ADVANCED HIGH ENTROPY MATERIALS

Abstracts of the VI School of Young Scientists "Advanced High Entropy Materials"

Belgorod State National Research University State Marine Technical University, October 2-3, 2024



Editors-in-Chief

Salishchev G.A. – Doctor of Technical Sciences, Professor, Head of the laboratory of the Belgorod State University; Povolyaeva E.A. – Junior Researcher, Belgorod State University.

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Тезисы VI школы молодых ученых «Перспективные высокоэнтропийные материалы»

Белгородский государственный национальный исследовательский университет Санкт-Петербургский государственный морской технический университет (2-3 октября, 2024 г.)



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Сборник содержит тезисы VI школы молодых ученых «Перспективные высокоэнтропийные материалы». В нем представлены доклады ведущих ученых, молодых ученых, аспирантов, студентов, представителей промышленности и освещены актуальные направления в области разработки высокоэнтропийных сплавов и покрытий, новых материалов, современных методов их изучения и технологий изготовления перспективных изделий.

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AL _X (NBTIZR) _{100-X} (X= 10; 25; 40 AT%)

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• A.Yu. Geller, Head of Project Management Department in Institute of Laser and Welding Technologies, Saint Petersburg State Marine Technical University.

TOPICS

Main directions of work:

- High-entropy metallic materials: structure, mechanical, and functional properties.
- Structure and properties of non-metallic high-entropy materials: ceramics, coatings,

etc.

• Advanced metallic and non-metallic materials and their applications.

• Additive and laser technologies, and other methods for processing of advanced materials.

• Theoretical and computational prediction of the structure and properties of advanced materials.

The school will be supported under Russian Science Foundation grant No. 19-79-30066 "Advanced alloys and technologies for the aerospace industry".

CONFERENCE ABSTRACTS

BCC-FCC PHASE TRANSFORMATION VIA HYDROGENATION OF THE ALLOYS IN Ti-V-Zr-Nb-Ta-Hf SYSTEM

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With growing demand for green energy sources, hydrogen storage is becoming an essential task. Among the ways to safely and efficiently store hydrogen along with unique mechanical and physical properties multi-principal-component alloys stands out. [1] This work is a continuation of our previous works [2-4] exploring the feasibilities and characteristics of single-phase multiprincipal-component alloys (MPC) for hydrogen storage applications. The Ti-V-Zr-Nb-Ta-Hf system alloys were developed using electron beam melting with pendant drop melt extraction (EBM-PDME). The synthesized alloys were characterized using powder X-ray diffraction (XRD). The hydrogenation behavior was investigated using a volumetric method. To gain a deeper understanding of the phase transformations and accompanying thermal effects during hydrogenation were carried out. In result, the EBM-PDME method produced BCC single-phase alloy with cell parameter 0.3328 nm. At hydrogen concentrations of less than 0.46 H/M, a single BCC solid solution phase is detected with slightly increase in cell parameter up to 0.3366 nm. After reaching a content of absorbed hydrogen of 0.46 H/M, a small amount of the FCC hydride phase with a cell parameter of 0.4465 nm emerged. This phase demonstrates relative volume expansion of 20.8% and can be attributed to dihydride typical as hydrogenation product of BCC MPC alloys. Further hydrogenation leads to a gradual increase in the FCC/BCC ratio with an increase in the lattice parameters of both the solid solution and the dihydride phase. The completion of hydrogenation occurs when the alloy has absorbed 1.76 H/M, and BCC phase has disappeared, and the FCC phase cell parameter has reached 0.4558 nm. Furthermore, at the same moment, the hydride phase exhibits a relative volume increase of 28.5%.

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1. Miracle D.B., Senkov O.N. A critical review of high entropy alloys and related concepts // Acta Mater. –2017. – Vol. 122. – P. 448–511.

2. Composition design, synthesis and hydrogen storage ability of multiprincipalcomponent alloy TiVZrNbTa / Zadorozhnyy V., Tomilin I., Berdonosova E. et al. // Journal of Alloys and Compounds. – 2022. – V. 901.

3. Production of multi-principal-component alloys by pendent-drop melt extraction / Korol A., Zadorozhnyy V., Zadorozhnyy M., et al. // International Journal of Hydrogen Energy. – 2024. – V. 54. – P. 161-175.

THE ROLE OF TWINNING AND γ→ε MARTENSITIC TRANSFORMATION IN STRAIN HARDENING OF MULTICOMPONENT Fe40Mn40C010Cr10 AND FeMnCoCrNi ALLOYS

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Deformation-induced microstructure and mechanical properties were studied in metastable $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ and stable FeMnCoCrNi (Cantor alloy) multicomponent alloys, which were strained in tension and cold rolling. Multicomponent alloys were obtained by a vacuum induction melting. Rectangular bars $10 \times 10 \times 25$ mm³ of cast material were subjected to multipass cold rolling with 80 % reduction at room temperature (RT) and 77K. A portion of as-rolled material was solution-treated at temperatures (1373-1473)K for 2h. For mechanical testing, flat dumb-bell shaped specimens with a gauge section of 12 mm \times 2.7 mm \times 1.3 mm were cut from the solution treated and as-rolled billets. Tensile tests were carried out in the temperature interval T = (77–673)K at initial strain rate of 10^{-4} 1/s.

In metastable Fe₄₀Mn₄₀Co₁₀Cr₁₀ alloy, RT-rolling is accompanied with a fragmentation of austenitic grains due to the development of dislocation slip, twinning and $\gamma \rightarrow \varepsilon$ martensitic transformation (MT). Highly misoriented dual-phase ($\gamma + \varepsilon$) ultrafine-grained microstructure is typical of Fe₄₀Mn₄₀Co₁₀Cr₁₀ alloy after rolling. In FeMnCoCrNi specimens rolled at RT, a misoriented grain/subgrain microstructure was formed: TEM data testify to the slip-dominated deformation with much lower activity of twinning and MT than that in metastable alloy. Much higher activity of twinning and $\gamma \rightarrow \varepsilon$ MT was observed in Cantor alloy after rolling at 77K, but it is still lower than that in Fe₄₀Mn₄₀Co₁₀Cr₁₀ alloy. It was noted that joint development of dislocation slip, twinning and deformation-induced $\gamma \rightarrow \varepsilon$ MT in Fe₄₀Mn₄₀Co₁₀Cr₁₀ alloy does not provide a superior strengthening over the Cantor alloy during rolling both at RT and 77K.

In tension, both alloys possess strong temperature dependence of the mechanical properties, and the highest strain hardening capacity is observed in low-temperature deformation of $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ metastable alloy, where $\gamma \rightarrow \epsilon$ MT acts as one of the dominating deformation mechanisms together with slip and twinning (at 77K). The higher deformation temperature the lower activity of MT and twinning and strain hardening of the $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ metastable alloy, so at T>200K both these deformation mechanisms have secondary importance or are completely suppressed at T > 300K. Cantor alloy shows strong temperature dependence of the strain hardening but deformation slip is a dominating deformation mechanism over the whole temperature range (77-673)K. In FeMnCoCrNi alloy, twinning activity is much lower than that in $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ metastable alloy, but this deformation mechanism support plastic deformation with high work hardening in Cantor alloy at 77K providing the perfect combination of strength/ductility in Cantor alloy in low-temperature deformation regime.

