



# ADVANCED HIGH ENTROPY MATERIALS

Abstracts of the IV International Conference  
and School of Young Scientists  
"Advanced High Entropy Materials"

Chernogolovka, Russia,  
September 26-30, 2022

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# ПЕРСПЕКТИВНЫЕ ВЫСОКОЭНТРОПИЙНЫЕ МАТЕРИАЛЫ

Тезисы IV Международной школы-конференции  
«Перспективные высокоэнтروпийные материалы»



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## TOPICS

### *Main directions of work:*

- High- and medium-entropy alloys, compositionally complex alloys: fabrication and processing methods, structure, mechanical, and functional properties, phase stability and phase transformations, deformation mechanisms, diffusion and ordering;
- High-entropy and compositionally complex coatings, methods of their production, structure, and properties;
- High-entropy ceramics, methods of their preparation, structure, and properties;
- New compositionally complex materials for technology and medicine, including nanostructured materials, microstructure design of multicomponent materials, practical applications;
- Advanced methods of fabrication and processing of metallic and non-metallic materials for structural and functional applications, including additive technologies, new methods of casting, powder metallurgy, welding, surface treatment;
- Other promising developments of new metallic and non-metallic materials;
- Computer modeling of the behavior of compositionally complex alloys, coatings and ceramics in different conditions.

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## CONFERENCE ABSTRACTS

### MACROKINETICS OF COMBUSTION OF GRANULAR MIXTURES (Ti+C)-Ni. EFFECT OF GRANULES SIZE

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One of the methods for obtaining metal-ceramic powders for applying protective coatings is self-propagating high-temperature synthesis (SHS).

Scaling up the process of obtaining composite materials from a mixture of powders of metals and nonmetals by the SHS method requires reproducibility of combustion parameters and predictability of the properties of the resulting products. As shown in [1], in the synthesis of titanium carbide with a nickel binder, it is possible to achieve stabilization of the process and the phase composition of the products by using a mixture granulated with an alcohol solution of polyvinyl butyral (PVB). However, the influence of such important characteristics of mixtures as the size of granules on the combustion process has not been studied.

In the present work, for granular mixtures, a study was made of the regularities of combustion with a change in the size of the granules.

Table 1. Burning rates of  $(100\%-x)(\text{Ti}+\text{C})+x\text{Ni}$  granules of different fractions in the absence of a gas flow.

<i>N</i>	<i>Fraction, mm</i>	<i>d, mm</i>	<i>U, mm/s, x=10%</i>	<i>U, mm/s, x=20%</i>
1	0,4-0,8	0,6	51	34
2	0,8-1,2	1,0	64	37
3	1,4-2,0	1,7	78	43
4	0,6-1,6	1,1	63	40

The phase composition of the combustion products, according to X-ray phase analysis, does not change with a change in the size of the granules.

The products of the synthesis of the studied granular mixtures, in contrast to powder mixtures, were highly porous brittle particles that were easily ground to a powder state.

It has been established that the burning rates of granular mixtures for fractions of 0.4÷0.8; 0.8÷1.2; 1.4÷2 and 0.6÷1.6 mm higher than for powder mixtures of the same composition, due to a change in the combustion mechanism.

Based on the experimental burning rate of the mixtures, the burning rate of the granule substance was calculated and it was shown that it is much higher than both the burning rate of the powder and the burning rate of the granular mixture.

The time of combustion transfer from granule to granule is determined by calculation.

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# CHEMICALLY ACTIVATED COMBUSTION SYNTHESIS OF ALON UNDER HIGH NITROGEN PRESSURE

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This work explores the synthesis of aluminum oxynitride (AlON) by combustion of Al/Al<sub>2</sub>O<sub>3</sub> mixtures under a high-pressure nitrogen atmosphere. The effect of nitrogen pressure and addition of Mg(ClO<sub>4</sub>)<sub>2</sub> and excess Al on the exothermicity of the mixture, kinetics of the combustion process, and phase composition of combustion products is studied. The addition of Mg(ClO<sub>4</sub>)<sub>2</sub> in combination with over-stoichiometric Al increases the combustion temperature of the green mixtures up to 1850 °C, enabling the synthesis of phase-pure AlON powder with 1 – 2 μm grains size. Additionally, engineering of bimodal particle distribution in SHS products is possible by using Al powders with different particle sizes.

In particular, we reveal the dependence of the AlON lattice parameter on nitrogen pressure (and, correspondingly, nitrogen content in the combustion products). The nitrogen content and the lattice parameter increase up to 4.1% and 7.952 Å, correspondingly at PN<sub>2</sub> = 60 MPa.

Addition of yttria results of smoothing of the particle surface in combustion products, which is likely conducive to structural refinements of the products during the ball milling.

Finally, we demonstrate the possibility of the fabrication of transparent AlON ceramics from the combustion products. Pressureless sintering of SHS AlON powders produced transparent ceramics with up to 70 % of light transmittance, hardness H=17.7±2.0 GPa, Young's modulus E= 320±29 GPa, and elastic recovery up to 62.8%.

## EFFECT OF Sn AND In ON THE MICROSTRUCTURE AND STRENGTHENING IN THE ALUMINUM Al-Cu-Si BASED ALLOYS

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The Al-Cu-Si based aluminum alloys are one of the most widely used in industry due to the good combination of mechanical and technological properties. The strengthening in these types of alloys comes from the predominant formation of the metastable plate-like  $\theta'$  phase [1, 2]. One should note that while the high temperature strength (at temperatures of up to 250 °C) of these alloys can be accepted as high enough, the room temperature properties are relatively low. However, it is well known that the microaddition of the low melting elements Cd, Sn and In allows one to dramatically increase the strength properties of the Al-Cu based alloys. The increase in the strength is due to the phenomenon of refining of the  $\theta'$ -phase precipitation structure. However, owing to the exceptional toxicity of Cd, this alloying cannot be used in most productions. On the other hand, according to numerous reports [3-4], tin (Sn) and indium (In) trace additions can be a promising substitute for cadmium. In the studies, we analyzed the separate influence of the 0.1 wt.% of Sn and In trace addition on the microstructure and precipitation hardening response in the Al-Si-Cu based alloy. For the study, the various advanced techniques such as thermodynamic calculations, transmission electron microscopy (TEM) and atom probe tomography (APT) analyzes, and others were used. The data obtained revealed that the peak hardness of the alloy with trace solutes is about 20 % higher compared to the trace solutes free alloy (135-140 vs 115 HV) and the peak hardness is achieved in a much shorter time of aging (2-4 h. vs 10 h.). Analysis of the precipitation structure using both TEM and APT revealed that the trace solutes lead to substantial refinement of the  $\theta'$  phase platelets with the increase in the number density. Using obtained data, the mechanisms of the influence of the trace solutes were proposed.

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**SYNTHESIS OF MATERIALS BY THE COMBUSTION**

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ISMAN – founder and scientific leader in the field of material-forming combustion processes and explosion, structural macrokinetics and self-propagating high temperature synthesis (SHS). The ISMAN team has competence in the field of technologies for the synthesis of powders of nitrides, carbides, and other composite powders and materials based on them. ISMAN activities cover fundamental research and the implementation of development results for industrial production, as well as the training of specialized young scientists. The main areas of ISMAN research and development include the following directions of key importance both for the fundamental development of materials sciences and for solving the most of current importance problems of the development of society and the country.

Development and production of powders and powder materials using combustion processes (SHS).

The SHS method makes it possible in the shortest possible time to organize pilot and, if necessary, large-scale production of hundreds of items of critically important powders and nanopowders, both on the basis of existing universal SHS reactors and proven technologies, and on advanced developments. Powders of AlN, Si<sub>3</sub>N<sub>4</sub> (alpha and beta phases), BN, SiC, SiALON, MoSi<sub>2</sub> produced in ISMAN, especially refractory carbides and borides, including multicomponent ones, silicides, selenides and others are in great and growing industrial demand. New chemical compositions and structures of powder materials are being developed, for example, high-entropy alloys and compounds, ultra high melting temperature materials.

Study of the fundamental aspects of explosion welding of dissimilar materials and the production of large-sized bimetallic and multilayer structural materials (steel-titanium plates, etc.) for the chemical industry, nuclear engineering, military equipment.

Development of methods for centrifugal SHS technology of new cast alloyed and modified alloys. One of the main tasks of this direction is the development of cast alloys and coatings based on tungsten for solving problems of nuclear and thermonuclear power engineering. It is planned to develop the production of electrodes for electric spark and electric arc surfacing using SHS-extrusion methods for the obtaining functional coatings. The results of these investigations are used in heavy engineering, transport and other industries for the reconstruction and extension of the service life of various structural elements and machine parts.

Development of special devices to ensure the safety of work and prevent explosions of installations with nuclear reactors, based on the use of SHS gas absorption processes.

## **BIOCOMPATIBLE POROUS SHS TINI-BASED MATERIAL WITH A MICROPOROUS SURFACE OF PORE WALLS**

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Porous TiNi-based alloys are a universal material with a wide range of functional and structural properties due to the possibility of using a wide range of technological methods in the process of its production and processing, they use various temperature-time regimes, they are synthesized in the presence of ultrasonic treatment, different SHS modes and different ratios of components in the initial powder mixture are used. The design of the pore space that interacts with cellular structures during implantation of a TiNi-based construct into the human body is a promising direction for optimizing the biocompatibility parameters of the TiNi SHS material. The use of a pronounced phase inhomogeneity of the SHS material opens up the prospect of increasing the specific surface area by etching particles of the  $Ti_4Ni_2(O,N,C)$  oxycarbonitride phases. These particles are located on the surface of the pore walls along the grain boundaries and less often in the body of the grain. In this regard, the development of a chemical etching technique for creating a microporous structure of the surface of pore walls is an urgent task and is of practical importance.

Porous SHS materials based on TiNi are obtained at a synthesis start temperature of 400 °C. The surface of the pore walls was treated with solutions based on nitric, hydrofluoric, sulfuric, hydrochloric, acetic, orthophosphoric acids at various temperature and time intervals of exposure.

The study of the influence of the proposed treatments on the structure of the pore space was carried out by scanning microscopy. The structural features of the surface of the pore walls and the distribution of particles of secondary phases  $Ti_4Ni_2(O,N,C)$  were studied in comparison with the initial untreated material. It has been established that one of the promising compositions for modifying the pore space is a composition based on an aqueous solution of hydrofluoric and nitric acids. The proportion of micropores smaller than 50 nm increased from 59 to 68%, and the number of pores larger than 1 μm doubled from 11 to 22%. At the same time, the porosity and average pore size of the SHS material practically do not change.

Excessive etching leads to disruption of the regular porous structure. By layer-by-layer etching of the TiNi matrix and etching of titanium-enriched particles, new pore wall surfaces with micropores are formed. Macropores significantly increase in size, and the metal matrix degrades due to active processes of etching of interpore bridges. Dead-end pores were found on the surface of the pore walls, inside which there are micropores. It is necessary to carry out chemical treatment with the shortest duration in order to avoid etching of the TiNi compound.

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# AlCoCrFeNi HIGH ENTROPY ALLOY: FABRICATION TECHNIQUES AND MECHANICAL PROPERTIES UNDER THE EXTREME CONDITION

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AlCoCrFeNi, first reported in 2007, it belongs to the Al<sub>x</sub>CoCrFeNi system, which is one of the most well-developed HEA systems. AlCoCrFeNi shows a high compressive and good ductility at temperature around 500 °C. This high strength at elevated temperatures makes it a promising structural material. In this report, first, different fabrication methods of AlCoCrFeNi will be discussed. AlCoCrFeNi was successfully fabricated by casting, shock consolidation and self-propagation high temperature synthesis (SHS) techniques. Effect of these methods on the microstructure and mechanical properties was investigated. In shock consolidation technique, the starting powders were mixed by mechanical alloying and then the shock wave was imposed to the compacted powders by explosion. Mixtures of metal oxide powders (Co<sub>3</sub>O<sub>4</sub>, Cr<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and NiO) and Al powder was used in SHS technique. The shock consolidated sample showed the nano-structure with hardness of 715 HV. SHS samples showed the elongated microstructure.

Secondly, the mechanical properties and microstructure evolution of as cast AlCoCrFeNi (HEAs) was investigated under the extreme condition (high strain rate and high temperature). High strain rate experiments were performed by the split-Hopkinson pressure bar (SHPB) at strain rates of 500-2500 s<sup>-1</sup> at three different temperatures (27, 250 and 500° C). And Gleeble machine was used to applied the test at elevated temperatures of 500, 750 and 900 ° C under quasi-static condition. Obtained stress-strain curves were analyzed. At high strain rate loading of the AlCoCrFeNi, no recrystallization or any phase transformation was observed even at temperature of the 500° C. The flow stress increases with more strain rate from 500s-1 to 2500 s-1. It is observed that the cracks are formed and propagated in the sample at all tested temperature under the dynamic loading condition, however number of the cracks are decreased with increasing the temperature. At quasi-static loading at temperatures higher than the 500° C DRX was observed with necklace structure.



## CHEMICAL MODIFICATION OF THE PORE SPACE OF SHS MATERIALS BASED ON TiNi

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Porous SHS materials based on TiNi are actively used to create implantable devices used in oncology, traumatology, maxillofacial surgery, etc. It is known that the process of interaction of an organism with a porous implant is determined by the morphochemical parameters of the surface of its pore space. Depending on the initial temperature conditions of SHS synthesis, after the passage of a combustion wave, it is possible to obtain a material with different porosity, the fraction of inclusions of particles of secondary phases, and the thickness of the oxycarbonitride layer on its surface. A method for modifying the pore space of such materials is possible by exposing the sample surface to a liquid solution of acids in order to etch out the layer of oxycarbonitrides and particles of secondary phases. The choice of this method is associated with a limited effect of modification methods on the surface of the material due to its porous structure.

The subject of the study is alloys based on TiNi-based alloy obtained by the SHS method at synthesis temperatures of 400 °C and 600 °C. Structural features of the pore space of porous samples were studied by SEM, chemical composition – EDX. To modify the surface of the samples, an aqueous solution of acids was used: hydrofluoric, nitric, hydrochloric.

In the course of the work, it was shown that a porous TiNi-based alloy obtained at a temperature of the beginning of synthesis of 400 °C is characterized as fine-porous (MP), and at 600 °C – coarse-pored (CP), each of which has a uniform macrostructure of the pore space and a rough microporous surface of the walls since. It has been established that the amount of secondary phases on the surface of the pore space in the SHS MP material is maximum, which is due to the concentration inhomogeneity of the melt and the thermal inhomogeneity of the reaction front during synthesis.

The effect of etching with an aqueous solution of acids of SHS alloys (CP and MF) on the pore space and the possibility of creating a microporous surface of the pore walls is established. It is shown that the chemical treatment of the SHS material is divided into three stages: removal of the oxycarbonitride layer, interdendritic layers with particles of secondary phases, and the layer with the dendritic segregation zone. After etching, the number of micropores in the MT material increases, which makes a significant contribution to the surface roughness of the pore walls. The CP-treated SHS material had a less pronounced rough microporous surface compared to the MT due to the lower initial number of secondary phases. It has been established that the surface structure of the pore walls obtained after etching has higher adhesive properties for interaction with cell cultures.

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**THE EFFECT OF NITROGEN ALLOYING ON STACKING FAULT ENERGY,  
DISLOCATION ARRANGEMENT AND DEFORMATION MECHANISMS  
OF HIGH-ENTROPY CANTOR ALLOY**

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The effect of nitrogen-alloying on the microstructure and deformation mechanisms of the high-entropy FeMnCrNiCo alloy (Cantor alloy) has been explored in the temperature interval (77-473)K. After homogenization treatment (cold rolling and anneal at 1473K), alloys with nitrogen contents of 0.8, 1.4 and 1.6 at. % possess a single-phase coarse-grained austenitic structure with the grain size  $\approx 200 \mu\text{m}$ . Uniaxial tensile tests are performed at the initial strain rate  $5 \times 10^{-4} \text{ s}^{-1}$ . Dislocation arrangement and deformation mechanisms are revealed using transmission electron microscopy.

It has been experimentally shown that nitrogen-alloying provides strong solid-solution hardening of the Cantor alloy. The solid-solution strengthening of the yield strength describes by a linear dependence  $\Delta\tau \sim C_N$  with high accuracy. At room temperature deformation regime, the dislocation glide mode changes from the planar to wavy one, which is typical of plastic deformation of Cantor alloy; twinning activates after substantial strain and acts as a secondary deformation mechanism. At lower temperatures, the activation of the mechanical twinning and more planar dislocation glide both support strain hardening, providing high strength properties and elongation of the Cantor alloy. Strain-hardening capacity of the interstitial-free Cantor alloy concedes those for interstitial alloys. At room temperature, the increase in strain hardening of the FeMnNiCoCr-N alloys relative to the Cantor alloy is associated with the change in dominated slip mode. The higher nitrogen content, the more planar dislocation arrangement and the higher strain hardening of the alloy. The planar dislocation glide in alloys with 1.4 at. %N and 1.6 at. %N is accompanied with the formation of the high dense dislocation walls, which effectively hinder dislocation glide in intersecting slip planes. Nitrogen-containing alloys show lower activity of the mechanical twinning relative to the interstitial-free Cantor alloy despite their close values of the stacking fault energy (SFE  $\approx 30 \text{ mJ/m}^2$ ). At room temperature deformation, twinning is completely suppressed, but in low-temperature deformation regime it develops complementary to the dislocation slip. Decrease in test temperature and nitrogen-alloying both enhances the tendency to the planar glide, which is responsible for extremely high strain hardening (up to the G/30). Analysis of the dislocation arrangement testifies that, additionally to the small SFE, large shear modulus, high atomic size misfit parameter, and high solute concentrations, the short-range ordering plays an important role in deformation behavior of FeMnNiCoCr-N alloys.

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## THE EFFECT OF SEVERE PLASTIC DEFORMATION ON STRUCTURE AND PROPERTIES OF MEDIUM ENTROPY ALLOYS WITH TWIP/TRIP EFFECTS

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High-entropy and medium-entropy alloys (HEAs and MEAs) with TWIP/TRIP effects attract great interest due to a good balance of ultimate strength and ductility; in some cases, decreasing temperature to cryogenic conditions even improves their mechanical properties [1,2]. However, such alloys possess rather low yield strength at room temperature, an increase of which can be achieved via deformation treatment.

One of the well-known and effective methods of material deformation is high-pressure torsion (HPT). HPT at room temperature can be used to obtain a nanocrystalline structure of the material. Information on the effect of HPT on the structure and mechanical properties of medium entropy alloys with TWIP/TRIP effects is practically absent.

In this work, medium-entropy iron-rich alloys obtained by vacuum-arc melting were used as program materials. The alloys were rolled to 80% at room temperature and then annealed at 900°C for 10 minutes with further water cooling. In the recrystallized state, the alloys were subjected to high-pressure torsion at room temperature on Bridgman anvils at a speed of 0.5 rpm. The anvil rotation angles were 180° (N0.5), 360° (N1), and 1080° (N3) at a pressure of 6.5 GPa.

The study of the initial recrystallized structure showed that the alloys have a two-phase structure consisting of the fcc matrix and «islands» of the martensitic BCC phase. Particles of the M<sub>23</sub>C<sub>6</sub> carbides were found inside the grains and along boundaries. The HPT treatment leads to the formation of an inhomogeneous, strongly deformed microstructure with (sub) grains of ~ 50 nm after 0.5 revolutions (180°). The samples after three revolutions (N3) showed the maximum values of microhardness; in addition, attractive mechanical properties of the alloys were revealed during tensile tests at room and cryogenic temperatures. The relationship between the HPT parameters, structure and mechanical properties of alloys is discussed.

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## PHASE COMPOSITION AND MECHANICAL PROPERTIES OF NITROGEN-CONTAINING HIGH-ENTROPY ALLOYS

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The mechanical properties in 20Fe-20Mn-20Cr-20Ni-20Co (at. %, 0N-HEA), 20Fe-20Mn-20Cr-20Ni-19.2Co-0.8N (0.8N-HEA), 20Fe-20Mn-20Cr-20Ni-18.6Co-1.4N (1.4N-HEA) and 20Fe-20Mn-20Cr-20Ni-18.4Co-1.6N (1.6N-HEA) alloys have been investigated. The cast billets were subjected to a thermal-mechanical treatment which consisted of annealing at 1200°C for 2 hours, cold rolling with a 80 % reduction and annealing at 1200°C for 2 hours with a final water-quenching. All HEAs have been investigated using the X-ray diffraction analysis, light, transmission and scanning electron microscopy, tensile testing at temperatures 77 K and 297 K.

It has been shown that all alloys possess single-phase austenitic structure with FCC crystal lattice. Interstitial nitrogen causes a distortion of a crystal lattice and increases a lattice parameter of austenite from 3.598 Å in nitrogen-free alloy, to 3.604 Å, 3.607 Å and 3.608 Å in alloys with nitrogen concentration of 0.8, 1.4 and 1.6 at. %, respectively.

The 0N-HEA is characterized by high tensile strength and ductility. The maximum values of the yield strength (YS) and elongation to failure ( $\delta$ ) are observed at temperature  $T = 77$  K (390 MPa and 92 %). The mechanical properties of this alloy decrease with the increase in the test temperature. Despite this fact, at room temperature the YS and ductility of 0N-HEA remains high (YS = 192 MPa, 61 %).

Nitrogen-alloying increases the YS and ultimate tensile stress (UTS) of FeMnCrNiCo alloy, and these values are dependent on nitrogen concentration. At  $T = 77$  K, the yield strength YS = 513 MPa, 632 MPa and 560 MPa, UTS = 2044 MPa, 1930 MPa and 970 MPa for 0.8N-HEA, 1.4N-HEA and 1.6N-HEA, respectively. An increase in test temperature to 297 K promotes a decrease in the value of YS down to 250 MPa, 288 MPa and 375 MPa for 0.8N-HEA, 1.4N-HEA and 1.6N-HEA, respectively. The  $\delta$ -values in nitrogen-alloyed alloys in low-temperature deformation regime are much lower than that in 0N-HEA ( $\delta = 61$  %, 49 % and 9 % for 0.8N-HEA, 1.4N-HEA and 1.6N-HEA). However, at 297 K nitrogen-alloying weakly influences this value.

The results demonstrate that doping of FeMnCrNiCo high-entropy alloy with nitrogen atoms promotes the formation of a single-phase solid solution based on the FCC crystal structure and improves the strength characteristics of the initial alloy.

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## CHARACTERISTICS OF POROUS MATERIALS FROM BASALT FIBERS

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The use of porous materials based on ceramic fibers as the basis of high-entropy and filter materials is determined by the possibilities of operation at high (over 15 MPa) pressures, temperatures (up to 700 °C) while maintaining strength in long-term operation. Unlike the known woven materials (fiber basalt slabs), the size and pore configuration of the created porous materials remain constant during operation due to the rigidity of the frame. This technical solution makes it possible to increase the stability of the characteristics.

In general, basalt fibers are superior to glass fibers in terms of thermal, physical, electrical and acoustic characteristics, as well as chemical resistance.

It is shown in that during radial pressing, the dispersed medium changes its density only as a result of structural rearrangement of particles, which occurs fairly uniformly throughout the entire volume of the pressed body. Thanks to this, it is possible to form a technologically durable product from particles with a low plasticity resource. This feature of radial pressing makes it possible to obtain long-length porous pipes made of basalt fiber with a length-to-diameter ratio of more than 35.

In the pressure range of 20-50 MPa, billets in the form of pipes with dimensions of – 19 mm, length – 100 mm and samples in the form of cylinders Ø19 mm and a height of 10 mm were formed from a charge based on basalt fiber BS16-6-76 by radial pressing, followed by sintering in air in dia-temperature range 1050-1150°C. Pressing pressure –45 MPa.

The characteristics of the obtained prototypes were determined according to standard methods adopted in powder metallurgy and materials science.

In result of the conducted researches it is established that the sintering of basalt fiber at 1050 C and 1100°C forms a porous material with a structure characterized by a porosity of 52-75%, an average pore size of 8-18 µm, the permeability coefficient of (42-55) 10<sup>-12</sup> m<sup>2</sup>, compressive strength 7-13 MPa. The results obtained prove that a porous material made of basalt fiber is capable of providing the specified characteristics when used as a basis for high-entropy materials.

# STRUCTURAL AND COMPOSITIONAL TRANSFERABILITY OF DEEP LEARNING POTENTIALS: EXAMPLE OF GdTbDyHoSc

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The deep learning potentials based on neural networks are usually considered to have poor compositional and structural transferability. This consideration is based on the fact, that neural networks are not able to extrapolate well. But if neural networks are trained only at the melt configurations, and deep learning potential is able to describe some properties of crystalline phases, then such potential is said to demonstrate good structural transferability.

Another kind of transferability is compositional one: the ability of potential to describe compositions which differ from those included in training set. This is extremely important for the case of high-entropy alloys. For example, is it possible to describe five four-component derivative equiatomic alloys with the potential trained only at one mother five-component equiatomic alloy? If so, such potential demonstrates good compositional transferability.

These two types of transferability are investigated at present work on the example of GdTbDyHoSc alloy. Density functional theory via VASP [1] is used for creating dataset consisted of only melt configurations. DeePMD-package [2] was used to generate deep learning potential and LAMMPS [3] was used to perform classical molecular dynamics simulations with generated potential. For estimation of transferability quality two DeePMD-potentials were trained with different initialization, and during the simulation deviation in forces between them were computed. For analysis of the structural transferability of this potential, simulations of HCP crystal were performed. For estimation of compositional transferability, five different derivative equiatomic four-component melts were simulated.

The results demonstrate that such potentials have good structural transferability (from liquid to crystal), and satisfactory transferability to 4 component melts.

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## SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS (SHS) TECHNOLOGY FOR DISPOSAL OF RADIOACTIVE WASTE

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Further advance in atomic power engineering is closely associated with the problem of safe handle and burial of radioactive wastes, especially high-level radioactive wastes (RW) with a half-life period of million years. A best way to go about solving the problem is the incorporation of RW into the crystal lattice of mineral-like compound. In this context, of current importance seems to be the SHS of new ceramics based on rock-forming minerals highly resistant to the action of chemical agents, heat, and radiation and suitable for burial of RW.

1. Immobilization of the entire spectrum of RW — SrO, Cs<sub>2</sub>O, actinoids, rare earths.

In this case, SHS-produced perovskite CaTiO<sub>3</sub> and zirconolite CaZrTi<sub>2</sub>O<sub>7</sub> were found capable of immobilizing nearly all (except for Cs) components of RW (including plutonium). Cs could be immobilized in polluzite CsAlSi<sub>2</sub>O<sub>6</sub>.

2. Immobilization of <sup>14</sup>C graphite (T<sub>1/2</sub> = 5730 years) + fuel spillage (actinides, Cs, Sr).

Carbon, including <sup>14</sup>C, is immobilized in the form of stable carbide TiC.

Fuel spillage was immobilized in SHS-produced perovskite CaTiO<sub>3</sub>, garnet Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>, and hibonite (Ca,Ce)(Al,Ti)<sub>12</sub>O<sub>19</sub>. Volatile <sup>137</sup>Cs can be immobilized in the form of Cs<sub>2</sub>Ti<sub>6</sub>O<sub>13</sub>.

3. Long-living wastes: Actinide/Zr/rare earth group.

Such waste was immobilized in SHS-produced pyrochlore Y<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub>. Replacement of Ti by Zr improves the oxidation/radiation resistance of Y<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub>.

4. Combined use of SHS and hot pressing: Forced SHS compaction of large-sized (M=1750g) ceramic blocks.

Immobilization was carried out in steel cartridges used in the practice of waste burial.

Conclusions

Solid RW were immobilized into mineral-like ceramics produced by the technique of forced SHS compaction. Synthesized materials were found to exhibit high hydrolytic stability and mechanical strength.

## STRUCTURES AND MECHANICAL CHARACTERISTICS OF LAYERED COMPOSITE MATERIAL BASED ON TiB/TiAl/Ti

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The fabrication of layered structures can be achieved in many ways. Known methods of their manufacture include rolling, diffusion welding, explosion welding, plasma spraying, hot pressing, etc. An alternative to these traditional methods is the unrestricted SHS compression method [1]. This method is a process of obtaining compact materials in the mode of self-propagating high-temperature synthesis, compaction, and molding of combustion products under the influence of a constant pressure of the order of 10–50 MPa without the use of special molds on a hydraulic press. The method of unrestricted SHS compression allows one to manufacture layered composites in one technological stage in tens of seconds with a given set of properties [2].

In this work, a TiB/TiAl/ $\alpha$ -Ti layered composite material has been obtained from the initial components of titanium, boron, and aluminum by the unrestricted SHS compression method. It has been found that, as a result of combustion and subsequent high-temperature shear deformation (thermal and mechanical conditions of the unrestricted SHS compression method), ceramic (TiB) and intermetallic (TiAl, Ti<sub>3</sub>Al) microlayers are oriented along the direction of material flow and perpendicular to the applied load. The specific features of the structure of the ceramic and intermetallic layers and their phase compositions have been studied; the diffusion boundaries between the formed layers have been established and studied. It is shown that the synthesis of the material proceeds according to the solid/liquid-phase mechanism. The mechanical characteristics of the obtained layered composite material have been studied, namely hardness, microhardness, and fracture toughness. The mechanism of crack propagation has been established and the dependence of the values of the stress intensity factor on the orientation of microvolumes in the material has been determined.

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## SOLID-PHASE MECHANOCHEMICAL METHOD FOR OBTAINING METAL-POLYMER COMPOSITIONS

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Metal-polymer compositions and materials based on them are widely used in many fields of science and technology. The most interesting is the use of such compositions in the creation of high energy systems, where a high fullness of use of components is required. One of the effective ways for producing metal-polymer compositions is the solid-phase mechanochemical method, which consists in the joint mechanical processing of components in energy-intensive devices [1, 2].

The processes occurring in the high-energy devices are based on two main principles: first, the impulsive nature of the process (rotation of the processes of the appearance of the stress field and its relaxation); secondly, the local nature of the mechanical action on the matter (the stress field does not occur in the entire volume of a solid particle, but only at its contact with another particle).

The formation of a composition from a mixture of components is accompanied by their intensive dispersion and deformation mixing. The reaction mixture, having received a dose of energy, passes into a non-equilibrium state, in which external stresses do not act, and internal (residual) stresses relax, trying to transfer the matter to an equilibrium state. Relaxation of the stress arising as a result of mechanical action on a matter or mixture of matters is carried out with the release of heat, the formation of a new surface, the appearance of defects in crystals, the amorphization of the surface of a solid, and the formation of bonds.

That way, under mechanical impact on the polymer, intermolecular and intramolecular bonds are broken, and macroradicals appear that can react with the metal surface. Mechanical impact on the metal also leads to the formation of various types of active centers on its surface. As a result, the surface of both the metal and the polymer is activated, which promotes to adsorption-chemisorption interaction and the formation of a metal-polymer composition. Thermodynamically the process will be determined by the formation of a layer of a component with the lowest surface tension (polymer) on the surface of a particle of another component (metal).

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**ALUMINUM ALLOYS OF Al–Cu–Mn (Zr, Si) SYSTEM FOR WROUGHT PRODUCTS WITHOUT REQUIREMENT FOR SOLUTION TREATMENT AND QUENCHING**

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The principal possibility for obtaining wrought products (sheets, rods and wires) directly from cast ingots of Al-Cu-Mn (Zr, Si) alloys (without prior homogenization) has been justified. Electron microscopy (SEM, TEM and EMPA), X-ray diffraction, DSC and Thermo-Calc software simulation have been used for optimizing the alloy composition. Cold-rolled commercial AA2219 and model alloys Al-2%Mn-2%Cu and Al-2%Mn-2%Cu-0.4%Si-0.2%Zr (further 2Cu2Mn and 2Cu2Mn-SiZr correspondingly) alloys have been compared in phase composition, microstructure, physical and mechanical properties after different heat treatment routes. The as-cast structure of the model alloys proves to be similar to the homogenized structure of the branded alloy, providing a far shorter technological route required for obtaining a 95% cold reduction ratio. It has been shown that the as-cast structure of the model alloys has the minimum quantity of Al<sub>2</sub>Cu eutectic inclusions, and almost all manganese (and Zr) content and about 1.2% Cu are dissolved in the aluminium solid solution. This structure provides for a high plasticity that allows for deformation of ingots without their preliminary homogenization. The formation of the Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub>, Al<sub>20</sub>Mn<sub>3</sub>Si<sub>2</sub>, and Al<sub>3</sub>Zr (L<sub>12</sub>) nano-sized dispersoids (with total volume fraction of about 7 vol.%) has been found to provide for the retaining of the fibre-like (non-recrystallized) grain structure in the model 2Mn2Cu and 2Cu2Mn-SiZr alloy after annealing at 400 °C (3 h), despite a very high cold rolling reduction ratio. The model alloy exhibits a substantially higher strength performance after annealing at 400 °C in comparison with the AA2219 alloy. For example, the yield strength value of the model alloys is 200–210 vs ~90 MPa. This indicates a higher tolerance of the model alloys toward softening.

Model alloys were considered for obtaining a welded joint providing high mechanical properties and thermal stability by friction stir welding (FSW). Sheets with a thickness of 4 mm (reduction rate 90%) were obtained from a non-homogenized ingots (40 mm) on a laboratory rolling mill at a temperature of 350 °C. The structure and properties of the joining were studied before and after annealing at a temperature of 400 °C for 3 hours. It is established that the experimental alloys have high thermal stability, owing to which, after FSW in the zones of the weld and heat affected zone, the decrease in hardness does not exceed 10% and remains practically unchanged after annealing. It was shown that the FSW method makes it possible to obtain high quality welded joints, maintaining almost the initial strength (UTS = 280-290 MPa) with a significant increase in elongation (from ~3% to 12-16%).

Summing up the results, the 2Mn2Cu and 2Cu2Mn-SiZr model alloys show a potential to become the basis for designing new high-tech heat-resistant alloys as a sustainable alternative to 2xxx alloys.

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## MULTICOMPONENT ELECTROPLATING COATINGS FROM SOLUTIONS OF HETERONUCLEAR COMPOUNDS

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Obtaining data on the creation of high-entropy alloys is known to be relevant, and there are no electrochemical methods among the methods for obtaining them [1,2].

The production of electrochemical coatings based on high-entropy alloys is an unexplored field of electroplating.

In our opinion, the creation of scientific bases for obtaining the materials in question in electroplating can be based on the joint electrodeposition of metals, and in some cases nonmetals from solutions of heteronuclear or heteroligand compounds. In heteronuclear and heteroligand complexes, the donors of the resulting material are in the same chemical compound, which favorably affects the process of their joint electrochemical recovery.

The paper presents data on the chemical and electrochemical behavior of zinc(II)-chromium(III)-nickel(II)-glycine-water and zinc(II)-chromium(III)-cobalt(II)-glycine-water systems. The compositions and stability of complex compounds have been established by various methods. Based on the obtained distribution diagrams of complex compounds, the compositions of solutions for obtaining highly corrosion-resistant electroplating coatings have been developed.

The results of the element analysis by the X-ray photoelectron spectroscopy method showed the presence in the coatings not only of the corresponding metals, but also of other components of electrolytes. Depending on the composition of the electrolyte and the mode of electrodeposition, it is possible to achieve almost equal amounts of components in the resulting coatings of a certain thickness.

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## THE EFFECT OF PRIOR AUSTENITE GRAIN SIZE ON MICROSTRUCTURE AND TENSILE PROPERTIES OF TEMPERED LOW ALLOYED 0.2C STEEL

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The preferred microstructure of recent developed advanced high-strength steels (AHSS) contain significant fraction of lath martensite. The initial austenitic structure has a significant effect on the dispersion of the elements of the quenched and tempered martensite. The mechanical properties of these steels are correlated with the size of the units of formed martensite.

In the present study the low-alloy steel Fe-0.25%C-1.6%Si-1.47%Mn-0.51%Cr-0.27%Mo was investigated. The steel was subjected to homogenization annealing at a temperature of 1150°C. The steel was quenched from 900°C and tempered at 200°C; 280°C; 400°C; 500°C for 1 hour. To decrease the size of prior austenite grains the hot rolling processing at 1000°C, followed by cooling in air was performed. Tensile tests were carried out at room temperature on an Instron 5882 machine.

The deformation of the steel in the austenitic region is an effective way to refine the austenitic structure, which results in a decrease of the size of the elements of the martensitic structure after subsequent quenching. However, for high-strength low-alloy steels, the effect of such treatment on the structure and mechanical properties during subsequent quenching and tempering has not yet been studied.

