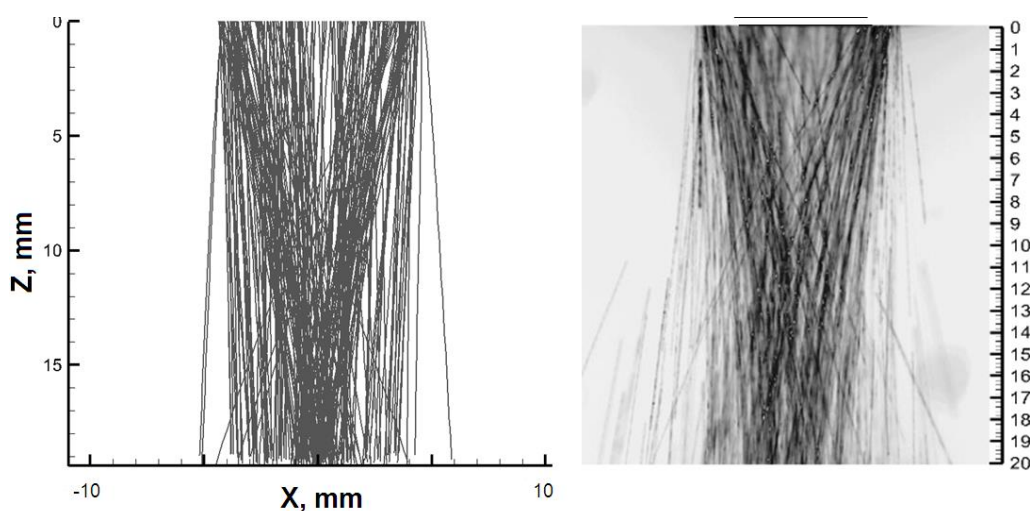


Figure 1. Comparison of particle tracks obtained numerically and experimentally [1].

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# SILICA NANOPARTICLES EFFECT ON THE LIQUID AND ITS PROPERTY MEASUREMENTS

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Nanotechnologies have a high growing temp and may be combined with additive technologies. Different nanoparticles may be applied to modify known additive materials or may be additive materials. Such role nanoparticle also plays in other technologies. So the problem of interaction between nanoparticles and medium is of great relevance. The carrier medium for nanoparticles often is a liquid. So we demonstrate some nanoparticles effect on liquid evidence from hydrophilic silica nanopowders in distilled water.

First of all the effective viscosity of the nanoliquid based on distilled water and different silica nanopowders much higher than in the case of distilled water with such volume concentration of micron particles which can be calculated by classical Einstein and Batchelor law [1] (Fig. 1.a). That is known fact for most of nanofluids but in earlier works this fact just was stated and some empirical dependencies were presented for experimental data descriptions [2]. We demonstrated that the assumption about some adsorbed layer on the nanoparticle surface let to use the Einstein and Batchelor formula with some effective volume concentration to describe nanofluids viscosity [3].

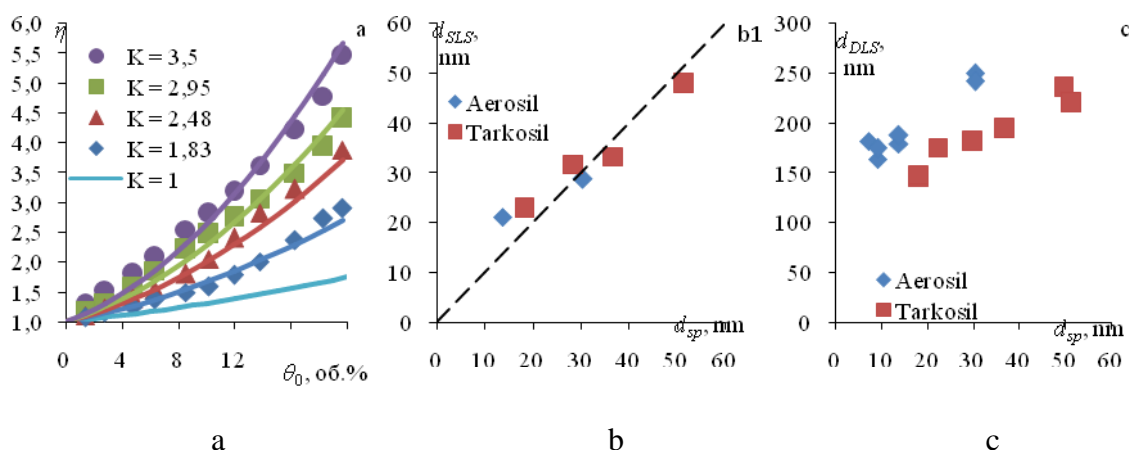


Figure 1. a) The relative viscosity of nanofluids  $\eta$  depends on nanoparticles volume concentration  $\theta_0$  in compare with Einstein and Batchelor law with rise up to  $K$  volume concentration. b, c) The particle size measurements results by SLS  $d_{SLS}$  (b) and DLS  $d_{DLS}$  (c) in compare with  $d_{sp}$  calculated from specific surface area of used nanopowders of different group – Aerosil and Tarkosil.

Similar concept of fixed water layer was previously applied to describe extraordinary water flow resistance of narrow quartz channels in compare with theoretical resistance of Poiseuille flow in such channels. High-precise experiments demonstrate the absent of any thixotropic effect in water even in low tangential stress. So the model of raise viscosity in water layer around hydrophilic quartz channel surface was developed to completely describe the experimental data [4, 5].

The molecular structure of water layer which lay around quartz surface was investigated in [6]. So it was demonstrated that water molecular dipoles structured out of hydrophilic surface and along of hydrophobic surface. The water molecules structuring isn't immobilizing but it may effect on a dynamic water characteristic – viscosity for examples.

So it is clear that the model of adsorbed layer on the nanoparticle surface in the problem of nanofluids viscosity is some effective model and the thickness of this layer is the thickness of water layer with raise viscosity around hydrophilic silica nanoparticles surface. This conclusion also let to solve come antinomy results of measurement of nanoparticles size in water solution by Static Light Scattering (SLS) and Dynamic Light Scattering (DLS) methods (Fig. 1.b, c).

The nanoparticles in liquid scatter the light. So the intensity of transmitted thought nanoliquid light decreases. If the nanoparticles size much lower than wavelength then the nanoliquid transmitted spectrum is described by Rayleigh law. That's let to calculate the nanoparticles size. The results of such SLS measurements  $d_{SLS}$  correlate with the size of particles  $d_{sp}$  which can be calculated from the specific surface area of nanopowders which are used to prepare nanofluids (Fig. 1.b).

The DLD method based on dynamic characteristics of scattered by nanoparticles light. Autocorrelation function of scattered light may be associated with mobility of particles. The theory Einstein-Smoluchowsky of Brownian motion usually is used to calculate the particles size from its mobility. The results of such DLS measurements  $d_{DLS}$  are much large then the size  $d_{sp}$  (Fig. 1.c). Here we see the antinomy between DLS and SLS methods. Such results of DLS measurements usually are interpreted as agglomeration of nanoparticles. But SLS clearly demonstrate that nanoparticles isn't agglomerated. Solvation of this antinomy is inapplicability of the Einstein-Smoluchowsky theory of Brownian motion. This theory does not take into account that the water viscosity around nanoparticles surface much higher than in volume water so the mobility of nanoparticles is really reduced.

So we can conclude that the applicability of nanotechnology for additive technology strongly require the fundamental investigations of the problem of the nanoparticles effect on its medium as well as the accuracy in applications of different analytical methods to measure characteristics of nanosystems.

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