The research was supported by the Russian Science Foundation (project No. 20-19-00261), https://rscf.ru/project/20-19-00261/. The studies were carried out using the equipment of the Nanotech Center (ISPMS SB RAS, Tomsk).

CRACKING BEHAVIOUR OF NIKEL-BASED SUPERALLOY DURING DIRECT LASER DESPOSITION

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Today nickel-based superalloys are actively used in the aviation, power and shipbuilding industries and are the main materials for the production of engine components operating at elevated temperatures. However, the production of complex-shaped parts using traditional methods such as casting, directional crystallization and powder metallurgy often requires subsequent deformation and heat treatment, which complicates the production process. Additive manufacturing (AM) of nickel-based alloys, namely direct laser deposition (DLD), allows for the production of large-sized parts or the repair of critical components with the requisite surface quality and desired mechanical properties. However, it is widely known that nickel-based superalloys containing a high fraction of strengthening γ' phase are highly susceptible to cracking during AM processes.

In this work, the effect of deposition mode parameters on the microstructure, mechanical properties and cracking behavior of the nickel-based superalloy was investigated. Two thin walls were fabricated by DLD under different conditions. The results of the alloy microstructure study demonstrated successful fabrication of crack-free wall at a low-energy process with power of 200 W, and a scanning speed of 3 mm/s. At the same time, increasing the energy input to 1800 W power and 10 mm/s scanning speed resulted in the development of two types of cracks: solidification hot cracking and ductility-dip cracking caused by σ -phase particles. The microstructure of the crack-free wall consisted predominantly of fine equiaxed grains with an average size of ~ 19 µm, while in the cracked wall the epitaxial grain growth led to the formation of a coarse columnar structure with long, straight high-angle boundaries, that provide more possibilities for fast crack propagation.

This research was supported by the Russian Science Foundation, grant no. 23-19-00622 (https://rscf.ru/en/project/23-19-00622/).

TEMPERATURE DEPENDENCE OF MECHANICAL PROPERTIES IN CoCrFeMnNi AND CoCrNi ALLOYS

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The materials used in this study were $Co_{20}Cr_{20}Fe_{20}Mn_{20}Ni_{20}$ high-entropy Cantor alloy and $Co_{33,4}Cr_{33,3}Ni_{33,3}$ alloys. The cast alloys' billets were subjected to a thermal-mechanical treatment (TMT): an annealing at a temperature of 1200°C for 2 hrs, cold rolling (80% reduction) and final annealing at temperature of 1200°C for 2 hrs followed by water-quenching for Cantor alloy, and cold rolling (80% reduction) and annealing at temperature of 1150 °C for 1 hr followed by the water-quenching for CoCrNi alloy. Both alloys have been investigated using the X-ray diffraction analysis, transmission and scanning electron microscopy, tensile testing in a temperature range from 77 to 297 K.

It has been shown that the investigated alloys have a misoriented coarse-grained structure. The average grain size of the CoCrFeMnNi alloy is 215 μ m, and that of the CoCrNi alloy is 57 μ m. Both alloys possess single-phase austenitic structure with FCC crystal lattice. Due to the absence of Mn and Fe in three-component alloy, its lattice parameter a = 3.563 Å is lower than that of the CoCrFeMnNi alloy (a = 3.598 Å).

The temperature dependence of the mechanical properties of the alloys has been established. With a decrease in temperature from 297 to 77 K, the yield strength (YS) and the ultimate tensile strength (UTS) of both alloys increases. The maximum values of YS and UTS are observed in the CoCrNi alloy: respectively 435 MPa and 2115 MPa at T = 77 K (at room temperature YS = 254 MPa and UTS = 1396 MPa). For the CoCrFeMnNi alloy, the mechanical properties at cryogenic and room temperatures are lower (YS = 385 MPa and UTS = 1908 MPa at T = 77 K; YS = 179MPa and UTS = 851 MPa at T = 297 K), but at liquid nitrogen temperature it has high elongation to failure values El = 97 % (63 % at room temperature). For the CoCrNi alloy El = 83 % at 77 K and El = 91 % at 297 K. The mechanical properties of the alloys are determined by their deformation mechanisms. At T = 297 K, dislocation slip is a dominating deformation mechanism of the five-component alloy. The absence of Mn and Ni in the CoCrNi alloy does not significantly change deformation mechanism of the alloy relative to the Cantor alloy. The dislocation density in fractured samples of both alloys is high ($\rho = 10^{14} - 10^{15} \text{ m}^{-2}$). Along with dislocation slip at T = 77 K, mechanical twinning is activated in both alloys. Despite similar deformation mechanisms, additional strengthening in the CoCrNi alloy compared to the Cantor alloy occurs due to smaller grain size (grain boundary strengthening).

The analysis of the fracture surface of the samples demonstrates that at 297 K and 77 K both alloys are characterized by ductile transgranular fracture. Numerous dimples are observed on the fracture surfaces of the alloys.

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CORRELATION BETWEEN MICROSTRUCTURE AND YIELD STRENGTH OF HIGH-STRENGTH STEEL SUBJECTED TO QUENCHING AND PARTITIONING HEAT TREATMENT

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Quenching and partitioning (Q&P) steels have been attracted much attention due to excellent combination of strength and ductility. The actual Q&P microstructures may contain the primary martensite (PM), bainitic ferrite (BF), secondary martensite (SM) and retained austenite (RA). Hence, it is difficult to predict the yield strength of Q&P steels since it requires prior knowledge of the yield strength of each constituent phase.

The samples of Fe-0.33%C-1.85%Si-1.44%Mn-0.58%Cr steel after homogenization at 1150°C were subjected to quenching in water and tempering at a 350°C for 10 min (Q&T), Q&P treatment, and isothermal treatment at 350°C for 1 hour (IT). Q&P treatment consists of quenching in a salt bath to temperatures of 190, 210, 230, 250 and 270°C and partitioning in a salt bath at 350°C for 10 min.

The phase fractions determined by X-ray diffraction and dilatometric analysis, as well as the mechanical properties of steel after heat treatments are presented in Table 1.