The investigation of the quenched steel structures in the initial state and after hot rolling operation showed that the average size of the PAGs was 32.7 μm and decreased to 16.6 μm in the pre-rolled steel. The subsequent tempering of quenched steels does not lead to a significant change in the dimensions of the structural elements of lath martensite. The results of tensile tests showed that decrease in the PAG size by ~2 times leads to a significant increase in the yield and tensile strength. The maximum increase in tensile strength of +28% was observed in the quenched steel. The yield strength of the pre-rolled steel after quenching at 900°C and tempering at 200, 280, 400, 500°C was 1360 MPa, 1330 MPa, 1330 MPa, 1250 MPa and 1090 MPa, respectively. With an increase in the tempering temperature, the increase in the tensile strength decreases and after tempering at 500°C it is 70 MPa. The ductility of the steel with a smaller average size of the initial austenitic structure insignificantly decreases, by 1–2% on average. Thus, the use of pre-rolling for high-strength low-alloy steels makes it possible to reduce the size of the PAGs and significantly increase the strength characteristics without a pronounced decrease in ductility.

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## PHYSICO-CHEMICAL PROPERTIES OF NITROGEN-CONTAINING BIOCOATINGS BASED ON TITANIUM

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Recently, work has been actively carried out to study the structure and properties of biocompatible coatings, among which titanium oxynitride is distinguished. The development of medical materials based on titanium seems promising. In practice, both titanium dioxide and nitrogen-doped titanium oxide ( $TiN_xO_y$ ) coatings are used with technological substitution of oxygen for nitrogen atoms [1].

In this work, coatings were obtained by the RMR method, with different weight contents of nitrogen and oxygen. The samples obtained were studied by optical microscopy, infrared and gas spectroscopy, and Raman spectroscopy (RAMAN). To assess the corrosion properties of coatings, modern spectral analysis methods, gas chromatography and a polarization method for studying the anodic behavior of the Ti-O-N ternary system were used. It has been established that the biocoating has a number of unique properties: chemically and thermally stable, corrosion-resistant in various biological media. Upon contact with model biological fluids, compounds with an N-O bond are released from the coating, forming nitrogen nitrite / nitrate, which in turn is an important compound for a living organism [2,3].

The electronic spectra of coating samples are characterized by absorption in the region of 500-550 nm. The broadened band in this region allows us to conclude that there are areas of concentration of the metal complex in the film. In addition, the spectrum obtained in the study of the elemental composition of the film has pronounced peaks belonging to the compound with the N-O bond [3].

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## DEEP OXIDATION OF CO AND PROPANE ON Co-CATALYSTS PRODUCED BY LOW-TEMPERATURE COMBUSTION ON MODIFIED SUPPORTS

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Catalytic reactions of deep oxidation of carbon monoxide and hydrocarbons are of great industrial and environmental importance. They underlie the processes of neutralization of vehicle exhaust gases and gas emissions from various enterprises. These reactions are also used to remove combustible components from the gas environment of fire and explosion hazardous industries, in flameless heat sources, fuel cells, etc. [1].

The method for the synthesis catalysts consists in impregnating the support with a calibrated mixture of solutions of metal nitrates and urea, followed by drying, roasting in an inert atmosphere, and stabilization with a hydrogen peroxide solution. Silica gel (GOST 8984-75) was chosen as the support. The support was modified by burning a mixture of aluminum nitrate and urea. The original modified 10% Al<sub>2</sub>O<sub>3</sub> support was calcined in a muffle furnace at temperatures of 500, 600 and 700°C, and then an active phase containing 10% Co in terms of metal was applied to the modified supports by the same method [2].

The samples were studied by XRF, SEM with elemental analysis. The specific surface area was measured by BET on nitrogen. The best ones turned out to be samples based on an uncalcined carrier and calcined at a temperature of 600°C, where 100% CO conversion was achieved at a temperature of 300°C. A propane conversion of more than 70% was achieved at a temperature of 450°C. Thus, obtained catalysts are promising for further research.

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# OBTAINING OF SPHERICAL TITANIUM POWDER FOR USE IN ADDITIVE TECHNOLOGIES

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Additive technologies, which have been actively developing in recent years, make it possible to manufacture products of complex shape, significantly reduce the production time and consumption of the material used. Despite this, the main disadvantage of the technology is the high cost of the raw materials used – spherical powders with a low content of impurities. For domestic consumers, initial powders are a serious problem. For comparison, the approximate price of powder materials on the foreign market is next: pure titanium – 230 € per kg, Ti-6Al-4V – 200 € per kg. They cost Russian consumers at least twice, as a rule – three times more expensive [1].

A promising method for producing titanium powder, which has a low cost, is the method of SHS hydrogenation and dehydrogenation. The low cost of the starting material, the reduction of energy consumption due to the exothermic reaction and the high purity of the final product makes this technology very popular. However, the resulting powders have a fragmentary shape, which must be transformed into a spherical one.

The paper proposes a method for obtaining spherical titanium powder of a given fractional composition by SHS-hydrogenation and thermal dehydrogenation followed by plasma spheroidization. To obtain titanium powder, a titanium sponge of the TG-100 brand with a purity of 99.7 wt. % was used as a starting material. Hydrogenation was carried out in a SHS reactor at a hydrogen pressure of 20 atm. Next, the hydrogenated sponge was crushed in a drum-ball mill and a fraction of 40-70 microns was separated. To remove hydrogen, titanium hydride powder was isothermally annealed at a temperature of 750 °C in vacuum. After dehydrogenation, titanium powder of polygonal fragmentation form with a purity of 99.5 wt. % was obtained. To give titanium particles a spherical shape, a plasma spheroidization system of powder materials was used by IMET RAS. The resulting spherical titanium powder has a particle size of 40-70 microns, and the degree of their spheroidization reaches 95 %.

As a result of the conducted experimental studies, the possibility of obtaining spherical titanium powder by SHS-hydrogenation and thermal dehydrogenation with subsequent plasma spheroidization has been established. The obtained powders response the requirements necessary for application in additive technologies.

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# INFLUENCE OF ELEMENTAL POWDER RAW MATERIALS ON THE FORMATION OF THE POROUS SKELETON OF THE MAX PHASE $Ti_3SiC_2$ WHEN OBTAINED BY THE NON-VACUUM SHS METHOD

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The porous skeletons of the MAX-phase  $Ti_3SiC_2$  obtained by the SHS method can be used as filters, catalyst carriers, biocompatible bone implants, as well as a porous ceramic billet (base) for the production of ceramic-metal materials (cermets) by spontaneous infiltration of the metal melt [1-3]. Studies were conducted on the effect on the phase formation, micro- and macrostructure of a porous skeleton based on the MAX-phase  $Ti_3SiC_2$  obtained by the SHS method in air in backfill from river sand when using various common grades of titanium powders (TPP-7, PTS-1, PTM-1), carbon (C-2, GLS-1, T 900, P 701) and silicon powder (Kr0). The fractional composition (particle size) of the initial powders of titanium and carbon, as well as the form of powdered carbon (graphite, soot) significantly affect the macrostructure of porous SHS skeletons of the MAX-phase  $Ti_3SiC_2$  obtained by burning mixtures of initial powders in the open air, that is, using non-vacuum SHS technology. The density and porosity of the SHS samples obtained from the Ti-Si-C system is most influenced by the shape of the carbon powder. The SHS frame, obtained using a large titanium powder of the TPP-7 brand and graphite powder of the C-2 brand, has the highest of the presented density and compressive strength indicators – 2.41 g/ cm<sup>3</sup> and 104 MPa, respectively, and also with a high content of the MAX phase  $Ti_3SiC_2$  – 66% relative to carbide titan. When using titanium powders of large fraction (TPP-7) and titanium of small fraction (PTM-1) together with graphite powders (C-2) in the initial charge by self-propagating high-temperature synthesis (SHS), the highest indicators in the amount of MAX phase were obtained – 68% and 66%, respectively.

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## EFFECT OF AGEING TIME ON THE TENSILE PROPERTIES OF THE LOW-CARBON 9% CR MARTENSITIC STEEL

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The structure and tensile properties of the tempered martensite/ferrite lath structure of the low-carbon 9 wt.% Cr heat-resistant steel with high N and low B contents after ageing at 650°C for 3000 h were investigated. Previously, the steel investigated was heat treated consisting normalizing at 1200°C with following tempering at 750°C for 3 h. After heat treatment, tempered martensite/ferrite lath structure of the 9%Cr steel with a mean size of martensitic laths of 297±30 nm and dislocation density in lath interior of  $4.6 \times 10^{14} \text{ m}^{-2}$  was revealed. The boundaries of martensitic laths were stabilized by fine “Cu”-rich particles with a mean size of 55±10 nm and TaX carbonitrides with a mean size of 11±2 nm. The tensile tests were carried out at room temperature. The yield strength and ultimate tensile strength were 670±40 MPa and 750±40 MPa, respectively, after heat treatment. Ageing at 650°C for first 500 h insignificantly decreased the yield strength on 40 MPa that was related to annihilation of dislocations. An increase in ageing time up to 1000 h led to a decrease in the yield strength on 110 MPa that related to the depletion of W from the ferrite matrix. After 3000 h of ageing, the coarsening of laths and TaX particles took place that led to a decrease in yield strength by 6%, while the tempered martensite lath structure retained without subgrain formation. No significant softening during 3,000 h of ageing at 650°C was observed that indicated high thermal stability of tempered structure of the low-carbon 9%Cr steel at thermal exposure. The relationship between structure and tensile properties of samples aged with different times was evaluated; the effect of ageing time on the strengthening mechanisms was discussed.

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# EFFECT OF CARBON CONTENT ON THE STRENGTH OF HSLA STEELS AFTER TEMPFORMING

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Currently, special attention is paid to the development of structural steels and alloys adapted to the climatic conditions of the Far North, as well as for various constructions exploited at low temperatures. The first generation of HSLA steels showed a yield strength above 350 MPa [1]. The yield strength of modern HSLA steels has increased to 700 MPa as a result of dispersion strengthening provided by microalloying and grain refinement owing to thermomechanical treatment [2]. The strengthening by grain size also plays an important role in the strength of high-strength steels with ultrafine-grained ferrite.

The objects of the study were three steels of the following chemical compositions: 1) Fe-0.15C-1.32Mn-1.42Cr-0.45Mo-0.42Cu-0.17Ti; 2) Fe-0.26C-0.23Si-0.54Mn-0.42Cr-0.44Mo-0.06Ti; 3) Fe-0.36C-0.4Si-0.56Cr-0.57Mn-0.54Mo (all in wt %). Tempforming was chosen as a promising treatment of HSLA steels. The samples were tempered at temperatures of 550, 600, or 650°C for 1 h followed by multiple rolling at a tempering temperature to a total strain of 1.5.

Tempforming is an effective method to increase the strength without remarkable degradation of plasticity. The yield strength of steel with 0.15%C increases from 810 MPa to 1140 MPa, while total elongation decreases from 18.3 to 10.5% with a decrease in tempforming temperature from 650 to 550°C. The yield strength of steel with 0.26%C increases from 985 MPa to 1160 MPa with a decrease in tempforming temperature from 650 to 600°C, elongation decreases from 11.3 to 9.2%. The yield strength of steel with 0.36%C increases from 1180 MPa to 1510 MPa with a decrease in tempforming temperature from 650 to 550°C, elongation decreases from 13.3 to 7.2%.

Therefore, with an increase in the carbon content from 0.15 to 0.36%, the yield strength increases by about 25% along with corresponding decrease in plasticity.

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# STUDY OF THE STRUCTURE OF AN ALLOY WITH CALCIUM BASED ON THE AL-MG-CA-ZN-FE-SI-MN MULTICOMPONENT

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The paper presents a study aimed at finding improvements in the Al-Mg system casting alloys, which are of very limited use and also require the use of pure materials for production. Recent works on the study of the Al-Ca-X system have shown that it is possible to use in alloys a stock with a high content of impurities Fe, Si, Mn. The experimental composition contains 6% Mg, 2% Ca to increase the proportion of eutectic and reduce the liquidus temperature, up to 2% Zn to increase strength, as well as 1% Mn and 0.5% Fe and Si.

According to the calculation of the liquidus surface, the  $\text{Al}_6\text{Mg}_2\text{Ca}_2\text{Zn}_{0.5}\text{Fe}_{0.5}\text{Si}_1\text{Mn}$  alloy during crystallization falls into the region of formation of primary crystals of the  $\text{Al}_6\text{Mn}$  phase. To get into the region of the primary aluminum solid solution, according to calculations, the limiting concentration of Mn should not exceed 0.3-0.4%. At the same time, with an increase in the cooling rate, it is possible to predict the expansion of the crystallization region (Al), and hence the absence of primary crystals. It can be seen from the polythermal cross sections that the addition of manganese significantly increases the liquidus temperature (by 50 °C) in comparison with alloys without Mn, and the calculation does not contain the  $\text{Al}_{15}(\text{Fe},\text{Mn})_3\text{Si}_2$  compound, which is probably due to the incorporation of silicon into the  $\text{Mg}_2\text{Si}$  compound with magnesium. Also, in this system there is a peritectic reaction, according to which the  $\text{Al}_3\text{Fe}$  phase is dissolved and the  $\text{Al}_6(\text{Fe},\text{Mn})$  phase is formed. According to calculations, the total mass fraction of the second phases is about 30% in cast and 15% in hardened states. In the cast structure of the alloy, elongated eutectic colonies and (Al) stand out, as well as individual precipitates, needle-shaped. Quenching leads to slight spheroidization and fragmentation of inclusions, but does not radically change the general appearance of the structure. Since aluminum and magnesium have similar atomic weights, and the structural components have dimensions not exceeding 10–15  $\mu\text{m}$ , it is difficult to reliably determine the phase composition. Slow cooling with an oven provokes a significant increase in structural components. Three types of crystals stand out against the background of an aluminum solid solution: acicular, skeletal and compact. Acicular elongated crystals correspond to the  $\text{Mg}_2\text{Si}$  compound. The skeletal shape of the crystals corresponds to the  $(\text{Al},\text{Zn})_4\text{Ca}$  phase. And more compact ones are divided into  $(\text{Al},\text{Zn})_3\text{Mg}_2$  and  $\text{Al}_2(\text{Mg},\text{Ca})$ . Large regular-shaped crystals corresponding to the  $\text{Al}_6(\text{Fe},\text{Mn})$  compound are also distinguished. The hardness of the experimental alloy in the cast and quenched ( $T = 440\text{ }^\circ\text{C}$ ,  $\tau = 3$  hours) state remains practically unchanged: 104.3 and 106.6 HV, respectively.

*This work was supported by the Russian Science Foundation grant no. 21-79-00134.*

# MATHEMATICAL MODELLING OF MULTICOMPONENT NUCLEI GROWTH USING NONEQUILIBRIUM THERMODYNAMICS METHODS

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The paper is devoted to some aspects of the high-entropy materials formation. A solution with  $m$  components  $\mathbf{R}_1, \dots, \mathbf{R}_m$  is considered. Let  $m$  chemical reactions occur in the solution:

$$n_{i1}\mathbf{R}_1 + \dots + n_{im}\mathbf{R}_m = \mathbf{P}_i, \quad (i = 1, \dots, n)$$

where the products  $\mathbf{P}_1, \dots, \mathbf{P}_n$  are insoluble in the initial solution, but not limitedly soluble in each other. A system consisting of two phases is considered: the solution phase  $\Upsilon$  and the growing particle phase  $\Omega$  consisting of  $\mathbf{P}_1, \dots, \mathbf{P}_n$  components. The particle grows due to the chemical reactions on its surface.

The following equations were obtained:

- the local mass balances equations of the phases components,
- the global mass balances equations of the system components,
- the system components motion equations,
- the local balances of the internal energy equations,
- the global balance of the internal energy equations,
- the equations of the local balances entropy,
- the equations of the global balances entropy,
- the phenomenological equations.

As a result, a comprehensive system of the equations was obtained, which is a broad basis for creating various mathematical models of the nucleus growth processes of a new phase, depending on our knowledge of the physico-chemical characteristics of a particular system of interest.

The advantages of this approach – the construction of mathematical models of the process based on the preliminary description by the nonequilibrium thermodynamics methods – in comparison with the direct construction of a mathematical model are as follows: gives guidelines and the correct organization of building a mathematical models; allows us to evaluate the impact of each assumption and simplification on the adequacy of the created model to the real process.

This paper shows an example of such an approach.

# THE MATHEMATICAL MODEL FOR CRYSTALS GROWTH IN MULTICOMPONENT MELTS WITHOUT THE LOCAL EQUILIBRIUM ON THEIR SURFACE

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High-entropy alloys crystallization [1] is determined by the processes of the new phase nuclei growth from the initial multicomponent solution, taking into account the influence of interrelated the thermal and the diffusion processes. If the crystallization occurs at a high rate, the rates of the diffusion flows do not completely determine the movement of the phase boundary between the nucleus and the solution. Therefore, the principle of the local equilibrium at the surface of growing nuclei is not always fulfilled [2] and special approaches to describe the growth of the nuclei are required.

In this paper, on the basis of the previously studies [3], a mathematical model of the growth of a nucleus in multicomponent melt is constructed. The interrelated thermal and diffusion processes in the phases of the nucleus and the solution have described by nonequilibrium thermodynamics methods. The processes at the interface of the phases have been considered as chemical reactions of the nucleus components formation from the initial components of the solution. When the local equilibrium between the phases is not realized, it is incorrect to apply the traditional linear assumption about the relationship of the thermodynamic forces and the thermodynamic flows. Therefore, a new variational approach had been developed [3], which made it possible to obtain the equations to describe the growth rate of the nucleus, taking into account the possible local deviation of the system from equilibrium.

The possible applications of the theory for the crystallization without the local equilibrium are considered. The mathematical models for the most common cases such as the crystal growth from solid solutions and the crystal growth in eutectic melts are constructed. The mathematical models have been used to study the growth of  $\alpha$ -Fe (Si) nanocrystals during annealing of the amorphous  $\text{Fe}_{73,5}\text{Cu}_1\text{Nb}_3\text{Si}_{13,5}\text{B}_9$  alloy [3], as well as to study the eutectic supercooled melt composition  $\text{Fe}_{83}\text{B}_{17}$  [4].

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# CREEP BEHAVIOR OF ADVANCED 9-12%CR MARTENSITIC STEELS WITH INCREASED BORON AND DECREASED NITROGEN CONTENTS

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The creep resistance is the main critical requirement to the 9–12% Cr martensitic steels which are widely used in the fossil power plant industry as materials for boilers, pipes, turbines, rotors, and blades, etc. The minimum long-term creep rupture strength of steels on the base of 100,000 h should be 100 MPa or higher at 650 °C for power units with ultrasupercritical conditions. One of the most effective ways to enhance the creep resistance was suggested by researchers at the National Institute for Materials Science in Japan. The method consists of increasing the B content and decreasing the N content. This work presents an overview of the creep strength and related microstructural features of the 9% Cr and 10–12% Cr martensitic steels with high B and low N contents in comparison with similar steels with conventional B/N contents.

The approach to alloying by the increased B (80–150 ppm) and decreased N (30–100 ppm) contents is successfully applied to advanced 9% Cr, as well as 10–12% Cr martensitic steels. The predicted long-term creep rupture strength at 650 °C for 100,000 h attained 80–110 MPa for the 9% Cr steels, such as the MARBN, G115, and SAVE12AD steels, 110 MPa for the 10% Cr experimental steel, and 65 MPa for the 11–12% Cr steels, such as Super VM12 steel. These values are sufficiently higher than that for the previous steels with conventional B (~50 ppm) and N (~500–600 ppm) contents.

A positive effect of high B and low N contents on the creep strength is associated with their strong effect on the dispersion strengthening by  $M_{23}C_6$  carbides and MX carbonitrides. Optimal ratio of high B and low N contents provides the full utilization of soluble boron in the matrix and  $M_{23}(B,C)_6$ -type carbides during tempering. Under creep conditions, fine and highly coarsening-resistant  $M_{23}C_6$  carbides impede the recovery of the lath structure in the vicinity of prior austenite grain (PAG) boundaries and, hence, retard the local deformation in the PAG boundary regions. Stable carbides, in turn, can provide the slower coarsening of Laves phase particles. The MX phase also demonstrates high resistance to coarsening and to transformation to the coarse Z-phase (CrVN). Despite the low N content, precipitation of small MX particles during the transient stage of long-term creep effectively reduces the creep rate and increases the time to rupture, as was shown in some steels. The increased dispersion strengthening provides a stable lath structure over a long creep time and prevents the transformation of the lath structure into a subgrain structure.

Therefore, the modification of alloying by high boron and low nitrogen can be successfully used in order to increase the creep resistance of martensitic steels in the long-term region in combination with other strengthening factors.

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# **STUDY ON HYDROGEN PRODUCTION TECHNOLOGIES WITH ASSOCIATED CO/CO<sub>2</sub> UTILIZATION BASED ON THE ANALYSIS OF PATENT AND NON-PATENT INFORMATION**

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In 2022 the FIPS Project Office did a complex research project titled “Study on hydrogen production technologies and associated CO/CO<sub>2</sub> utilization based on the analysis of patent and non-patent information”.

Main objectives of the work included studying, systematizing and generalizing modern technologies for producing hydrogen with associated CO / CO<sub>2</sub> utilization based on an analysis of predominantly patent and, to a certain extent, non-patent information.

High relevance of this topic is achieved due to the crucially important need of creating effective expert and analytical support mechanisms for the implementation of governmental plans regarding hydrogen energy/economy (“Concept for the Development of Hydrogen Energy in the Russian Federation”).

The key objectives of the study included 1) expert development of a comprehensive knowledge model; 2) collection and organization of relevant patent documents for comprehensive analysis; and 3) multidimensional analysis of trends, geography, companies, universities and applications within the patent landscape.

As part of the research project, an extensive analysis of non-patent literature and industry-specific Internet resources was carried out, expert brainstorming sessions were held in the interests of developing a domain-specific knowledge model.

As part of the research project, a search strategy has been developed, including a separate search by types of raw materials, energy sources, processes for producing hydrogen, as well as methods of subsequent using, transporting and storing hydrogen.

As part of the research project, a comprehensive analysis of trends, geography, companies, universities and applications was carried out, as well as a separate analysis for five "colour types" of hydrogen: green, pink, orange, blue and turquoise.

The key results of the research project include the Patent Landscape “Hydrogen production with associated CO/CO<sub>2</sub> utilization” itself, as well as a provided brand-new feature methodology of the FIPS Project Office for working with super-large (>5 thousand patent families) patent collections in the patent landscapes development process.

Important outputs of the research project are also an extensive digital invoice and a final report on the project, describing the processes and assumptions in the performance of conducting such a complex study.

The results of the research project could be used in the interests of expert and analytical support for the implementation of the governmental hydrogen energy plans in the Russian Federation, and in a broader context, a wide range of tasks for the implementation of Russia's environmental agenda.

At the applied level, the results of research project can be used for information and analytical support by Russian companies, universities and research organizations.

# CREEP BEHAVIOR OF LOW-CARBON 9% CR STEEL STRENGTHENED BY TAX NANOPARTICLES

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9-12% Cr martensitic steels are prospective materials for new generation of fossil power plants with steam temperature of 620°C. High creep strength is accompanied with a formation of the tempered martensite lath structure with high density of free dislocations. The main structural changes during creep at elevated temperature are considered to be the increase in width of the martensitic laths and decrease in the dislocation density that is accompanied with the growth of  $M_{23}C_6$  carbides and Laves phase. The aim of present work is to report on creep behavior of the low-carbon 9% Cr steel in comparison with the 9% Cr steel with 0.1% C. Two steels with the different C content (0.02%C-9% Cr and 0.1%C-9% Cr) were heat treated as normalizing at a temperature of 1200 (for 0.02%C-9% Cr) and 1050°C (0.1%C-9% Cr) during 1 hour, cooling in air, with following tempering at temperature of 750°C during 3 hours, air cooling. Flat specimens with a gauge length of 25 mm and a cross section of 7 mm × 3 mm were crept until rupture at 650 °C under the applied stresses of 120, 140 and 160 MPa. The structural studies were carried out by transmission electron microscopies.

Heat treatment of the steels led to the formation of the tempered martensite lath structure in both steels with different prior austenite grain size: about 200 μm for 0.02%C-9% Cr steel and 20 μm for 0.1%C-9% Cr steel. The size of the martensite laths was about 300 nm in both steels, and the dislocation density was very high of  $(2-4) \times 10^{14} \text{ m}^{-2}$  in both steels. The phase composition of 0.02%C-9% Cr steel includes ferrite and such secondary phase particles as TaX, Laves phase and “Cu”-rich particles, whereas 0.1%C-9% Cr steel contents ferrite,  $M_{23}C_6$  carbides, Nb(C,N) and V(C,N) carbonitrides. So, the creep strength of these steels is provided by different type of secondary phase particles. The 0.02%C-9% Cr steel at the applied stress of 160 MPa demonstrates a significant increase in the rupture time by a factor of 3 compared to the 0.1%C-9% Cr steel due to increasing the duration of the primary creep stage by a factor of 3 and decreasing the minimum creep rate by two orders of magnitude. Under 140 MPa, the rupture time is similar for both steel and comprises about 1800 h. Under 120 MPa, the creep specimen of the 0.02%C-9% Cr steel has not been broken yet, but behavior of creep curve is similar with that of the 0.1%C-9% Cr steel. So, the high level of threshold stresses from TaX carbonitrides gives the increment in creep strength under the high applied stresses, only, whereas  $M_{23}C_6$  carbides provide the strengthening effect until their size retains below 100 nm at volume fraction of 2%.

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## STUDY OF MECHANICAL AND TRIBOLOGICAL PROPERTIES OF FE-CO-NI-CR-CUX HIGH-ENTROPY ALLOYS

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High Entropy Alloys (HEAs) are a class of materials that are alloys of 5 or more components, the volume fractions of which are in the range of 5-35%. HEAs based on the Fe – Co – Ni – Cr system are widely used in structural, magnetically soft and magnetically hard materials, catalysts, materials with high corrosion resistance, etc.

One of the alloying additions, similar in physicochemical properties and lattice type to Fe – Co – Ni – Cr alloys, is copper. The most common methods for obtaining HEA systems Fe – Co – Ni – Cr – Cu are arc melting, vacuum induction melting and other foundry methods. As a rule, when using these technologies, copper is separated as a separate phase, which leads to a decrease in the strength of the material. In this work, HEAs based on Fe – Co – Ni – Cr – Cu are obtained by powder metallurgy methods: mechanical alloying (ML) of powder mixtures with their subsequent hot pressing. Thus it is possible to obtain single-phase metastable solid solutions despite the limited solubility of the components (in particular copper in iron, cobalt and chromium).

Fe, Co (carbonyl), Ni, Cr (reduced), and Cu (electrolytic) metal powders were used as starting materials. The equiatomic Fe-Co-Ni-Cr alloy was chosen as the base composition. It was additionally alloyed with copper in the amount of 5-20 at. %. Mixing of the components was carried out in mixers of two types: a ball mill (SHVM) and a planetary centrifugal mill (PCM).

It has been established that HEAs based on Fe – Co – Ni – Cr -(Cu), made from ML powder mixtures, had a high level of flexural and tensile strength (1800 – 2500 MPa), which is 2 times higher than that of samples made with the help of SHVM. The high mechanical properties of the alloy in this case are due to the uniformity and fineness of the microstructure. HEAs with a copper content of up to 10% had the highest mechanical properties; in this case, copper was completely included in the composition of the solid solution with the fcc structure. With an increase in the copper concentration to 20%, the strength decreases, which is associated with the formation of an undesirable two-phase structure.

To assess the wear resistance, tribological tests were carried out according to the "rod-disk" scheme. According to the test results, it was found that the addition of 5% copper leads to a decrease in wear during friction in a pair with a ball of sintered Si<sub>3</sub>N<sub>4</sub> by more than 10 times – from 8.6 to 0.6\*10<sup>-5</sup> mm<sup>3</sup>/H\*m. The reason for this is the high hardness of the HEA, which is achieved due to the solid solution hardening of the Fe – Co – Ni – Cr matrix with copper.

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## ASPECTS OF HIGH-SPEED PHOTOGRAPHY OF SHOCK-WAVE LOADING OF STEEL TUBE

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Currently, the complex technology including SHS, shock-wave loading and extrusion is one of the promising technologies for the production of metal-intermetallic composites.

This study presents the results of high-speed photography of experiments on compression of a steel tube by an oblique shock wave. For high-speed photography, an electron-optical camera Nanogate 22/16 with a shooting frequency of 1 million frames /s was used. The experiments were carried out according to two schemes using ammonite as an explosive. According to the first scheme, a hollow steel tube with a diameter of 14 mm and a length of 70 mm was compressed; in the second scheme, a tablet from a reactive Ni-Al powder mixture with a height of 10 mm was placed inside the lower part of the tube. The schemes provided for strong joint of the tube and the upper plate on which it was installed with the lower plate. The thickness of the lower plate was equal to 2 and 12 mm in the schemes No.1 and 2, respectively. To align the detonation front, a conical steel plug was installed in the top of the tube.

Processing and analysis of images obtained as a result of high-speed photography allowed us to determine the approximate velocity of the detonation front in both experiments (~ 3.5 km/s). In addition, according to the images and analysis of the surface of the lower plate, during the collapse of the inner void space of the tube, a dense flow of heated gases and fine particles of steel is formed in it. Besides, the rate of departure of the clot formed by this flow from the lower part of the tube in experiment No. 1 is higher by ~ 1 km/ s, which is due to the presence of a Ni-Al tablet in experiment No. 2. As a result of the collision of the upper and lower plates, a concentric wave shape is formed on their surface. In the center of the lower plate (experiment No. 2) a 2.5-mm deep crater was formed. Ni-Al intermetallic compounds within the crater were found. High pressure and temperature led to the initiation of the synthesis process in the Ni-Al tablet.

The outer diameter of the tubes decreased by about 30% after high-speed compression. Thus, a loss of stability of the walls led to the tube collapsed almost to the rod state.

Based on the results obtained, it can be concluded that it is promising to carry out further experimental work to study the characteristics of a high-speed flow of particles and gases formed as a result of shock-wave loading of the tube outer surface. The results obtained will be used to develop the technology of shock-wave extrusion of intermetallics and cermets in a metal shell.

# NANOSTRUCTURED COATINGS BASED ON AMORPHOUS CARBON AND CARBON-DOPED WITH GOLD, SILVER AND NITROGEN OBTAINED BY THE PULSED VACUUM-ARC METHOD

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According to the data of [1] annealing in a vacuum at 600 °C allows initiating the formation of nanocrystallites with an ordered structure in the amorphous matrix of ta-C coatings. It was found that the size of nanocrystallites depends on the initial state of the carbon matrix and, first of all, on the content of the  $sp^3$  phase and the level of internal stresses in it, which is determined by the conditions of coating formation. Another method of forming nanostructures based on the amorphous carbon matrix is the addition of elements that do not form stable chemical bonds with carbon atoms (Au, Ag, Pt, Cu) to the coating [2]. The addition of N to the carbon coating formed by the pulsed vacuum-arc method and its subsequent annealing to increase the electrical conductivity leads to the formation of nanoclusters with sizes of the order of 3-10 nm [3].

Nanostructured coatings based on amorphous carbon and carbon-doped with Au, Ag and N were obtained by the pulsed vacuum-arc method. Carbon coatings have been annealed in a vacuum as well as treated with Ar ions. The alloying of carbon coatings with elements that do not form chemical bonds with the carbon matrix (Ag, Au) leads to the formation of Au or Ag nanocrystallites with sizes of 2-20 nm in the matrix of amorphous carbon, whose density depends on the concentration of the doping element. Annealing of C:Ag coatings leads to the formation of islands on the surface with the size of the order of micrometers. This is due to the Ag diffusion and coalescence of small islands into larger ones. The HRTEM method discovered the effect of twinning in carbon nanocrystallites after vacuum annealing as well as Ag and Au in the initial state in the amorphous carbon matrix. Analysis of Raman spectra of pure C coating and C:Ag showed that the addition of Ag leads to a decrease in  $sp^3$ -phase in the carbon matrix. The addition of N in the carbon coating leads to an increase in the  $sp^2$  – phase fraction, and additional annealing leads to a significant increase in the D – peak intensity and formation of clusters of the order of 5-15 nm, which are not localized but fill the entire space. Analysis of the coating a-C:Au irradiation with Ar ions shows that the number of nanoinclusions decreased, simultaneously decreased surface roughness degree, besides, decreased electrical conductivity of the coating as a result of decreased Au content.

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# EFFECT OF HIGH-PRESSURE TORSION TEMPERATURE ON THE MICROSTRUCTURE, PHASE COMPOSITION, AND MECHANICAL PROPERTIES OF A HIGH-ENTROPY FeMnCrNiCo ALLOY

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In recent years, high-entropy alloys (HEAs) have gained wide popularity due to their high strength and ability to exhibit a number of fundamental properties that distinguish them favorably from conventional materials with one or two basic elements. High-entropy alloys are alloys that consist of five or more elements, while the atomic concentration of each of them varies from 5 to 35%. One of the first investigated high-entropy alloys is Kantor's alloy 20Fe-20Mn-20Cr-20Ni-20Co (at. %), which is quite well studied [1]. The alloy has high ductility, toughness and is able to maintain a face-centered cubic structure in a wide temperature range, but has low strength. One way to increase the strength of FCC WES is to refine their grain structure. High strength is observed in the Kantor alloy after high-pressure torsion (HPT), which ensures the formation of a nanoscale structure. Further hardening in this case can be achieved due to the decomposition of the solid solution during additional annealing at a fairly low temperature up to 450°C [2]. On the other hand, the processes of dynamic recovery and precipitation of a nano-sized multiphase structure under severe plastic deformation have not been practically studied.

In this work, we studied the effect of the temperature of severe plastic deformation by torsion on the microstructure, phase composition, and mechanical properties of a high-entropy FeMnCrNiCo alloy. The ingots were smelted by vacuum-arc remelting. Grain refinement was carried out by HPT at room temperature and 300°C. Using X-ray diffraction and X-ray phase analysis, it is shown that the alloys after all the studied treatments have an austenitic structure. TEM methods have been used to study the features of changes in the microstructure. Microhardness measurements showed that the alloys after HPT significantly increase their hardness to 535 HV compared to the initial state of 347 HV. This is accompanied by an increase in the yield strength, tensile strength, and strain hardening coefficient of the high-entropy alloy. The mechanism of hardening is discussed.

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**THERMAL STRENGTHENING OF HIGH-ENTROPY RARE-EARTH ALLOYS WITH YTTRIUM AND SCANDIUM**

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The purpose of this work is to identify the role of the atomic size factor on structure formation and functional properties of multicomponent rare earth alloys (REA) high-entropy systems (HES) GdTbDyHoSc (REA-Sc) and GdTbDyHoY (REA-Y). It is assumed that the combination of magnetic REA with non-magnetic elements (yttrium or scandium) with different atomic radii will allow the formation of a crystal structure of materials with many defects, which makes it possible to properly trace the role of the atomic size factor on the structure formation of REA HES and their functional characteristics. It is worth noting that REA materials containing scandium have been studied extremely poorly due to the high cost of this element. However, it is known that doping with scandium often improves the quality of materials. Four magnetic lanthanides Gd, Tb, Dy, and Ho were used in this work as the basic elements of the HES, the main characteristics of which (lattice structure, atomic radius, lattice parameters, electronegativity) are so close to each other that the Hume-Rothery rules was clearly satisfied, therefore, in many works, such alloys are considered thermally stable. The following components were chosen as the fifth component: yttrium with structural characteristics close to the base metals and scandium with significantly lower radius. The addition of Sc, due to the significant difference in the size of the atoms, leads to an increase in the distortion of the crystal lattice. This should reduce the stability of the HES when going from four-components to a five-components system, and on the other hand, increase the stability, since the entropy factor increases. All metals of  $\geq 99.9\%$  purity was melted by Uporov et al [1] using the arc furnace under Ar (99.99%) to protect the metals from high temperature oxidation. To reach a homogeneous distribution of the initial components, the samples were repeatedly remelted (6 melts) [1]. The relationship between the compositions, structure, and hardness of five-components HES, including elements with different atomic sizes was established. It is shown that heat treatment (HT) of REA-Sc and REA-Y alloys significantly increased the hardness of the studied HES. For example, the REA-Sc alloy after HT showed a denser structure with better formed grains, especially in the near-surface oxidized layers, this was confirmed by an increase in the REA-Sc hardness from 220 HV to 550 HV (almost by 3 times). It is found that annealing significantly affects the microstructure and mechanical properties of the alloys but does not reduce the HES stability.

*The work is supported by the Russian Science Foundation grant 21-43-00015.*

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# HIGH VOLTAGE CONSOLIDATION OF POWDER MATERIALS

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The main features of the method of high-voltage consolidation of powder materials and the resulting advantages and limitations of this method are considered. The electrothermal processes at the contacts between powder particles and on the macroscale of the entire consolidated sample are analyzed. The results of calculations of the dynamics of the closure (collapse) of interparticle pores in the process of consolidation of the powder material are presented. The experimental results of high-voltage consolidation of powders are discussed on the example of hard alloys and heavy alloys based on tungsten [1].

The results of a study of the macro- and microstructure of consolidated materials and mechanical tests of the obtained samples are presented. Tests at room temperature for compression in the studied range showed that all consolidated samples withstand compressive stress without failure. High-voltage sintering contributes to the maintenance of the initial fine-grained structure, more uniform distribution of the iron-nickel binder, and the almost complete absence of porosity in the consolidated materials.

A criterion has been established that determines the range of optimal technological parameters for creating high-density materials. Possible directions for further research into the process of high-voltage consolidation of powder materials are proposed.

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# STRUCTURE AND PRACTICAL OF WELD CLADDING OF THE NON-EQUIATOMIC Al-Co-Cr-Fe-Ni HIGH-ENTROPY ALLOY SYSTEM ONTO ALUMINUM ALLOY A5083

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Aluminum and its alloys have many applications because of their low density, high specific strength, and good atmospheric corrosion resistance. However, they have poor wear resistance, which could lead to shorter life of components due to damage. Coatings can improve tribological characteristics of the aluminum parts and prolong their performance duration. Nowadays, there are few methods that can be used for coatings' deposition on aluminum alloys: thermal spray processes (such as cold spraying, plasma spraying and high-velocity oxyfuel spraying), weld cladding (cold metal transfer and laser cladding) and combination of methods (cold spray + laser melting).

High-entropy alloys (HEAs) have many promising properties as coatings on aluminum alloys. HEAs are alloys in general consist of five or more principal elements with the content of each component varying from 5 to 35 at. %. Some HEA-systems as Al-Co-Cr-Fe-Ni reveal high hardness, good wear and corrosion resistance that makes them attractive as defense coatings.

This study showed the possibility to fabricate wear resistant coating from Al-Co-Cr-Fe-Ni high-entropy alloy onto A5083 aluminum substrate via weld cladding. The following results could be drawn:

1. The obtained coating has poor cohesion with the substrate and visible cracks on the surface and inside layers which formed during solidification.
2. The coating has the microhardness 6 times higher than that in the substrate and the wear rates comparable to the other coatings from high-entropy alloys obtained in the previous studies.
3. The chemical elements' distribution inside the layer is quite homogeneous and the microstructure represents matrix and the second phase with the size of  $4 \pm 2 \mu\text{m}$ . The microstructure of the substrate near to the boarder with the clad coating has star-shaped and needle-shaped grains with the sizes of  $4.4 \pm 0.1 \mu\text{m}$  and  $3.2 \pm 0.2 \mu\text{m}$ , consequently.

Although the obtained high-entropy alloy coating has poor cohesion with the aluminum alloy substrate, the method has showed its applicability. Therefore, the future investigations should be focused on the searching those compositions of the high-entropy coatings that will form FCC crystal structure and better dilute with the substrate.

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# MICROSTRUCTURE CHARACTERIZATION AND DYNAMIC BEHAVIOR OF NBZRTITA REFRACTORY HIGH ENTROPY ALLOY

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Refractory high entropy alloys (RHEAs) are newly developed candidate materials for high temperature applications. However, few researches focus on the dynamic behavior of RHEAs, which would restrict its application as structural material. In the present work, microstructure and dynamic behavior of equiatomic NbZrTiTa RHEA are investigated. The phase structure of NbZrTiTa RHEA is examined by X-ray diffraction. Microstructure and elements distribution of the alloy are characterized by electron back-scattered diffraction, scanning electron microscope. Microstructure characterization results show that this alloy exhibits a single-phase body-centered cubic structure. Equiaxed grain with homogenous orientation was observed. In addition, the mechanical behavior of the NbZrTiTa RHEA is systematically investigated through material testing machine and split Hopkinson pressure bar system. This alloy exhibits high strength 1100MPa and good ductility 40% under strain rate  $0.001\text{s}^{-1}$ . Significant strain rate effect is observed from  $0.001\text{s}^{-1}$  to  $4600\text{s}^{-1}$ . Finally, the relationship between microstructure and mechanical properties is revealed. This work would support for the application of RHEAs in the environment required to suffer high strain rate loading.

**Keywords:** *Refractory high entropy alloys; Microstructure; Dynamic behavior; Strain rate effect.*



# THE EFFECT OF NITROGEN ALLOYING ON HYDROGEN EMBRITTLEMENT OF HIGH-ENTROPY CANTOR ALLOY

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The influence of nitrogen alloying on characteristics of hydrogen embrittlement in high-entropy FCC alloys was studied. The following alloys were chosen for investigation: 20.0Fe-20.0Cr-20.0Mn-20.0Ni-20.0Co (HEA), 20.0Fe-20.0Cr-20.0Mn-20.0Ni-19.2Co-0.8N, (0.8N-HEA) and 20.0Fe-20.0Cr-20.0Mn-20.0Ni-18.6Co-1.4N (1.4N-HEA) (at.%). The cast alloys were thermomechanically treated to form a single-phase solid solution. Electrochemical hydrogen-charging was carried out in the 3% NaCl water-solution containing 3 g l<sup>-1</sup> of NH<sub>4</sub>SCN as recombination poison at the current density of 10 mA cm<sup>-2</sup> for 50 h. Uniaxial static tensile tests were performed on LFM 125 electromechanical testing machine at the initial strain rate 5 × 10<sup>-4</sup> s<sup>-1</sup>.

It was shown by X-ray diffraction analysis and electron microscopy that all specimens have a single-phase coarse-grained austenite structure. Due to the nitrogen alloying, the lattice parameter of austenite in specimens increases from 3.598 Å in nitrogen-free alloy to 3.602 Å and 3.607 Å in 0.8N-HEA and 1.4N-HEA respectively.

All hydrogen-free specimens had a good elongation:  $\delta_{\text{HEA}} = 64\%$ ,  $\delta_{0.8\text{N}} = 66\%$  and  $\delta_{1.4\text{N}} = 71\%$ , but hydrogen-charging reduced it. The hydrogen embrittlement index  $I_{\text{H}}$ , which describes the hydrogen-induced loss elongation, equals to 14 % in nitrogen-doped specimens and 25 % in HEA specimens. This can be due to the different hydrogen diffusivity and distribution in the alloys both during the hydrogen-charging process and subsequent tensile deformation. After hydrogen-charging, the yield strength  $\sigma_{0.2}$  increases in all specimens by  $\Delta\sigma_{0.2}^{\text{HEA}} = 5$  MPa,  $\Delta\sigma_{0.2}^{0.8\text{N}} = 5$  MPa and  $\Delta\sigma_{0.2}^{1.4\text{N}} = 21$  MPa, which is associated with a solid-solution hardening of austenite by hydrogen atoms.

Scanning electron microscopy revealed a brittle hydrogen-assisted surface layers in hydrogen-charged specimens of the alloys. The thickness of the brittle layer in HEA specimens is  $W_{\text{H}}^{\text{HEA}} = 70 \pm 21$  μm and it decreases to  $W_{\text{H}}^{0.8\text{N}} = 38 \pm 12$  μm and  $W_{\text{H}}^{1.4\text{N}} = 45 \pm 14$  μm for 0.8N-HEA and 1.4N-HEA respectively. Transgranular and intergranular fracture elements are observed on the fracture surfaces of the hydrogen-charged specimens. Moreover, with an increase in nitrogen concentration in alloy composition, the contribution from transgranular brittle elements also increases.

Alloying with nitrogen leads to a decrease in the thickness of the brittle hydrogen-assisted surface layer and an increase in the hydrogen embrittlement resistance of high-entropy alloys specimens.

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Self-propagating high-temperature synthesis (SHS) is an efficient method for producing composite powders. For more than 35 years, the O.V. Roman Powder Metallurgy Institute (PMI) has been intensively researching and developing technological processes to obtain composite powders and porous materials from titanium powders by the SHS method.

The thesis presents some research results, created thermodynamic and physical models, which made it possible to develop technologies for obtaining a wide range of composite SHS powders of the "metal binder / refractory compound" type, including a number of materials with new performance properties. Metals, alloys (based on iron, nickel, aluminum) and intermetallic compounds ( $Ni_xAl_y$ ,  $Fe_xAl_y$ ,  $Ti_xNi_y$ ,  $Ti_xAl_y$ ) are usually used as a matrix. As refractory compounds, as a rule, titanium, chromium, silicon carbides and their combinations, as well as aluminum, titanium and chromium oxides are used.

The synthesized powders characteristic features are: the presence of a metallurgical bond between the components of the composite; fine-grained structure with a fine volumetric distribution of the; constancy of the phase composition, regardless of the particle size of the powder; low cost (due to the low energy intensity of the process compared to other production methods). In some cases, preliminary mechanical activation of the reaction mixture in an attritor is used, which makes it possible to carry out mutual grinding of the initial powders to the required size and to form composite particles with a uniform distribution of the initial reagents over the volume. Synthesized powders are divided into the following groups.

Metal-ceramic powders of the "carbide-metal" type.

The most popular are SHS compositions based on titanium carbide. The grain size of the carbide phase is, as a rule, from 0.5 to 2...5  $\mu m$ . The microhardness of the material depends on the type and relative content of the metal matrix (binder) and averages about 950 HV<sub>0.5</sub> with a range of values from 300 to 2000 HV<sub>0.5</sub>

Composite powders "intermetallide/oxide".

They can be obtained on the nickel, iron and titanium aluminides basis, which provide a significant reduction in weight due to the low density of aluminides compared to traditionally used superalloys while maintaining high mechanical and corrosion properties of the compositions. Such compositions are obtained in two ways: by the synthesis of intermetallic compounds from elements in the presence of dispersed oxide particles and by the formation of compositions using aluminothermic reactions. NiAl/15%  $Al_2O_3$  composite powder is obtained by mechanically activated synthesis of nickel monoaluminide from elements in the presence of dispersed particles of aluminum oxide. The micro hardness of the composite powder is 350-650 HV<sub>50</sub>. Varying the content of the oxide phase makes it possible to effectively control the coefficient of linear thermal expansion (CLTE) of the material.

The developed composite powders are successfully used for applying protective wear- and corrosion-resistant coatings by plasma, detonation, and high-speed flame spraying. Compared to the mechanical mixtures and conglomerated powders traditionally used for thermal spraying, the synthesized powders ensure the preservation of the phase composition of the composition during the spraying process, uniform distribution of the solid phase in the coating volume, an increase in the utilization rate of the sprayed material (by 10-30%), as well as higher wear resistance. coatings. The most promising are  $Cr_3C_2/Me$ ,  $TiC/Me$ , and  $Cr_3C_2/TiC/Me$  composites, which are used to produce high-quality wear-resistant coatings that are not inferior to the best wear-resistant coatings based on tungsten carbide composites. At the same time, the specific cost per m<sup>2</sup> of the coating is

significantly lower due to the lower density of the material, the lower cost of the feedstock, and the increased utilization of the material during spraying.

In the field of obtaining porous materials from titanium powders, two processes have been developed and mastered: SHS in a nitrogen atmosphere of all-ceramic elements from titanium oxynitride and SHS in air with the formation of a composite structure "titanium matrix – oxynitride coating of the surface of metal particles". The first method produces disk porous inserts of vacuum faceplates to hold silicon wafers during their grinding and polishing in the production of microelectronic devices. The second is porous tubular chokes of emergency fire extinguishing systems resistant to gas-dynamic shock and high temperatures. The original development of the scientists of the Institute was the self-extinguishing SHS process initiated by an electric discharge, which forms a porous composite with a ceramic surface and a metal frame. At the same time, due to the introduction of aluminum additives into the initial charge, it's possible to form intermetallic compounds and MAX phases in the coating, which increase the resistance of the composite to high temperatures and increase the specific surface area, which is promising for use in catalyst carriers.

The report also presents other innovations in this area.

**POWDER ROUTINE AND FIRST RESULT OF THE STRUCTURE  
AND PHYSICO-MECHANICAL PROPERTIES HOT PRESSURE FABRICATED  
Fe-Cr-Ni-Mo-W SPECIMENS**

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The presented data are the results of a new work at IMET RAS in the field of obtaining and certifying the structure and properties of high-entropy powders alloys.

The purpose of this work was to study the process of obtaining powders of high-entropy alloys of the Fe-Cr-Ni-Mo-W system, analysis of the phase composition, distribution of chemical elements for subsequent use in consolidation processes and study of the effect of powder charge preparation technology on the properties and structure of consolidated samples of high-entropy powder alloys of the Fe-Cr-Ni-Mo-W system.

In accordance with the experimental plan, the pressing charge was obtained by both mechanical mixing and mechanical grinding in a planetary mill.

To obtain mixtures of 35, 30 wt.% Fe – 30 wt.% Cr – 20 wt.% Ni – 10 wt. % Mo – 10, 5 wt.% W used a mixer of the "drunk barrel" brand Turbula. The mixing time was 320 minutes. As well as the Retsch PM-400 planetary mill with 500 ml steel cups and a 1:5 ratio of powder weight to grinding balls with a diameter of 5.5 mm made of bearing steel was used for mechanical alloying. The grinding time varied from 1, 2.5, 5 and 10 hours.

The prepared powder mixture was pressed at room temperature on a hydraulic press in a cylindrical mold with a diameter of 25 mm at a pressure of 100 MPa to obtain consolidated blanks with a relative density of 50-60%. Then the resulting blanks were consolidated on a hot press at a pressure of 30 MPa, a temperature of 1200 ° C, for 30 minutes. The HP20-3560-20 (Thermal Technology Inc.) unit was used for hot pressing.

For the specimens after hot pressing, the density change was evaluated depending on the method of charge preparation. The specimens after mechanical mixing had the maximum density during hot pressing.

For the specimens after hot pressing, the hardness was evaluated by the Rockwell method on the C scale and microhardness by the Vickers method. After mechanical alloying for 2.5 hours, the specimens had a maximum hardness of 59±1 and 56±1 HRC, respectively, for compositions with 35 and 30 wt.% Fe.

Cylindrical specimens with a diameter of 4 mm and a height of 10 mm were prepared for the study of compressive strength by electric spark wire cutting. The strength analysis was performed on the Instron 3382. Loading speed was 1 mm/min. For specimens of the composition of 30 wt.% Fe compressive strength increases from 1350 ± 50 to 1500 ± 50 MPa, respectively, for specimens obtained from the charge prepared by mixing the initial powders and from the charge after grinding in a planetary mill for 5 hours.

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## STRUCTURE AND SHAPE MEMORY EFFECTS IN POROUS TINI ALLOYS

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Monolithic and porous TiNi alloys in the pre-martensitic state and in the process of its realization are distinguished by a number of unique properties. A striking and practically important property is the shape memory effect. Control of the SME parameters (hysteresis loop width, the value of the total cumulative strain, the temperatures of the beginning and the end of transformation) is possible by changing the concentration of titanium, nickel and additional alloying. The release of secondary phases of various shapes and sizes in the process of alloying leads to a change in the chemical composition of the TiNi(B2) matrix. The isolated particles can control the nucleation and growth of martensitic crystals, strengthen the austenitic B2 matrix, act as stoppers in the movement of the interface, as well as being the preferred sites for the nucleation of martensite crystals. For monolithic TiNi alloys, there is a sufficient number of works in which it is shown that Cu is one of the common and effective additives, the introduction of which leads to a change in the sequence of martensitic transformations and SME parameters. Similar works showing the possibility by introduction of Cu into the composition of porous alloy TiNi directionally and systematically control the characteristics of SME have not been found. This fact makes the present study promising and practically significant. Porous alloys TiNi(Cu) with 1, 3, 6, 10 at. % Cu were obtained by the SHS method. The structure was analyzed on a scanning electron microscope. The characteristics of the SME were determined by the curves of accumulation and strain reversal under the multiple shape memory effect. The structure of the alloys is represented as: matrix phase TiNi(Cu), individual  $Ti_2Ni$  particles, dendrites surrounded by interdendritic interlayers  $Ti_2Ni(Cu)$ . When doped with 1-3 at. % Cu, the dendritic regions are separate and shallow, filling most of the matrix volume. With an increase in Cu up to 10 at. %, large dendrites are formed, located in separate parts of the matrix, and an increase in the volume of the B2 phase is observed. Analysis of the MSME curves showed that during alloying up to 10 at. % Cu the value of the total accumulated strain, consisting of elastic, martensitic and plastic strain components, increases. The plastic component of strain, as well as the elastic strain, does not change in the process of alloying during MSME. The martensitic strain is determinative for all alloys. In alloys with 6 and 10 at. % Cu with a small number of large dendrites, the increased volume of the B2 phase results in the transformation taking place in a larger volume of material as compared to alloys with 1 and 3 at. % Cu. MT in alloys with 6 and 10 at. % Cu are also realized through the  $B2 \leftrightarrow R \leftrightarrow B19'$  sequence, as in monolithic alloys. In the beginning, the R phase is formed from B2 during cooling under load. At further cooling under load the R phase transforms into B19' martensite with the plate orientation favorable with respect to the applied load. As the alloying additive increases up to 10 at. % Cu, the hysteresis loop width increases.

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# ESTIMATION OF ENTROPY OF MULTICOMPONENT 'HIGH ENTROPY' CERAMICS

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Multicomponent carbides, nitrides, borides and diborides, or the so-called 'high entropy ceramics' (HEC), are extensively studied as promising functional materials and coatings for a variety of applications including ultrahigh temperatures [1]. This term is adopted from high entropy alloys (HEAs), which are fcc or bcc substitutional solid solutions containing at least 5 metallic elements in equal molar fractions [2].

The entropy in HEC, and particularly, the role of excess entropy, which is supposed to contribute to thermal stability of HEC, is a subject of discussion in literature [3]. Typically, the configurational entropy (the ideal entropy of mixing) of HEC, same as of HEAs, is described by simple formula  $S_{id} = -R\sum x_i \ln x_i$  [3], where  $R$  is the universal gas constant,  $x_i$  is the atomic fraction of  $i$ -th component. This formula follows from the Boltzmann's equation originally derived for gases. But, when applied to crystalline solids, it is valid only for the situation when all the atomic positions are equivalent, which is true only for disordered substitutional solutions such as HEAs.

Since nitrides, carbides, borides and diborides of most metals are interstitial phases, they are typically considered as composed of two sublattices where metal atoms (in the case of several metal species) are distributed chaotically over the 1st sublattice while the non-metal atoms (C,N,B) and the so-called structural vacancies ( $v$ ) occupy the 2nd sublattice. In this situation, their thermodynamic functions, including configurational entropy, should be described using the compound energy model (the extension of Hillert-Staffanson model), which include the site fractions of  $i$ -th metal on the 1st sublattice ( $y'_i$ ) and the site fraction of non-metal atoms ( $Nm$ ) and structural vacancies on the 2nd sublattice ( $y''_{Nm}$  and  $y''_v$ ). To evaluate the role of excess entropy, the so-called mismatch entropy [4] was considered, which is connected with the atomic size difference of metal species on the 1st sublattice, and is known to be the only type of excess entropy applicable to condensed matter. Calculations have shown that even for 6 metals, the configurational entropy of carbides/nitrides and diborides is actually low, and increases to values typical of medium-entropy alloys ( $1R < S_{id} < 1.5R$ ) only at strong deviation of the content of a non-metal from the stoichiometric composition.

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## FEATURES OF THE MICROSTRUCTURE OF POROUS-PERMEABLE TINI – BASED ALLOY PRODUCED BY SHS AND SINTERING METHODS

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Obtaining new data from fundamental studies of the structure and properties of porous TiNi-based alloy contributes to its wide use in various fields of medicine. The solution to the problem of integrating porous-permeable TiNi-based alloy into the tissues of the body lies, first of all, in the choice of the method of obtaining, since its main characteristics are responsible for the formation of the porous space of the material. Taking into account the interaction of cells and tissues of the body with the pore space of the material during its implantation into the body, its microstructural characteristics, namely the morphology of the surface of the walls of pores in the micron and submicron range of sizes (micropores, terraces), which lead to a radical improvement in the biocompatibility of the material, become important. The present study is devoted to the study of the formation of the microstructure of the porous space of TiNi-based alloy obtained by SHS and sintering. Since the SHS and sintering methods are characterized by different mechanisms of structure formation, the formed pore space has distinctive features. It is shown that the developed rough structure of the pore walls and interpore space, containing a large number of micropores, is a necessary condition for the active integration of the implant with body tissues.

Porous-permeable TiNi-based alloy with a developed rough terrace-like morphology of the surface of the pore walls, which is characterized by the presence of a stepped relief of various curvature, has been obtained by sintering TiNi-based powder. The appearance of this relief is dictated by the processes of bulk diffusion of atoms, surface diffusion of adatoms, and their interaction with substrate defects (kinks, steps, grain boundaries, dislocations, twins, secondary inclusions) during melt crystallization. The presence of a complex microrelief of the surface of the pore walls, along with secondary phases on the surface, allows us to conclude that the rough surface of the pore walls of the material is developed.

The SHS method was used to obtain a porous material with a high porosity index and a characteristic pore wall morphology. The phase-chemical composition of the material is extremely heterogeneous. A surface layer of a complex oxycarbonitride composition was found. The developed rough surface of the pore walls has in its structure, in addition to a complex oxycarbonitride layer and secondary precipitates, also many nano- and micropores. The sizes of such pores are in the range from 0.01 to 1  $\mu\text{m}$ . The presence of micropores and their size distribution was confirmed by mercury porosimetry data.

It has been experimentally established that the process of integration of cell cultures in the pore space of the material is of great importance to the size factor – the size of the pores, the distribution of pores by size and the morphology of the surface of the pore walls.

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# SYNTHESIS OF POROUS CERAMIC MATERIALS BASED ON SILICON CARBIDE FOR MICROFILTRATION OF LIQUIDS

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The work is devoted to obtaining porous materials based on SiC for microfiltration of liquids. At present, silicon carbide is a widespread ceramic material with many industrial applications. It has a wide range of unique properties, including high hardness and strength at elevated temperatures, chemical resistance to oxidation, high erosion resistance, etc. The combination of these characteristics makes ceramic porous materials based on silicon carbide one of the most promising in filtration processes for cleaning hot gases, filtration of aggressive liquids and for use as a substrate for catalytic converters in processes of petrochemical synthesis [1].

In general, the technology of ceramic filters production involves the synthesis of materials with their porosity on the order of 30% to 60% and effective permeability. However, depending on the actual operational requirements of the filters, their pore size can significantly vary. The pore size of most porous ceramic materials used in industry ranges from 100 microns to 100 nm. One of the problems is obtaining effective microporous and nanoporous ceramics, since reducing the pore size is usually accompanied by a decrease in the permeability of the material. The search for solutions in this direction is associated with the need to synthesize a porous ceramic material possessing an optimal combination of pore structure and permeability characteristics for microfiltration processes.

In this work for obtaining the porous material was used SiC powder with a particle size ~ 7 microns, synthesized by the SHS method, which allowed to obtain a highly developed surface (up to 20 m<sup>2</sup>/g). A mixture of powder additives based on magnesium oxides, silicon, etc. was introduced into the charge as a sintering binder. Pressing of the finished mixture was carried out at a pressure of 70 MPa followed by sintering at a maximum temperature of 1300 °C in an air atmosphere.

The characteristics of the porous material were studied using an Autopore IV 9500 mercury porosimeter by mercury intrusion into the material. The open porosity of the material was 58.5%, mean pore size ~ 384 nm, liquid permeability ~ 0.76 mD, and pore channel tortuosity ~ 59.

X-ray analysis showed the presence of a dominant  $\beta$ -SiC phase as well as SiO<sub>2</sub>, a significant portion of which was formed as a result of SiC oxidation during sintering of the material. The study of the microstructure showed that the synthesized material has a nanoporous structure with a highly developed pore space surface. Thus, the material can be used for production of filters effective in microfiltration processes.

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# QUALITATIVE EVALUATION OF THE PLASTICITY OF HIGH-ENTROPY ALLOYS BY ARTIFICIAL NEURAL NETWORK

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The typical strategy for developing metallic alloys is associated with the selecting one or two principal components and further tuning desirable microstructure and properties via the addition of minor elements. Unlike the traditional design concept, high-entropy alloys (HEAs), proposed by Yeh et al. [1] and Cantor et al. [2], have five or more principal elements with equiatomic or near-equiatomic concentrations (the percentage of each element in HEAs varies from 5 to 35 at. %). The HEA paradigm provides an enormous number of possible alloys compositions, which makes highly inefficient the standard trial-and-errors procedure. Therefore, methods for predicting structure and properties of HEAs depending on their chemical composition are urgently needed

An artificial neural network (ANN) approach was used in the present work for prediction of tensile ductility. A dataset was constructed using experimental data reported in peer-reviewed research articles and included 154 alloys. The alloys in the dataset were classified as ‘plastic’ (fracture elongation > 10%) and ‘non-plastic’ (fracture elongation < 10%). The neural network showed an accuracy of 92% in classification of alloys on ‘plastic – non-plastic’ on validation set.

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# MACROKINETIC ANALYSIS OF THE COMBUSTION PATTERNS IN THE TRANSITION FROM POWDER TO GRANULATED MIXTURES BY THE EXAMPLE OF 5Ti+3Si AND Ti+C COMPOSITIONS

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In self-propagating high temperature synthesis processes, even a slight change in the amount of impurity gases adsorbed in initial components can lead to a change in the combustion mode and characteristics of the desired products. The influence of both impurity gas release and particle size of the initial components on the combustion velocity of 5Ti + 3Si and Ti + C (graphite) mixtures is considered. Experimental results are analyzed using a convective-conductive combustion model [1], which explains the strong effect of impurity gas release on combustion velocity. The approach used in the work made it allowed to reveal for the first time the suppressing effect of gases released during heating of titanium particles of different sizes on the velocity of the combustion wave in the mixtures. The conditions of warming up of initial components in the preheating zone of the combustion wave in powder mixtures were formulated in the work. This made it possible to predict the behavior of the combustion rate of the studied mixtures during the transition from powder to granulated mixtures. Unlike powder mixtures, granular mixtures are characterized by a monotonous decrease in the burning rate with an increase in the total particle size of the initial components. This is due to the leveling of the influence of impurity gas evolution on the combustion rate of granular mixtures. The values of combustion velocities of granulated mixtures are approximated by similar power dependence on the total particle size of the initial components for both compositions; it corresponds to the linear law of interaction of the initial components in the theory of flame front propagation in a condensed heterogeneous medium.

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**THE EFFECT OF HIGH-CURRENT PULSED ELECTRON BEAM TREATMENT ON THE ELEMENTAL DISTRIBUTION IN AL-CO-CR-FE-NI HIGH-ENTROPY ALLOY**

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High-entropy alloys consist of at least five principal elements in equal or close to equal atomic ratios. Due to the high entropy effect, severe lattice distortion and sluggish diffusion the innovative concept of HEAs results in a significant number of alloys with extraordinary properties. In this study we fabricated Al-Co-Cr-Fe-Ni high-entropy alloy by wire-arc additive manufacturing using cable-type feeding wire composed of three different filaments: pure Al wire (99.95 wt. % Al), Cr-Ni wire (20 wt. % Cr, 80 wt. % Ni), and Fe-Ni-Co wire (17 wt. % Co, 29 wt. % Ni, Fe – balanced). High-entropy alloy was layer-by-layer deposited on an AISI 1020 steel in Ar (99.99%) atmosphere. The operation parameters were constant: a wire feed speed – 8 m/min, voltage – 17 V, a torch travel speed – 0.3 m/min, a gas supply speed (Ar) – 14 L/min. Then samples were irradiated by a high-current pulsed electron beam: the energy of accelerated electrons 18 keV, the density of an electron beam (10, 20, 30) J/cm<sup>2</sup>, the duration of a beam pulse 50 μs, the pulse repetition frequency 0.3 s<sup>-1</sup>, a number of irradiation pulses 3. TEM analysis demonstrated that the initial HEA sample has dendritic microstructure. The dendrites are enriched with Al, Ni, and Fe, while the interdendritic areas mostly contain Cr. The most liquating element of the alloy is Cr, whose liquation coefficient is  $\delta = 27.5$ , while the least liquating element is Co ( $\delta = 5.9$ ). A modification of the surface by high-current pulsed electron beams remelts the dendrites and forms a cellular structure. The processes of fast melting and crystallization that proceeds at the rates of 10<sup>5</sup>–10<sup>6</sup> K/s induces the homogenization of the elemental distribution. An energy beam of 10 J/cm<sup>2</sup> reduces the range of the Cr liquation from 27.5 to 4.6, while the most liquation element is Al ( $\delta = 10.4$ ). Further increase in the energy of electron beam up to 20 J/cm<sup>2</sup> leads to an increase the homogeneity of distribution of Al content ( $\delta = 2.7$ ). The most liquating element in this case is Cr ( $\delta = 5.4$ ) and the least liquating element is Co ( $\delta = 1.9$ ). When input of an energy density of electron beam rises up to 30 J/cm<sup>2</sup> the temperature gradients also increase, that leads to excessive evaporation of some elements and deteriorates homogeneity of the elemental distribution.

To sum up, the modification of Al-Co-Cr-Fe-Ni high-entropy alloy fabricated by wire-arc additive manufacturing with high-current electron beams leads to the homogenization of the element's distribution in the modified layer. The highest homogenization degree of chemical elements obtained when the energy density of an electron beam was set 20 J/cm<sup>2</sup>.

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# ESTIMATION OF THE IMPACT PRESSURE OF THE IMPACTOR PLATE WITH A BRONZE BARRIER ACCELERATED BY THE EXPLOSION ENERGY

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The study of the formation of spall cracks in bronze alloys BrAZH9-4 and BrAMc9-2 made it possible to study the processes of plastic deformation under shock-wave loading. Shock waves caused by the energy of the explosive creates significant pressure in the material. The impact pressure estimation will make it possible to determine the impulse load level for changing the structure of bronze materials.

To estimate the pressure arising from the collision of an aluminum impactor plate with the surface of a barrier made of a bronze cylindrical sample, the equation from the work [1] was used. This equation is used in the case of a normal collision during explosion welding of two plates, when at the moment of impact, the plates touches the entire area simultaneously and plane shock waves propagates from the contact surface:

$$p_k = \frac{\rho_2 v_0^2}{\left( \sqrt{1 - \rho_2 / \rho_2'} + \sqrt{(1 - \rho_1 / \rho_1') \rho_2 / \rho_2'} \right)} \quad (1)$$

where  $v_0$  – impact velocity;  $\rho_1$  и  $\rho_2$  – initial plate densities;  $\rho_1'$  и  $\rho_2'$  – densities at pressure  $p_k$ .

It is possible to calculate the impact pressure of the impactor plate with the bronze barrier according to the formula (1) only if the velocity  $v_0$  and the equation of state of metals are given. The impact velocity was experimentally determined in [2] and is 1.5 km/s, which correlates with theoretical calculations. Impact adiabat is known for most metals in the pressure range up to 400 GPa. Also, it should be noted that the equations of state of metals have at some point  $\rho'$  a bend corresponding to the transition of the medium from an elastic to a plastic state.

The equations of state of metals for the impactor plate and the barrier have the following form:

$$p = A_1 \left[ \left( \frac{\rho}{\rho_1} \right)^{n_1} - 1 \right], \quad p = A_2 \left[ \left( \frac{\rho}{\rho_2} \right)^{n_2} - 1 \right] \quad (2)$$

where  $A_1$  и  $A_2$  – constant pressure dimensions;  $n_1$  и  $n_2$  – dimensionless constants.

The values of the impact pressure of the aluminum impactor plate, accelerated by the energy of the explosive ammonite 6ZHV, with a bronze barrier, obtained by the equation (1), are about 15-16 GPa. This pressure is several times higher than the shear strength of the studied material.

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# MATHEMATICAL MODELING COMBUSTION OF LAYERED CONDENSED MEDIA TAKING INTO ACCOUNT THE DIFFUSION MIXING OF THE REACTANTS

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Earlier studies on combustion wave propagation along the interface in bilayer systems [1, 2] have shown that critical conditions for combustion of such systems are defined by bilayer thickness and heat sink into environment.

In this communication, we report on mathematical modeling of combustion wave propagation in between two reactive layers with due regard for melting and interdiffusion of reagents and special emphasis on critical conditions for steady wave propagation, interdiffusion of melted reagents, and onset of oscillation combustion modes.

It is assumed that one-stage combustion reaction compounds  $C_1$  and  $C_2$  yields product  $C$ . Chemical reaction gets started only after the interdiffusion of melted reagents, i.e. reaction rate is defined by reagent concentrations  $C_1$ ,  $C_2$  and running temperature  $T$ . After ignition with a coil warmed up to temperature  $T_m$ , a combustion wave propagates along the interface between compounds  $C_1$  and  $C_2$  at burning velocity  $u$ . Each layer undergoes melting at one and the same melting point  $T_m$ . The melt is characterized by the function  $\eta$  defined as the volume fraction of liquid phase per a volume unit. For  $T < T_m$ , the interdiffusion is negligibly low; whereas for  $T > T_m$ , diffusivity  $D$  spasmodically grows. The problem will be formulated without explicit definition of interface as suggested in [3, 4]. Phase transformation is replaced by non-isothermic “chemical” reaction when the process of phase formation takes place within a “narrow” layer.

It was found that the melted domain moves ahead along with the combustion front and then stops at the sample butt because of reduced temperature. Lateral heat losses lead to the formation of unreacted layer that acts as a heat insulator for the inner high-temperature domain. Combustion wave propagation in between two reactive layers of a bilayer structure can steadily propagate until some critical bilayer thickness ( $l_{cr}$ ).

An oscillation combustion mode arising above  $l_{cr}$  yields a product with periodic in homogeneities in its structure. Strong heat losses lead to encapsulation of combustion process into a shell of unreacted mixtures.

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# THE INFLUENCE OF THE ELECTROLYSIS CURRENT ON THE ThO<sub>2</sub> CONCENTRATION IN THE CRYSTALLINE SOLID SOLUTION UO<sub>2</sub>–ThO<sub>2</sub>

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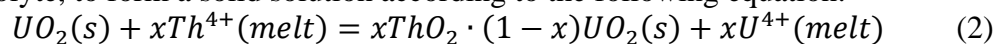
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The crystalline deposits of UO<sub>2</sub>-ThO<sub>2</sub> were obtained on the cathode during the electrolysis of the molten electrolyte (NaCl-KCl) equiv-UO<sub>2</sub>Cl<sub>2</sub>-ThCl<sub>4</sub>. They are formed as follows. Initially, at the inert cathode, when the current is turned on, uranyl ions are reduced with the release of an UO<sub>2</sub> phase according to the following equation:



Then the dioxide enters react in an exchange reaction with thorium ions, which are present in the molten electrolyte, to form a solid solution according to the following equation:



The thorium dioxide content in the cathode deposit increases with a decrease in the initial current density to 0.08 A / cm<sup>2</sup>, which is explained by the decrease in the rate of the electrochemical reaction. With a decrease in the electrolysis current density from 0.08 to 0.04 A/cm<sup>2</sup> the quantitative composition of the oxide phase remains unchanged. In this case, the concentrations of uranium and thorium dioxides in the cathode deposit are the same and are equal to 50 mol%.

It is found that with an average ThO<sub>2</sub> content of less than 50 mol%, its concentration initially increases (see picture), and reaches a maximum ( 50 mol%). This is a consequence of the fact that the rate of the electrochemical reaction decreases faster than the rate of the chemical reaction.

## OBTAINING AND STUDYING HIGH-ENTROPY COMPOSITIONS BASED ON BIMEVOX

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Entropy stabilization of crystal structures in oxide systems has received great attention recently. Success in the discovery of new materials with complete phase disorder but, at the same time, improved properties and better compatibility with the environment, provides a basis for the development of new technologies and products. To date, the results of studies of the properties of various high-entropy oxide (HEO) systems are presented in literature, as well as attempts to generalize the experience of obtaining such phases. At the initial stage of research, attention was paid to relatively simple systems, for example, based on rock salt. Later, works appeared devoted to high-entropy oxide systems with a more complex structure. One of the directions for the development of HEO can be the study of the possibility of multicomponent doping in previously studied and certified materials. In the present work, a family of substituted bismuth vanadates was chosen for this purpose.

Samples of  $\text{Bi}_4\text{V}_2\text{-xM}_x\text{O}_{11-\delta}$  (Me=Fe, Ni, Zn, Mg) solid solutions were synthesized using standard ceramic technology. The observation and analysis of phase formation processes occurring during the synthesis of samples at temperatures close to the temperature of synthesis was carried out using the XRD method. With a stepwise increase in temperature, a number of series-parallel stages are observed; metal vanadates, complex oxides of bismuth, calcium, zinc, and nickel are observed as intermediate synthesis products.

The total electrical conductivity of the samples was studied by impedance spectroscopy in the temperature range 1073–473 K in the cooling mode. Based on the results of impedance measurements and analysis of the impedance hodographs, dependences of the total electrical conductivity of the samples on temperature in the coordinates  $-\lg\sigma=f(1000/T)$  were plotted. Plots were found to be straight falling lines with a slight change in slope. The observed change in the slope can be related to phase transitions of structural modifications which are typical for BIMEVOX. The activation energies of electrical conductivity for the compositions studied were found to lie in the range of 0.4–1.7 eV, which is quite consistent with those for typical oxygen-ion conductors and other members of the BIMEVOX family.