Sample		Q&T	Q&P					IT
			190	210	230	250	270	11
Phase fraction	РМ	1	0.82	0.78	0.72	0.64	0.56	-
	RA	-	0.09	0.10	0.08	0.11	0.10	0.11
	SM	-	0.03	0.05	0.06	0.07	0.10	-
	BF	-	0.06	0.07	0.14	0.18	0.24	0.89
Mechanical properties	YS, MPa	1560	1340	1300	1290	1280	1250	1170
	δ, %	5.9	9.1	10.3	11.0	11.3	10.5	11.8

Table 1. Phase fraction and mechanical properties of the studied steel after heat treatment

After quenching and tempering, the steel microstructure composed of lath martensite. Such a microstructure provides high yield strength, but relatively low ductility. Q&P treatment leads to the formation of a multiphase microstructure, which provides a good combination of strength and ductility. Isothermal treatment at 350°C for 1 hour provide completed bainitic transformation. Steel in this state demonstrate the lowest tensile strength (1170 MPa) and the highest total elongation (11.8%). Theoretical calculations and experimental data allow us to conclude that primary martensite makes the highest contribution to the yield strength, while steel samples with a high fraction of bainitic ferrite and retained austenite demonstrate lower yield strength, but higher ductility.

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

POSITIVE EFFECT OF INCREASING THE QUENCHING TEMPERATURE ON THE CREEP RESISTANCE OF HIGH CHROMIUM STEEL WITH LOW NITROGEN AND HIGH BORON CONTENT

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One of the promising materials for power unit components are martensitic steels with chromium content of 9-12%. The aim of the study is to reveal the effect of quenching temperature on creep resistance of high chromium steel additionally alloyed with cobalt, tungsten, molybdenum and copper, with low nitrogen and high boron content. Heat treatment of the steel consisted in hardening from temperatures of 1050°C or 1100°C for 1 h, cooling in air, followed by tempering at temperatures of 750-770°C for 3 h. Flat specimens with a working length of 25 mm and a cross section of 7 mm \times 3 mm were tested for long-term strength at 650°C at nominal stresses of 200-140 MPa in steps of 20 MPa. Structural studies were carried out by transmission electron microscopy (Jeol JEM 2100).

Modification of heat treatment ($1100^{\circ}C+750^{\circ}C$) led to an increase in the average size of initial austenitic grains by 25% due to a decrease in the volume fraction of MX carbonitrides, as well as to a decrease in the average size of M₂₃ C₆ carbide by 38% with a simultaneous increase in their numerical density along the boundaries by 16% and a decrease in the average size of NbX particles by 3 times.

Modification of heat treatment resulted in a significant increase in creep resistance for all applied stresses. The increase in time to failure was 3 to 4 times after application of 1100°C+750°C treatment. The minimum creep rate decreased by an order of magnitude for all applied stresses. Moreover, the slope of the long-term strength curve decreased from 0.083 after 1050°C+770°C treatment to 0.066 after 1100°C+750°C treatment.

Carrying out the treatment with higher quenching temperature reduces the rate of martensitic laths enlargement by 2.4 times during creep, which is accompanied by a 3.5-fold decrease in the rate of enlargement of behind-the-border particles of the Laves phase. The growth rate of M_{23} C₆ carbides remains low 7E-13 μ m⁴ ×s⁻¹ after both treatments. The lattice structure of tempering troostite stabilised by 50-nm M_{23} C₆ carbide particles and 20 nm NbX particles after the modified heat treatment, as well as the release of more small Lavez phase particles with sizes up to 50 nm along the low-angle martensitic lath boundaries due to the increased extent of such boundaries, provides high stability against short-term creep.

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EFFECT OF QUENCHING TEMPERATURE ON LOW-CYCLE FATIGUE AT ROOM TEMPERATURE OF 10%CR STEEL WITH HIGH BORON CONTENT

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High-chromium steels are a promising material for the manufacture of power plant components that will operate at ultra-supercritical steam parameters (temperature 600–620 °C, pressure 25–30 MPa) [1]. At present, there is a transition to ultra-supercritical steam parameters, which will increase the efficiency of power plants to 45% [2; 3].

Ingots of 10%Cr-3%Co-2W-0.5Mo-0.8Cu-0.2Re-0.003N-0.01B steel were homogenized at 1200°C for 16 h. Heat treatment of the steel included normalization at 1050°C or 1100°C for 1 h and tempering at 770°C for 3 h.

With amplitude of 0.2% for two quenching temperatures, the number of cycles before failure exceeds the conventional low-cycle fatigue (LCF) threshold of 50 thousand cycles, and a decision was made to stop testing before the sample failed. With an increase in the deformation amplitude from 0.3 to 0.6%, the number of cycles to failure decreases by ~ 7 and 18 times for a quenching temperature of 1050 and 1100 °C, respectively. At stress amplitude of 0.3%, the number of cycles to failure of the sample quenched at 1100°C is approximately twice as high as at 1050 °C. With a further increase in the deformation amplitude from 0.6 to 1%, the number of cycles to failure drops by ~ 4 times for both quenching temperatures. With the increase in deformation amplitude, the contribution of the plastic component increases, both at a temperature of 1050 °C and at a temperature of 1100 °C.

At deformation amplitude of 0.3%, softening is observed only after 40 and 50% of the total number of cycles to failure at a quenching temperature of 1050 and 1100 °C, respectively.

After the LCF test at room temperature, the structure of the tempered martensitic lath structure is preserved, and the transformation of the laths into subgrains does not occur. The dislocation density after the LCF test changes at the error level, and no dependence on the deformation amplitude is observed. The width of the laths increases slightly.

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EFFECT OF GRADIENT STRUCTURE ON MECHANICAL BEHAVIOR OF TI-STABILIZED AUSTENITIC STEEL

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New thermo-mechanical processing was proposed. The method included cold rotary swaging up to 95% and post-deformation annealing at 600-700°C. In the current work, the mechanisms of microstructure and texture formation during swaging and subsequent annealing, as well as mechanical behavior of the obtained gradient structure, were investigated. During cold rotary swaging, dislocation sliding and mechanical twinning of the rod was realized in the workpiece center. As a result, a twin-matrix microstructure was formed. At the rod edge, because of more intense plastic deformation, the shear banding was dominant, which led to the formation of an ultra-fine grained microstructure. The difference in the mechanisms of plastic deformation of the center and edge of the workpiece affected the texture development. An axial <001>/<111> texture was detected in the rod center. At the same time, a shear B/\overline{B} texture was observed at the edge of the rod. Post-deformation annealing at 600-700°C had virtually no effect on the microstructure and texture in the rod center. However, the small amount of recrystallized grains was detected at the edge after annealing at 700°C. In this case, the shear texture was preserved. The effect of the structural and textural gradient on mechanical behavior was clearly observed after annealing at 600-700°C. So, the gradient-structured material performed increased yield strength and attractive ductility.

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THERMAL AND DEFORMATION MARTENSITE FORMATION IN THE MEDIUM-ENTROPY ALLOY Fe40Mn40C010Cr10

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The microstructure, phase composition and microhardness of cast, rolled and homogenized $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ samples were studied. The ingots of the $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ alloy were produced by vacuum induction melting and casting in a pure Ar atmosphere. The homogenization was performed by cold rolling to 65% thickness reduction, followed by annealing at four different modes, where temperature (T) and time (t) of the annealing were varied: mode I (T = 1200 °C, t = 2 h), mode II (T = 1200 °C, t = 1 h), mode III (T = 1100 °C, t = 2 h), mode IV (T = 1100 °C, t = 1 h).