## POWDER HIGH-ENTROPY ALLOY SYSTEM Cr-Fe-Co-Ni FOR ADDITIVE MANUFACTURING

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Alloys based on several chemical elements, called high- and medium-entropy alloys, represent a new class of materials with higher mechanical properties compared to traditional stainless structural alloys [1]. This group of alloys is especially important due to the possibility of their use in the field of additive technologies [2]. One such special alloy is a high-entropy alloy (HEA) of the Cr-Fe-Co-Ni system with a chemical composition represented by the main elements in equal proportions.

The task was to obtain a metal powder of this alloy in order to manufacture samples by 3D printing to further determine and compare mechanical characteristics with the corresponding properties of traditional AISI 316 L steel.

To obtain a metal powder the technology of gas atomization of the melt prepared in an open induction furnace was used. The resulting powder was sifted into a fractional range of 20-56  $\mu\text{m}$ . As an analysis of the powder the following were determined: bulk density, fluidity, tapping density. The samples were printed on a 3D printer, which allows change the laser power, beam speed, sintering area overlap, etc.

The samples formed according to different modes were tested for porosity by the metallographic method. Mechanical tests of samples were carried out in the temperature range from  $-150\text{ }^{\circ}\text{C}$  to  $300\text{ }^{\circ}\text{C}$ . Additionally, the values were determined by the hardness of the obtained samples.

The results of the work showed that the samples obtained from the HEA powder demonstrate improved mechanical characteristics compared to the 316L alloy sample. The tensile strength and ductility of the investigated alloy after printing are comparable to traditional hot-rolled alloys in the temperature range from  $-150$  to  $300\text{ }^{\circ}\text{C}$ .

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**PRODUCTION OF HIGH-ENTROPY Al-Co-BASED INTERMETALLIDE  
BY SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS  
AND SPARK PLASMA SINTERING**

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High-entropy B2 AlCo-based  $\text{CoNi}_{0.3}\text{Fe}_{0.3}\text{Cr}_{0.15}\text{Al}$  intermetallic materials were produced from elemental powder mixtures by self-propagating high-temperature synthesis (SHS) and reactive spark plasma sintering (RSPS) methods. Reactive composite particles, which were used as initial materials for SHS and RSPS, were produced by short-term high-energy ball milling of elemental powders. It is considered how the structure of powders changes during the preparation of composite particles from a 3-component system to a high-entropy 5-component one. It was shown that the use of a short-term high-energy ball milling allowed reaction initiation with the possibility of synthesizing a high-entropy material in a multicomponent AlCoNiFeCr mixture, which usually does not burn. Utilization of the treated powder for reactive spark plasma sintering made it possible to simplify and shorten the manufacturing cycle for production of high-density high-entropy alloy (HEA). Bulk HEA with 97% relative density was consolidated at a relatively low sintering temperature ( $< 0.7 T_m$ ) and less than 15 minutes of total processing in the mill and spark plasma sintering unit. X-ray diffraction analysis of the bulks obtained at 1000 °C according to the SHS+SPS and RSPS schemes revealed formation of the two-phase B2+BCC structure in both samples. Based on complex of the XRD, SEM and EDS data was confirmed formation of high-entropy intermetallic phase  $\text{CoNi}_{0.3}\text{Fe}_{0.3}\text{Cr}_{0.15}\text{Al}$  with B2 structure. Interaction between components during heating up to 1600 °C was investigated by DSC and possible reaction mechanism was discussed. Compression tests at room and elevated temperatures showed that the alloy obtained by RSPS method has a good level of mechanical properties that surpasses many known alloys in this system. The combustion reaction during spark plasma sintering allowed material plasticity to increase while maintaining a high-yield strength at low temperatures, the compressive strength of the RSPS sample at room temperature was 2572 MPa and increased to 2790 MPa at 400 °C. Possible reasons for this behavior were also considered.

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## EFFECT OF TA CONTENT ON MECHANICAL PROPERTIES AND ADHESION OF TI-AL-TA-SI-N COATINGS

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Coatings based on the Ti-Al-N system are widely used to protect parts and mechanisms in various industries due to their high hardness and wear resistance, increased thermal stability and oxidation resistance [1]. However, at 800-900 °C the cubic AlN transforms into the stable hexagonal AlN phase that induces a sharp decrease in the hardness and wear resistance. To further improve the mechanical and tribological characteristics, thermal stability and oxidation resistance the Ti-Al-N coatings have been alloyed with silicon. The limited solubility of Si in the Ti-Al-N solid solution leads to the precipitation of an amorphous Si<sub>3</sub>N<sub>4</sub> phase at the Ti-Al-N grain boundaries, which contributes to a decrease in the grain size and the formation of a harder nanocomposite structure. Ti-Al-Si-N coatings are characterized by high hardness, which can exceed 40 GPa, and increased thermal stability up to 1100°C. At the same time, poor adhesion and low toughness reduce the potential of using Ti-Al-Si-N as a material for protective coatings. One of the possible solutions to this problem is alloying the Ti-Al-Si-N coatings with additional chemical elements. In particular, it has been shown that introduction of Ta into Ti-Al-N coatings significantly increases their crack resistance and adhesion. Thus, the aim of this work is to study the effect of Ta content on the structure, mechanical properties, and adhesion strength of coatings based on the Ti-Al-Si-N system at various Ta concentrations.

The Ti<sub>1-x-y-z</sub>Al<sub>x</sub>Ta<sub>y</sub>Si<sub>z</sub>N coatings were deposited on Ti and Si substrates by DC magnetron co-sputtering. The Si content in the coatings was maintained at 5 at.%, while the Ta content was changed from 0 to 7.5 at.%. The X-ray diffraction studies revealed that an increase in the Ta content in the Ti<sub>1-x-y-z</sub>Al<sub>x</sub>Ta<sub>y</sub>Si<sub>z</sub>N coatings from  $y = 0$  to 0.15 resulted in increasing the lattice parameter and the change of the preferred orientation of crystallites from (200) to (111). It was found that the coatings were characterized by compressive residual stresses, which increase with the Ta content, reaching -6.8 GPa in Ti<sub>0.35</sub>Al<sub>0.40</sub>Ta<sub>0.15</sub>Si<sub>0.10</sub>N. Using the nanoindentation method, it was found coating hardening with an increase in the Ta content, while their elastic modulus decreased. The scratch testing revealed a monotonic increase in the crack resistance of the Ti<sub>1-x-y-z</sub>Al<sub>x</sub>Ta<sub>y</sub>Si<sub>z</sub>N coatings with increasing the Ta content. At the same time, the maximum adhesion strength was found in the Ti<sub>0.36</sub>Al<sub>0.44</sub>Ta<sub>0.10</sub>Si<sub>0.10</sub>N coatings, while the further increasing of  $y$  up to 0.15 resulted in earlier coating delamination due to high residual compressive stress.

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## SHS DENSIFICATION OF Ti-Al-Si ALLOY: STRUCTURE AND PHASE FORMATION

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The purpose of this study is to obtain an alloy based on the Ti–Al–Si system by self-propagating high-temperature synthesis (SHS) with densification and study its structure and properties. The SHS-densification of an intermetallic alloy based on Ti-Al-Si allowed to reduce the porosity of the synthesized materials from 41.5 to 2.7%. The microstructure of the synthesized sample is represented by grains of the  $Ti_{20}Al_3Si_9$  phase with the size of 5-10 microns. Individual pores are also distinguishable, with a size not exceeding 1 micron. According to the EDA data obtained from the surface of the  $1 \times 1 \text{ mm}^2$  area, the chemical composition of the alloy is 8.83 at. % Al, 27.52 at. % Si, and 63.65 at. % Ti, which corresponds quite accurately to the composition of the  $Ti_{20}Al_3Si_9$  phase. The content of the main  $Ti_{20}Al_3Si_9$  phase was 87 wt. %, while the content of the  $Ti_3Al$  phase was 13 wt. %. The increased microhardness values of  $9905 \pm 450 \text{ MPa}$  are due to the formation of the  $Ti_{20}Al_3Si_9$  phase with a high Si content of about 28.13 at. %. The fracture surface of  $Ti_{20}Al_3Si_9$  grains mainly corresponds to a brittle type of intragranular fracture without noticeable signs of plastic deformation with the formation of facets of intergranular and, partially, transgranular fracture. The  $Ti_3Al$  phase, shaped like  $\beta$ -Ti particles in the form of parallel plate-shaped particles or rounded particles at the grain junctions, could not be identified. Considering that the solubility of Si in Ti is less than 0.7 at. % at a temperature of 800 °C, 3.35 at. % at a temperature of 1000 °C and 5.0 at. % at a temperature of 1200 °C, it can be assumed that with an increase in the temperature of the SHS reaction, solid-state diffusion of Si into Ti occurs. The initiation of the SHS reaction occurs at the temperature of the formation of the liquid phase Al, which wets titanium particles Ti and Si. The dissolution of  $\beta$ -Ti in the Al melt leads to the formation of the intermetallic phase  $Ti_3Al$ . The formation of this phase is possible because the  $Ti_3Al$  compound is formed at a temperature of 1125 °C by the  $(\beta\text{-Ti}) \leftrightarrow Ti_3Al$  reaction, which corresponds to the temperature in this system. The formation of the  $Ti_{20}Al_3Si_9$  phase occurs on the base of the  $Ti_5Si_3$  phase with the substitution of silicon with aluminum and the formation of  $(TiSi)Al$ . The presence of the  $Ti_3Al$  phase is due to the excessive content of aluminum in the reaction mixture, which reacts with unreacted titanium.

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## OBTAINING COMPOSITE POWDERS FOR GAS-THERMAL SPRAYING OF RAM

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In the last few years, due to the rapid development and widespread use of microwaves in the gigahertz range for electronic and telecommunication systems such as mobile telephone and radar systems, and due to their potential application in commercial areas, the demand for various types of radar absorbing materials (RAMs) for electromagnetic interference reduction is increasing [1, 2]. Due to the transformation at high temperatures, the permeability of most magnetic materials is sharply reduced, which limits their scope. For this reason, materials with high heat resistance are the most suitable for use as RAMs for high temperature applications.

Promising from this point of view are cermets consisting of a dielectric matrix and a high-temperature conductor, which have a high operating temperature and excellent erosion resistance.

The use of the material based on alumina as a high-temperature dielectric in cermets can ensure the operability of the radio-absorbing material up to a temperature of 1400 °C. For this reason, composite ceramics based on alumina have recently been actively studied in the form of radio absorbing coatings applied by gas-thermal spraying [3].

Powders of the high-temperature conductor FeCrNiAl were obtained by gas-spraying methods. Master alloy for FeCrNiAl alloys was obtained using the method of self-propagating high-temperature synthesis (SHS). Later, after joint grinding of the resulting alloy and high-temperature dielectric (Al<sub>2</sub>O<sub>3</sub>), composite powders (CP) for gas-flame spraying of RAM were obtained by granulation. The structure and properties of CPs and RAMs deposited from them were studied in this work.

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# THE ROLE OF SN TRACE ADDITION ON THE PRECIPITATION BEHAVIOR AND STRENGTHENING OF THE AL-CU-MN ALLOY

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Among various classes of precipitation-strengthened Al alloys, wrought Al–Cu alloys (2xxx series) are amongst the most popular in the industry. The aforementioned alloys derive a significant measure of their strength from the controlled precipitation of the  $\theta'$  phase, which is characterised by the number density, crystal structure, size distribution of precipitates [1,2]. One of the areas of works is on the manipulation of precipitation hardening throughout microalloying with additives including Sn, In, Cd, Bi, Ag, Au. They have been found effective in accelerating the precipitation and contribute to a significant increase in the strength of the Al–Cu alloys by modifying the precipitation structure [1-4]. Moreover, among the above-mentioned additions, it is safe to say that tin is of particular scientific and industrial interest, due to availability, content in secondary raw materials and the degree of the effect at extremely low concentrations.

In this work, we intensively investigated the influence of tin trace addition on the microstructure and mechanical properties of the Al–Cu–Mn based alloy and dramatical increase in precipitation hardening after deformation, solution heat treatment and aging was found out.

Using computational (Thermo-Calc) and experimental research techniques, including hardness tests, electrical conductivity measurements, electron microscopy methods (SEM, TEM), the effect of a (Sn) trace addition on the structure and precipitation hardening of the Al<sub>4</sub>Cu<sub>1.5</sub>Mn alloy after deformation and aging was studied. It is shown that the Sn trace addition in the Al<sub>4</sub>Cu<sub>1.5</sub>Mn alloy leads to an increase in the peak hardness of 33 % (~120 HV). According to TEM, it is shown that the hardness effect of Sn is related to the modification of precipitation structure; this is shown in the decrease in the linear size and increase in the number density of the metastable  $\theta'$ -phase precipitates.

*The study was carried out with the financial support of the grant of the Russian Science Foundation (Project N° 20-79-10373).*

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## COMPARATIVE INVESTIGATION OF MACROKINETIC PARAMETERS OF DILUTED (Ti+C)-BASED POWDER AND GRANULAR MIXTURES

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For the first time, the macrokinetic parameters of “gasless” combustion were studied comparatively upon dilution of powder and granular mixtures of Ti+C. As in the work [1], combustion velocities of powder mixtures (Ti+C)+20%Me (Me = Ni, Cu) turned out to be higher than those of Ti+C mixtures, despite the lower temperature of combustion. This contradicts theoretical models of the combustion velocity dependence on the maximum temperature in condensed heterogeneous media [2]. When diluting the Ti+C mixture with Ti or TiC powders, such a contradiction does not occur. Using the convective-conductive combustion model [3], the experimental data were explained by the strong influence of impurity gas release from titanium ahead the combustion front. The emission of volatile impurities in a powder mixture depends on the warming up the mixture particles ahead the combustion front. The applicability of the heating conditions formulated by the authors was confirmed for powder mixtures under study. The values of the combustion velocity inside the granules and the time of the transfer of combustion between the granules were obtained. The combustion inside the granules is similar to combustion in the powder mixture without impurity gases effect. Therefore, a ratio of the combustion velocity inside the granules to the combustion velocity in a powder mixture gives a quantitative assessment of the decelerating effect of impurity gases in powder mixtures without a direct measurement of their volume.

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# PRODUCTION OF BRIQUETTES AND METALLURGICAL COMPOSITES FROM METAL-WORKING WASTE

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The technology proposed for implementation in this work includes the following technological operations:

- separation of waste in order to remove foreign objects, crushing, batch preparation by mixing components;
- non-oxidizing heating of the charge to temperatures of incomplete hot deformation (700-800 °C) in an atmosphere of products of thermal sublimation and pyrolysis of the oil component of the cutting fluid (coolant);
- formation of a pyrocarbon coating on the surface of metal particles, which protects the metal from oxidation and acts as a lubricant in the hot pressing process;
- combustion of oil vapors together with natural gas, flue gas cleaning in a Venturi scrubber, catalytic afterburning of CO, condensate collection and hydrocarbon regeneration;
- supply of hot charge into the mold with minimal heat loss (no more than 15-20 °C);
- pressing on a hydraulic press under a pressure of 470-500 MPa in a mold with a movable matrix that activates the action of lateral friction forces directed towards the current pressing force;
- production of briquettes and metallurgical composites with a density of at least 90% of the density of castings of the same chemical composition.

The technical and economic result of the proposed technology is the complete removal of residual moisture and organic impurities from metal waste, the elimination of decarburization and waste metal, its fine fractions (up to 1 mm), the use of sludge powders, slag-forming and alloying additives in the composition of briquettes, the production of high-quality briquettes – substitutes for overall lump scrap and cast billets, reduction of energy and transport costs for the production of briquettes and their processing. Dense and high-strength briquettes with a predetermined and demanded chemical composition are sold at a higher selling price.

The technological module for hot briquetting includes a hydraulic press with a force of 800 tf, a trestle, two small-sized suspended heating units that do not require significant heating costs at the beginning of a work shift, and a two-position hot briquetting stamp. The charge is loaded into the storage bins of the module by two bucket elevators, and the briquettes are unloaded using pneumatic devices and a chain conveyor. Hot briquetting molds are equipped with systems of internal and external cooling, automatic ejection of briquettes, removal of metal powder spills from the internal working cavity. The module capacity in two-shift operation (hour/day/year) is 3t/20t/6000t.

**INTENSIFICATION OF THE DIFFUSION WELDING PROCESS BY PRELIMINARY LASER NANOMODIFICATION OF DETAILS SURFACES**

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Achieving the required physical and mechanical properties of welded joints, obtained by diffusion welding, is possible by ensuring the course of diffusion exchange between the welded surfaces and by the formation of a sufficient diffusion zone. For each pair of materials, it will have its own magnitude, which will be optimal in terms of ensuring the strength characteristics of the joint.

The process of formation of the diffusion zone can be intensified by varying the process parameters, and also – by using different methods: – application of intermediate layers; – introduction of ultrasonic vibrations into welding zone; – influence of an electrostatic field; – preliminary formation of various ordered structures on the surfaces to be welded by exposing them to electron-beam, ion-plasma or laser radiation [1].

In recent years, the simplest in terms of execution technique and demanded in terms of the results achieved is the method of activation of welded surfaces by laser radiation.

Positive results have been obtained, when a scanning beam of a frequency-pulse nanosecond ultraviolet (UV) laser was used to influence the metal. A variety of nano- and microstructures appear which dramatically change the adhesive properties. Such heat treatment preserves the geometry of the detail.

A new phenomenon – the optoplastic effect, was discovered at the IEE RAS [2]. It manifests itself when a metal is exposed to a high-power laser pulse, the intensity of which is somewhat lower than the optical damage threshold and manifests itself in the appearance of high-temperature plastic deformation in the near-surface layer.

It is shown that the preliminary heat treatment of ChS57 alloy samples by UV laser pulses results in a significant improvement in the mechanical properties of joints obtained by diffusion welding. The process temperature is reduced by 160°C, while maintaining the mechanical characteristics of the welded joint.

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## A NOVEL SYNTHESIS PROCESS OF HIGH-ENTROPY (HFZRTINBTA)N POWDER VIA SILICON THERMAL REDUCTION NITRIDATION

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High-entropy nitride powder is one of prerequisite materials for the preparation of high-performance high-entropy nitride ceramics. High-entropy (HfZrTiNbTa)N powder was synthesized *via* silicon thermal reduction nitridation at high temperatures. The phase and microstructure samples were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) with energy disperse spectroscopy (EDS), high-resolution field emission transmission electron microscopy (HRTEM), respectively. The results show that the metal oxides, HfO<sub>2</sub>, ZrO<sub>2</sub>, TiO<sub>2</sub>, Nb<sub>2</sub>O<sub>5</sub> and Ta<sub>2</sub>O<sub>5</sub> all can be transformed into the corresponding metal nitrides at 1973 K, and the solid solutions of the metal nitrides occur as the temperature increases from 2173 K to 2373 K. High-entropy nitride powder with a single face-centered cubic structure can be formed readily when the Hf molar content decreases from 0.20 to 0.18 or 0.16. The high-entropy (Hf<sub>0.18</sub>Zr<sub>0.205</sub>Ti<sub>0.205</sub>Nb<sub>0.205</sub>Ta<sub>0.205</sub>)N powder synthesized at 2373 K has the particle sizes of 0.2-2.0 μm with a quasi-spherical particle morphology and a uniform elemental distribution. The mechanism of the synthesis reaction was also analyzed based on thermodynamic calculation and atomic diffusion theory.

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**JOINT OF ALUMINOSILICATE GLASS With MOLYBDENIC ALLOY**  
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Diffusion welding is the only bonding technology which allows obtaining qualitative one-piece joints of metallic materials with nonmetallic materials in various combinations [1,2]. In particular, it is applied to the bonding of aluminosilicate glass S48-3 (C48-3) and molybdenic alloy TsM-2A (LM-2A), widely used in precision instrument-making industry, in electronic and electric devices.

The main requirements for such devices (besides achievements of the strength of joint at the level of the strength of bonded materials) are:

- provision of hermeticity of the joint at the level from  $5 \cdot 10^{-12} \text{ m}^3 \cdot \text{Pa/s}$  to  $1 \cdot 10^{-10} \text{ m}^3 \cdot \text{Pa/s}$  for all the operating period and also after thermocycling in the temperature range from minus  $65^\circ\text{C}$  to  $+55^\circ\text{C}$ ;

- value of residual thermal stress in the welded joint shall not be more than 10 MPa;

- parts after welding shall not have changes of their geometric sizes.

Diffusion welding of the parts made of the above materials may be carried out by two technology process schemes:

1. Bond the glass with the alloy directly to each other. In this case, the qualitative joint is formed at the temperature  $T > 700^\circ\text{C}$ , which is close to the temperature of glass softening ( $T_s = 810^\circ\text{C}$ ). But even at a minimum force of squeeze ( $P = 0.5 \text{ kgf/mm}^2$ ), the glass begins to deform plastically.

2. Apply an interfacial layer of 0.1 mm thickness aluminum foil in order to lower the welding process temperature. At that, of course, in the zone of joining will form intermetallic compounds of the Al-Mo system. As the welding temperature increases, there also increases the formation speed and amount of intermetallic compounds in the welded joint. Microfractures and nonsolid areas appear in the zone of intermetallic compounds formation. They have a negative impact upon the hermeticity of joint.

Nevertheless, the diffusion welding mode with  $T=600^\circ\text{C}$ ,  $P>5 \text{ kgf/mm}^2$  and  $t=30 \text{ min}$  provides fulfillment of the announced requirements. It is connected with the fact that the applied welding pressure deforms plastically the intermetallic compounds and provides seizing with the main material.

The same effect can be achieved if foils of titanium and aluminum are used as interfacial layers; at that, at first, a diffusion welding of the titanium foil with the molybdenic alloy is to be carried out, and then the aluminum foil is to be placed in between the welded titanium foil and the glass, and the diffusion welding is to be carried out with the above given parameters.

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# MECHANICAL AND FRACTURE PROPERTIES OF Al-Mg-Si ALLOY IN THE CG AND UFG STATE

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Aluminum alloys of the Al-Mg-Si system used in industry are distinguished by a high level of technical characteristics after hardening heat treatment (quenching and aging). In addition to the methods of thermal hardening and aging, methods of severe plastic deformation (SPD) are used to improve the properties of metallic materials. These methods are interesting due to the deep change in the structure, physical and mechanical properties and phase composition and the acquisition after their application by the material of a multiple increase in strength characteristics while maintaining high plasticity[1]. The issue of fracture in the ultrafine-grained (UFG) state remains poorly understood [2, 3].

In the presented work, the interrelation between the change in the microstructural characteristics of the alloy 6101 of the Al-Mg-Si system and its physical and mechanical properties after deformation-heat treatment, including ECAP-C, is established.

Alloy Al 6101 shows higher strength during preliminary grinding of the grain structure by the ECAP-K method. The fracture surface of a coarse-grained (CG) alloy contains a large percentage of Fe, many times higher than its average content in the alloy. Coarse particles of FeAl<sub>3</sub> intermetallic compounds are areas of nucleation of pores and subsequent fracture in the CG state under tension, and intermetallic particles based on Fe in a large content are on the fracture surface. The Fe content on the fracture surface of the UFG state after ECAP-C treatment is noticeably lower. The fracture surface is pitted and has a uniform structure. The size of the pits on the surface of fractures for samples with a CG structure can be estimated up to 10 μm in size, and for samples with a UFG structure, up to 5 μm. The highest concentration of pores is located in the center of the sample, which corresponds to the region of maximum stress concentration. The short-circuit state corresponds to pores with a size of 3-7 μm, for UFG – 1-2 μm. ECAP-K treatment leads to the refinement of FeAl<sub>3</sub> particles from 7 to 2 μm in alloy 6101, relative to its coarse-grained (CG) state.

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[3] Letters on materials 5 (2), 2015 pp. 202-206 Structure and properties of aluminum alloy system Al-Mg-Si after processing by the method of Multi-ECAP-Conform E.I. Fakhretdinova1†, E.V. Bobruk1,2, G.Yu. Sagitova1, G.I. Raab1,3

## PROSPECTS FOR SHORT-PULSE LASER APPLICATION FOR OBTAINING HIGH-ENTROPY ALLOY COATINGS

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The high-speed laser melting technology (HLM) is an advanced version of the selective laser melting (SLM) additive technology. Our studies show the HLM-produced coatings with unique strength and tribological characteristics [1-3]. Even if a small diameter of the laser beam is used, the ultrasonic pulse repetition rate (20 kHz) makes it possible to apply coatings or grow products using the HLM method with a productivity sufficient for many industrial applications. The main difference between HLM and SLM is the use of highly dispersed powders and lasers with pulse durations of tens of nanoseconds or milliseconds. For example, a short-pulse high-frequency ytterbium laser with a pulse power of 3-50 W and a spot diameter of 30 μm provides an instantaneous power of up to 25 kW. As a result of high-speed heating, temperatures up to 3500 °C are reached on the surface. Subsequent intense cooling results in the high-speed solidification with the strongly non-equilibrium states formation, amorphization and supersaturated solid solutions. Therefore, unique prospects open up if using the HLM technology for the high-entropy alloy (HEA) coatings formation, for which it is especially important to ensure the conditions for obtaining closely-packed single-phase disordered substitutional solid solutions with strong lattice distortions and, at the same time, for suppressing the formation of brittle intermetallic phases. Most products are destroyed from the surface (wear, corrosion, cavitation, fatigue cracking), so the use of expensive HEAs in the form of thin laser coatings will provide significant savings.

Using the HLM technology by the method of four-layer slip laser cladding applying an ytterbium fiber laser with a wavelength of 1.065 μm, a pulse duration of 3.5 ms, and a frequency of 20 kHz, multicomponent coatings were obtained based on the CrFeNi equiatomic alloy with various hardening additives of chromium carbide, tungsten carbide, tungsten boride, as well as cobalt and nickel. Depending on the composition, the microhardness of coatings varied within 200-850 HV. The structural-phase state of the coatings was studied by transmission electron microscopy.

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# THE EFFECT OF THE TELLURIUM VAPOR ON THE STRUCTURE FORMATION AND DIELECTRIC PROPERTIES OF A MULTI-COMPONENT SYSTEM BASED ON SODIUM-POTASSIUM NIOBATE

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The paper presents the results of studying the effect of paratellurite vapor during sintering on the dielectric properties of a multicomponent system based on sodium–potassium niobate ceramics. Samples of modified ceramics based on sodium–potassium niobate (mKNN) with the general formula  $(\text{Na}_{0.5}\text{K}_{0.49}\text{Li}_{0.05}\text{Sr}_{0.05})(\text{Nb}_{0.9}\text{Ta}_{0.05}\text{Ti}_{0.05})\text{O}_3$  were obtained by solid-phase synthesis. Two batches of samples were produced; one was introduced with an impurity of  $\text{TeO}_2$  ~ 5 wt %. The presence of  $\text{TeO}_2$  during sintering the mKNN ceramic samples leads to a decrease in the temperature of solid-phase synthesis.

The inclusion of paratellurite in mKNN ceramics changes the shape and increases the grain size by an order of magnitude. Thus, if grains containing only mKNN material have a cubic shape, then the presence of tellurium leads to the formation of grains in the form of sufficiently long tubes (when the length is several times greater than the diameter) with a porous internal structure.

Temperature studies carried out in the frequency range from 1 Hz to 15 MHz have shown that the addition of  $\text{TeO}_2$  to the mKNN composition leads to the disappearance of the additional maximum observed for mKNN on the temperature dependence of the permittivity in the region of 220–250°C. This maximum corresponds to a structural phase transition in KNN ceramics.

An anomalous behavior of the permittivity at high frequencies (1 – 15 MHz) was revealed. With the introduction of paratellurite, a distinct peak appears in the modified mKNN ceramics, the shape of which is similar to that of the piezoelectric resonance–antiresonance. In the absence of a paratellurite impurity in the mKNN ceramic sample, the resonant component of the peak is absent, and only a weak antiresonance minimum takes place.

## CATALYTIC REFORMING OF BIOGAS TO OBTAIN SYNTHESIS-GAS

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Catalytic reforming of renewable raw material – biogas plays a decisive role in petrochemistry for the production of hydrogen-containing fuels. Basically, biogas consists of 60-65% methane, 30-35% carbon dioxide, 1% nitrogen, and less than a percent of other gases such as H<sub>2</sub>S, H<sub>2</sub> and steam. As a model for research, a mixture of methane with carbon dioxide was used, which was subjected to the process of carbon dioxide conversion of methane (DRM). Nickel-based catalysts are used to achieve high product yields. Nickel-containing catalysts are highly active, but they are prone to coke formation and sintering. In order to reduce the formation of coke, Mn, Cr, Fe, Co are added as promoters. The catalysts obtained by the SHS and SCS methods are of high quality and do not require additional processing. The main advantage of this method is the saving of energy and time, as well as the occurrence of self-propagating exothermic red-ox reactions, which result in the formation of nanocrystalline and high-purity solid nanomaterials.

In the presented work, Ni-Cr-Mg-Al catalysts were studied in the DRM process. The catalysts were tested in a flow catalytic unit at a space velocity of 2500 h<sup>-1</sup> at a ratio of CH<sub>4</sub> : CO<sub>2</sub> : Ar = 33% : 33% : 34%, in the temperature range 600 – 900°C.

As a result of the data obtained, it was found that at 800°C the conversion of both CH<sub>4</sub> and CO<sub>2</sub> reaches its maximum value and at 900°C it is: K<sub>CH<sub>4</sub></sub> = 99.5% and K<sub>CO<sub>2</sub></sub> = 98%. With an increase in temperature, the yields of H<sub>2</sub> (39.4%) and CO (59.6%) rise, as well as the ratio of H<sub>2</sub>/CO in the range of 1.5÷1.7. The catalysts were characterized by XRD, SEM and TEM methods. Spinels MgNiO<sub>2</sub> (JCPDS, 24-712), NiCrO<sub>3</sub> (JCPDS, 22-748), as well as mixed metal oxides NiO (JCPDS, 4-825), CrO<sub>2</sub> (JCPDS, 9-332) and intermetallic compounds Al<sub>9</sub>Cr<sub>4</sub> (JCPDS, 2 -1193).

Thus, Ni-Cr-Mg-Al catalysts show high activity for producing synthesis gas by carbon dioxide reforming of methane, which is mainly due to the formation of spinels and resistance to carbon deposition.

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# STRUCTURAL-PHASE TRANSFORMATIONS IN HIGH-ALLOYED STEELS UNDER VARIOUS TYPES OF LOADING

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The development of technologies for creating structural materials leads to the spread of multicomponent high-alloyed steels in the industry. Some of them have low structural stability and may undergo structural-phase transformations under the influence of various technological and operational factors, such as temperature and deformation [1]. As a result of such transformations, the physical, mechanical and service characteristics of the steel change as well.

This report investigates the structural-phase transformations that take place in high-alloyed steels during their deformation caused by mechanical tests. Austenitic steel 10Kh13G12N2S2D2B and steel 23Kh15N5AM3Sh with a two-phase austenitic-martensitic structure were studied. Tensile and instrumented indentation tests of these steels were performed. Stress-strain curves and indentation diagrams were recorded.

An analysis of the obtained diagrams made it possible to reveal the plastic deformation features of such high-alloyed steels. In both studied steels, a phase transformation of metastable austenite into martensite ( $\gamma \rightarrow \alpha$  transformation) occurs. As the plastic strain degree increases during loading, the amount of martensite grows. Structural-phase transformation is the reason that at a certain stage of testing, a new structure is deformed, which has different mechanical properties compared to the original one. This causes a change in the values of mechanical characteristics of the material: the Young’s modulus, hardness, strength and strain hardening parameters. The exponents change in the Meyer and Ludwik-Hollomon equations, which characterize the material strain hardening for ball indentation and tension test, respectively.

The phase transformations in the studied steels during deformation were confirmed by studying the structure of the deformed material using transmission and scanning electron microscopy, as well as by measuring its magnetic characteristics.

The obtained results must be taken into account when establishing the relationship between indentation diagrams and stress-strain curves, which underlie some methods for determining the mechanical properties of materials from hardness characteristics.

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# SYNTHESIS OF NOVEL HIGH-ENTROPY INTERMETALLIC COMPOUNDS (NiCoFeCuMn)Zn<sub>3</sub> AND (NbTaVNiTiFe)Al<sub>3</sub>

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In this study, we investigated the possibility of fabricating single phase HEIC (high-entropy intermetallic compounds) in (NiCoFeCuMn)Zn<sub>3</sub> and (NbTaVNiTiFe)Al<sub>3</sub> systems.

The (NiCoFeCuMn)Zn<sub>3</sub> and (NbTaVNiTiFe)Al<sub>3</sub> HEICs were synthesized by melting of high purity precursors.

As a result of the analysis of the X-ray spectra of powders, it can be seen that (NiCoFeCuMn)Zn<sub>3</sub> crystallizes into an almost uniform cubic structure of the type of  $\gamma$ -brass (D8<sub>2</sub>, spatial group *I43m*) with minor inclusions of BCC (Fe-rich) and HCP (Co-rich, determined from the analysis of the EDS map). The EDS analysis indicated that the sum of atomic ratios of Ni, Co, Fe, Cu, and Mn elements are 25.11%, marginally higher than the range reported for  $\gamma$ -brass structure forming in Co–Zn system. However, it should be also considered that the segregation of Fe and Co brings the chemical composition of (NiCoFeCuMn)Zn<sub>3</sub> in the proper range for formation of  $\gamma$  brass structure.

The XRD spectra of (NbTaVNiTiFe)Al<sub>3</sub> HEIC indicated higher amount of secondary phases. The main XRD peaks could be indexed as a D0<sub>22</sub> HEIC phase (based on TaAl<sub>3</sub> structure, space group *I4/mmm*) with Ni<sub>4</sub>Ti<sub>3</sub>, FeNi, and BCC (Fe-rich) as the main inclusions. In (NbTaVNiTiFe)Al<sub>3</sub> HEIC, the segregation and precipitation of secondary phases make it hard to precisely judge about its atomic configuration.

The back scattered-electron scanning electron microscopy (BSE SEM) images of the ball milled HEIC powders shown us that both powders consist of particles with typical irregular morphologies of ball milled powders. The particles sizes of (NiCoFeCuMn)Zn<sub>3</sub> and (NbTaVNiTiFe)Al<sub>3</sub> HEICs are in the range of 5–40  $\mu\text{m}$  (average particle size of 17.3  $\mu\text{m}$ ) and 10–60  $\mu\text{m}$  (average particle size of 25.4  $\mu\text{m}$ ), respectively. In the BSE-SEM of (NbTaVNiTiFe)Al<sub>3</sub> HEIC, two distinct areas with bright and gray contrasts could be clearly detected, suggesting elemental segregation and multiphase nature of this sample. EDS elemental maps indicate a homogenous distribution of the constituent elements in (NiCoFeCuMn)Zn<sub>3</sub> HEIC, in spite of minor Fe and Co inclusions.

On the other hand, EDS maps indicated a clear segregation of Ta and Nb in the bright contrast regions, and Fe and Ni in the gray contrast regions. The chemical composition of the powders indicates some Mn losses for (NiCoFeCuMn)Zn<sub>3</sub> and Ti losses for (NbTaVNiTiFe)Al<sub>3</sub> HEICs. However, a relatively good agreement between EDS analysis and nominal composition of the samples still exists.

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## ORGANIZATION OF FUNCTIONAL LAYERS OF COMPOSITE COATINGS FOR TRIBOTECHNICAL APPLICATION

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The variety of plain bearings used in the modern equipments units is ensured by their design features. However, another promising solution for expanding the range and scope of plain bearings is the use of special materials for their manufacture. These materials can adapt to changing conditions and allow minimizing the effects of wear, as well as guaranteeing the working of the friction unit before scheduled repairs. This scientific direction development can be ensured through the organization of functional layers of composite coatings for tribotechnical application. Moreover, such materials consist of a steel base that perceives the load, intermediate technological layers that provide the required values of adhesive strength, as well as layers formed directly in the synthesis process of the composition and layers with a composite structure that have the required level of tribological properties.

This paper presents the results of comprehensive studies of the developed compositions based on structural steels with functional layers of composite materials based on aluminum and tin, produced by the liquid-phase arc surfacing process with a non-consumable tungsten electrode in argon. The influence of the functional layers organization and technological parameters of synthesis process on their structure and operational characteristics is shown. Particular attention is paid to the interfaces between the functional layers. The tribotechnical properties of the manufactured composite coatings were evaluated under conditions of dry sliding friction in a wide range of loading parameters. This made it possible to characterize the performance of the obtained materials under extreme operating conditions, characterized by boundary lubrication or even its absence.

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**FUTURE OF HIGH TEMPERATURE OXIDE-FIBRE/HEA-MATRIX COMPOSITES**

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The invention of high entropy alloys (HEAs) [1] opened a new chapter in the development of new high temperature composites since, first, among such alloys there can be found those characterized by high melting point and high oxidation resistance [2,3] and, secondly, being multi-component and equiatomic HEAs they would not provide troubles in wetting ceramic fibres that is a necessary condition to design fibrous composites characterized by both high creep resistance and high fracture toughness.