In the cast state, the studied $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ alloy had a coarse-grained dendritic structure, represented by two phases: austenitic (γ -phase) and thermally-induced martensitic (ϵ -phase). Using the dispersive spectroscopy method, it was found that the dendritic regions of the specimens were depleted in manganese (up to ≈ 35 at. %), which contributed to the martensitic transformation at room temperature.

The deformation of cast specimens of the alloy by cold rolling led to the formation of deformation martensite as a result of plastic deformation. The microhardness H_{μ} of the rolled specimens of the Fe₄₀Mn₄₀Co₁₀Cr₁₀ alloy significantly increased compared to the cast state: 3.4 GPa and 1.4 GPa, respectively. The reason for this is the formation of a structure with a high density of dislocations and martensitic plates, revealed by transmission electron microscopy.

The phase composition of the samples homogenized in modes I–IV, according to the results obtained by X-ray structural analysis and electron backscatter diffraction, is represented only by the austenitic phase. The average grain sizes were $148 \pm 77 \ \mu\text{m}$, $103 \pm 47 \ \mu\text{m}$, $40 \pm 20 \ \mu\text{m}$, $35 \pm 17 \ \mu\text{m}$ for the samples subjected to thermomechanical treatment in modes I–IV, respectively. The formation of finer grains in such samples relative to the cast state caused grain boundary strengthening and, as a consequence, an insignificant increase of microhardness H_{μ} : for samples homogenized in mode I – 1.7 GPa, in mode IV – 2 GPa.

Thus, the inhomogeneous distribution of chemical elements, which is depletion of dendrites in manganese, contributes to the martensitic transformation in the cast state of $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ alloy at room temperature. Plastic deformation assists to the formation of deformation martensite, which is accompanied by an increase of the microhardness H_{μ} by more than two times relative to the cast specimens. The application of thermomechanical treatments leads to the formation of a single-phase austenitic homogeneous structure with a large grain size and microhardness of ≈ 2 GPa.

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MACHINE LEARNING-BASED PREDICTION OF THE OXIDATION RATE OF HIGH-ENTROPY ALLOYS

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The compositional space of high-entropy alloys is incomparably larger than that of conventional alloys. Therefore, developing the methods for forecasting of a structure, mechanical, and functional properties of the new high-entropy alloys is a challenge of a modern material science.

Machine learning algorithms show high efficiency for predicting mechanical properties (yield strength, plasticity), phase composition. In this work, machine learning was used to predict the oxidation behavior of HEAs. The trained surrogate model showed good accuracy in predicting the oxidation rate over a wide temperature range.

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EFFECT OF PROCESS PARAMETERS OF WIRE-ARC ADDITIVE MANUFACTURING ON STRUCTURE AND PROPERTIES OF LOW-ALLOY STEEL

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Additive manufacturing is a promising approach for fabrication of metal components with complex geometry in a short production time for various industries. Additive technologies based on the use of an electric arc as a heat source and a filler material as a wire demonstrate a number of advantages, such as rapid forming speeds, higher deposition efficiency, and lower costs of equipment and the components, which is especially important for the production of large-sized parts for the heavy industry. Wire arc additive manufacturing (WAAM) technology has a high potential for increment process performance, which can be realized through increasing the electric arc energy. However, this may be complicated by a significant increase in technological defects, internal stresses, and loss of geometric stability. In addition, complex thermal history during highenergy WAAM process including repeated thermal cycles, rapid heating and cooling rates, can negatively affect the mechanical characteristics, in particular, due to the formation of specific microstructure and a large number of structural defects. In this study, the influence of parameters during WAAM process on the microstructure and mechanical properties of the 0.08%C-1.0%Cr-1.2%Mn-0.55%Si-0.6%Mo-0.25%V steel was investigated. Features of the structure and phase composition of steel was studied by using scanning electron microscopy and EBSD analysis. The optimal parameters (power, current, wire feed speed, shielding gas flow rate) of the process for fabrication of low-alloy steel thin walls was determined.

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DIRECT LASER METAL DEPOSITION OF HIGH-ENTROPY ALLOY OF Ti-Nb-Cr SYSTEM

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High-entropy alloys (HEAs) are a type of alloy consisting of several elements with the same or similar atomic ratio, where the atomic concentration of each element is approximately 5 to 35 %. The term 'high entropy' indicates the high configurational entropy in the alloy that results from the random distribution of these elements in the crystal lattice.

Previously, traditional methods such as melting and sintering have been used to create highentropy alloys (HEAs). However, obtaining consistent bulk HEAs by arc melting is a challenging task that requires additional processing such as remelting. As a more modern and efficient alternative to traditional methods, three-dimensional (3D) printing or additive manufacturing is considered as a promising technology for producing HEAs with fewer defects.

Among all additive manufacturing technologies for the creation of HEAs, the direct laser metal diposition (DLD) method stands out. This state-of-the-art technology allows the creation of complex three-dimensional metal structures through the gradual melting and deposition of metal powder onto a workpiece using a laser. This process gives the possibility to precisely control

temperature gradients and cooling rates, which directly affects the formation of the microstructure and material characteristics.

In this work we focus on the study of direct laser metal diposition of high-entropy alloys based on NbTiCr. We will analyse how the main parameters of the DLD process affect the structure of the obtained alloys, investigate their microstructure and phase composition, and discuss the potential of their application in various fields of engineering.

During the research process, data was collected that can help further develop high-entropy alloys and additive technologies, opening new perspectives for the development of innovative materials with improved properties.

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DIRECT ENERGY DEPOSITION OF FeCoNiCr-Al-AlTi HIGH-ENTROPY ALLOYS FROM PURE POWDERS

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High-entropy alloys (HEA) are a type of alloy composed of several major elements with the same or close atomic ratio, where the atomic concentration of each element is approximately 5% to 35%. The term "high-entropy" indicates the high configurational entropy in the alloy, which is created by the random colonization of lattice sites by several species at significant concentrations on a single solid solution lattice.

Several conventional methods, including melting and sintering, have previously been used to produce HEAs. However, fabrication of homogeneous bulk HEAs by arc melting is challenging because several subsequent treatments (e.g., remelting) are required. As an advanced alternative method to overcome the limitations of traditional processing methods, three-dimensional (3D) printing or additive manufacturing (AM) is considered as a promising technology for manufacturing HEAs with fewer defects.

Among all AM techniques for HEAs fabrication, Direct Energy Deposition (DED) deserves special attention. DED is an advanced additive manufacturing technique that enables the creation of complex three-dimensional metal structures by stepwise melting and depositing metal powder with a laser. This process allows precise control of temperature gradients and cooling rates, which affects the formation of the microstructure and material properties.