Despite creep resistance is a main property of a high temperature material only scarce publications on creep of HEAs are known and they are mainly concerned with relatively low temperature alloys (see i.e., [4,5]). Hence, we should agree with the authors of [6]: “Creep, a fundamental high-temperature property, serves as an important standard to evaluate service life, safety, and reliability of engineering components. Despite the excellent properties of HEAs at room and cryogenic temperatures [...], their creep behavior has not been studied systematically by far”

Creep of metal matrix composites containing high strength brittle fibres can be easily evaluated provided mechanical properties of the components and fibre/matrix interface are known [7, 8]. The latter depends on chemical compositions of the components.

A huge number of possible HEAs [9] and a large number of single crystalline and eutectic oxide fibres produced by the internal crystallization method that can be a base for the industrial technology of such fibres sufficiently cheap for their usage in structural materials [10,11] make prospects of high temperature oxide-fibre/HEA-matrix composites clear. Preliminary experiments [12] support this statement.

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## **EFFECT OF HIGH TEMPERATURE TEMPERING ON CREEP BEHAVIOR OF A 10% CR MARTENSITIC STEEL**

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High-chromium martensitic steels are the creep-resistant materials at elevated temperatures. Their high creep resistance is provided by the tempered martensite lath structure formed during normalizing and tempering. Parameters of the lath structure can significantly vary depending on the tempering temperature and affect the creep strength.

In order to estimate the optimal tempering temperature of advanced 10% Cr martensitic steel with high B and low N contents, its creep behavior after tempering at 790°C for 3 h was examined. Creep rupture tests were carried out at temperature of 650°C under different applied stresses from 120 to 160 MPa.

It was found that high-temperature tempering resulted in a lower creep resistance of the 10% Cr steel in the whole stress range as compared with tempering temperature of 770°C. The long-term creep rupture strength was roughly predicted by approximation of experimental data and comprised less than 100 MPa at 650°C for 100,000 h.

Times to rupture are lower about one order of magnitude as compared with tempering temperature of 770°C. This is due to the fact that the minimum creep rates at the apparent steady-state creep stage are two orders of magnitude higher. It should be noted that the steel demonstrated the significantly larger off-set strain of about 5% at which the minimum creep rate was attained. This is in accordance with high total elongation of creep samples of about 20-30%.

The tertiary creep stage is characterized by the continuous increase in the creep rate.

The lower creep resistance of the 10% Cr steel tempered at 790°C can be probably caused by the more equilibrium lath structure or subgrain structure, lower dislocation density, and larger precipitates of  $M_{23}C_6$  and MX phases in the as-tempered state in comparison with lower tempering temperature of 770°C. The reasons for lower creep strength should be studied further.

The results obtained can be useful for the development of heat treatment of 10% Cr steel studied and other martensitic steels with the same composition.

*This work was carried out using equipment of the Joint Research Center of Belgorod State National Research University «Technology and Materials».*

## WELDING TECHNIQUES OF Ti<sub>2</sub>AlNb-BASED ALLOYS

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Nowadays, plasma, laser, diffusion, and electric resistance welding of Ti<sub>2</sub>AlNb-based alloys attract greatest interest. Various techniques for welding of such alloys are considered and proposed in the work. A defect-free weld with an optimal structure is formed due to the high welding speed and the concentration of thermal energy or the absence of the need to melt the metal. The welding modes are selected and the technological features of plasma, laser, diffusion, and electric resistance welding with obtaining the optimal structural state and strength properties up to 90% of the base metal.

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## HYPEREUTECTIC ALLOYS BASED ON Al-Ca-Mn- (Ni, Ce) SYSTEMS AS AN ALTERNATIVE TO HYPEREUTECTIC SILUMINS

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Hi-load pistons operating at temperatures up to 300 °C are mainly made of alloys A390.0 and FM180. In fact, they are natural metal matrix composites (NMMC) composed of solid (Si) crystals distributed over a eutectic matrix. However, they have a number of fundamental disadvantages, such as fragility and the need for a modification operation for grinding the primary and eutectic (Si) phases. Natural Metal-Matrix Composites (NMMC) based on the multicomponent hypereutectic Al-Ca-(Mn)-(Ni, Ce) alloys were studied in as-cast, annealed and rolled conditions. Thermo-Calc software and electron microscopy (SEM, TEM and EDS) observations were utilized for analyzing the equilibrium and experimental phase composition of the alloys including the solid phase distribution in the Al-Ca-Mn-(Ni, Ce) systems. A previously unknown  $Al_{10}Mn_2Ca$  compound was discovered by both EDS and X-ray studies. The hot-rolled sheets were obtained from flat ingots of alloys Al-8Ca-2Mn-1Ni and Al-6Ca-3Mn-2Ce at a temperature of 550 °C. The total degree of strain was about 90%. The structure of rolled products is fully consistent with the structure of "natural composite". It contains compact small (less than 50  $\mu m$ ) primary crystals, evenly distributed in a fine-crystalline eutectic matrix. The rolling process caused the FM180 alloy specimen failure due to coarse silicon crystals in its structure. New alloys showed competitive thermal expansion coefficient values and tensile properties which were remarked to be dependent on the strength characteristics of the primary crystals. It is shown that a compact morphology can be achieved by conventional casting without using any refining agents. Novel hypereutectic Al-Ca NMMC materials encountered primary solidification of the compact  $Al_{10}Mn_2Ca$  compound have the best ductility and strength and thus, may be reasonably proposed as a structural material for hi-load pistons. In addition, it was clearly shown using direct thermal analysis and DSC that the operating temperatures of aluminum-calcium alloys can be significantly higher than that of silumin.

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## THERMAL STABILITY AND CORROSION RESISTANCE OF ULTRAFINE-GRAINED HIGH-ENTROPY ALLOY Fe<sub>30</sub>Ni<sub>30</sub>Mn<sub>30</sub>Cr<sub>10</sub>

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The thermal stability and corrosion resistance of a high-entropy Fe<sub>30</sub>Ni<sub>30</sub>Mn<sub>30</sub>Cr<sub>10</sub> alloy [1,2] subjected to high pressure torsion deformation (HPT) [3] have been studied. The influence of additional heat treatment on the microhardness and corrosion resistance of HPT samples was investigated.

TEM studies of the HPT samples have revealed the formation of an ultrafine-grained structure with an average size of 300 nm, containing microtwins.

Thermal stability studies have shown that the maximum microhardness of 6009 MPa was achieved on a sample subjected to HPT 300°C after annealing 450°C, 90 minutes, which is more than 3 times higher than the strength of the sample in the initial state (1915 MPa). The maximum microhardness of the sample subjected to HPT 20°C after annealing 450°C for 90 minutes was equal to 4208 MPa.

Electrochemical tests have shown that the Fe<sub>30</sub>Ni<sub>30</sub>Mn<sub>30</sub>Cr<sub>10</sub> alloy sample subjected to HPT 300°C and thermal treatment at 450°C has a high corrosion resistance in an aqueous solution of 3.5% NaCl, comparable to the corrosion resistance of stainless steel 304L.

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# MECHANISM OF MICRO/NANOSTRUCTURED SURFACE FORMATION IN HIGH-ENTROPY ALLOYS AT ELECTRON BEAM TREATMENT

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Formation of surface submicro- and nanostructures in high entropy alloys AlCoCrFeNi and CrMnFeCoNi irradiated with an electron beam with the energy density of 10-30 J/cm<sup>2</sup> with regard to the hydrodynamic concepts about the occurrence of combined thermocapillary, concentration-capillary and thermoelectric instability at the melt/plasma boundary has been studied. It has been established that growth of E<sub>s</sub> results in increase of crystallization cells sizes as well as of interlayers between them. It is conditioned by the fact that, under E<sub>s</sub> > 10 J/cm<sup>2</sup> the power density losses by evaporation of the substance become significant and they reduce the value of the temperature gradient. Analysis of the dispersion equation at low-frequency approximation without taking into account the influence of the thermoelectric field under the boundary conditions for tangential stresses allowed establishing that the wave length values under which the maximum growth rate of disturbances is observed and which are closest to the experimental data in terms of crystallization cell sizes are 186 nm for AlCoCrFeNi alloy and 430 nm for CrMnFeCoNi under E<sub>s</sub> = 30 J/cm<sup>2</sup>.

Dispersion equation:

$$\begin{aligned} & \left( (-C^2 + 2C_4 + 2(z^2 - 1)(z + 1)) \text{Pr} - 2(z^2 - 1) \right) C_2 + \\ & + \left( 2C_4 \text{Pr} - C^2 \text{Sc} + 2(z^2 - 1)(z + 1) \text{Sc} - 2(z^2 - 1) \right) C_3 - \\ & (2C_5 + (z^2 - 1)(z^2 + 2z + 3)) \text{Pr} C_4 - 2(z^2 - 1) C_5 + (z + 1)^2 (C^2 + (z^2 + 1)^2 - 4z) = 0 \end{aligned} \quad (1)$$

$$\text{where } C = \frac{\omega_c}{\omega_v}, \quad C_2 = \frac{\omega_T}{2\omega_v}, \quad C_3 = \frac{\omega_C}{2\omega_v}, \quad C_4 = \frac{\omega_p}{2\omega_v}, \quad C_5 = \frac{\omega_E}{2\omega_v}. \quad \omega_T = \frac{\sigma_T G_0}{\rho v}, \quad \omega_C = \frac{\sigma_C G_1}{\rho v},$$

$$\omega_v = \nu k^2, \quad \omega_c^2 = \frac{\gamma_m k^3}{\rho} - \frac{\varepsilon \varepsilon_0 E_0^2}{\rho} k^2, \quad \omega_p = \frac{p'_v G_0}{\rho \nu k}, \quad \omega_E = \frac{\varepsilon \varepsilon_0 E^2}{\rho v}, \quad \delta = \text{Pr}/(1-\text{Pr}), \quad \delta_1 = \text{Sc}/(1-\text{Sc}),$$

Pr = ν/χ – Prandtl number, Sc = ν/D – Schmidt number. Let us take Re(z) > 0 and Re(ω) > 0 as the criterion of the instability of disturbances. It being fulfilled, the disturbances will increase without limit at the rate of α = α(λ). The wavelength λ<sub>m</sub>, under which the maximum of this dependence is observed, will determine the size of the formed crystallization cells.

With consideration to the thermoelectric field under the boundary conditions λ<sub>m</sub> = 189 nm for AlCoCrFeNi alloy and for CrMnFeCoNi it grows up to 454 nm.

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# THE EFFECT OF AGE-HARDENING ON HYDROGEN EMBRITTLEMENT OF CoCrFeMnNi HIGH ENTROPY ALLOY

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The influence of hydrogen charging on the mechanical properties and fracture mechanisms of a high-entropy alloy before and after age-hardening was investigated. A high-entropy alloy Co<sub>20</sub>Cr<sub>20</sub>Fe<sub>20</sub>Mn<sub>20</sub>Ni<sub>20</sub> was chosen as an object for the study. The cast alloy was subjected to a thermomechanical treatment, including solid-solution treatment (1200 C, 2 h), cold rolling (80% reduction) and final SST (1200 C, 2 h). Some of the specimens were studied in the initial state after this treatment (I-HEA), and the other part was subjected to age-hardening at 900°C for 1 hour (A-HEA). Electrochemical hydrogen charging was carried out at a current density of  $j_H = 10 \text{ mA/cm}^2$  for 50 h at room temperature in a 3% aqueous NaCl solution in the presence of 3 g/l NH<sub>4</sub>SCN.

Electron microscopic studies have shown that I-HEA specimens are characterized by single-phase austenitic structure. Age-hardening leads to the precipitation of the intermetallic  $\sigma$ -phase, enriched with chromium, along the grain boundaries in A-HEA specimens. An analysis of room temperature tensile properties shows close values of the yield strength ( $\sigma_{0,2}$ ) and elongation to failure ( $\delta$ ) for I-HEA and A-HEA specimens:  $\sigma_{0,2}^{\text{I-HEA}} = 160 \pm 5 \text{ MPa}$ ,  $\sigma_{0,2}^{\text{A-HEA}} = 155 \pm 5 \text{ MPa}$ ,  $\delta^{\text{I-HEA}} = 62 \pm 2\%$ ,  $\delta^{\text{A-HEA}} = 65 \pm 2\%$ . Hydrogen charging is accompanied with significant change in mechanical properties. The yield strength increases ( $\Delta\sigma_{0,2}$ ) in the hydrogen-charged specimens, which is caused by solid-solution hardening. Moreover, the  $\Delta\sigma_{0,2} = 60 \pm 5 \text{ MPa}$  in A-HEA is higher than that in the I-HEA specimens ( $\Delta\sigma_{0,2} = 25 \pm 5 \text{ MPa}$ ). The index of hydrogen embrittlement ( $I_H$ ), which describes the hydrogen-induced loss of plasticity, for I-HEA specimens is 23%, that is 2 times higher than that for A-HEA specimens,  $I_H = 12\%$ .

All hydrogen-free specimens fractured via a ductile dimple micromechanism. Hydrogen-charging leads to the formation of brittle surface layers that undergo intensive cracking during tensile tests at room temperature. An analysis of the fracture surface showed that intergranular fracture of the hydrogen-assisted brittle layers occurred, but transgranular elements were also seen in I-HEA and A-HEA specimens. The intergranular facets in the A-HEA specimens have uneven, "grainy" morphology. This indicates the crack propagation along the interphase (particle/matrix) boundaries and testified the predominant accumulation of hydrogen atoms along such boundaries. The thickness of hydrogen-assisted brittle layer ( $W_H$ ) for A-HEA specimens ( $W_H = 28 \pm 7 \text{ }\mu\text{m}$ ) is much smaller than for the I-HEA specimens ( $W_H = 70 \pm 21 \text{ }\mu\text{m}$ ).

Therefore, specimens of the CoCrFeMnNi alloy after age-hardening at a temperature of 900°C for 1 hour are more resistant to hydrogen embrittlement, compared with the initial single-phase specimens.

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## STUDY OF ALMG6+TITANIUM WELD INTERFACE AFTER EXPLOSIVE WELDING

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Composite materials based on aluminum alloys and titanium, due to the combination of high specific strength and corrosion resistance are widely used in aircraft building, shipbuilding, petrochemical engineering, etc. Aluminum alloys and titanium have a significant difference in physical and mechanical properties. Therefore, the brittle intermetallic phases  $Ti_xAl_y$  are formed at the weld interface, which reduce the mechanical properties of composite materials [1]. In view of the fact, explosive welding (as solid state welding method) is widely used to produce the composite materials consisting of aluminum alloys and titanium.

The aim of the study was to examine in detail the microstructure of the weld interface and the mechanical properties of AlMg6 + titanium composite material produced by explosive welding.

In the experiment, commercially available plates of AlMg6 and titanium were used. The explosive used in the experiment is a mixture of microporous ammonium nitrate and diesel oil (ANFO). The detonation velocity was about 2500 m/s. As a result, a composite material with a size of  $(4+5) \times 200 \times 300$  mm was produced. The weld interface was flat along the entire length of the composite material and did not have pores. Also, a transition zone 10–20  $\mu\text{m}$  thick was formed on the AlMg6 side, consisting of Al (87.32%), Mg (6.70%) and Ti (5.26%). The microhardness of this zone was 300...330HV. According to EBSD data, the transition zone has a crystalline structure with local areas of an amorphous structure. Also, adiabatic shear bands were found in AlMg6. They are located at an angle of 30 to 45° to the weld interface. It was determined that they consist of the deformed and recrystallized grains (6...15  $\mu\text{m}$ ). The significant part of the recrystallized grains was most probably formed after explosive welding, when the composite material was cooled down. The tear strength of the weld seam after explosive welding was  $130 \pm 50$  MPa, which is almost half the tear strength of the AlMg6 alloy. Although the maximum tear strength of some samples reached 180 MPa, which is close to the optimal strength threshold.

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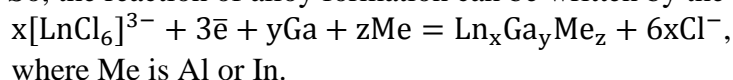
# ELECTROCHEMICAL FORMATION OF LN-GA-AL AND LN-GA-IN (LN = LA, PR, DY) ALLOYS IN LIQUID METAL / MOLTEN SALT SYSTEM. THERMODYNAMICS OF TRIPLE COMPOUNDS

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Nuclear power, as a kind of "green and clean" energy, is currently considered as a promising candidate for the future because of its enormous efficiency, cost-effectiveness and lack of greenhouse gas generation. A limiting factor in its development is the lack of modern technology for the disposal of spent nuclear fuel and high-level nuclear waste. Currently, research is underway for creation a closed nuclear cycle with using molten salts. It is known that lanthanides are neutron poisons, and therefore they must be separated and concentrated from the basic components of the fuel during its reprocessing. Therefore, these investigations are aimed for studying the electrochemical processes of the alloys formation and determining principal thermodynamic characteristics of triple compounds in "liquid metal-molten salt" system.

Cyclic and square wave voltammetry were used for determination the reaction of electrochemical reduction of Ln(III) ions in different molten salts on inert and active electrodes. It was established that the process of alloy formation proceeds with depolarization, the value of which reaches 0.4 – 0.6 V. The effect of current density on the composition of the cathode product was investigated and the conditions for production of individual intermetallic compounds were found. So, the reaction of alloy formation can be written by the following scheme:



For calculation of the principal thermodynamic characteristics of the alloy formation processes open-circuit potentiometry were used. Three-dimensional graphs on the basis of the functional dependence of the apparent electrode potentials of the metals and alloys, temperature and the atomic number of the elements in the Periodic Table were obtained. The universal mathematical Maple 17 software was used for this purpose. Low values of the activity coefficients ( $10^{-12} - 10^{-15}$ ) show strong interaction between lanthanides and the liquid metal compositions. It was found that the activity coefficients decreased with the increasing of the atomic number of the element in the Periodic Table and the risen of the temperature shifted the system towards more ideal behavior.

Analysis of the data obtained showed that Ga–In and Ga–Al alloys are the prospective media for application in partitioning technologies of spent nuclear fuels and nuclear wastes treatment.

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# OXIDATION RESISTANCE OF (NiCoFeCuMn)<sub>3</sub>(AlTi) HIGH ENTROPY INTERMETALLIC COMPOUND

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High entropy intermetallic compounds (HEICs) are the most recent poster child of high entropy materials (HEMs) with potentially intricate properties since they combine the high entropy effects of solid solutions and the ordered crystal structures of intermetallic compounds. Recently, several HEICs with C14 Laves phase [1, 2], D0<sub>22</sub>-phase [3], B2-ordered structure [4, 5], and  $\sigma$ -phase [2] have been developed. While research on HEICs is still in its infancy, several promising properties such as superior strength and thermal stability [3, 5], and exceptional oxidation resistance [6] have been already reported. Compared with binary intermetallics, HEICs even provide greater opportunity to further tailor their mechanical properties. For example, an interesting approach to provide better room-temperature ductility in intermetallics is production of two-phase or three phase microstructure with a mixture of ductile and ordered intermetallic phases, as we recently demonstrated in (NiCoFeCuMn)<sub>3</sub>(AlTi) HEIC [2].

In this work, we study high temperature oxidation resistance of (NiCoFeCuMn)<sub>3</sub>(AlTi) HEIC to further evaluate its performance for practical applications. (NiCoFeCuMn)<sub>3</sub>(AlTi) were fabricated by milling-melting technique. A two phase B2+D0<sub>3</sub> structure with clear segregation of Cu, Fe and Mn was obtained for this HEIC. The oxidation resistance of (NiCoFeCuMn)<sub>3</sub>(AlTi) was studied at 900 °C for 10 h. The sample indicated a continuous weight gain without exhibiting a plateau region on the dynamic oxidation curve. The scale layer was mainly consisted of Mn oxide (Mn<sub>3</sub>O<sub>4</sub>) which could not provide an effective protection against high temperature oxidation. Finally, it can be concluded that while phase segregation is effective to improve ductility of HEICs, it may have detrimental effect on their high temperature oxidation resistance.

*The work was supported by the Russian Science Foundation, project No. 21-73-00086.*

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# EFFECT OF HARDENING WITH BORIDES ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF TiNbZr AND Al<sub>5</sub>Nb<sub>24</sub>Ti<sub>40</sub>V<sub>5</sub>Zr<sub>26</sub> ALLOY-BASED COMPOSITES

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The use of titanium alloys is often limited by their other characteristics: relatively low strength, hardness and wear resistance. A significant improvement in strength characteristics can be achieved by using a combination of strengthening strategies, for example, due to a significant modification of the chemical composition and transition to the so-called medium- and high-entropy (MEA and HEA) compositions with high solid solution hardening, as well as by creating metal-matrix composites with ceramic reinforcing components. The best choice for titanium-based alloys is the use of TiB particles as a hardener, which adheres well to the titanium matrix without the formation of a transition region and has a similar coefficient of thermal expansion; in addition, due to its good thermal stability, TiB can provide strength even at elevated temperatures. The structure of composites with an MEA/HEA matrix will depend on many variables, for example, the production method and conditions (casting or powder metallurgy) and the volume fraction of borides. These issues have hardly been studied for metal-matrix composites with an MEA/HEA matrix. Therefore, the detailed analysis of the dependence of the structure and properties of composites with MEA and HEA matrix on the volume fraction of borides, and the conditions for obtaining blanks is undoubtedly relevant, and can be of high practical importance.

In this work laboratory-scale NbTiZr and Al<sub>5</sub>Nb<sub>24</sub>Ti<sub>40</sub>V<sub>5</sub>Zr<sub>26</sub> alloys-based composites ingots (40 g) were melted by vacuum arc remelting with a non-consumable electrode. For melting, pure (with a purity of at least 99.9%) elements that make up the matrix, as well as TiB<sub>2</sub> (titanium diboride) powder with an average particle size of 3-8 microns, were used. The initial microstructure of the both synthesized metal-matrix composites with different amounts of TiB<sub>2</sub> in the range from 0.2 to 4 wt. % was composed of bcc matrix and complex needle-like monoborides (Ti, Nb) B. Alloying base alloys with borides led to an increase in tensile strength by ~ 20% and a slight decrease in ductility.

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## NEW REFRACTORY HIGH-ENTROPY ALLOYS WITH A BCC-B2 STRUCTURE

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Intermetallics are generally used to strengthen a disordered and soft matrix in many high-performance alloys, such as nickel-based superalloys. In turn, alloys with an intermetallic matrix are strong yet brittle. For instance, in the recently developed refractory high-entropy alloys (RHEAs), a mixture of an intermetallic B2 matrix and coherent bcc particles ensure high strength at elevated temperatures but low room-temperature ductility. However, several works report that the B2 phase can be ductile at room temperature. These compounds have a high melting point but show low yield strength. In this study, we obtained balanced mechanical properties at ambient and high temperatures thanks to a soft refractory intermetallic B2 matrix and hard bcc particles. The composition-structure-property relationships and possibilities for further improvements in properties are discussed.

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# MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AL5NB24TI40V5ZR26 ALLOY-BASED COMPOSITES, REINFORCED WITH BORIDE PARTICLES

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The issue of increasing the strength characteristics, hardness and wear resistance of beta titanium alloys while maintaining or reducing the elastic modulus is relevant, since this would significantly expand the scope of these materials. A radical improvement in the strength characteristics of titanium alloys can be achieved by using a combination of different hardening strategies, both through a significant modification of the chemical composition and the transition to the high-entropy compositions, and through the creation of metal-matrix composites with ceramic reinforcing components. Among a variety of reinforcements TiB seems to be the most suitable option due to very similar properties (density, thermal expansion coefficient, good crystallographic match) with the Ti matrix. Metal-matrix composites can be produced using traditional metallurgical methods through addition of B or compounds of boron and titanium into melted metal matrix.

In this work the Al<sub>5</sub>Nb<sub>24</sub>Ti<sub>40</sub>V<sub>5</sub>Zr<sub>26</sub> alloy-based composites with different amounts of reinforcing component TiB<sub>2</sub> were produced by vacuum arc melting in a high purity argon atmosphere. For melting, pure (with a purity of at least 99.9%) elements that make up the matrix, as well as TiB<sub>2</sub> powder with an average particle size of 4 microns, were used. The weight proportion of TiB<sub>2</sub> in cast composite billets was 1, 2 and 3 wt. %. In the as-cast condition composites had a single phase bcc structure. The microstructure of the alloy in the initial condition consisted of mainly equiaxed grains with a size of ~ 150 μm. In the as-cast condition composites had a single phase bcc structure with heterogeneously distributed boride particles. According to SEM-EDS mapping results revealed that, formed borides enriched in niobium and titanium. A significant refinement of the microstructure was found with an increase in the proportion of borides. Alloying the base alloy with 1 wt. % TiB<sub>2</sub> led to an increase in strength from 760 MPa to 840 MPa. Increasing the content of TiB<sub>2</sub> to 2 and 3 wt. % led to a sharp drop in ductility (0.5 and 0 %, respectively) and a slight increase in strength to 890 and 900 MPa, respectively.

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# EFFECT OF ROTARY SWAGING AND SUBSEQUENT ANNEALING ON THE STRUCTURE AND MECHANICAL PROPERTIES OF A METASTABLE AUSTENITIC STAINLESS STEEL

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Metastable austenitic stainless steels (MASS) possess high ductility, impact toughness, and corrosion resistance but reduced yield strength. Obtaining a gradient structure resulted in an increase in strength without a noticeable decrease in ductility and impact toughness. Using laboratory techniques and small samples, gradient structures with 200 microns in depth are usually attained. Thereby, it is difficult to apply these approaches for real products. However, rotary swaging with high strain might result in the bulk structural gradient formation. Subsequent heat treatment can be accompanied by reverse martensitic transformation, polygonization, recrystallization, and precipitation of nanoparticles that can change mechanical properties in the wide range. Thus, the present work aims to study the effect of rotary swaging and subsequent heat treatment on the structure and mechanical properties of a metastable austenitic stainless steel.

During cold rotary swaging of the AISI 321 steel rod with a strain of 60% or more, the formation of the strong gradient structure occurs that is associated with a gradual increase in the  $\alpha'$ -phase fraction in the radial direction from the rod center to the edge. The most pronounced gradient structure is formed after a strain of 90%. Meanwhile, the texture gradient of austenite is also obtained. So, in the center, the strong two-component axial 111//rod axis (RA) and 001//RA texture is found that sufficiently diffused towards the edge. After cold rotary swaging, UTS and YS increase from 610 to 1410 MPa and from 270 to 1405 MPa, respectively. Yet, ultimate elongation and impact toughness decrease from 67 to 11.5% and from 1.58 to 0.7 MJ/m<sup>2</sup>, respectively.

During low-temperature annealing (500-650 °C), partial reverse martensitic transformation, uncompleted recrystallization and nanocarbide precipitation are observed. At the same time, the structural and textural gradients, as well as the gradient of the  $\alpha'$ -phase fraction, are unchanged. Medium-temperature annealing (700 °C) is accompanied by a decrease in the  $\alpha'$ -phase fraction to the level of the undeformed condition (2-5%), but recrystallization is also not finished. After high-temperature annealing (800-900 °C), the reverse martensitic transformation and recrystallization are completed and the equiaxial austenitic structure is formed. It should be noted that the excellent mechanical properties are received after low-temperature annealing at 500 and 650 °C. After annealing at 500 °C, the strength characteristics and impact strength increase by ~ 10% and 2 times, respectively, in comparison with the cold-swaged condition. However, after annealing at 650 °C, the improved level of strength characteristics, plasticity and toughness is observed.

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## INVESTIGATION OF MICROSTRUCTURE AND RESIDUAL STRESS IN LASER-SHOCK-PEENED TI-6AL-4V

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Due to its excellent combination of low density and high strength, Ti-6Al-4V is widely used in the aerospace industry. In many applications, enhanced high-cycle fatigue (HCF) performance is also often desired. In view of the major effect of material surface condition on HCF behavior, a number of techniques have therefore been developed to improve surface properties and fatigue resistance. One recent innovation in this area has been the development of laser shock peening (LSP). LSP represents a surface treatment that introduces compressive residual stresses with high penetration depth in critical regions susceptible to fatigue phenomena. During propagation of the shock wave, the material experiences severe deformation at very high pressure (GPa level), ultra-high strain rate, and ultra-short duration. These attributes give rise to significant compressive residual stresses in the surface layer which is beneficial in retarding the initiation of fatigue.

Samples of Ti-6Al-4V titanium alloy with a thickness of 6 mm, cut from a hot-rolled plate were used in this study. LSP was conducted using a Q-switched Nd:YAG laser operating at 10 Hz with a wave length of 1064 nm and a pulse duration of 10 ns. A diffractive optic was used to deliver 5 J in a square spot of 1 mm x 1 mm on a specimen surface covered with a steel foil, which led to a laser power density of 25 GW/cm<sup>2</sup>. 1 and 3 numbers of overlapping LSP treatment were carried out.

The resulting distributions of (surface/near surface) compressive residual stresses were quantified using the incremental-hole-drilling method, and microstructures were determined using EBSD and TEM techniques. Microstructural analysis suggests that LSP-induced deformation is characterized by limited cross slip and climb of dislocations associated with the extremely short timescale of the process. The large mechanical energy gives rise to the stresses within the processed material whereas the very short duration of LSP prevents stress relief via dislocation cross-slip. LSP-processed material contains twins and poorly-developed dislocation boundaries whose traces often lay close those for slip planes. It was found that one overlap of laser shock peening led to an increase in the value of residual stresses in comparison with the initial state, in which practically zero values of residual stresses were determined. It is shown that an increase in the number of LSP overlaps from 1 to 3 led to an increase in the magnitude of compressive residual stresses: – 200 MPa versus – 300 MPa on the surface; – 500 MPa versus – 700 MPa at a depth of 0.5 mm.

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## STRUCTURAL-PHASE STATES OF MULTICOMPONENT ALLOYS NANOPARTICLES FABRICATED BY EXPLODING WIRES

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Nanoparticles of multi-component (high-entropy) alloys garner increasing researchers' attention as these nanoparticles open opportunities for obtaining materials with novel or advanced functional properties. Wider opportunities for the fabrication of new materials stipulate the necessity for developing new and highly efficient techniques for producing nanoparticles of multi-component alloys. In the development of new multi-component alloy fabrication techniques, high-speed (explosive) physical processes are given preference. Such processes have higher nanoparticle cooling rates, which in some cases makes it possible to rule out segregation of metals with limited mutual solubility in the nanoparticle volume.

Electrical explosion of wires (EEW) is a high-speed process ( $10^{-6}$  s), which suggests its potential applicability in the fabrication of nanoparticles of multi-component alloys. That is why we suggested a new approach to nanoparticle fabrication and it is based on the electrical explosion of wires of dissimilar metals or alloys. The capabilities of EEW in the fabrication of nanoparticles with different structural-phase states were determined by the example of AlCoFeNiCu, FeNiMoCuCo and AlCoCrFeNiCu systems with non-equiatomic compositions ( $\Delta S_{\text{mix}} \geq 1.5R$ ). It was determined that varying the quantitative content of metals between 5 and 35 per cent allows for the fabrication of single-phase samples based either on bcc or fcc structures. At  $\Delta H_{\text{mix}} \geq 0$  kJ/mol for AlCrFeNiCu and AlCoFeNiCu nanoparticles, particle surface enrichment with copper atoms is observed, which is caused by the segregation of metals in the nanoparticles volume. All the investigated nanoparticle samples are characterized by log-normal distribution by size where number-average nanoparticle size varied between 40 and 50 nm.

The research findings have shown that joint electrical explosion of wires of different metals/alloys can produce nanoparticles of multi-component alloys with a pre-determined structural-phase state.

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# EFFECT OF COLD ROLLING ON THE MICROSTRUCTURE EVOLUTION AND MECHANICAL PROPERTIES OF AL<sub>5</sub>NB<sub>24</sub>Ti<sub>40</sub>V<sub>5</sub>ZR<sub>26</sub> ALLOY-BASED COMPOSITES

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A significant improvement in strength characteristics of titanium alloys can be achieved by using a combination of different strengthening strategies, for example, due to a significant modification of the chemical composition and transition to the so-called medium- and high-entropy (MEA and HEA) compositions with high solid solution hardening, as well as by creating metal-matrix composites with ceramic reinforcing components. The best choice for titanium-based alloys is the use of TiB particles as a hardener, which adheres well to the titanium matrix without the formation of a transition region and has a similar coefficient of thermal expansion. The structure of composites with an MEA/HEA matrix will depend on many variables, for example, the production method and conditions (casting or powder metallurgy) and the volume fraction of borides. The detailed analysis of the dependence of the structure and properties of composites with MEA and HEA matrix on the volume fraction of borides, and the conditions for obtaining blanks is undoubtedly relevant, and can be of high practical importance.

In this investigation laboratory-scale Al<sub>5</sub>Nb<sub>24</sub>Ti<sub>40</sub>V<sub>5</sub>Zr<sub>26</sub> alloys-based composite ingots (~ 50 g) were melted by vacuum arc remelting with a non-consumable electrode. For melting, pure (with a purity of at least 99.9%) elements that make up the matrix, as well as TiB<sub>2</sub> (titanium diboride) powder with an average particle size of 3-8 microns, were used. Prismatic samples measuring were cut out from the homogenized cylinders using. The samples were then rolled to a total thickness strain of 5-80 % thickness reduction.

The initial microstructure of the both synthesized metal-matrix composites with different amounts of TiB<sub>2</sub> in the range from 0.2 to 1 wt. % was composed of bcc matrix and complex needle-like monoborides (Ti, Nb) B. Cold rolling resulted in some elongation of the matrix grains toward the metal flow direction, elongated TiB particles also became aligned with the rolling direction. Cracks formed in TiB particles could possibly initiate secondary cracks in the bcc matrix. The evolution of microhardness of alloys depending on the strain and a quantitative analysis of the contribution of various mechanisms to hardening of alloys were discussed.

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## CATALYTIC GROWTH OF CARBON NANOMATERIALS ON MULTICOMPONENT ALLOYS PRODUCED BY JOINT ELECTRIC EXPLOSION OF WIRES

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The synthesis and study of carbon nanomaterials (CNMs) is one of the promising areas of modern materials science. The structure of CNM and their functional properties are determined by the nature of carbon-containing raw materials, the parameters of the catalytic decomposition process, and the composition of the used catalyst. The basis of the catalysts used for CNM synthesis are mainly metals of the iron triad (Fe, Co, Ni) capable of synthesizing carbon filaments by the carbide cycle mechanism. Alloy additives (Pd, Cu, Mo, etc.) are often used to increase the activity and stability of catalysts, which also significantly affect the structural characteristics of CNM.

The aim of this work was to develop a method for obtaining two- and five-component alloys and to compare their catalytic ability in the decomposition of hydrocarbons with the production of carbon nanofibers (CNFs). The key components in the composition of the alloys were Ni and Al. The alloy samples were obtained by joint exploding wires (EW). A mixture of C<sub>2</sub>-C<sub>4</sub> hydrocarbons was used as hydrocarbon raw material. The process temperature was 600°C, reaction time – 30 minutes.

Three series of two- and five-component alloys of the following composition were obtained: Ni<sub>95-75</sub>Al<sub>25-5</sub> (1), Ni<sub>33-25</sub>Fe<sub>29-23</sub>Cu<sub>23-8</sub>Al<sub>35-8</sub>Cr<sub>9-7</sub> (2), Ni<sub>32-23</sub>Fe<sub>31-21</sub>Cu<sub>25-19</sub>Al<sub>22-12</sub>Co<sub>9-6</sub> (3). The average particle size of the obtained samples ranged from 40 to 60 nm. The samples of series (1) were solid solutions based on the *fcc* lattice of nickel. Samples of series (2) and (3), according to XRD data, were solid solutions based on *fcc* or *bcc* lattice, depending on the aluminum content in the sample.

It was found that the catalytic decomposition of a mixture of C<sub>2</sub>-C<sub>4</sub> hydrocarbons on the samples studied resulted in the accumulation of CNM. The yield of CNM for the samples of series (1) varied in the range from 3.7 to 13 g/g<sub>cat</sub>, for samples of series (2) – from 0.5 to 14.8 g/g<sub>cat</sub>, for samples of series (3) – from 44 to 86 g/g<sub>cat</sub>. At the same time, the productivity of pure nickel (reference sample) obtained by the EEW method under these reaction conditions was 2.5 g/g<sub>cat</sub>. Thus, with increasing aluminum content in the sample (regardless of the number of components in the alloy), a decrease in the catalytic activity of the alloy is observed, which leads to a decrease in the yield of CNM.

Data on the morphology and structure as well as textural and macroscopic characteristics of the carbon nanomaterial obtained using five-component alloy catalysts will also be presented and discussed in the report.