In this paper we focus on the study of Direct Energy Deposition of high-entropy alloys of the FeCoNiCr, FeCoNiCrAl and FeCoNiCrAlTi systems. We will consider the influence of key parameters of the DED process on the structure and properties of the obtained alloys, and analyze their microstructure and phase composition. We will also focus on the mechanical properties of HEAs obtained via DED and discuss their potential applications in various branches of engineering.

The research has yielded data that can contribute to the further development of high-entropy alloys and additive manufacturing technologies, opening new horizons for the creation of innovative materials with improved characteristics.

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VACUUM-FREE ELECTRIC ARC SYNTHESIS OF TANTALUM-DOPED HIGHER TUNGSTEN BORIDE

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In recent years, there has been a rising interest in new materials that possess the strength, durability, and resistance required to withstand increasingly demanding operating conditions. A particularly promising area of research is the development of high-entropy materials. Higher tungsten borides have a hardness greater than that of industrial tungsten carbide, positioning them as a promising alternative for drilling technologies to replace WC-based hard alloys [1]. Previously, our laboratory successfully synthesized the higher tungsten pentaboride phase WB_{5-x} using a vacuum-free electric arc method [2]. It is promising to dope WB_{5-x} with other transition metals, such as tantalum, to synthesize the WTaB_{5-x} phase, which can significantly improve the mechanical properties of the material.

The vacuum-free electric arc method is regarded as a promising approach for synthesizing various carbides and borides [2-3]. This technique enables material synthesis in electric arc plasma in an open-air environment, made possible by the self-shielding effect of the reaction zone, created by the emitted carbon monoxide and carbon dioxide gases. Consequently, there is no need for a sealed chamber, vacuum equipment, or additional systems to maintain an inert atmosphere, which simplifies the arc reactor design and synthesis process while enhancing energy efficiency.

In this work, a series of experiments was conducted to produce tantalum-doped tungsten pentaboride with varying ratios of precursors, synthesized using a vacuum-free electric arc method.

The research was carried out with the support of the Ministry of Science and Higher Education of the Russian Federation within the framework of the scientific project FSWW–2023–0011. References:

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MICROSTRUCTURE, PHASE COMPOSITION AND MECHANICAL PROPERTIES OF (Fe40Mn40C010Cr10)100-x(N)x MEDIUM-ENTROPY ALLOY

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At this study, the microstructure, phase composition and mechanical properties of the multicomponent medium-entropy alloy $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ and nitrogen-doped alloys based on it were studied using scanning electron microscopy, X-ray diffraction analysis and uniaxial tensile testing.

According to the results of the electron back-scattered diffraction analysis, homogenization led to the formation of a single-phase large-crystalline structure in all alloys with average grain sizes of 151 μ m, 174 μ m and 180 μ m for Fe₄₀Mn₄₀Co₁₀Cr₁₀, (Fe₄₀Mn₄₀Co₁₀Cr₁₀)_{99.4}(N)_{0.6} and (Fe₄₀Mn₄₀Co₁₀Cr₁₀)_{98.8}(N)_{1.2}, respectively. Both X-ray diffraction analysis and electron microscopic studies indicate that an austenitic structure (a face-centered cubic crystal lattice) has formed in all homogenized alloys.

Alloying the Fe₄₀Mn₄₀Co₁₀Cr₁₀ alloy with nitrogen provided a significant increase in its yield strength ($\sigma_{0.2}^{0.N} = 180$ MPa, $\sigma_{0.2}^{0.6N} = 260$ MPa, $\sigma_{0.2}^{1.2N} = 270$ MPa) and ultimate tensile strength ($\sigma_{UTS}^{0N} = 500$ MPa, $\sigma_{UTS}^{0.6N} = 590$ MPa, $\sigma_{UTS}^{1.2N} = 610$ MPa), while there was a predicted decrease in plasticity ($\delta^{0N} = 80$ %, $\delta^{0.6N} = 74$ %, $\delta^{1.2N} = 65$ %).

The fracture structure of the tensiled samples mainly has a viscous transcrystalline appearance with a characteristic dimpled relief that corresponds to the plastic fracture of a material.

Thus, when alloying the $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ medium entropy alloy with nitrogen, its mechanical characteristics increase at room temperature due to the implementation of solid-solution hardening while maintaining high ductility and fracture toughness.

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"SUPERPLASTICS" MATERIAL FLOW DURING FRICTION-STIR WELDING OBSERVED BY THE "STOP-ACTION TECHNIQUE"

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This work was undertaken to provide an insight into microstructural processes and material flow during friction-stir welding. To this end, the stop-action technique was applied and a typical 6013 aluminum alloy was used as a program material. It was found that the microstructural evolution included several stages. Specifically, the initial material underwent the discontinuous static recrystallization in the heat-affected zone. With the approach of the rotating tool, the recrystallized grains experienced continuous dynamic recrystallization, which resulted in grain refinement. The subsequent transportation of material around the rotating tool provided no significant alterations in microstructure. This "superplastic-like" character of material flow was attributed to a dynamic balance between grain refinement and grain coarsening. It was also found that the stirred material experienced a secondary deformation induced by the rotating tool shoulder far behind the welding tool. The concomitant microstructural changes were most pronounced at the upper weld surface and gave rise to a fine-grained layer.

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STRUCTURE, MECHANICAL PROPERTIES, AND OXIDATION BEHAVIOUR OF REFRACTORY (HfNbTaTiZrX)84Si16 (X = M0; M0V; CrMoV) COMPLEX CONCENTRATED ALLOYS

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The development of alloys capable of operating at extreme temperatures beyond nickel superalloys and having an acceptable level of plasticity is highly demanded in aerospace sector. In this context, refractory complex concentrated alloys (RCCA), also called high/medium entropy refractory alloys (RH/MEAs), are very promising materials. Recently, special attention has been given to silicide-containing RCCAs, which are a group of ceramic-reinforced alloys that demonstrate prospective mechanical properties.

In current study, we presented a systematic investigation of structure, mechanical properties, and oxidation behaviour of a series (HfNbTaTiZrX)₈₄Si₁₆ (X = Mo; MoV; CrMoV) alloys. Preliminary results showed that certain alloys had a better combination of specific yield strength at 1200°C, fracture toughness and mass gain after 1 hour at 1200°C compared to most modern RCCAs.

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LASER WELDING OF TI-6AL-4V AND AL-5MG-SC ALLOYS USING HIGH ENTROPY INTERMEDIATE LAYERS

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A new method of laser welding of dissimilar alloys using intermediate layers of mediumand high-entropy alloys (HEA) to prevent the formation of intermetallic compounds in the weld is proposed. Experimental compositions of intermediate layers for laser welding of Ti-6Al-4V and Al-5Mg-Sc alloys are selected. The calculation of intermediate layers was carried out by the Thermo-Calc software according to the criteria of the required minimum interval of crystallization. Selected element compositions should predominantly form BCC solid solutions with Ti-6Al-4V and Al-5Mg-Sc alloys in a wide range of element concentrations and temperatures. So, TiNbZr, HfNbTaTiZr, and CoNbNiVCu systems of intermediate layers were determined. To find parameters of laser welding, preliminary studies have been conducted, including the selection of welding modes, microstructure characterization, and tensile mechanical testing of welded joints.

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THE EFFECT OF AGE-HARDENING ON HYDROGEN EMBRITTLEMENT OF HIGH ENTROPY CANTOR 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. A high-entropy Cantor alloy (Co₂₀Cr₂₀Fe₂₀Mn₂₀Ni₂₀) was chosen as an object for the study. The cast alloy was subjected to a thermomechanical treatment, including solid-solution treatment (SST, 1200 C, 2 h), cold rolling (80% reduction) and final SST (1200 C, 2 h). Some of the specimens were studied in the coarsegrained state after SST (HEA). Part of the specimens was subjected to age-hardening at 900°C for 1 hour (σ -HEA) to form a grain boundary σ -phase (σ -HES). Electrochemical hydrogen charging was carried out at a current density of $j_{\rm H} = 10$ mA/cm² for 50 h at room temperature in a 3% aqueous NaCl solution in the presence of 3 g/l NH₄SCN as a recombination poison.

The results of X-ray diffraction analysis showed the formation of an austenitic phase structure in HEA and σ -HEA specimens. However, electron microscopic studies have shown that age-hardening leads to the precipitation of the intermetallic σ -phase, enriched with chromium, along the grain boundaries in σ -HEA specimens. An analysis of room temperature tensile properties shows close values of the yield strength ($\sigma_{0,2}$) and elongation to failure (δ) for HEA and σ -HEA specimens. Hydrogen charging is accompanied with significant change in their mechanical properties. The yield strength increases ($\Delta \sigma_{0,2}$) in the hydrogen-charged specimens, which is caused by solid-solution hardening be hydrogen. The hydrogen embrittlement index (I_H), which describes the hydrogen-induced loss of elongation, for HEA specimens is 27%, that is 2 times higher than that for σ -HEA specimens, I_H^{A-HEA} = 12%.

The formation of the grain-boundary σ -phase particles, and, as a consequence, the creation of additional "particle/matrix" interfaces, helps to increase the resistance of the Cantor alloy to hydrogen embrittlement in terms of macromechanical behavior. 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. The main factors that determine the thickness of brittle surface zones formed during hydrogen charging and subsequent uniaxial tension of hydrogen charging specimens have been identified, and the micromechanisms of fracture have been established. It is shown that the formation of the grainboundary σ -phase particles increases the diffusion transport of hydrogen along the interfaces during plastic deformation, ensuring the formation of secondary cracks in the central part of the specimens, while the dislocation transport of hydrogen in heterophase specimens is suppressed.

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STRUCTURE, MECHANICAL PROPERTIES, AND OXIDATION RESISTANCE OF LIGHTWEIGHT AI-Cr-Ti-Fe-Mn COMPLEX CONCENTRATED ALLOYS

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The increasing demand for lightweight materials that can withstand high temperatures while maintaining strength is critical for high-performance engineering applications, such as the aerospace and energy sectors. In this context, complex concentrated alloys (CCAs), including high-entropy alloys (HEAs) attract special attention. Particularly, lightweight CCAs can stand as promising alternatives to nickel-based superalloys.

In this study, we presented a series of lightweight CCAs based on the Al-Cr-Ti-Fe-Mn system. By systematic and comprehensive investigations of the structure, mechanical properties, and oxidation resistance, we showed certain alloys to exhibit exceptional synergy between specific yield strength at 1000 °C and compressive ductility at room temperature, as well as a lower mass gain after 100 h at 1000 °C compared to both lightweight and the most oxidation-resistant refractory complex concentrated alloys (CCAs). The relationships between chemical composition, structure, and properties were thoroughly discussed.

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CORROSION RESISTANCE, WEAR RESISTANCE AND BIOCOMPATIBILITY OF TiNbZr-BASED COMPOSITES REINFORCED WITH BORIDES

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Knowing the undoubted advantages of medium-entropy alloys of the Ti-Nb-Zr system in the form of a low elastic modulus, high specific strength, excellent corrosion resistance and biocompatibility, the areas of application of these alloys are often limited due to their known weak points, which as low strength, hardness and wear resistance. One of the most suitable ways to strengthening for titanium-based alloys is the use of TiB particles (fibers) as a high-strength reinforcing component. The use of similar reinforcing components to enhance mechanical properties of titanium and its alloys such as absolute strength and hardness has been adopted by many researchers. It is already well known that TiB fibers, which has good relationship to the titanium matrix, similar coefficient of thermal expansion with Ti and good thermal stability. Despite the extensive work conducted on Ti(Ti-alloys, Ti-rich alloys)-TiB composites, very little is known about their biocompatibility which has so far precluded their use in bioapplications. There are also a fairly limited number of studies on the corrosion resistance and wear resistance of Ti(Ti-alloys, Ti-rich alloys)-TiB composites, which show the absence of a negative effect from the presence of borides in the titanium matrix. At the same time, a very interesting and more littlestudied issue is the effect of the addition of TiB particles on the corrosion resistance and wear resistance of the equiatomic Ti-Nb-Zr matrix, as well as on its biocompatible properties. Thus, in this work, attention is focused on the study of corrosion resistance, wear resistance and biocompatibility of composites based on equiatomic TiNbZr alloy with different content of borides.

TiNbZr-TiB composites were produced by vacuum arc melting; the weight amount of TiB₂ was 0.7 (Alloy A) and 4.0 (Alloy B) wt. %; the unreinforced TiNbZr alloy was also fabricated without the addition of TiB₂. Microstructure of the TiNbZr-TiB composites consisted of the TiNbZr β matrix and (Ti,Nb)B fibers, the fraction of which increases with an increase in the amount of TiB₂ in the charge mixture. The borides had a needle-like shape with the average diameter ~ 0.4 and ~ 2.0 µm for alloys A and B, respectively. The volume fraction of the borides in the structure of four conditions of the composites was found to be ~ 2.5 and ~ 12.4 % for Alloys A and B, respectively. The introduction of 4 % TiB₂ into the alloy reduces the corrosion resistance of the Alloy B in comparison with the unreinforced TiNbZr alloy and Alloy A. Alloy A and unreinforced alloy showed similar values of corrosion resistance. It was found that the addition of the TiB₂ to the TiNbZr alloy leads to a decrease in the friction coefficient; when adding 0.7 % TiB₂ to the alloy (Alloy A), the friction coefficient decreased from 1.15 to 1.13, and when the percentage of TiB₂ in the alloy increased to 4 % (Alloy B), the friction coefficient decreased by ~ 2 times from 1.15 to 0.58. Full biocompatibility of TiNbZr-TiB composites was demonstrated; no significant differences from the unreinforced state and alloy were found.