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## STUDY OF THE MOTION VELOCITY OF W AND TIC PARTICLES DURING HIGH-ENERGY TREATING OF A BARRIER

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In the process of high-energy interaction of detonation products in an explosive wave with powder particles, their entrainment occurs as a result of inelastic collision of detonation product molecules with particles. The collision of the particle flow with the barrier leads to the stoppage of the largest part of them in the near-surface zone and the formation of a coating on its surface. Structural changes in the studied material consist of the action of shock waves created when a particle flow collides with a barrier, and from the effect of particles embedded in the sample.

In the experiments, samples of U8 tool steel with a diameter of 25 mm and a height of 30 mm was used as a steel barrier, as well as powders of tungsten (10-16 microns) and titanium carbide (50-70 microns). Bulk density hexogen was used as an explosive.

After the detonation began, the shock wave and the detonation products of the explosive dispersed the powder particles ( $m = 5 \text{ g.}$ ) in the guide channel and together with them affected the barrier. The methodology of the experiment is given in the work [1]. An electron-optical camera Nanogate-4BP for high-speed photo shooting, which allows taking four frames in one experiment, was used. The delay time for the start of taking the first frame after the detonation of the explosive was 7 microseconds, which is necessary for the detonation products to reach the powder layer. The delay time between frames is 5 microseconds.

Analysis of high-speed photographs of the movement of powder particles and explosive detonation products in the guide channel showed that the flow of tungsten particles moves at an average speed of 2.4 km/s; the flow of titanium carbide particles at a speed of 4.9 km/s.

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# INFLUENCE OF ADDITIONAL INTERCALIATION COPPER ATOMS ON THE PHYSICAL PROPERTIES OF $\text{Cu}_y\text{-Cr}_{0.25}\text{HfSe}_2$

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For layered transition metal dichalcogenides with the general formula  $\text{TX}_2$  intercalated with 3d- transition elements, a transition from paramagnetic behavior to states with local or long-range magnetic order is possible with an increase in their content [1, 2].

Intercalation of the  $\text{HfSe}_2$  compound by chromium atoms leads to an increase in the magnetic susceptibility and a decrease in the electrical resistance, while maintaining the semiconductor type of conductivity. The  $\text{Cr}_x\text{HfSe}_2$  compounds in the entire concentration range  $0 \leq x \leq 0.25$  exhibit paramagnetic behavior at  $T > 2$  K. In addition to superexchange interlayer interaction between the 3d electrons of intercalated atoms, the formation of a magnetic state can be the result of an indirect exchange through conduction electrons of the RKKY type, which has a long-range and oscillating character [3].

To establish the role of such an indirect interaction in the  $\text{Cr}_{0.25}\text{HfSe}_2$  compound, copper atoms were additionally intercalated and  $\text{Cu}_y\text{Cr}_{0.25}\text{HfSe}_2$  ( $y=0.1; 0.2$ ) samples were synthesized, where copper atoms, upon their ionization, should increase the concentration of free electrons. As a result, it was found that, while maintaining the semiconductor nature of the conductivity, its value increases both in comparison with  $\text{Cr}_{0.25}\text{HfSe}_2$  and with an increase in the copper content. In this case, the magnetic susceptibility of copper-containing samples increases, which can also be caused by the partial contribution of copper ions  $\text{Cu}^{2+}$ , the presence of which was established from EPR data and also leads to a rather high value of the diamagnetic contribution. It has been found that the additional insertion of copper atoms, which is accompanied by an increase in the electron concentration, leads to a change in the sign of the paramagnetic Curie temperature from negative at  $y = 0$  to positive in copper-containing compounds and the appearance of a state of the cluster spin glass type in the  $\text{Cu}_{0.2}\text{Cr}_{0.25}\text{HfSe}_2$  compound at temperatures below  $T_f = 6.5$  K. These results indicate that the mechanism of indirect exchange interaction of the RKKY type has a significant effect on the magnetic state of layered compounds of the  $\text{TX}_2$  type intercalated by atoms of 3d elements.

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# EVOLUTION OF STRUCTURE OF CoCrFeNiCu HIGH-ENTROPY ALLOY DURING PROLONG ANNEALING

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High-entropy alloys (HEA) attract attention in terms of their properties. On their basis, it is possible to create thermoelectric converters having high efficiency ( $ZT > 3$  [1]) simultaneously with environmental friendliness and cheapness of production materials, corrosion-resistant coatings, as well as the creation of spintronics devices.

The concept of materials of this type is that all elements taken in equal or close molar fractions that are part of the alloy form one phase. Moreover, atoms of different types are randomly arranged in the crystal lattice, i.e. the HEA is a disordered solid substitution solution. With an increase in the content of components in the alloy, the configuration entropy increases and the stability of the structure increases [2].

The report discusses the results of studies of the thermal stability of a nanostructured CoCrFeNiCu high-entropy alloy. The stability of the CoCrFeNiCu HEA was studied after annealing lasting 1 day, 3 days, 10 days, 20 days and 204 days at a temperature of 800°C. X-ray phase analysis was carried out. Transmission electron microscopy was performed on cross sections prepared by the focused ion beam method. The elemental composition was obtained using energy-dispersive X-ray spectroscopy, and the phases were determined using diffraction of the selected region.

It is shown that the main structural changes occur on the first day of thermal annealing, but even prolonged annealing leads to further enlargement of the particles in the alloy.

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## MEDIUM ENTROPY FE-BASED ALLOY WITH TRIP EFFECT

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In new classes of materials, such as high- and medium-entropy alloys (HEAs and MEAs), the manifestation of TRIP effects are of greater interest. The TRIP effect in face-centered cubic (fcc) HEAs and MEAs, resulting in high strength in combination with high ductility, is usually associated with a decreased stability of the fcc phase. It is well known in some alloys with a high Fe content the fcc phase can become unstable, while a large fraction of Fe decreases the cost of the alloy. Interstitial elements such as C can also strongly affect the fcc phase stability; besides, carbon promotes additional strengthening mechanisms such as interstitial solid solution strengthening and/or precipitation hardening. In this work, we examined a new carbon-containing medium-entropy alloy of the Fe-Co-Ni-Cr-C system, which has a TRIP effect, with excellent mechanical properties during deformation at liquid nitrogen temperature (-196 C). The influence of cold rolling and test temperature on the microstructure and mechanical properties of the material is discussed. The data obtained during this study can expand the understanding of the mechanisms that determine the behavior of alloys with TRIP effects under various conditions.

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# POLYMETALLIC GRID CATALYSTS WITH ACTIVATED SURFACE FOR THE SYNTHESIS OF CARBON NANOTUBES FROM METHANE

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Currently, there are a huge number of different methods for the synthesis of carbon nanotubes(CNT). A significant part of them is based on technologies that use varieties of the most productive and flexible pyrolysis method [1]. Pyrolysis is subjected to both the simplest hydrocarbons of the paraffin series and unsaturated hydrocarbons. Saturated cyclic hydrocarbons are represented by cyclohexane, aromatic hydrocarbons by C<sub>6</sub>H<sub>6</sub>, C<sub>6</sub>H<sub>5</sub>CH<sub>3</sub>, (CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, polyaromatic hydrocarbons by polyphenylacetylene, pyridine and pyrene, ketones by acetone, alcohols by methanol and ethanol. There are ways to obtain CNT from hydrocarbons with such heteroatoms as O, N, S, and also Cl.

The synthesis of CNT is most often carried out using a powder catalyst of various compositions and fineness. Good catalytic properties in this process are exhibited by metals of the iron group Fe, Co, Ni[2].

In [3] was proposed preparation of an active phase by leaching and stabilizing complex intermetallic compounds formed during high-temperature saturation of the surface of polymetallic alloys consisting of catalytically active metals with aluminum using the example of chromium-nickel stainless steel grid. The active phase has a composition similar to the most commonly used nanotube growth catalysts.

In this work, we show the possibility of obtaining CNT on nichrom grid polymetallic catalysts received by a similar method. Methane was used as the feedstock for pyrolysis. Even at a low conversion of the initial methane in the temperature range of 600–700°C, the formation of a significant amount of CNT on the surface of the catalytic grid is observed. Obtained CNT have a specific surface area of 141.2 m<sup>2</sup>/g. The composition contains an admixture of nickel.

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## THE EFFECT OF CARBON ALLOYING ON TEMPERATURE DEPENDENCE OF THE MECHANICAL PROPERTIES OF HIGH-ENTROPY FeMnCrNiCo ALLOY

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The microstructure, phase composition, temperature dependence of the mechanical properties and deformation mechanisms in Fe<sub>19.9</sub>Mn<sub>19.9</sub>Cr<sub>20.0</sub>Ni<sub>20.0</sub>Co<sub>20.2</sub> (at. %, HEA-0C), Fe<sub>19.9</sub>Mn<sub>20.0</sub>Cr<sub>20.0</sub>Ni<sub>20.0</sub>Co<sub>19.0</sub>C<sub>1.1</sub> (HEA-1C) and Fe<sub>19.9</sub>Mn<sub>20.0</sub>Cr<sub>20.0</sub>Ni<sub>20.0</sub>Co<sub>17.3</sub>C<sub>2.8</sub> (HEA-3C) high-entropy alloys have been investigated. After homogenization, HEA-0C and HEA-1C specimens possess a single-phase austenitic structure. Carbon alloying contributes an increase in the crystal lattice parameter: from 0.3598 nm in master material to 0.3611 nm and 0.3612 nm in HEA-1C and HEA-3C, respectively. Despite the high carbon concentration in Cantor alloy containing 3 at. %C, its crystal lattice parameter slightly differs from that for the HEA-1C. The HEA-3C specimens possess complex microstructure: solid-solution-strengthened austenite and precipitates. A portion (about 1 at. %) of carbon is soluted in austenitic phase, but the rest carbide atoms are bonded in carbides. All alloys show a strong temperature dependence of the yield strength (YS), ultimate tensile strength (UTS) and elongation-to-failure (EL). HEA-0C possesses the highest mechanical properties in low-temperature deformation regime (YS=385 MPa, UTS=950 MPa and El=97 % at T=77 K). With an increase in the test temperature up to 483 K, there is a decrease both in El (53 %) and in YS (130 MPa). Carbon alloying with 1 at. % significantly increases the YS and UTS (YS and UTS are 720 and 1320 MPa at 77 K; 280 and 730 MPa at 473 K, respectively) and reduces the El of the alloy (62 % at 77 K to 47 % at 473 K). Increasing the carbon concentration up to 3 at. % contributes further increase of the strength properties (YS=830 MPa at 77 K; 315 MPa at T=473 K), but HEA-3C specimens have the smallest value of EL, which varies from 30 to 40 % in the temperature range of 77-473 K. The dislocation arrangement of the HEA-0C after tensile tests to fracture showed that the dislocation slip is the main deformation mechanism of the alloys. Analysis of the HEA-1C and HEA-3C indicates that carbon alloying does not influence the deformation mechanism and dislocation glide mode of the high-entropy alloy. Deformation diagrams for carbon-alloyed specimens are more parabolic at low strains in comparison with deformation behavior of carbon-free alloy. All alloys are ductile after tensile tests both at 77 K and 297 K – numerous dimples are observed on the fracture surfaces. In HEA-3C large carbide particles are observed in the large dimples. Therefore, large incoherent carbides do not affect the micromechanism of fracture in the HEA-3C alloy but reduce the total elongation of the alloy.

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# SYNTHESIS AND INVESTIGATION OF TiC-Ni AND TiC-Ni-Co CATALYSTS FOR CO<sub>2</sub> HYDROGENATION TO METHANE

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Catalytic hydrogenation of CO<sub>2</sub> to higher hydrocarbons has attracted much attention as a promising way to utilize CO<sub>2</sub> and produce fine chemicals. It is of interest to search for new methods for the synthesis of highly active catalysts on a ceramic substrate for hydrogenation processes.

The production of TiC and TiC-Ni by the SHS method has been well studied[1,2]. Also, highly efficient polymetallic hydrogenation catalysts without support were obtained[3,4]. In this work, was made an attempt to combine these two directions and get a highly effective catalyst with a metal active phase on a ceramic support.

A series of precursors of the composition TiC-Ni, TiC-NiAl, TiC-(Ni,Co)Al was synthesized by the SHS method from granulated mixtures. The catalysts obtained from them were characterized by XRD and BET methods. The catalytic activity in the process of CO<sub>2</sub> hydrogenation to methane was studied in the temperature range 150–400°C using model gas mixtures with different ratios of CO<sub>2</sub> to hydrogen. The maximum conversion of CO<sub>2</sub> reaches 65–69% for different samples. Selectivity for methane is 100% in the studied temperature range.

The obtained results indicate the prospects for further research in this area.

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# RESEARCH OF STABILITY OF HIGH-ENTROPY ALLOYS PRODUCED BY MECHANICAL SYNTHESIS

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The question of the stability or metastability of high-entropy alloys (HEAs) was raised shortly after the discovery of HEAs [1]. The prospects for the practical use of HEAs directly depend on the solution of this problem. Stabilization of a multicomponent solid solution due to the entropy factor is more pronounced at high temperatures and decreases as the temperature decreases; therefore, at moderate temperatures, one can expect the HEA to decay. But with a decrease in temperature, diffusion processes slow down sharply, which slows down or even stops the nucleation and growth of new phases. Due to this, the metastable phases of HEA can remain unchanged for a long time, that is, they can be stable from the point of view of the practical application of materials and products made from them [2]. Literature data on the results of medium and high temperature annealing of HEAs for a relatively short time, up to several hours, are contradictory [3].

In this work we present the results of a very long (up to 204 days) continuous isothermal annealing of the HEAs CoCrFeNiX, where X = Al, Ti, Cu, or Mn. All compositions were obtained by mechanical alloying in planetary mills. Annealing was carried out in vacuum at three temperatures: 600°C, 800°C, and 1000°C. The obtained samples were studied by XRD, SEM, EDS, TEM, and other methods.

Phase, structural and microstructural transformations occurred in all alloys during long-term annealing. The strongly disordered and partially amorphized crystal structure of the mechanically synthesized HEAs becomes more ordered; secondary intermetallic phases precipitate, and the corresponding change in the local chemical composition of the five-component matrix phase, which remains a high-entropy alloy, occurs during the first day of annealing. The HEA crystal lattice parameter decreases; in some alloys two fcc phases with very similar crystal cell parameters appeared. The evolution of the microstructure (collective recrystallization, grain growth), as well as the change in the ratio of the fcc and bcc phases, can continue throughout the long annealing.

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# MICROSTRUCTURES AND PROPERTIES OF HIGH-ENTROPY ALLOYS CoCrFeNiX<sub>x</sub> (X<sub>x</sub> = Al, Cu, Ti): A MOLECULAR DYNAMICS STUDY

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In recent years, a comprehensive experimental and theoretical study of high-entropy alloys (HEA) has been carried out to determine their structure, the nature of their properties, and methods of synthesis [1]. In this work, the structure and properties of the HEA obtained from the melt for CoCrFeNiX<sub>x</sub> (X<sub>x</sub> = Al, Cu, Ti) systems will be studied by the method of molecular dynamics simulation.

For molecular dynamics simulations, the embedded atom method (EAM) was chosen. It was chosen because EAM describes well the interaction between metals at the atomic level. The interaction potential was developed according to the method given in [2]. The data for the elements Co, Fe, Ni, Al, Cu, Ti were taken from [2] and for Cr from [3]. Molecular dynamics simulations were carried out using the Lammmps software package.

The simulated system composed of five thin layers of different metals from the list above in an equimolar ratio. The typical initial dimensions of the system were 87.5 nm × 87.5 nm × 1.4 nm and amounted to 1,000,000 atoms, that is, exactly 200,000 atoms of each type. Periodic boundary conditions were applied to all three axes. This layered system was heated to a temperature above the melting temperature in the NPT ensemble and kept in it until a completely mixed structure was achieved. This was followed by gradual cooling of the system to 300 K consisting of alternating stages of temperature decrease by 10 K in the NPT ensemble and stages of system relaxation in the NVE ensemble, each with a duration of 25 ps. The cooling rate was thus 0.2 K/ps = 2 · 10<sup>11</sup> K/s. Such a high cooling rate is due to the computational complexity of molecular dynamics simulation: on a 32-core computer for a system of a million atoms, one nanosecond of calculation takes two days of real time. Fragmentation of a multicomponent melt into separate components was not observed for any of the systems. Each system formed its own HEA structure. For the CoCrFeNiCu system, a crystal lattice of the FCC type is formed. Crystallization starts from several centers at once, which is due to the high cooling rate. The grain boundaries are clearly visible. In the CoCrFeNiAl system, a crystal structure is visible, but it is difficult to attribute it to one of the simple types (BCC, FCC, HCP). The structure obtained for the CoCrFeNiTi system differs the most: the crystal structure has the form of a solid solution.

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# STRUCTURE AND MECHANICAL PROPERTIES OF Al – Ti – Zr – V – Nb HIGH – ENTROPY ALLOYS OBTAINED BY ALUMATHERMY

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Two decades ago, a completely new concept appeared in the metallurgical field, it made possible to obtain a new type of materials – high-entropy alloys [1]. Since the development of high-entropy alloys, the number of components in them has gradually increased, and their ratio has varied from 5 to 35 at. %. It should be noted that high-entropy alloys have unique physical and mechanical properties, in many ways superior to known superalloys such as Inconel 718 and Haynes 230 [2]. This paper presents the results of obtaining a high-entropy Al – Ti – Zr – V – Nb alloy by the method of aluminothermic reduction of metals from their oxides ( $\text{TiO}_2$ ,  $\text{ZrO}_2$ ,  $\text{V}_2\text{O}_5$ ,  $\text{Nb}_2\text{O}_5$ ) [3-4].

The resulting alloy was studied by X-ray spectral microanalysis (XSMA), X-ray phase analysis (XPA). The microhardness of the samples was measured using the Vickers method. The load and exposure time did not exceed 200 kgf/mm<sup>2</sup> and 15 seconds, respectively.

The chemical composition of the alloy is determined to be equal to (at. %): 26.28Al-15.92Ti-8.23Zr-26.57V-23Nb. According to the XPA, the main phase is a solid solution  $\text{Ti}_{0.64}\text{V}_{0.52}\text{Al}_{0.84}$ . The  $\text{ZrVAl}$ ,  $\text{TiAl}$ ,  $\text{Ti}_{1.5}\text{Zr}_{1.5}\text{V}_2\text{Al}_3$ ,  $\text{Nb}_3\text{Ti}_2\text{Al}_3$  phases are also identified. The composition of the latter was confirmed by a full-profile analysis using the Rietveld method using as an initial model the structural parameters of the compound  $\text{Ta}_5\text{Al}_3$ , as closer than  $\text{Zr}_5\text{Al}_3$ . The microhardness of the alloy was determined to be 647.87 HV, which is 1.25 times higher than that of the alloy obtained by the vacuum-arc method from pure components [5].

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## SYNTHESIS OF SINGLE-PHASE NbSi<sub>2</sub> AND Nb<sub>5</sub>Si<sub>3</sub> IN THE MODE OF THERMALLY COUPLED SHS PROCESSES

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Currently, silicides are widely used in various fields of science and technology for the implementation of special technological processes and the creation of materials with the necessary properties. Materials based on niobium silicides have been actively considered in recent years as a replacement for heat-resistant alloys in high-temperature structures, due to the highest melting temperatures and lower density than those of nickel-based heat-resistant alloys in aviation technology. Silicides were chosen as the main material for the blades of high-pressure turbines. There are attempts to use niobium–silicon-based composites for the production of additives for additive technologies. The niobium–silicon system is considered a low-energy system and it is considered impossible to carry out self-propagating high-temperature synthesis without any preliminary preparation in the form of separate heating or mechanical activation.

In this work, from powder mixtures Nb+37.7 wt. %Si (NbSi<sub>2</sub>) and Nb+15.36 wt. % Si (Nb<sub>5</sub>Si<sub>3</sub>) cylindrical samples with a diameter of 12 mm, a weight of 5 g and a height of 14.5 and 12 mm, respectively, were formed by unilateral pressing. The initial reagents were powders Nb (particle size less than 45 microns) and Si (particle size less than 45 microns) with a frequency of at least 99.9%. Hollow cylinders with a diameter of 30 mm, a mass of 20 g and a height of 15 mm were formed from the powder mixture Ti+0.6 Si. Cylindrical holes with a diameter of 12.2 mm were formed along the axis of the samples, into which Nb–Si samples, wrapped in filter paper (d – 200 microns), were placed. Control of combustion temperature Nb c Si used tungsten-rhenium thermocouples with a junction diameter of 100 microns. Thermocouples were placed to a depth of 3 mm from the lower end of the samples. The synthesis was carried out in an argon medium at a pressure of 1 atm. The initiation of combustion samples from a mixture of Ti+ 0.6Si was carried out by a heated tungsten spiral from the upper end.

As a result of the experiments carried out, it was possible to carry out self-propagating high-temperature synthesis and obtain synthesized niobium silicides. The reaction products were easily daubed samples, the composition of which, according to X-ray phase and X-ray structural analysis, corresponds to single-phase products NbSi<sub>2</sub> and Nb<sub>5</sub>Si<sub>3</sub> of hexagonal structure. The analysis of the structure by X-ray phase and microanalysis of the obtained products showed the formation of single-phase products NbSi<sub>2</sub> and Nb<sub>5</sub>Si<sub>3</sub> hexagonal structure.

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**SHORT-RANGE ORDERING AND ITS EFFECTS ON MECHANICAL PROPERTIES  
OF HIGH-ENTROPY ALLOYS**

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High Entropy Alloys (HEAs), which are typically composed of five or more components in nearly equal concentrations, have attracted much attention for their unusual properties such as high hardness, good oxidation resistance, corrosion resistance, and outstanding structural stability [1, 2]. The properties of these alloys with a complex composition can be controlled over a wide range by changing the type and concentration of their components [1–3]. At an early stage of their appearance, HEAs are usually considered as random solid solutions, and their constituent atoms are randomly distributed over accessible lattice sites. However, more and more studies [4, 5] show that the arrangement of atoms in the HEA is not an ideal disordered state due to the variety of atomic radii and complex interactions between constituent elements. The inevitable development of structural stabilization processes already during solidification and/or heating usually leads in multi-element alloys to a change in the order of the arrangement of atoms. As a result, the observation of short-range order and its possible influence on the properties of alloys is increasingly being reported [6-9]. Controlling the short-range order in such alloys can be an effective way to form the HEA properties. However, the formation, characterization, and modeling of short-range order in these compositionally complex solid solutions are still far from being fully understood due to the chemical and structural complexity of such objects, as well as its effect on physical and mechanical properties. The report will consider modern approaches to the methods of research and modeling of short-range order, features of the effect of composition on its formation, deformation mechanisms and properties of alloys. Attention will also be paid to the prospects for future research.

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## **DEVELOPMENT OF THE SCIENTIFIC BASIS FOR HIGH-ENTROPY ALLOY COATING BY LASER CLADDING AND BY COMBINED ADDITIVE TECHNOLOGIES**

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The research is aimed at the development of scientific bases for the production of new structural coatings based on high-entropy alloys by methods of additive technologies (primarily by laser cladding and by combined additive technologies), as well as the development of fundamental bases for the processing and operation of such coatings. It was necessary to conduct a theoretical and experimental study of physical and chemical processes occurring during coatings formation based on high-entropy alloys using high energy impact, as well as the study of the properties of the obtained coatings and how these properties affect the parameters of the coating formation processes.

The following methods and approaches were used for the research:

1. Experimental manufacturing of the samples with the coatings by different methods under variation of the production conditions (compositions and geometrical characteristics of the powders, the energy and temperature characteristics of the coating formation).

2. Experimental study of the composition and structure of the coatings (by electron microscopy, X-ray microanalysis and X-ray phase analysis). The distribution of various elements in the microstructure of the coatings and their phase composition were investigated.

3. Experimental study of phase stability in the coatings. It was performed by DTA methods to determine the temperature limits of the phase stability of the obtained coatings based on HEA. In addition to the DTA study, phase stability was investigated in the course of experiments on long-term annealing of the obtained coatings in order to determine how many hours of exposure of samples at temperatures from the range of 800-1200 °C affects the elements distribution and the phase composition of the coatings.

4. Thermodynamic and kinetic modeling of the processes of high-entropy phases formation and their evolution in possible service conditions. In this part of the study, modern approaches were used for modeling that allows the implementation of modern software. In particular, thermodynamic modeling was performed using the methods developed within the CALPHAD approach. Thermo-Calc Software (including DICTRA and TC-PRISMA kinetic simulation software) and FactSage Software were used for this work. The simulation results allowed us to draw the conclusions necessary to improve the compositions of the used powders.

5. The results of the experiments were analyzed and compared with the literature data, as well as with the results of the simulation to determine the ways to optimize the coating processes, and determine the possibility of their optimal application.

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# MECHANICAL PROPERTIES AND HEAT RESISTANCE OF NiAl BASED ALLOYS PRODUCED BY CENTRIFUGAL SHS – CASTING

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Nickel aluminide is a promising heat-resistant alloy. The disadvantage of these alloys is low mechanical strength and ductility at room temperature, which leads to insufficient manufacturability during machining and the risk of fracture. In order to increase the fracture toughness, the following plasticizing additives are introduced into the alloys: Cr, Co, Hf, Mo, Nb, et al. At the same time, alloying additives affect the oxidation resistance of the alloy, which is an important parameter of the hot gas path parts. It was shown the possibility of enhancing the mechanical properties of the  $\beta$ -alloy at room temperature [1]. The ultimate compressive strength and offset yield strength of the cast alloy with 15% Mo the following values:  $\sigma_B = 1728 \pm 30$  MPa,  $\sigma_{0.2} = 1566 \pm 30$  MPa [2]. Heat treatment made it possible the further increase of strength and ductility:  $\sigma_B = 1916 \pm 30$  MPa,  $\sigma_{0.2} = 1634 \pm 30$  MPa. Alloying with Re significantly effects to microstructure modifying and thereby increase the strength properties to the values:  $\sigma_B = 1800 \pm 20$  MPa,  $\sigma_{0.2} = 1618 \pm 30$  MPa. Heat treatment provided an additional increase in ultimate tensile and yield strength till  $\sigma_B = 2267 \pm 30$  MPa,  $\sigma_{0.2} = 1740 \pm 30$  MPa. The influence of additives on the oxidation resistance of  $\beta$ -alloy obtained by centrifugal SHS-casting, elemental SHS, HIP, SLM methods was reported. Additive of Zr provides the best heat resistance due to the formation of  $Al_2O_3$ -based protective layer with nanoscale precipitates  $Zr_5Al_3O_{0.5}$  which block the oxygen and nitrogen diffusion. The effect of elements (Mo, Zr, Ta, Re, Ru, Ti) has a selective contribution on microstructure, mechanical properties as well as oxidation kinetics and mechanism.

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## PRODUCTION OF CAST HIGH-ENTROPY ALLOYS BY CENTRIFUGAL SHS-METALLURGY DISPERSION-HARDENED IN-SITU

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A distinctive feature of HEA, in addition to the multicomponent composition, is the formation of a predominantly single-phase thermodynamically stable solid solution, mainly with a face-centered cubic (fcc) or body-centered cubic (bcc) lattice [1]. Then, with the intensive development of this scientific direction, the studied metal compositions were continuously expanded, and in the structure of the HEA with the participation of active metals (Al, Ti, Zr, etc.), dispersion precipitates of the nanosized and micron range were already observed. The observed secondary phases, as a rule, were formed on the basis of double metal intermetallic compounds and the Laves phases.

Relatively recently, small additions of nonmetallic elements (C, B, N) began to be introduced to control the microstructure of the resulting HEAs [2, 3]. The concept of obtaining materials based on HEA has already expanded to the production of multicomponent high-entropy compounds (carbides, borides, intermetallics) [4].

Within the framework of work was successfully completed, including experimental testing of the production of cast HEAs based on: (1) the transition metals (Kantor alloy) and (2) the refractory metals using SHS- metallurgy [5].

Various HEA doping systems were considered directly in the process of synthesis (in-situ SHS) of cast HEAs. Three systems were investigated: 1-(Co-Cr-Fe-Ni-Mn) / Ti(Cr)-Si-B; 2-(Co-Cr-Fe-Ni-Mn) / Mo(Nb)-Si-B; 3-(Nb-Mo-Cr-V-Ti / Mo(Nb)-Si-B.

An analysis of the microstructure data of the obtained cast HEAs showed that all the target elements are present in the composition of the obtained materials and are relatively evenly distributed throughout the volume of the ingot, which ensures a uniform structure. On the stage of analysis of the structural components on the element distribution map, it was revealed that the HEAs consist of metal-matrix with precipitation of new structural elements based on borides or silicoborides Mo and Nb is observed. As the modifier elements increases, a change in the concentration and morphology of dispersed precipitates in the matrix material (HEA) is observed. A further increase in the concentration of the introduced additive leads to noticeable changes in the structure, which has a pronounced composite structure, which may be of practical interest.

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## TITANIUM POWDER METALLURGY. QUO VADIS?

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Technically pure titanium and titanium alloys are one of the most advanced and promising materials of modern technology, which successfully combine high corrosion resistance in most liquids and gases (including at elevated temperatures), high biocompatibility with human tissues, with a simultaneous weak bactericidal effect, low specific gravity (almost 2 times lower than steels), high mechanical strength and ductility, which can be easily controlled by alloying, pressure treatment, hydrogenation. A semi-finished product of titanium metallurgy – a sponge – is used not only for processing into ingots, rods, shaped rolled products, and other blanks, but also for obtaining relatively inexpensive (compared to spherical sprayed or electrolytic) spongy powders.

The thesis shows the formation and perspective directions of development of titanium powder metallurgy history. The first is a reduction in the cost of the initial powder obtained by the Kroll method. The TiROTM process has been developed, which is a kind of magnesium-thermal reduction and has a high productivity due to the reaction in a fluidized bed, and by introducing crushed aluminides, vanadium-containing ore concentrates into the fluidized bed, it becomes possible to immediately obtain homogeneously alloyed powders of titanium  $\alpha$ - $\beta$  alloys Ti-6Al-4V. The ITP/Armstrong Process has been developed, through which a stream of titanium tetrachloride  $\text{TiCl}_4$  ejects vapors of a  $\text{Na}_2\text{CO}_3$  solution, interacting with drops of which releases titanium. According to the technology, gaseous chlorides of other metals ( $\text{AlCl}_3$ ,  $\text{VCl}_4$ ) can be supplied to the ejector, which makes it possible to immediately obtain homogeneous powders of titanium alloys of the Ti-6Al-4V type in this process. The CHIP-process (CIP-Sinter-HIP) has been developed. Its essence is that a work piece is obtained by cold isostatic pressing, which is then sintered. The sintered billet is subjected to additional densification by hot isostatic pressing, extrusion or forging. The properties of the finished product comply with the requirements of the AMS 4928 standard. The process has been developed for direct rolling of a titanium charge (both homogeneously alloyed and in the form of a mechanical mixture). The powder is pre-rolled into a green sheet, which is then heated and rolled hot in a protective atmosphere. After annealing, the sheet is rolled again. The process is carried out continuously. The mechanical properties of the finished sheets are such that they can be used to enhance the protection of armored vehicles. In the field of porous materials from titanium powders, a number of original technologies have been developed in Belarus: electric pulse sintering; sintering a bidisperse mixture of spongy powders, a mixture of spongy and spherical powders; self-extinguishing SHS process initiated by an electric discharge, forming a porous composite with a ceramic surface and a metal frame.

The thesis also presents other innovations in this area.

# INFLUENCE OF Hf, Mo AND W DOPING ON THE MICROSTRUCTURE, MECHANICAL PROPERTIES AND OXIDATION RESISTANCE (TaTiNbZrX)C HIGH ENTROPY CERAMICS

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High-entropy carbide ceramic materials have recently attracted more and more attention due to the peculiarities of their properties. A large number of studies are being carried out to assess the prospects for use in the aerospace and nuclear industries. A significant improvement in performance characteristics can be achieved by changing the composition and concentration of elements of high-entropy carbides. Modification of the chemical composition within the conditions for the formation of a high-entropy structure will allow the necessary characteristics. HEC based on the composition of (TaTiNbZrHf)C shows fairly good resistance to oxidation at various temperatures [1] and even in the composition of a ceramic matrix composite [2]. However, for high-entropy carbide ceramics, the effect of composition on properties and oxidation resistance has been studied to a much lesser extent.

In this work, HEC powders were obtained by the method of self-propagating high-temperature synthesis and sintered by spark plasma sintering of bulk samples of compositions (TaTiNbZrX)C, where X= Mo, W, Hf. For the synthesis, pure powders of refractory metals and graphite, which are part of the HEC, were used. The SHS products were sintered in a SPS apparatus at temperatures up to 2000°C in a vacuum and argon atmosphere under uniaxial pressing with a force of up to 50 MPa. The microhardness values of the studied compositions ranged from 22.6 to 25.7 GPa (at 30 N), the maximum hardness value (25.7 GPa) was shown by the composition (TaTiNbZrHf)C. The study of static oxidation curves at 1200 °C showed that HEC oxidize according to a power law and a linear law, for each of which a function is determined that predicts behavior during prolonged exposure to an oxidizing environment. Based on SEM and XRD analysis of oxidized layers, the mechanism of oxygen diffusion and the limiting stages for each of the compositions were determined. Synchronous thermal analysis made it possible to more thoroughly study the process of HEC oxidation, as well as to determine the multi-stage process and calculate the activation energies for each stage.

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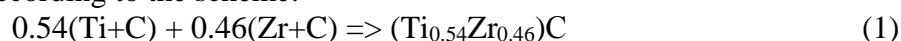
## PREPARATION of the TiC-ZrC COMPOSITES by ETE UNDER PRESSURE

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The ultra-high temperature composites are widely used in modern technologies of mechanical engineering, metalworking, and nuclear energy, aerospace. They include solid solutions based on titanium and zirconium carbides, having an extremely high melting point (more than 3000 °C), high mechanical strength, oxidant resistance and cyclic thermal loads.

The present paper the results of preparation by an electro-thermal explosion (ETE) under pressure ultra-high temperature composites based on the TiC–ZrC system. The exothermic synthesis was carried out according to the scheme:



The reaction mixture was prepared from titanium powders (PTM grade), zirconium (PCrK-1 grade) and carbon (P804-T grade) in the AGO-2 planetary mill.

It was shown that the optimal condition for preparation of a reaction mixture is a two-stage mechanical activation. At the first stage, the mixture of titanium and zirconium powders was activated for 10-90 min in a hexane in a high energy ball milling. In the second stage, carbon black was added to the prepared mixture and mixed for four minutes.

The resulting mixture was placed in a reaction mold and exothermic synthesis was carried out in the ETE mode under a pressure of 100 MPa. The effect of activated mixing (MA) duration on formation of phase composition and microstructure of the composites was studied.

The results of X-ray analysis showed that the phase content of composites depend on activated mixing duration. At ETE mixture activated for 10 minutes synthesized product contains three carbide phases:  $\text{Zr}_{0.14}\text{Ti}_{0.86}\text{C}$ ,  $\text{Zr}_{0.52}\text{Ti}_{0.48}\text{C}$  and  $\text{Zr}_{0.74}\text{Ti}_{0.26}\text{C}$ . Increasing the MA duration to 40 minutes leads to formation the single-phase solid solution  $\text{Zr}_{0.5}\text{Ti}_{0.5}\text{C}$ . At ETE mixture activated for 90 min composite contains two solid solutions:  $\text{Zr}_{0.14}\text{Ti}_{0.86}\text{C}$  and  $\text{Zr}_{0.74}\text{Ti}_{0.26}\text{C}$ . The difference of the phase composition composites is linked with duration of MA the initial powders and the ETE temperature.

The results obtained are consistent with the microstructural analysis data. It was found that when activated for 40 min, the average grains size is 3-5 microns, and at 90 min – 0.1–0.2 microns. The micro hardness of the composites synthesized from the mixture MA for 40 minutes is 13.2 GPa. When the composite syntheses from the mixture MA for 60 minutes – 18.53 GPa. It is important to note that the composite with submicron structure containing the  $\text{Zr}_{0.16}\text{Ti}_{0.84}\text{C}$  and  $\text{Zr}_{0.73}\text{Ti}_{0.27}\text{C}$  phases was formed directly during at the ETE.

Thus, for the first time, a dense ultra-high temperature composite based on the TiC–ZrC system with a submicron structure was synthesized from a mechanic activated mixture of titanium, zirconium and carbon black.

## THE EFFECT OF NITROGEN ON THE STRUCTURE AND MECHANICAL PROPERTIES OF THE Fe<sub>40</sub>Mn<sub>40</sub>Cr<sub>10</sub>Co<sub>10</sub>-BASED ALLOYS

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High-entropy alloys (HEAs) with a face-centered cubic (FCC) structure are currently considered as promising structural materials. The Co-Cr-Fe-Mn-Ni system alloys demonstrated encouraging properties, for example, high ductility and fracture toughness at room and cryogenic temperatures, but generally they have low strength. The Fe<sub>40</sub>Mn<sub>40</sub>Cr<sub>10</sub>Co<sub>10</sub> alloy is particularly interesting as a single-phase solid solution with impressive mechanical properties. Thermomechanical processing can be effectively used to tailor the microstructure and properties. In addition, alloying with interstitial elements, in particular nitrogen, can lead to significant hardening. Therefore, in this work the effect of nitrogen content (0; 0.5; 2.0 at.%) on the structure and mechanical properties of the Fe<sub>40</sub>Mn<sub>40</sub>Cr<sub>10</sub>Co<sub>10</sub>-based alloys after thermomechanical processing was studied.