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STRUCTURE, MECHANICAL PROPERTIES, AND OXIDATION BEHAVIOUR OF REFRACTORY NBTIZRX (X = V, Mo, Ta) COMPLEX CONCENTRATED ALLOYS

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Refractory complex concentrated alloys have garnered attention due to their high melting points and exceptional resistance to softening, making them ideal for high-temperature applications. The incorporation of multiple elements as base components offers a vast array of possible alloy combinations. However, exploring such an extensive compositional space is time-consuming. Analysis of published work shows that complex concentrated alloys often contain Nb, Ti and Zr. A promising equiatomic NbTiZr alloy has demonstrated remarkable mechanical properties: the alloy has a relatively low density and exhibits a balance of strength and ductility at room temperature. Unfortunately, the alloy has a significant drop in strength at elevated temperatures. Our current work aims to evaluate the effect of frequently used alloying elements and their combinations on the phase composition and mechanical properties of the NbTiZr alloy. In this study, we investigated the influence of refractory elements on the structure, mechanical properties, and oxidation resistance of Nb-Ti-Zr-X (X = V, Mo, Ta) alloys. The strong correlation between experimental and computational data underscores the effectiveness of using CALPHAD and machine learning to accelerate the development of complex-concentrated alloys.

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STRUCTURE AND MECHANICAL PROPERTIES OF GRADIENT-STRUCTURED METASTABLE STAINLESS STEEL SUBJECTED TO POST-DEFORMATION ANNEALING

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Microstructure and mechanical properties of a metastable austenitic stainless steel after cold rotary swaging and subsequent annealing are investigated. The gradient of microstructure and texture is detected after cold rotary swaging to an area reduction of ~90%. After annealing at 823-923 K, the martensitic mechanism of $\alpha' \rightarrow \gamma$ transformation is observed. Annealing at 973K and 1073K is accompanied by the diffusional $\alpha' \rightarrow \gamma$ transformation mechanism and static recrystallization. Annealing at 823-923 K results in the development of the axial (111) texture in the workpiece center. The axial (100) texture gradient is found after annealing at 973 K. After annealing at 823-923 K, the program material exhibits superior mechanical properties.

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INFLUENCE OF MAGNETRON SPUTTERING REGIMES ON THE STRUCTURE AND PROPERTIES OF THIN HIGH ENTROPY FILMS CoCrFeNiCu

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In recent years, the field of materials science has experienced a revolution with the emergence of high-entropy alloys (HEAs), a new class of metallic materials. These alloys consist of five or more principal components in concentrations ranging from 5% to 35%, giving them unique mechanical, electrical, and magnetic properties [1]. In recent work [2], it has been demonstrated that HEA films can be promising for use as resistive and thermoelectric materials. However, methods for controlling their electrical properties remain in the early stages of research.

The aim of this work was to extend the existing knowledge on controlling the electrical properties of HEAs, as they are currently in their infancy. For this purpose, the well-studied CoCrFeNiCu in bulk form was used. The CoCrFeNiCu HEA system is of particular interest due to its outstanding mechanical properties, such as high strength and wear resistance [3]. It also demonstrates attractive electrical and magnetic properties. This work presents a comprehensive study of the impact of sputtering time and power on the structure, elemental composition, and electrical properties of HEA films. The resulting films had a smooth structure with uniform elemental distribution, and their resistivity ranged from 23,000 to 34 $\mu\Omega$ ·cm, with the best temperature coefficient of resistance (TCR) reaching 240 ppm/°C. The CoCrFeNiCu-based films also showed a high power factor (PF) of 2.5 mW/(m·°C²) at 177 °C. Increased sputtering power and time were found to enhance the crystallinity of the HEA films.

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CRYOGENIC MECHANICAL PROPERTIES OF A MEDIUM ENTROPY Fe65C012.5Ni12.5Cr9.5C0.5 ALLOY PRODUCED BY LASER-BASED POWDER BED FUSION

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One of the most advanced methods for manufacturing parts with complex geometries using high- and medium-entropy alloys (HEAs/MEAs), is laser-based powder bed fusion (PBF-LB). PBF-LB enables the rapid production of high-quality alloy components, with high cooling rates during the process often leading to the formation of a fine-grained microstructure that typically enhances the material's mechanical properties.

This study examines the microstructure and mechanical properties of the medium-entropy $Fe_{65}Co_{12.5}Ni_{12.5}Cr_{9.5}C_{0.5}$ alloy, fabricated using the PBF-LB method. The alloy microstructure and cryogenic mechanical properties were analyzed both in the as- printed condition and after heat treatment (annealing at 800°C). In the as- printed condition, the alloy exhibited a face-centered cubic (fcc) single-phase microstructure. After annealing, body-centered cubic (bcc) martensite plates and $M_{23}C_6$ carbides were formed in the fcc phase matrix. Tensile testing at 77 K resulted in a significant improvement in the alloy's mechanical properties compared to room temperature testing. In both the as-printed and heat-treated conditions, the ultimate tensile strength (σ_B) was 1800 MPa vs 1850 MPa (four times higher than that at room temperature), while elongation to fracture (δ) remained consistent (26% and 25%, respectively). The reasons for the enhancement of the alloy's mechanical properties under cryogenic deformation are discussed in detail in this work.

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PRACTICAL APPLICATION OF HIGH-ENTROPY MATERIALS

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High-entropy alloys (HEA) are a new strategy for alloying alloys, which consists in creating a combination of several basic elements in equiatomic or close to it concentration. The main feature of this approach to alloying alloys is the creation of a multidimensional compositional space, and although several hundred alloys with properties superior to conventional ones have been developed to date, only small areas of it have been studied.

The vast majority of alloys developed to date are formed by three main groups of elements: 3d transition Co, Cr, Cu, Fe, Mn, Ni, Ti, V, etc., refractory Cr, Hf, Mo, Nb, Ta, Ti, V, W, Zr, rare earth Dy, Gd, Ho, Tb, Y, etc. Within these groups, with a certain combination of elements, substitutional or interstitial solid solutions can be created as a matrix base for alloys. These are alloys of the following systems: Co-Cr-Fe-Mn-Ni, Hf-Nb-Ta-Ti-Zr or Nb-Ta-Mo-W, as well as, for example, the Dy-Gd-Ho-Tb-Y system, respectively, fcc, bcc and hcp structures. We also consider alloys based on only three main elements, such as CoCrNi (fcc), NbTiZr (bcc), etc., or two with additional elements of lower concentration. Alloys of the Co-Cr-Fe-Mn-Ni system are close in structure and properties to austenitic steels, and those created based on refractory elements, due to the peculiarities of their characteristics, to heat-resistant alloys. Modification of the formation of, for example, TRIP/TWIP alloys, as well as dispersion-hardened alloys due to the formation of carbides, nitrides, oxides and intermetallic compounds. Using the features of alloying and phase transformations in these systems, multiphase alloys, single- and two-phase intermetallic alloys can be created.