The Fe<sub>40</sub>Mn<sub>40</sub>Cr<sub>10</sub>Co<sub>10</sub> alloy had dual-phase structure with FCC matrix and sigma phase precipitates, while N-doped alloys had single-phase FCC structure. Nitrogen alloying increases both strength and ductility at room temperature. Decrease in the testing temperature results in the pronounced increase in strength of the alloys. Annealing of the Fe<sub>40</sub>Mn<sub>40</sub>Cr<sub>10</sub>Co<sub>10</sub> alloy at 700 C in resulted in a formation of a partially recrystallized structure with high amount of a  $\sigma$ -phase. Sigma phase fraction decreased with the increase in the annealing temperature. The addition of nitrogen results in the decrease in the volume fraction of the  $\sigma$ -phase in the Fe<sub>39.5</sub>Mn<sub>40</sub>Cr<sub>10</sub>Co<sub>10</sub>N<sub>0.5</sub> alloy and some M<sub>2</sub>N type nitrides were found in Fe<sub>38</sub>Mn<sub>40</sub>Cr<sub>10</sub>Co<sub>10</sub>N<sub>2</sub> alloy after annealing at 700°C. After annealing at higher temperatures, the alloys had a single-phase FCC structure. The strength of the alloys increases with increasing nitrogen concentration and decreases with increasing annealing temperature. The microstructure-mechanical properties relationships and effect of nitrogen strengthening mechanisms is discussed.

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## TECHNOLOGY OF OXID-FREE HEATING OF CHIP-POWDER DISPERSIONS OF FERROUS METALS IN HOT BRIQUETTING FURNACES

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Heating and briquetting of metalworking wastes for the purpose of cleaning from coolant and remelting is associated with the issues of protecting the metal from oxidation, reducing waste and obtaining high-quality castings. In the technology proposed for implementation, heating is carried out in small-sized continuous muffle furnaces, the distinguishing feature of which is preheating in a gas burner flame to a temperature of 450...550 °C, then in a low-frequency inductor to a temperature of 700...800 °C. In this case, the metal is heated in an atmosphere of products of thermal sublimation and pyrolysis of the coolant without the access of an oxidizer with the release of coolant vapor into the burner flame. The pyrocarbon coating 0.1-0.2 mm thick formed on the surface of metal particles, in addition to protecting the metal from oxidation, acts as a lubricant in the hot pressing process. The high-calorific oil component of the coolant, entering the furnace furnace, burns together with natural gas.

The chips enter the induction heater in a practically dry form – the oil content is 4-5 wt.%. The charge is heated by direct contact heat exchange with the walls of the muffle, radiation and convection in the space of the muffle, which is especially effective when heating paramagnetic and non-magnetic materials that are part of metallurgical composites. Metal particles are also subject to electromagnetic influence. Due to the fact that contact heat exchange plays the main role in the heating process, the use of an energy-saving low-frequency inductor leads to significant energy savings. The metal comes out of the furnace completely free of coolant.

The economic efficiency of hot briquetting is ensured by reducing the cost of electricity for the operation of the briquetting press, due to a decrease in the resistance of the metal to deformation by 2.0-2.2 times. The cost of natural gas is 10-12 times lower than the cost of electricity, and the content of the oil component of the coolant in the composition of the charge ensures a reduction in natural gas consumption by up to 30%. The formation of a pyrocarbon coating (lubricant) on the surface of metal particles reduces the work of a deformation seal by 10-15%. The release of solid carbon reduces the total amount of furnace gases and simplifies the task of their complete combustion.

The economic efficiency of hot briquetting increases many times with the use of sludge powders in the composition of the chips, as well as alloying additives in the production of composite charge materials of a given chemical composition.

The technological module of hot briquetting, in addition to the pressing equipment, includes two small-sized heating installations that do not require significant costs for heating at the beginning of the shift. Their total productivity (hour / day / year) – 3t / 20t / 6000t corresponds to the performance of the briquetting press. The design of the heating devices excludes the emission of flue gases into the atmosphere. Exhaust gases enter the wet cleaning unit, condensate collection and oil products regeneration.

# INFLUENCE OF MECHANICAL ACTIVATION ON COMBUSTION PARAMETERS OF POWDER COMPACTS OF HF-PTFE AND HF-PTFE-AL SYSTEMS

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Reaction materials are mixtures of metallic and non-metallic powders that, when subjected to high-speed impact or heating, release a large amount of thermal energy. These materials are used in the military field as cumulative liners and in the civil field for perforating oil wells. [1]. Promising reaction materials include an aluminum/polytetrafluoroethylene (PTFE) powder system with high-density additives (W, Hf, Ta, etc.). The aim of the work was to study the effect of mechanical activation on the combustion parameters of Hf-PTFE and Hf-PTFE-Al powder systems.

Hf (HFE-1), Al (ASD-1), and PTFE (Fluralite) powders were used in the work. The ISMAN-THERMO program was used to calculate thermodynamic characteristics in two- and three-component systems Hf-PTFE and Hf-PTFE-Al, where aluminum is used as an activating additive. Mechanical activation (MA) was carried out on an AGO-2 setup for 2 min at a speed of 2200 rpm. The ignition temperature ( $T_{ig}$ ) was determined on compacts with a diameter of 3 mm and a height of 1.5-2 mm. The combustion rates and temperatures of the compositions were studied on the parallelepiped-shaped compacts with dimensions 5 mm × 5 mm × 20 mm and a relative density of 0.95. Selected compositions, densities ( $\rho$ ), adiabatic combustion temperatures ( $T_{ad}$ ), ignition temperatures ( $T_{ig}$ ) before and after MA are presented in Table 1.

Table 1. – Selected compounds and their properties

No.	Compositions mas. %	$\rho$ , g/cm <sup>3</sup>	$T_{ad}$ , °C	$T_{ig}$ , °C before MA	$T_{ig}$ , °C after MA
1	65Hf–35PTFE–0Al	4.54	2381	680	650
2	62Hf–33PTFE–5Al	4.38	2642	680	570
3	58.5Hf–31.5PTFE–10Al	4.20	3327	660	590
4	55Hf–30PTFE–15Al	4.02	3368	690	630
5	52Hf–28PTFE–20Al	3.91	3127	620	620

It has been established that as a result of mechanical activation, the reactivity of the compositions increases. As evidenced by a decrease in the ignition temperature from 680 to 570 °C, an increase in the burning rate from 1.1 to 5.5 mm/s and temperature from 2100 to 2650 °C in composition No. 2. Mechanical activation improves the efficiency of the energy material due to the complete combustion of the components.

The study was financially supported by the Russian Foundation for Basic Research within the framework of the scientific project No. 20-08-00640-A

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## STRUCTURE AND MECHANICAL PROPERTIES OF A MEDIUM-ENTROPY TRIP ALLOY PRODUCED BY SELECTIVE LASER MELTING

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Recently, multicomponent alloys (high-entropy and medium-entropy (HEAs and MESs)) with close to equiatomic element amounts have attracted considerable attention caused by unusual mechanical properties. In particular, some MEAs demonstrate TRIP effect, which provides significant work hardening and, as a result, very high values of tensile strength and ductility; in some cases, decreasing temperatures to cryogenic conditions even improves these characteristics [1].

However, most of the studies were devoted to the study of HEAs and MEAs, including those with the TRIP effect, obtained by the casting. Selective laser melting (SLM), in comparison with to traditional manufacturing methods, enables obtaining complex geometry parts directly from powder, with control all production parameters. The formation of a fine-grained microstructure with a uniform chemical composition and a high level of mechanical properties can be obtained by high crystallization rate during SLM. Meanwhile, there are no systematic data on the effect of SLM on the structure and mechanical properties of carbon-containing MEAs with the TRIP effect.

In this work, we consider the structure and properties of a new TRIP MEA obtained by selective laser melting. The particle sizes of the spherical powder varied from 5 to 60  $\mu\text{m}$ . The samples were manufactured using the following regime: laser output power – 200 W; scanning speed – 1600 mm/s. The samples were produced under a nitrogen atmosphere to minimize oxidation. Surface defects were not detected on the samples. The alloy after SLM had a structure consisting of the both fcc and bcc phases similar to specimens produced by traditional melting. The average grain size after SLM was  $\approx 30 \mu\text{m}$ . After SLM, an increase in hardness from 180 HV to 220 HV is observed compared to the as-cast state. In addition, attractive mechanical properties were found during tensile tests at room and cryogenic temperatures. The relationship between the production parameters, structure and mechanical properties of the alloy is discussed.

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# **TECHNICAL CERAMICS (TC): PROBLEMS, PRINCIPLES AND MECHANISMS OF SCIENTIFIC AND PRACTICAL DEVELOPMENT AND IMPLEMENTATION IN PRODUCTION**

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From the position of the completed and implemented R&D (R&D) GPNI in the field of technical ceramics, a fundamentally objective analysis of the state of the main and secondary current problems of creating and applying specialized TC in various scientific and practical fields and industries was carried out; hypotheses and scientific ideas were put forward and analyzed, determining the choice of the path: achieving the main and additional goals and tasks; development of objects and subjects of research, strategies and tactics, methodology and logistics of research (minimizing financial, time and search costs). The concept of creating a TC (for various scientific and technical purposes) has been formed from the perspective of physico-chemical processes and mechanisms of structural engineering – structuring profiled materials science and technology objects (MTO) – materials – technologies – products – productions of structural, functional, electrical, ceramic – refractory, etc. purposes and, in particular, for energy, space, aviation and rocket engineering. The developed innovative MTO, based on oxide and oxygen-free chemical compounds – at the level of compositions from mineral powder systems, have passed production tests and quality testing at enterprises of the Republic of Belarus and the Russian Federation. RB – JSC "BMZ – UKH – "BMK", Zhlobin – ceramic supporting rollers for the production of metal cord and wire rod; double-layer corundum – dioxycirconium dosing cups (SD) for the milled CML; JSC "NPO Center" of the National Academy of Sciences of Belarus – refractory funnels for dosing into a centrifuge melts of cast iron, aluminum, black, stainless steels and alloys; RF – NPP "Izumrud", St. Petersburg – capillary – permeable diaphragms (efficiency) without and with thin-film (ion – plasma and magnetron) functional coatings for electrochemical reactors (ECR) energy treatment and structuring of water and aqueous solutions into physico-chemical reagents for household, agricultural, medical (antibacterial, antiviral) and other appointments; JSC "Gormash", Belgorod – high-temperature electrical insulators for vacuum furnaces (Ipson, Germany) of gas-thermal hardening (1050oCx10.5 hours) of drilling tools (loading weight – 2 tons) in the medium of dissociated acetylene; JSC OKB Fakel, Kaliningrad – high-energy fuel electrodes (thermoemitters) for space plasma jet engines and others.

## THE ISSUES AND RESULTS OF TECHNICAL CERAMICS PRODUCTS MANUFACTURING FOR INDUSTRY

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Technical ceramics is a relatively new type of ceramics and its annual output growth rates (from 15 to 25%) significantly exceeds similar indicators for metals. Due to its unique set of properties, it is widely used. Its main components are oxides, carbides, nitrides, solid solutions based on them, complex chemical compounds.

The solution of the producing high-quality ceramic materials-products problem requires an integrated approach and consideration of many factors at all stages of the technological chain. Due to the complexity of the transformations occurring simultaneously, the search for the desired result is impossible by solving optimization problems and requires numerous experimental studies. We have developed basic principles for the creation of ceramic materials-products, including: an analytical flowchart for the directional selection of ingredients for the creation of constructive, functional, refractory and other ceramic materials-products; the choice of optimal chemical compositions and granulometric composition of each of the components of the charge; activation; selection of shaping parameters and methods; temperature modes of sintering and final finishing processing.

In BNTU there was created a laboratory and technological area, developed technologies for creating about 20 new innovative specific ceramic materials-products based on oxide and oxygen-free chemical compounds which are widely used in the Republic of Belarus and the Russian Federation.

The keys of the technology are the choice of chemical composition, granulometric composition and the thermal sintering cycle. A particularly important point is the of nanoscale particles with a large number of structural defects receiving. Ones play a significant role in the sintering process, as a source of vacancies, while the action of the vacancy diffusion mechanism. It is found out that the particle size affects the features of the pressing process, shrinkage and density of the final product.

Abnormal behavior was observed in the region of relatively small particle sizes less than 70 microns. Compacts made of such compositions have maximum changes in density, porosity, size and mass during pressing and sintering. This should be taken into account at all stages of ceramic production.

One should note that predicting the results in the case of multicomponent state diagrams is much more difficult than for two-component ones.

Thus, the analysis of the state of the issue shows the necessity of comprehensive thorough approach at all stages of the technological chain from the selection of ingredients, their granulometric composition, methods of charge activation, molding, sintering to mechanical or other finishing processing within the framework of the implementation of a given program – methodical formula (scheme) "composition — structure – property –material projected".

# INVESTIGATION OF THE COMPOSITIONAL SPACE OF MULTICOMPONENT HIGH-ENTROPY CARBIDES VIA MACHINE LEARNING

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It is known that the use of ab-initio methods for calculating the properties of high-entropy carbides (HEC) with more than 5 metallic elements is difficult due to the impossibility of reproducing the heterogeneity of the atomic distribution with preserving of the homogeneity of the composition [1]. This problem can be solved by using machine learning (ML) to predict the properties of materials with a certain accuracy, when the first principles calculations is impossible or irrational [2, 3].

This work proposes a descriptor based on the properties of monocarbides in the composition of a multicomponent solid solution, that were used to create and verify the ML model for predicting the stability of the HEC, and then, to develop an active ML system to predict the mixing enthalpy, Young's modulus, hardness, and fracture toughness.

The known descriptor Entropy Forming Ability (EFA) [1] was selected as a predictable criterion for the stability. Data on 145 carbides of Hf, Nb, Ta, V, Ti, Zr, Mo, W, Cr containing simultaneously 3, 4 and 5 metallic components and the Materials Project database were used to build an active model that can generalize available information about compositions to predict the properties of HECs with more components.

The developed system was applied to predict the values of EFA, mixing enthalpy, Young's modulus, hardness, and fracture toughness of solid solutions of Hf, Nb, Ta, V, Ti, Zr, Mo, W, Cr monocarbides, i.e., 126 equimolar compositions based on 5 metals, 84 compositions based on 6 metals, 9 compositions based on 8 metals. The obtained results allow us to conclude that the obtained model makes it possible to correctly rank compositions according to the probability of formation of a solid solution, estimated within the framework of the entropy stabilization descriptor (EFA), and to determine the most probable range of mechanical properties for previously unexplored compositions.

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# STRUCTURE OF MULTICOMPONENT ALLOYS SYNTHESISED BY ARC MELTING

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Multicomponent high-entropy alloys are expected to be one of the emerging advanced structural materials [1]. This work presents calculated data on the values of the difference in the chemical elements atomic sizes ( $\delta$ ), the generalized thermodynamic parameter ( $\Omega$ ), the mixing enthalpy and entropy ( $\Delta H_{mix}$  and  $\Delta S_{mix}$ ) for the  $Ti_{20}Zr_{20}Hf_{20}V_{20}Nb_{20}$  alloy and its five various four-component modifications with an equiatomic composition. The calculation is made according to method described in [2]. According to the literature survey and calculation results the alloys were chosen for investigation. The ingots of  $Ti_{25}Zr_{25}V_{25}Nb_{25}$  and  $Ti_{20}Zr_{20}Hf_{20}V_{20}Nb_{20}$  alloys were obtained by arc melting in inert gas (Ar) chamber under the same conditions. For alloys structure characterization the X-ray phase analysis, metallographic and scanning electron microscopy examination were performed. According obtained data there was made conclusions about appropriateness of arc melting mode and possibility of single-phase structure formation for the alloys under consideration.

*The work was carried out within the framework of the state task of the IMET UB RAS.*

*The results were obtained using the equipment of the Ural-M Central Research Centre.*

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## DIFFUSION-HARDENING COMPOSITE MATERIAL Ga-In-Sn-Cu-Ti

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Traditional soldering requires the use of fluxes, which leads to contamination of the seam, and the temperature of soldering and soldering of such a seam are close. The connection at a temperature above 300 °C leads to the formation of internal stresses and a decrease in strength. The use of composite diffusion-hardening solders, including those based on gallium, makes it easier in many cases to solve technical issues of connecting dissimilar materials, to connect not only metal materials, but also glass, ceramics, quartz, etc., both among themselves and with metals [1]. The peculiarity of soldering with diffusion-hardening composites is the use of multicomponent fusible alloys and powders of various metals, after isothermal exposure (50-150 °C) they form new phases with significantly higher melting temperatures (solid solutions, intermetallic compounds, etc.). To improve the properties of such solders, solid powdered fillers are used, which leads to a change in properties composite. Studies have shown that repeated heat treatment at an elevated temperature close to the melting temperature leads to an increase in the mechanical properties of the composite. The basis of the composite material consisted of, %: Ga-20,1; Cu-66,4; In-6,2; Sn-7,3, to which 5, 10 and 15% Ti were added. The results of measuring the microhardness of the obtained compositions, depending on the composition and temperature treatment, are shown in the table.

Table – Microhardness of samples

№	Composite alloy	Heat treatment temperature, °C	
		150	500
		Microhardness, HV, GPa.	Microhardness, HV, GPa.
1	Base – 5% Ti	420	960
2	Base – 10% Ti	1330	1950
3	Base – 15% Ti	680	1400

*The work was carried out in accordance with the state task and plans of the Research Institute of IHTT of the Ural Branch of the Russian Academy of Sciences.*

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## SIALON-BESED HETERO-MODULUS CERAMIC COMPOSITES BY CS AND SPS

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Hetero-modulus ceramic composites (HMCCs) represent a ceramic matrix with a high Young's modulus (300–800 GPa) with inclusions of a phase with a markedly lower Young's modulus (15–20 GPa) such as  $sp^2$ -structured carbon or boron nitride. Unusually high tolerance of HMCCs to high-speed impacts and thermal shock is caused by their inherent ability to absorb and dissipate the elastic energy released during crack propagation and their capacity to blunt and divert a propagating crack [1]. SiAlON ceramics have high hardness, good strength, and excellent wear/corrosion resistance [2]. The h-BN ceramics possess such properties as high electric resistance, low friction coefficient, low wettability with molten metals and salts [3]. The HMCCs formed by SiAlON and h-BN are capable of combining these properties in desirable proportions and recognized as promising multifunctional materials for high-temperature applications. Up to now, the development of new simple and efficient methods for production of SiAlON–BN HMCCs is a hot subject of research.

In the present paper, the obtaining of SiAlON–BN HMCCs was experimentally investigated according two schemes based on combustion synthesis (CS) and spark plasma sintering (SPS): – direct infiltration-assisted CS in one stage under high-pressure nitrogen gas; – SPS of CSed raw powders. For both schemes, the key technological parameters and optimal conditions for obtaining HMCCs with controllable phase composition and high-density structure were determined. The main properties (hardness, modulus of elasticity, flexural and compressive strength, electrical conductivity, tribological characteristics, etc.) and its correlation with phase composition and structural parameters of obtained samples were established. The obtained SiAlON–BN HMCCs were found have extremely high resistance to thermal shock and corrosion in metallurgical melts. These properties are combined with their excellent machinability by conventional tools.

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## OBTAINING A COMBINATION OF HIGH STRENGTH, DUCTILITY AND CREEP RESISTANCE OF Ti<sub>2</sub>AlNb-BASED ALLOY

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The intermetallic alloys, based on the orthorhombic Ti<sub>2</sub>AlNb phase, has been receiving attention as potential materials for aircraft engine applications due to their high strength-to-weight ratio, greater fracture toughness, and better workability than conventional intermetallic alloys such as TiAl-based and Ti<sub>3</sub>Al-based alloys. For practical application of Ti<sub>2</sub>AlNb alloys, their ductility, toughness, strength and creep resistance have to be optimized by means of appropriate microstructural design. The microstructural design requires detailed knowledge of the relationship between microstructure and mechanical properties. In this work were studied the microstructure evolution during hot deformation and heat treatment, as well creep and tensile properties of Ti<sub>2</sub>AlNb-based alloy.

The Ti<sub>2</sub>AlNb-based alloy in hot rolled condition was subjected to recrystallization annealing and forging in ( $\alpha+\beta$ ) phase field. As a result, a homogeneous fine-grained structure was obtained. To form a colonial structure characterized by more optimal mechanical properties in a wide temperature range, a two-stage heat treatment was carried out. Combine the regimes of the first and second stages of heat treatment made it possible to obtain various combinations of mechanical properties. Dependences of mechanical properties on such microstructural parameters as grain size of  $\beta$ -phase and thickness of O-phase particles were plotted. The yield strength was sensitive to the thickness of O-phase particles, increase in the aging temperature led to a decrease in strength and an increase in ductility. The creep rate strongly dependent on the grain size of  $\beta$ -phase, the increase of which led to its decrease. The relationship between tensile properties, creep resistance of alloy and microstructural features, such as grain size of  $\beta$ -phase and thickness of O-phase particles was also discussed.

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## PROTECTIVE SHS-COATING FOR STEEL PRODUCTS

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Chemical inertness and thermal stability open up the possibility to use oxide bronzes as anticorrosive coatings. In recent years quite an extensive range of domestic and foreign materials that are declared to be highly effective for various working conditions has appeared in the Russian market. However, there is no data on protection efficiency and durability in real conditions, in particular in climatic conditions of Siberia and the Far North, for most of them.

In the present work, potassium titanium oxide bronze, obtained by the SHS method according to the procedure [1], was used as the base of the protective coating. The suspension of the coating components was applied to the steel surface by the method we patented earlier [2]. Then, we investigated a set of coating physicochemical characteristics.

The adhesion of the protective coating was controlled by a mechanical adhesimeter Constanta. The average adhesion value for the tested samples was 2.3 MPa.

To calculate the protective coating service life, climatic tests of coating were carried out: samples were placed in a humid atmosphere and kept at temperature  $t = 40 \pm 2$  °C and relative humidity  $97 \pm 3$  % for 2 hours, then the heating was turned off and kept in the chamber for 2 hours more. Then the samples were transferred to a low-temperature thermostat chamber and incubated at  $t = -30$  °C for 6 h. From the cold chamber, the samples were transferred to the drying cabinet and incubated at  $t = +60$  °C for 5 h. Then, the samples were transferred to the cold chamber and incubated at  $t = -60 \pm 3$  °C for 3 h. Then the samples were kept in the air at room temperature and relative humidity of no more than 80% for 6 hours. This constituted the first test cycle. Specimens were inspected after 15, 30, 50 and 100 test cycles.

According to a set of tests in an aggressive atmosphere, simulating a cold, moderately cold climate of the regions of the Russian Federation, it is noted that the protective coating after 100 test cycles does not show cracking, peeling, bubble formation and signs of corrosion of the base metal.

Service life prediction of the protective coating was carried out in accordance with the recommendations of State Standard 9.401-91 (Annex 11). It is calculated that the protective coating is able to provide corrosion protection of body steel in conditions of moderate-cold and cold climate not less than 18 years.

The corrosion resistance of the obtained coatings was evaluated by the rate of corrosion of the samples. The rate of corrosion was taken as the change in sample weight over time. It was shown that the corrosion rate of coated samples was three times less than the corrosion rate of uncoated steel.

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# THE EFFECT OF MICROSTRUCTURE PARAMETERS ON STRENGTH AND DUCTILITY OF $\beta$ -SOLIDIFIED $\gamma$ -TiAl BASED ALLOY

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$\gamma$ -TiAl based alloys are considered as promising high-temperature lightweight materials for the manufacturing of aircraft engines due to a favorable combination of specific strength, stiffness, and creep resistance [1]. To increase the room temperature ductility, alloying and refine structure were used [2, 3]. For instance, some alloying elements like boron, carbon, silicon or rare-earth elements allow to significantly refine structure of as-cast alloys [4, 5]. Colony size and interlamellar spacing can be controlled by heat treatment, including quenching from  $\alpha$ -phase field and subsequent aging at temperatures of ( $\alpha_2+\gamma$ )-phase field or slow cooling from  $\alpha$ -phase field to room temperature [6].

The effect of microstructure parameters on mechanical properties of the  $\beta$ -solidified Ti-44Al-2V-1Nb-1Cr-0,1Gd alloy under tension was studied. Ingot in the as-cast condition was subjected to thermomechanical and subsequent two-step heat treatment to obtained conditions with different parameters. The interlamellar spacing ( $\lambda$ ) was ranged from 70 to 1000 nm, remaining the same colony size of 70  $\mu$ m. The tensile stress-strain curves and the resulting mechanical properties were obtained. The alloy with  $\lambda \sim 150$  nm demonstrated the maximum values of ductility under tensile – 1.5 %. Both increasing and decreasing of interlamellar spacing resulted in significant drop in the ductility. The alloy with lower colony size of 15  $\mu$ m and  $\lambda \sim 80$  nm achieved much more strength and ductility, was 750 MPa and 4.5 %, respectively.

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**DEVELOPMENT OF BCC/B2-STRUCTURED HIGH ENTROPY ALLOYS**

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The so-called high entropy alloys (HEAs) have attracted considerable attention from the materials scientists worldwide due to their unique structures and properties. Although initial studies strongly emphasized the need for alloys with a single solid solution structure, later it was revealed that a better strength-ductility combination can be achieved in precipitation-strengthened alloys. Large progress has been made in the development of precipitation-strengthened HEAs composed of transition metals based on face-centered cubic (FCC) structure. Meanwhile, the precipitation-strengthened transition metals HEAs with a body-centered cubic (BCC) structure remain much less explored.

In the present study, the focus is made on the 3d transition metals HEAs with BCC structure. It was revealed that addition of Al to the non-equiatomic Fe-Mn-Cr-Ni HEAs with high Fe content resulted in (i) gradual transformation of the structure from FCC-based to BCC-based (ii) formation of the ordered B2 precipitates. As the result, in alloys with high Al percentage (10 at.% and more) dual-phase microstructure with BCC matrix with embedded nanoscale B2 precipitates was found. Addition of Ti has resulted in the formation of L2<sub>1</sub> phase. CALPHAD calculations correctly predicted FCC-BCC transition and B2 phase formation but not L2<sub>1</sub> phase formation. Therefore, high-throughput CALPHAD calculations were used to obtain new BCC/B2 structured HEAs with improved structure stability. The experimental verification confirmed improved stability of the BCC/B2 structure of the new alloys. Mechanical characterization has revealed that some of the alloys possess high strength at  $T \leq 600^\circ\text{C}$ .

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## USING THERMO-CALC SOFTWARE TO SIMULATE THE WELDING PROCESS OF HIGH-ENTROPY ALLOYS

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One of the understudied aspects worthy of consideration in the context of research of high-entropy alloys (HEAs) [1, 2] is their behavior when exposed to the thermal cycle of welding [3]. Information about the processes occurring in the welding process is necessary to solve the technological problems that can be expected during the introduction of HEAs into production.

As a rule, the assessment of weldability and the study of various welding methods of HEA is conducted by trial and error, based on the results of experimental laboratory studies [3]. This is a costly and labour-intensive way. At the same time, the use of theoretical methods, including methods of thermodynamic and kinetic modeling using modern specialized Thermo-Calc software, is useful for understanding the metallurgical processes in welding HEAs.

Thermo-Calc, among other things, makes it possible to calculate thermokinetic diagrams, which, depending on the heating temperature and cooling rate of the metal, predict the kinetic parameters of the phase transformation and the possible structure, on which the mechanical, technological and operational properties of the welded joint largely depend.

The task of our work was to construct thermokinetic diagrams for a number of HEAs based on the Cantor alloy. In the process of research, the capabilities of the built-in Thermo-Calc modules DICTRA and TC-PRISMA as well as the thermodynamic and kinetic characterization databases of TCHEA5 and MOBHEA2 were used. The results of the performed calculations are compared with the literature experimental data.

The results of the work demonstrate the productivity of the approach related to the construction of thermokinetic diagrams for HEAs by means of Thermo-Calc to reveal the dependences of the influence of thermal cycles of welding on the structure and mechanical properties of the HEA weld joint, which is useful for rational basis the choice of technology and modes of welding of HEA.

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# FABRICATION OF NOVEL REFRACTORY CARBONITRIDE CERAMICS FOR HIGH-TEMPERATURE APPLICATIONS BY COMBUSTION SYNTHESIS AND SPARK PLASMA SINTERING

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Carbides and nitrides of transition metals of IV and V groups are characterized by extremely high melting points, high mechanical properties, and high oxidation resistance. The synergy of properties makes these compounds promising candidates for protection of the most heat-loaded units and structures operating at temperatures above 2000°C. In addition, some transition metal carbides and nitrides may form a continuous series of solid solutions, which are superior in properties to binary compounds. For example, according to the studies of Savvatimsky et al. [1], the melting point of Ta<sub>0.80</sub>Hf<sub>0.20</sub>C double carbide is 4300 ± 80 K, which is higher than that of pure TaC and HfC. Theoretical calculations by Qi-Jun Hong and Axel van de Walle [2] showed that the incorporation of nitrogen atoms into the HfC and (Hf,Ta)C lattice will improve their mechanical and thermophysical properties. But there was no experimental confirmation.

In this work, UHTCs based on hafnium carbonitride Hf (C, N) and (Ta, Hf) CN were obtained by CS and SPS with a relative density above 98 % and a hardness of 21.3 and 19.4 GPa, respectively. The incorporation of nitrogen atoms into the HfC lattice contributed to an increase in the melting point from 4200 to 4300 °C, thermal conductivity by 75 %, and oxidation resistance by 40 %. Tantalum-hafnium carbonitride with a high hafnium content demonstrated higher oxidation resistance in a high-temperature gas flow (mass oxidation rate – 0.15 mg / s; linear oxidation rate – 0.8 μm/s) compared to tantalum hafnium carbide (0.67 mg/s, 2.38 μm/s).

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# INFLUENCE OF FRICTION STIR PROCESSING ON THE MICROSTRUCTURE, PHYSICAL AND MECHANICAL PROPERTIES OF A CU-CR-ZR ALLOY

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A Cu-0.3%Cr-0.5%Zr alloy (mass %) was chosen as the research material. The alloy was forged at a temperature of 900 °C to the total strain of  $\epsilon = 1$ . Then the alloy was subjected to supersaturated the solid solution at a temperature of 920 °C for 1 hour with cooling in water and aging at 450 °C for 1 hour. The plates of 3 × 70 × 95 mm were subjected to a friction stir processing (FSP). The processing tool was fabricated from tungsten carbide and consisted of a shoulder measuring 14.5 mm in diameter and a semi-spherical pin of 2.5 mm in diameter. The FSP had the following parameters: the tool rotation speed – 500 rpm; the tool travel speed – 2 mm/s. The processing temperature was measured using K-type thermocouples placed into the plate in closely proximity to the stir zone and reached ~600 °C.

An increase in electrical conductivity and hardness compared to the base material was observed after FSP in the heat-affected and the thermo-mechanically affected zones. The maximum electrical conductivity of 70.9% IACS was achieved in the stir zone, while the hardness in the stir zone decreased and approached the values of the base material of 115 HV. The maximum hardness of 145 HV was observed in the thermo-mechanically affected zone. The average grain size decreased from 17.3 in the base material to 1.04  $\mu\text{m}$  in the stir zone after FSP. An equiaxed grain structure with a fraction of high-angle boundaries of 0.87 was formed in the stir zone. In the thermo-mechanically affected zone chains of dynamically recrystallized grains with a size of about 1  $\mu\text{m}$  separated deformed regions of Cu-Cr-Zr alloy.

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# INVESTIGATION OF THE EFFECT OF ALUMINUM ON THE STRUCTURE AND MECHANICAL PROPERTIES OF REFRACTORY $\text{Al}_x\text{Nb}_{40}\text{Ti}_{40}\text{V}_{20-x}$ MEDIUM-ENTROPY ALLOYS

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High/medium-entropy alloys based on refractory elements (RH/MEAs) represent a new class of alloys with promising high-temperature properties. Currently, the influence of individual elements on the structure and mechanical properties of RH/MEAs is being studied actively. For example, the addition of Al was shown to result in the formation of a B2 ordered structure. Many RH/MEAs with the B2 structure demonstrate high strength but extremely low ductility, even under compression tests. One of the ways to achieve more balanced properties is the variation of the chemical composition. Specifically, previous works reported that reducing the Al content or adding of Zr to some RH/MEAs increased compressive ductility by lowering the degree of B2 ordering. In this work, we studied the effect of Al on the structure and mechanical properties of  $\text{Al}_x\text{Nb}_{40}\text{Ti}_{40}\text{V}_{20-x}$  ( $x = 0; 5; 10; 15; 20$  at.%) RMEAs. It was shown that additions of Al up to 15 at.% led to the formation of the B2 structure and provided a higher strength and ductility in tension at  $T = 22$  and  $500^\circ\text{C}$  compared to bcc  $\text{Nb}_{40}\text{Ti}_{40}\text{V}_{20}$ . At higher temperatures, the positive effect of the B2 ordering on strength is retained only for the alloy with 20 at.% of Al. Relationships in the triangle "chemical composition-structure-properties" were discussed.

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## **EFFECT OF AUSTENITIZATION TEMPERATURE AND TIME ON THE MECHANICAL PROPERTIES OF A CARBON STEEL**

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The selection of appropriate materials and their treatments for medical instruments is always an important task for materials scientists. In particular, the materials for rotary instruments (for example, dental burs) must possess manufacturability and safety. The former includes good workability, and the latter means compliance with the declared mechanical properties throughout the entire service life. The present study is aimed to clarify the influence of temperature and time of austenitization on the hardness of prospective carbon steel after tempering.

In this work, the hardness of A75 type steel was studied, which was tempered after austenitization for different time (10-120 min.) at 750, 790, or 800°C. After austenitization for 10-40 min at 750°C (740°C is A3 temperature according to the phase diagram), incomplete quenching is observed that leads to relatively low hardness irrespective of tempering temperature. In contrast, longer austenitization at 750°C provides the hardness well above 450 HV even after tempering at 400°C, although this hardness is little lower than that after austenitization at 790°C or 800°C. After austenitization for 10 minutes at 750, 790 or 800°C and then tempering at 350°C, hardness of the present steel samples comprises 236, 510 or 514 HV, respectively.

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**NEW HIGH-ENTROPY OXIDE PHASES WITH MAGNETOPLUMBITE STRUCTURE**

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The fundamental scientific problem of this work was to study of the influence of high entropy of mixing of multicomponent ionic systems on the possibility of formation and stabilization of high-entropy crystalline solid solutions in such systems. As part of solving this problem, in the course of the study, to study the processes and results of the formation of such solutions in complex oxide systems with a magnetoplumbite structure were carried out, and to study the structure and properties of such systems.

The specific tasks of the work were:

1. Obtaining samples of high-entropy phases with a magnetoplumbite structure of previously unknown compositions.
2. Development of methods for obtaining high-entropy phases with a magnetoplumbite structure.
3. Study of the composition and structure, as well as a wide range of properties of the obtained samples.
4. Analysis of the obtained experimental data in order to create methods for the synthesis of phases of this kind with a given level of properties, and to obtain highly efficient functional materials on their basis.

The conclusions of the work are based, first of all, on our own experimental data and theoretical developments. The work shows not only the fundamental feasibility of controlling the properties of high-entropy phases with the magnetoplumbite structure, but also put the theoretical basis for systematic research in this area, which will be aimed at both obtaining fundamental knowledge and applications.

In phases formed by complex oxides, the effect of the influence of high entropy of mixing on the stability of solid solutions has been rarely studied, as well as the effect of high configurational entropy of mixing in such structures on their properties, which allows us to say that the theoretical level of expected results is comparable with the world level. and even ahead of similar foreign and domestic developments in this field of science.

The discovery of new stable solid solutions with a magnetoplumbite structure, a detailed study of their structure, a study of the temperature and concentration boundaries of their stability, an experimental study of their electrophysical / magnetic / catalytic characteristics – all these works and their results have an absolute scientific novelty.

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# SOLUTION COMBUSTION SYNTHESIS OF CoCuFeNi HIGH ENTROPY ALLOY AND THE FOLLOWING SPARK PLASMA SINTERING

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A solution combustion synthesis (SCS) is a chemical approach for facile one-step synthesis of a huge range of nanoscale materials. Until recently, SCS was used solely for the preparation of oxide-based materials. In a course of the method evolution, a new approach had appeared: a so-called sol-gel combustion synthesis (SGCS) [1]. This method has advantages upon the traditional (SCS) in a perspective of the process controllability. The SGCS allowed to synthesize a number of new for this method non-oxide compounds that include pure metals [2], binary metal alloys [3], nitride [4] and carbide [5] ceramics. Currently, the preparation of nanoscale multimetallic alloys such as high entropy alloys (HEA) [6] is one of the active directions of SGCS.

In this work, by implementing the SGCS approach, we have synthesized the CoCuFeNi ternary HEA with the unique microstructure. Typically, the Cu constituent is mobile and tends to form independent clusters in a HEA matrix [7]. Due to unique characteristics of SGCS, including homogeneous mixing of reagents at the molecular level, high combustion temperature and short reaction times (seconds) we have synthesized the CoCuFeNi HEA with the equally distributed constituents along all the volume of the as synthesized samples. For the preparation of the ready to use material the sintering process is often needed. To minimize the high temperature treatment which brings the increase of Cu mobility we studied the spark plasma sintering (SPS) process for further HEA compaction and sintering. At his point under the 800 °C sintering temperature and 10 minutes dwelling time the Cu clustering was already observed.

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## PERSPECTIVES APPLICATIONS PRODUCTS FROM AMORPHOUS MICROWIRES FOR MEDICINE

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Due to the unique combination of physical, mechanical, tribological, corrosion properties, radiopacity, and biocompatibility, amorphous microwires are perspective materials for medical applications.

IMET RAS is searching for new compositions of ferromagnetic amorphous alloys and studying their structure and properties. The original technologies for obtaining amorphous microwires of various compositions by the Ulitovsky-Taylor method with diameters of 50-150  $\mu\text{m}$ , microwires with a variable cross section [1], as well as microspirals based on them has been developed [2]. The strength of amorphous microwires exceeds the strength of high-strength stainless steels and nitinol currently used in medicine. The smooth mirror microwires surface does not need machining and provides a low coefficient of friction. Microwires have superelasticity (elastic stretching up to 4%) and the ability to cold plastic deformation due to the amorphous structure [3, 4]. Microwires withstand elastic twisting up to 1 rev/cm without change in geometry. Herewith the microwire, even in a deformed state, is able to withstand significant stresses. Due to such unique properties, it is possible to manufacture from microwire stents, catheters, cava filters, medicaments delivery microrobots and etc. Amorphous microspirals can be used in the manufacture of guides, embolus, magnetic applique napkins, for catheters reinforcement, etc. The magnetic properties of microwires allow them to be used as electromagnetic markers. For example, to identify test tubes, non-contact detection and accounting of surgical napkins and swabs, etc.

According to a some of leading experts, amorphous microwires can be great practical interest for the manufacture new types medical products

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## METHOD FOR THE SYNTHESIS OF HIGH-ENTROPY CARBIDE IN AN IONIC MELT

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High entropy alloys including high entropy carbides are of interest from scientific and practical point of view [1]. So, this paper describes novel method of synthesis of high entropy carbide.

The low temperature synthesis of high-entropy carbide ( $\text{Ti}_{0.2}\text{Zr}_{0.2}\text{Nb}_{0.2}\text{Hf}_{0.2}\text{Ta}_{0.2}$ ) C was performed in the ionic melt. The method is based on the phenomenon of no-current transfer of metal to carbide of another metal through the molten salt.

In a series of standard electrode potentials relative to the reference chlorine electrode in the NaCl-KCl melt (1:1 mol.) at 1000 K [2], niobium and tantalum take the places of the most electropositive metals, zirconium and hafnium are the most electronegative ones. Titanium takes a position between these pairs. Thus, it is possible to create conditions, described in [3], for the step-by-step transfer to titanium carbide first of niobium and tantalum, and then of zirconium and hafnium. In order to receive one phase material the powder obtained during the synthesis was heated twice in a vacuum furnace to a temperature of 1733K.

Bulk pellets were made out from the high-entropy carbide powder with the addition of 6% cobalt powder. Vacuum sintering of pressed mixed powders was performed at a temperature of 1673K. Sintered high entropy alloy was studied by scanning electron microscopy and microhardness measurements.

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# FEATURES OF MICROSTRUCTURE AND THERMOELECTRIC PROPERTIES OF TWO-PHASED MATERIAL DERIVED FROM INITIAL HIGH-ENTROPY Bi-Sb-Te-Se-S SYSTEM

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Developing high-entropy and medium-entropy alloys is prospect approach of thermoelectric materials science, which can be applied to prepare high-effective thermoelectric materials. Owing to effective scattering of phonons by lattice disorder, these alloys possess intrinsically low lattice thermal conductivity that favors to enhancing thermoelectric figure-of-merit. The aim of this work is to examine features of microstructure and thermoelectric properties of material, derived from initial high-entropy Bi-Sb-Te-Se-S system. To prepare the material under study, method of reactive spark plasma sintering (RSPS) of mixture consisting of elemental Bi, Sb, Se, Te and S powders was applied. This method combines the synthesis and densification stages. The synthesis stage corresponds to self-propagating high-temperature (SHS) synthesis of material desired, and common spark plasma sintering of preliminary SHS-synthesized material is the second stage. In accordance with data of scanning electron microscopy and X-ray analysis, RSPS-prepared material was found to be two-phased. First phase correspond to high-entropy BiTeSeSb<sub>0.5</sub>S<sub>0.5</sub> material, whereas second phase is wide band-gap Sb<sub>2</sub>S<sub>3</sub> semiconductor. This semiconductor happened to be heavily doped with Bi and Se. The high-entropy phase is dendrite-like structure. The wide band-gap semiconductor phase is mainly located in interdendritic space. Separate dendrite crystals are characterized by length of a few dozens of microns and lateral size of a few microns. Temperature behavior of all the thermoelectric properties (specific electrical resistivity, Seebeck coefficient, total thermal conductivity) of two-phased material being studied, measured from room temperature up to ~530 K, are typical for the Bi<sub>2</sub>Te<sub>3</sub>-based alloys. Main features in these properties are due to change in electrical conductivity mechanism from degenerate semiconductor conductivity (low temperatures) to intrinsic electrical conductivity (high temperatures). The total thermal conductivity of two-phased material is low enough (~ 0.65 W·m<sup>-1</sup>·K<sup>-1</sup> at ~400 K). Reducing in the thermal conductivity can be attributed to effective phonon scattering by different inhomogeneous in the material, related to lattice disorder in high-entropy BiTeSeSb<sub>0.5</sub>S<sub>0.5</sub> phase and to interphase boundaries. Owing to low lattice thermal conductivity, thermoelectric performance of the material under developing is promising enough. Highest thermoelectric figure-of-merit equal to ~0.18 is observed at ~450 K.

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## THE REASON FOR THE INCREASE IN COMBUSTION RATE OF THE POWDER MIXTURE Ti+C DILUTED WITH COPPER

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In the work a comparative study of the combustion of powder and granular mixtures of Ti+C and Ti+C)+20%Cu (wt.) was carried out with granules of different sizes with varying Ti particle sizes from 31 to 142 microns.

It was found that the combustion rate of the powder mixture (Ti+C)+20%Cu is higher than the mixture Ti+C, despite the lower combustion temperature (experimental combustion temperatures were  $2550 \pm 50$  K and  $2950 \pm 50$  K, respectively). The use of the theory of «gasless combustion» [1,2] to determine the kinetic parameters of the process by the combustion rate of the powder mixture leads to a negative value of the apparent activation energy, which shows the inapplicability of the traditional approach. The obtained results are explained in the framework of the convective-conductive combustion model [3] by the inhibitory effect of impurity gases released during heating of component particles in front of the combustion front. Using the values of the combustion rate of granular mixtures with granules ranging in size from 0.6 to 1.7 mm, the values of the combustion rate of the substance of the granules are calculated, that is, the combustion rate of the powder mixture, in which the influence of impurity gases is leveled. Using the values of the combustion rate of granular mixtures with granules from 0.6 to 1.7 mm in size, the values of the combustion rate of the substance of the granules is calculated, that is the combustion rate of the powder mixture, in which the influence of impurity gases is eliminated.

Therefore, it is impossible to reliably determine the effective kinetic characteristics of high-temperature interaction based on experimental dependences of the combustion rate of powder mixtures on the maximum temperature and particle size for two reasons: 1) due to the strong influence of impurity gas release on the combustion rate; 2) due to the inapplicability of the conductive model of "gasless combustion", linking the combustion front velocity with the rate of the chemical reaction of the interaction of the initial reagents.

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## PREDICTION OF ELONGATION TO FRACTURE OF HIGH-ENTROPY ALLOYS USING NEURAL NETWORK

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High entropy alloys (HEAs), sometimes also called multi-principle element alloys, were originally discovered by Yeh [1] and Cantor [2]. In contrast to traditional alloys, which are based on one principal element, HEAs were defined as alloys with five or more principal elements in equal or near-equal atomic percentages (5-35 at.%). Due to the high mixing entropy effect, solid solutions of FCC or BCC crystal structures with multiple elements were generally formed instead of intermetallics or complex phases. HEAs can possess many interesting mechanical and physical properties, including high strength, remarkable ductility, good wear and corrosion resistance [3].

The machine learning approaches (especially artificial neural network (ANN)) are promising methods for designing new high-entropy alloys with optimal properties [4]. In the present work the ANN approach was used for prediction of compressive ductility of high-entropy alloys at room temperature. The dataset with 153 alloys was used for training ANNs with different configurations. The model with 2 hidden layers and 14 neurons in each one was shown standard deviation of 4.3%.

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## THE EFFECT OF AGING ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF VIT-1 ALLOY

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Search for materials that will meet stringent performance requirements is the most important task in the aircraft engine industry. In this regard, alloys based on orthorhombic titanium aluminide ( $Ti_2AlNb$ ) due to an excellent combination of good workability and oxidation resistance, as well as high specific strength [1]. Meanwhile, one of the main problems hindering the practical application of such alloys is the lack of systematic data on the relationship between their structure and mechanical properties. In particular, the structural and phase transformations during the aging of alloys have not been adequately studied based on orthorhombic titanium aluminide and their effect on mechanical properties [2]. In this regard, the purpose of this work was to study the effect of heat treatment by aging on the evolution of the microstructure and hardness of the VIT-1 alloy.

The object of study was an alloy with a nominal composition Ti–18,3Al–22,8Nb–1,3Zr–0,28Mo–0,33Ta–0,7W–0,2Si–0,07C (at. %). The material was subjected to forging and the consequent quenching (975 °C,  $\tau=2$  h, air cooling). Aging was carried out at temperatures (750–850°C) and holding times (0.5;1;2;4;6;8;12;24;48 h).

As a result of the study, it was found that the aging conditions have a significant effect on the hardness of the hardened alloy. At all temperatures, a noticeable increase in hardness is found at the initial stage of aging. As the holding time increases, the hardness begins to decrease.

An analysis of the results of structural studies showed that after quenching, the alloy consists of a matrix  $\beta$ -phase and equiaxial particles  $\alpha_2$ . The grain size and volume fraction of particles were 3.4  $\mu m$  and 27%, respectively. Heating and subsequent exposure contributed to the formation of the O-phase of various morphology. At short exposures over the entire temperature range, the precipitation of a finely dispersed acicular O-phase was observed, as well as the formation of layers of the O-phase along the boundaries of  $\alpha_2$  particles. The rise of temperature and exposure time contributed to: i) coarsening of the acicular O-phase and its transformation into a lamellar one; ii) the appearance of a lath and globularized O-phase along the boundaries and in the body of  $\beta$ -grains, respectively. There has been a decrease in the size and volume fraction of  $\alpha_2$  particles, as a result of the formation and growth of O-phase layers, along the boundaries of  $\alpha_2$ . The report discusses the relationship between structure evolution and hardness during aging.

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# ORIENTATION DEPENDENCE OF THE CRITICAL RESOLVED SHEAR STRESS FOR SLIP AND TWINNING IN SINGLE CRYSTALS OF THE COCRFENIMO HIGH-ENTROPY ALLOY

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Temperature dependence of the critical resolved shear stresses (CRSS)  $\tau_{cr}$  for slip and twinning and the strain hardening coefficient was studied on [001]-,  $[\bar{1}11]$ -,  $[\bar{1}44]$ -oriented crystals of the fcc  $\text{Co}_{24}\text{Cr}_{24}\text{Fe}_{24}\text{Ni}_{24}\text{Mo}_4$  (CoCrFeNiMo) (at.%) high-entropy alloy (HEA) under tension. The CoCrFeNiMo (at.%) HEA was obtained by alloying with Mo atoms up to 4 at.% by reducing the concentration of each element of the fcc equiatomic CoCrFeNi HEA in equal atomic percentages. The stacking fault energy of the CoCrFeNiMo (at.%) HEA was equal to  $\gamma_0=0.027 \text{ J/m}^2$ .

It was shown that initial yield behavior along studied orientations of the CoCrFeNiMo (at.%) HEA associated by dislocation slip. The CRSS for slip  $\tau_{cr}^{sl}$  were independent of crystal orientation and the Boas-Schmid law was fulfilled. Alloying with Mo atoms up to 4 at.% of the CoCrFeNi system led to solid solution strengthening at which the CRSS  $\tau_{cr}$  increased relative single crystals of the equiatomic CoCrFeNiMn HEA.

Studies of the mechanical behavior of flow curves showed that twinning developed only at  $T=77 \text{ K}$  in  $[\bar{1}11]$ -,  $[\bar{1}44]$ - oriented crystals. Two types of twins (nanotwins and macrotwins) were found in  $[\bar{1}11]$ -,  $[\bar{1}44]$ - single crystals of the CoCrFeNiMo HEA. Nanotwins developed in  $[\bar{1}11]$ -,  $[\bar{1}44]$ - oriented crystals after low strain level ( $\epsilon \geq 5\%$ ) and were detected only by TEM. Nanotwinning developed in two systems in  $[\bar{1}11]$ - oriented crystals and in one system in  $[\bar{1}44]$ - oriented crystals simultaneously with slip. The interaction nanotwins in several systems in  $[\bar{1}11]$ - crystals and nanotwins in one system in  $[\bar{1}44]$ - crystals with slip led to a sharp increase in the strain hardening coefficient  $\Theta = 2000 \text{ MPa}$ . Macrotwins developed after a significant strain by slip of 20 and 60% in  $[\bar{1}11]$ -,  $[\bar{1}44]$ - oriented crystals, respectively. Transition from nanotwinning to macrotwinning, which developed predominantly in one system, led to decrease in  $\Theta = 950\text{--}1050 \text{ MPa}$ . Macrotwinning was determined metallographically on the surface of deformed crystals in  $[\bar{1}11]$  orientation. In  $[\bar{1}44]$  orientation, macrotwinning was found both optical metallography and X-ray studies on the surface of deformed crystals and at the precession of the crystal axis, respectively. Twinning was not detected in [001]- crystals. CRSS  $\tau_{cr}^{tw}$  for twinning in single crystals of the CoCrFeNiMo HEA depended on the crystal orientation.

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## PREPARATION AND TRIBOLOGICAL PROPERTIES OF (MoTaTiV)<sub>4</sub> CERAMICS

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High entropy ceramics (MoTaTiV)<sub>4</sub> were prepared by SPS sintering with mono-carbide. The phase, density, microstructure and tribological properties of (MoTaTiV)<sub>4</sub> were investigated. The results show that the minimum temperatures of (MoTaTiV)<sub>4</sub> forming single phase and densification were 1800 °C. At 1800 °C to 2100 °C, densification and grain growth of (MoTaTiV)<sub>4</sub> were promoted by heating, and the density of (MoTaTiV)<sub>4</sub> sintered at 2100 °C was 98.7 %. Ti element segregation existed in (MoTaTiV)<sub>4</sub> sintered at different temperatures. The hardness of (MoTaTiV)<sub>4</sub> remained about 20 GPa. (MoTaTiV)<sub>4</sub> showed good wear resistance, with an average specific wear rate of less than  $2 \times 10^{-5}$  mm<sup>3</sup>/Nm. The reason of low wear rates of (MoTaTiV)<sub>4</sub> might be due to that the friction oxide layer with antiwear effect generated on the worn surface of the ceramics.

## EFFECTS OF COLD ROLLING AND ANNEALING TREATMENT ON MICROSTRUCTURE AND PROPERTIES OF COFENIMNV HIGH-ENTROPY ALLOY

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In this study, a combination of cold rolling (CR) and annealing treatment is used to investigate the evolution of the microstructure and mechanical properties of CoFeNiMnV high-entropy alloys (HEAs) fabricated by powder plasma arc additive manufacturing (PPA-AM). The as-deposited CoFeNiMnV HEAs exhibit a single-phase face-centered cubic (FCC) structure with a small amount of accompanying V-Mn-rich precipitates. After 50% cold rolling, the microstructure of CoFeNiMnV HEAs is severely deformed along the rolling direction, which the crystal structure is obviously refined, and the hardness and strength of the alloy are significantly improved (compared to the PPA-AM deposited sample, the hardness is about 1.9 times higher and the tensile strength is about 1.4 times higher). The rolled CoFeNiMnV HEAs is annealed at 500 °C, 700 °C and 900 °C, respectively, for 60 min. The results show that annealing at 500 °C has little effect on the microstructure and dislocation density of the alloy, and the alloy still maintains high strength and hardness. After annealing at 700 °C and 900 °C, The combined effects of the significantly decrease dislocation density, the gradual dissolution of V-Mn precipitates, obviously coarsened recrystallization grains, and significant coarsening of annealing twin size decreased with the annealing temperature decrease the strength of the alloy, but the elongation improved significantly. CoFeNiMnV HEAs annealed at 700 °C after cold rolling exhibit an excellent high strength-toughness combination.

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# INVESTIGATION ON THE MICROSTRUCTURE AND TRIBOLOGICAL RESPONSES OF NOVEL CoCrCu<sub>0.2</sub>FeMo<sub>x</sub> HEA COATING USING PLASMA ARC CLADDING

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High entropy alloy coatings show great potential for wear resistance applications, but most of them are not enough to apply in practice. In this study, we prepared CoCrCu<sub>0.2</sub>FeMo<sub>x</sub>(Mo<sub>x</sub>) high entropy alloy coatings on the surface of 304 substrate by using plasma arc cladding technology. By using scanning electron microscope, X-ray diffractometer, microhardness tester, three dimensional laser measurement microscope, we analyzed the effect of Mo element on the phase structure, microscopic morphology, microhardness and wear mechanism of the coatings and the strengthening mechanism of their wear resistance. The experimental results show that the CoCrCu<sub>0.2</sub>FeMo<sub>x</sub> high entropy alloy coatings are composed of Face-Centered Cubic phase (FCC phase) and  $\delta$  phase (rich Mo, Cr). The microscopic morphology of the alloy changes gradually from branch crystal to eutectic organization with the increase of Mo content. With the increase of Mo element, the hardness of the coatings increase, while the wear volume, wear rate and average surface roughness decrease. When the Mo content is increased to 0.3, the delamination of the oxide layer is effectively reduced. The wear mechanism changes from abrasive wear, oxidation wear and fatigue spalling to abrasive wear.

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# IMPROVED MECHANICAL PROPERTIES OF $\text{AlCo}_x\text{CrFeNi}$ HIGH ENTROPY ALLOYS WITH HIGH CO CONTENT FABRICATED BY LASER MELTING DEPOSITION

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$\text{AlCo}_x\text{CrFeNi}$  ( $x=2.2, 2.8$ ) high entropy alloys (HEAs) were successfully prepared by multi-layer and multi-channel laser melting deposition (LMD). The tensile properties of the LMD-fabricated  $\text{AlCo}_x\text{CrFeNi}$  HEAs were investigated for the first time. The phase evolution of these alloys was examined by X-ray diffraction and compared with existing models. The microstructure of the alloys was characterized using scanning electron microscopy and electron backscatter diffraction. It is found that Co element can promote the phase transformation from BCC phase to FCC phase in the as-deposited  $\text{AlCo}_x\text{CrFeNi}$  HEAs, and the volume fraction of FCC phase increases from 51.4% to 74.6% as the Co content increases from 36.2 at% to 40.8 at%. With the increase of Co content, the grain size of BCC phase in the alloys decreases and a larger amount of fine needle-like BCC phase appears in the FCC matrix. Tensile testing shows that higher Co content in the deposited  $\text{AlCo}_x\text{CrFeNi}$  alloy can enhance its plasticity without significantly compromising its strength. As the Co content increases, the fracture strain increases from 5.9% to 15.4%, while the yield strength only reduces from 450 MPa to 360 MPa and the ultimate tensile strength increases from 734 MPa to 739 MPa. The variations in tensile properties of the  $\text{AlCo}_x\text{CrFeNi}$  alloy result from phase structure changes and microstructure evolution. Through this research, it is demonstrated that enhancement of the tensile properties of the LMD-fabricated  $\text{AlCoCrFeNi}$  HEAs can be realized by increasing the content of Co element.

# INTERCONNECTED EFFECTS OF Sm-DOPING ON GRAIN STRUCTURE AND TRANSPORT PROPERTIES OF THE TEXTURED $\text{Bi}_{2-x}\text{Sm}_x\text{Te}_{2.7}\text{Se}_{0.3}$ COMPOUNDS

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Textured  $\text{Bi}_{2-x}\text{Sm}_x\text{Te}_{2.7}\text{Se}_{0.3}$  compounds with  $x = 0; 0.005; 0.01; 0.02; 0.05; 0.1; 0.2$  and  $0.3$  were prepared by using solvothermal synthesis and spark plasma sintering of starting powders. Sm-doping results in several interconnected effects. First of them is reducing in size of particles in starting powders with increasing  $x$ . This effect is attributed to increasing in ionic bonding fraction in polar covalent Bi(Sm)-Te bonds, which occurs at increasing Sm content due to difference in electronegativity of Bi and Sm. As result, under solvothermal synthesis in polar solution, dissolution process, which competes with growth process, becomes more effective, limiting growth of the  $\text{Bi}_{2-x}\text{Sm}_x\text{Te}_{2.7}\text{Se}_{0.3}$  particles for all  $x$  values. With increasing  $x$ , reducing in the size grains in the bulk samples, which is governed by relevant changing in the particles size in the starting powders, takes place, too. This effect also results in enhancing in texturing degree in samples at gradual increasing  $x$ . Finally, grain size effects on the specific electrical resistivity and the thermal conductivity are observed in bulk samples with different grain size. With increasing the grain size, the resistivity increases, whereas the total thermal conductivity decreases. These features are due to electron scattering by grain boundaries. Grain size effects on the specific electrical resistivity and the total thermal conductivity were found in the bulk samples with different grain sizes. These effects were measured parallel or perpendicularly to the texturing axis (direction of mechanical loading at spark plasma sintering). With increasing the grain size, the resistivity abruptly increases, whereas the total thermal conductivity and the electron thermal conductivity abruptly decrease. These features are connected with ability of grain boundaries act as scattering centres for electrons.

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# PRODUCTION OF HIGH-ENTROPY CARBIDE (Ti,Zr,Hf,Nb,Ta)C WITH MINIMUM ZrO<sub>2</sub> OXIDE CONTENT

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High-entropy carbides (HEC) surpass the individual monocarbides of which they are composed in terms of their strength properties. This is due to the thermodynamic effect of high entropy, as well as large distortions of the crystal lattice due to differences in the sizes of the atoms of the metal components that form the HEC.

In the present work, a HEC of composition (Ti,Zr,Hf,Nb,Ta)C was obtained by hot pressing at a temperature of 2100°C for 30 min under a pressure of 35 MPa in a graphite mold. To minimize the oxidation process, the consolidation process was carried out in a vacuum of the order of  $10^{-2}$  –  $10^{-3}$  mm Hg. Art. Prior to hot pressing, TiC, ZrC, HfC, NbC, and TaC powders in a ratio of 1:1:1:1:1 were subjected to high-energy ball milling in a Fritsch Pulverisette 7 planetary ball mill. The ratio of ball mass to powder mass was 6:1. Drum and ball lining – ZrO<sub>2</sub>.

X-ray phase analysis established that after high-energy grinding of monocarbides, the (Ti,Zr,Hf,Nb,Ta)C phase is not formed. Only individual reflections from TiC, ZrC, HfC, NbC, and TaC are present, the lines of which are rather broad. At large angles  $2\theta$ , the formation of an amorphous halo is observed. The ground powders were characterized by a bimodal particle size distribution with distribution centers at 0.4 (~20% of powder particles) and 6.1  $\mu\text{m}$  (~80% of powder particles).

Hot pressing at 2100°C made it possible to form virtually single-phase (Ti,Zr,Hf,Nb,Ta)C, which is distinguished by sharp and thin high-intensity lines on the X-ray diffraction pattern. However, the structure also contained a phase of the ZrO<sub>2</sub> type of the monoclinic modification in an amount of about 3% wt.

Compact (Ti,Zr,Hf,Nb,Ta)C was characterized by the following properties: lattice parameter  $a = 0.4509$  nm, microhardness at a load of 4.9 N – 20.2 GPa, crack resistance coefficient  $K_{IC} = 4.6$  MPa  $\sqrt{\text{m}}$ , porosity  $P = 11\%$ , grain size  $d = 8.82$   $\mu\text{m}$  (geometric mean).

Thus, using the technology of mechanical grinding of Ti, Zr, Hf, Nb, and Ta monocarbides, followed by hot pressing of the powder mixture at a temperature of 2100 °C, a high-entropy carbide (Ti,Zr,Hf,Nb,Ta)C with a minimum content of ZrO<sub>2</sub> oxide (<3 wt. %) and homogeneous chemical compositions.

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## SIMULTANEOUS INCREASE IN STRENGTH AND DUCTILITY OF REFRACTORY MEDIUM-ENTROPY ALLOYS DUE TO B2 ORDERING

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Apart from the obvious density reduction and somewhat improvement of oxidation resistance, Al additions to recently introduced refractory high/medium-entropy alloys (RH/MEAs) result also in B2 ordering. This phenomenon affects the mechanical properties in the way that most of Al-containing RH/MEAs appear to be strong yet brittle at ambient and intermediate temperatures, even under compression. The existing data suggest several ways to balance the mechanical properties of these RH/MEAs, including a decrease in a degree of B2 ordering or the B2 matrix-to-B2 particles transition. In any cases, achieving even a minor increase in plasticity was followed by a notable strength deterioration. Moreover, tensile ductility for B2 RH/MEAs was reached in extremely rare cases, and these alloys showed a classical strength-ductility trade-off. In this work, we demonstrated that controlled additions of Al, which induced the certain degree of B2 ordering, increased both strength and uniform elongation of a NbTiZr RMEA by ~ 35%. The underlying mechanisms responsible for such an improvement of the mechanical performance were discussed.

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## INFLUENCE TEMPERATURES OF TEMPERING ON MECHANICAL PROPERTIES OF STEEL 60Si2CrVNb

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Spring steels 60Si2CrV, 60Si2CrVA are widely used for production of agricultural equipment. The steel for such application must possess a high level of strength, hardness, and wear resistance. However, the durability of the above spring steels does not meet modern requirements due to low wear and fracture resistance. Recently microalloying with Nb was suggested to improve mechanical properties of spring steels. It is well known that carbonitrides of niobium prevent coarsening of prior austenite grains (PAG) during austenizing. Small PAGs provide homogeneous martensitic structure with low distance between boundaries of lath, block and packets and may increase strength and wear resistance of the steels. Decomposition of martensite during tempering leads to decrease strength of the steels. Therefore study effect of tempering on mechanical properties is important to find optimum balance between strength and plasticity. The work focuses on the detail research of the influence of tempering temperature on mechanical properties of 60Si2CrVNb steel.

The cast ingot of 60Si2CrVNb steel was hot-forged at temperature of 1150°C. Then, specimens were austenitized for 40 min at 900°C and subsequently quenched into water. Tempering was carried out at temperatures ranging from 200 to 500°C for 60 min. After tempering, the specimens were cooled in air. Tensile properties and Rockwell hardness were studied in accordance with GOST 1497-84 and GOST 9013-59, respectively.

The yield strength and ultimate tensile strength of quenched samples are measured to be 1289 MPa and 1630 MPa, respectively. The Rockwell hardness is 61. It should be noted that quenched specimens are ruptured before reaching maximum stress because extremely low elongation to failure of 0.5%. The tempering at a temperature of 200°C provides an increase in the yield strength and ultimate tensile strength by 220 MPa and 640 MPa, respectively. Elongation to failure increases up to 4.2%. The hardness of steel changes insignificantly. Optimal combination between yield strength of 1783 MPa, ultimate tensile strength of 2190 MPa and elongation to failure of 6.2% are obtained after tempering at temperature of 280°C. Further increase in tempering temperature leads to a decrease in tensile strength, hardness, and increase in the plasticity.

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## OXIDATION BEHAVIOUR OF EUTECTIC Al-Cr-Nb-Ti-Zr REFRACTORY HIGH-ENTROPY ALLOYS

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The increasing necessity for improved economic efficiency of aircraft engines requires new materials with superior performance. Particular interest exists in amplifying the thrust-to-weight ratio by using of light alloys capable of operating at 600-1000°C. The recently introduced refractory high-entropy alloys (RHEAs) concept offers wide opportunities to satisfy this need. Specifically, RHEAs based on the Al-Cr-Nb-Ti-(V)-Zr system with densities  $< 6.5 \text{ g/cm}^3$  have higher specific strength at  $T \leq 1000^\circ\text{C}$  than Ti-based intermetallics and Ni-based superalloys. However, compared to mechanical properties, the oxidation behaviour of these alloys is less studied. The existing data cover only limited number of lightweight RHEAs and give diverse information about the relations between oxidation resistance and chemical composition, especially with non-equiatomic alloys. Structure is another factor that can affect the oxidation behavior greatly, but it remains almost unexplored. In this work, we systematically studied the oxidation behaviour of the B2/Laves phase hypoeutectic  $\text{Al}_{23}\text{Cr}_{20}\text{Nb}_{15}\text{Ti}_{32}\text{Zr}_{10}$ , eutectic  $\text{Al}_{28}\text{Cr}_{20}\text{Nb}_{15}\text{Ti}_{27}\text{Zr}_{10}$ , and hypereutectic  $\text{Al}_{33}\text{Cr}_{20}\text{Nb}_{15}\text{Ti}_{22}\text{Zr}_{10}$  (at.%) RHEAs during the interrupted tests in the air at 800-1000°C and exposures up to 100 h. The oxidation mechanisms and suggestions for improving the oxidation/spallation resistance were discussed thoroughly.

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## THE SYNTHESIS, STUDY OF THE STRUCTURE AND PROPERTIES OF HIGH-ENTROPY INTERMETALLIC COMPOUNDS

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Intermetallic compounds are typically undesirable phases in high-entropy alloys, in which the high configurational entropy of mixing minimizes the Gibbs free energy of the solid solution and consequently stabilizes a single high-entropy solid solution phase rather than a mixture of low-entropy phases (which include intermetallic compounds). However, it is sometime desirable to deliberately tailor the properties of a high-entropy metallic matrix by formation of intermetallic inclusions within its volume.

The formation of single phase high-entropy intermetallic compounds (HEICs), where one sub-lattice satisfies the high-entropy criteria, is a relatively new concept. The interest in intermetallics, their formation, properties, and applications is constantly growing. The number of works directly devoted to the preparation of HEICs is very limited (at the moment there are no more than ten articles), which gives broad prospects for research in this direction.

The scientific problem to be solved by this work is the synthesis and characterization of a new class of high-entropy phases – HEICs, i.e. multicomponent phases with close to equimolar concentrations of the main components in which a homogeneous crystalline intermetallic structure is stabilized by high entropy of mixing.

The specific tasks to be solved by the study are:

1. The synthesis of samples of a new class of high-entropy systems – high-entropy intermetallic phases.
2. The investigation of the composition and structure, as well as the complex of properties of the obtained samples.
3. The analysis of the obtained experimental data in order to formulate the general laws of the formation of high-entropy intermetallic phases, which include the criteria for the stability of such phases.
4. The study of the possibility of using the obtained phases as functional and/or structural materials.

An analysis of the experimental data that are obtained during the implementation of the research work will make it possible to lay a theoretical basis for the creation of high-entropy intermetallic compounds. At the same time, specific systems are selected to study the patterns of formation of high-entropy intermetallic compounds, in such a way to increase the probability of obtaining materials that are interesting from an applied point of view, and at the same time to obtain new information about systems that are currently not or little studied.

An analysis of the results obtained made it possible to draw conclusions regarding the regularities related to both a new class of materials – intermetallic high-entropy phases, and, in general, to high-entropy systems.

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# INFLUENCE OF THE Co CONTENT ON THE THE COMPOSITION AND STRUCTURE FORMATION OF CAST ALLOYS Co-Cr-Nb-W-Mo-Al-C IN THE PROCESS OF CENTRIFUGAL SHS-METALLURGY

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Co-based cast alloys have high strength at high temperatures and are used in gas turbine engines [1]. In this paper, the task is to investigate the possibility of obtaining alloys with different ratios of Co and alloying additives (Cr-Nb-W-Mo-Al-C) by centrifugal SHS metallurgy and controlling their composition and structure.

To obtain cast alloys, mixtures of  $(\text{Cr}_2\text{O}_3/\text{Nb}_2\text{O}_5/\text{WO}_3/\text{MoO}_3/\text{Al/C}) + x (\text{Co}_3\text{O}_4/\text{Al})$  with  $x$  from 0 to 100 wt.% were used. Under atmospheric conditions, the combustion of mixtures is accompanied by an intense dispersion of combustion products. The effect of overload suppresses the spread and allows you to get cast alloys with different ratios of cobalt and alloying elements (Cr, Nb, Mo, W, C and Al). With an increase in the content of  $\text{Co}_3\text{O}_4$  from 0 to 100% wt. in the mixture, the combustion rate increases ~5 times, and the yield of target elements into the ingot approaches the calculated values, the Co content in the alloy increases monotonically, the content of Nb, Mo, W, C decreases and the Al content passes through a maximum.

In cast alloys, a Co-based matrix is formed in which the NbC is distributed. The change in the phase composition is accompanied by a change in the microstructure from globular to dendritic.

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## STRUCTURE AND PROPERTIES OF HIGH-ENTROPY ALLOY-BASED METAL-MATRIX COMPOSITES

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Medium-entropy alloys (MEAs) of the Ti-Nb-Zr system have low elastic modulus, high specific strength, excellent corrosion resistance and biocompatibility. However, wider use of these alloys in industry and medicine can be limited due to relatively low strength, hardness and wear resistance. A significant increase in strength characteristics can be achieved by using an integrated approach comprising both modification of the chemical composition and creating metal-matrix composites with high-strength ceramic reinforcements. For titanium-based alloys the use of TiB particles is very attractive, since these reinforcements forms in-situ during sintering; adheres well to the titanium matrix without the formation of a transition region; has similar to Ti coefficient of thermal expansion; possess good thermal stability. However, interaction of boron with two other elements (Nb, Zr) of the TiNbZr alloy is not sufficiently studied.

In this investigation the NbTiZr-based metal-matrix composites with different amounts of TiB (1.0 vol.% (alloy A), 6.8vol.% (alloy B) and 4.4vol.% (alloy C) were produced by vacuum arc melting. The initial microstructure of the synthesized composites for all iterations was composed of bcc NbTiZr matrix and needle-like monoborides (Ti, Nb) B. Occasionally particles of the  $\omega$  phase ( $\sim 10$  nm in diameter) were found in the matrix of all composites. EBSD analysis suggested microstructure refinement with an increase in the amount of borides. Higher amount of borides expectedly resulted in an increase in strength. The Alloy A showed the lowest value of the yield strength 690 MPa and the highest elongation  $\sim 20$  %. Samples of the Alloy B showed the yield strength of 810 MPa and elongation  $\sim 5$  %. The highest strength properties were observed for the Alloy C – the yield strength reached 900 MPa, however, the specimen failed at 0.5 % strain. The Orowan strengthening contributed mainly to the overall strength of the composite. Analysis of biocompatibility suggests some decrease in the proliferation rate of mesenchymal stem cells with an increase in the borides amount.

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# **EFFECT OF BORIDES ADDITION ON THE OXIDATION BEHAVIOR OF TA<sub>25</sub>W<sub>25</sub>MO<sub>25</sub>NB<sub>25</sub> ALLOY**

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Two kind of boride TiB<sub>2</sub> and ZrB<sub>2</sub> were added in Ta<sub>25</sub>W<sub>25</sub>Mo<sub>25</sub>Nb<sub>25</sub> high entropy alloy for improve oxidation behavior of alloy. Single phase body-centered cubic alloys were prepared by hot pressing sintering at 1950°C. The oxidation behavior of alloys at 1023K to 1223K are investigated. The addition of borides increase the hardness of alloys. The oxidation kinetics of alloys followed parabolic law after oxidation at 1073K for 4hr, while followed liner law when oxidation temperature increased up to 1173K. The mass gain per unit surface area during oxidation at 1073K decrease 42% and 52% respectively comparing alloys without borides. The addition of TiB<sub>2</sub> and ZrB<sub>2</sub> to high entropy alloy decreased the oxidation rate significantly.

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