The results of numerous studies show the possibility of developing high-strength alloys based on high-melting elements, alloys with special properties: cold-, corrosion-, wear- and radiation-resistant, heat-resistant, magnetic and catalytic. Examples of alloys of practical interest are given.

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SYNTHESIS OF CAST RHEAS BASED ON THE Mo-Nb-Ta SYSTEM BY SHS METALLURGY FOLLOWED BY MAKING OF COMPOSITE MATERIALS ALLOY-MATRIX/OXIDE-FIBER

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Among the family of high entropy alloys (HEAs), the alloys consisting predominantly of refractory elements (Mo, Nb, Ta, W, Ti, Cr, Zr, Re, Hf, V etc.) have attracted special attention. Such alloys are called refractory high-entropy alloys (RHEAs). RHEAs demonstrate a high melting point and, as a result, high strength characteristics at elevated temperatures, which makes them potential materials for replacing nickel superalloys in new-generation turbine-type power plants, where an increase in operating temperatures is required above those established for modern nickel superalloys (up to 1100 C). However, when developing RHEAs, difficult problems arise associated with the conflict between strength and crack resistance. These difficulties can be overcome in the case of a combination of fiber composites and a metal matrix.

It should be noted that the high melting point of the constituent elements of RHEAs creates many technological problems in their production. There are many laboratory methods for obtaining RHEAs (VAR, VIR, EBM, SPS, MA+SPS, 3D AT etc.). The main common disadvantages of these methods are the need to use pure metals, high power-intensity of the processes and very low productivity, which can only be satisfied at the stage of scientific research.

Previously, the authors successfully tested the possibility of obtaining cast HEAs and metal composites based on 3D metals (Co–Cr–Fe–Ni–Mn) by centrifugal SHS metallurgy methods [1]. This method is based on the use of oxide raw materials (oxides of target metals) and chemical energy released during highly exothermic reactions in the combustion wave of the original powder components. This approach is significantly cheaper than the synthesis of such alloys by melting pure components, in addition, the high synthesis temperature (above 2200 C°) and the effect of centrifugal forces (overload) allow achieving maximum dissolution of the alloy components.

In frame of the presentation, we will present a brief review of previous research and latest results on obtaining cast RHEAs based on the Mo-Nb-Ta system alloyed with Cr, V, Zr, Hf, Ti metals by centrifugal assisted SHS metallurgy. The synthesis of cast RHEAs of such a composition was studied for the first time. Based on the synthesized alloys, methods for forming composite materials alloy-matrix/oxide-fiber were tested. These studies are at an early stage and the results obtained will also be covered in the report.

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COMPARATIVE RESULTS OF COMPUTER MODELLING OF TiVZrCrAI HIGH-ENTROPY COATING DEPOSITION AND THE EXPERIMENT

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At present, increasing the reliability and service life of compressor blades of gas turbine engine (GTE) is achieved through the use of special protective coatings that provide the necessary set of performance characteristics during operation.

To develop a coating with all the properties (erosion, corrosion resistance, ability to work at high temperatures), it is necessary that nitrides of various metals are formed in the functional layer of the coating. This can be achieved by coating high entropy alloys, which are also known as multi-component or multi-base alloys. Group IV-VI transition metals (Ti, Zr, V) in coatings provide higher hardness, erosion resistance, wear resistance, the introduction of Cr and Al leads to the formation of protective oxides on the alloy surface to ensure its resistance to oxidation.

This paper compares experimental results of vacuum-arc deposition of high entropy alloy TiVZrCrAl with the data of computer modelling in the COMSOL Multiphysics software package.

Experimental investigations of deposition of high-entropy coatings of TiVZrCrAl composition on titanium alloy Ti-6Al-4V substrate under variation of technological parameters

have shown that the distribution of elements is Ti(21.1-23.8%) Al(8.5-14.9%) V(16.3-21.4%) Zr(21.2-25.8%) Cr(20.8-27.3%), and the distribution of coating thickness is $1.7-6.2 \mu m$.

According to the results of calculations and computer modelling, the distribution of coating thickness on the substrate surface is $6.5-7.5 \,\mu\text{m}$ and the distribution of elements in the single layer coating is Ti(17%)Al(41%)V(14%)Zr(12%)Cr(15%).

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EFFECT OF NITROGEN AND VANADIYM ON STRUCTURE AND MECHANICAL BEHAVIOR OF HIGH ENTROPY CO-CR-FE-NI-MN SYSTEM

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High-entropy alloys are considered to be one of the currently promising classes of materials. One of the most studied is the so-called Cantor CoCrFeMnNi alloy, which has a single-phase FCC structure. This alloy exhibits high ductility, impact toughness, and fracture toughness, with a decrease in test temperature to 77K contributing to an increase in mechanical properties. However, this alloy has a number of disadvantages: high content of expensive elements (Ni and Co) and low strength at room temperature ($\sim 180 - 200$ MPa in recrystallized state). Modern alloy design approaches allow changing the composition to maintain and/or increase the mechanical properties and reduce the concentration of expensive elements. Thermomechanical processing can be effectively used to improving the mechanical properties. In addition, alloying with interstitial elements, in particular nitrogen, can lead to significant hardening. However, a large amount of nitrogen in the matrix can lead to material softening. To prevent this behavior, it was proposed to "bond" nitrogen atoms into nitrides by adding a strong nitride-forming element, vanadium.

In this work, in present work we have studied the effect of nitrogen doping on the structure and mechanical properties alloys based on CoCrFeMnNi with reduced concentrations Ni and Co were alloyed with nitrogen and vanadium. Vanadium was added in the ratio with nitrogen 1:2 to obtain stable nitrides. Such alloying led to the release of vanadium nitrides. The yield strength of the alloys increased proportionally with nitrogen and vanadium content at room and cryogenic temperature. The as-cast alloys were cold rolled to a thickness reduction of 80%, followed by annealing in the temperature range from 700 to 1000°C. Relationships between chemical composition, processing conditions, structure, and mechanical properties were discussed.

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

STRUCTURE AND PROPERTIES OF GRADIENT MEDIUM-ENTROPY MATERIAL OBTAINED BY DIRECT LASER DEPOSITION

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Recently, metal scientists have been tasked with the development of technological processes for obtaining and processing materials with a high set of mechanical and operational properties, including at temperatures below room temperature. One of the promising areas of development can be associated with functionally graded materials with unique mechanical, technological and special properties. To date, there are many strategies for the formation of a gradient structure in metallic materials, starting with traditional casting or plastic deformation and ending with a relatively new direction - additive technologies. Of particular interest is the production of gradient medium-entropy materials based on Fe-Cr-Co-Ni-C alloys by additive technologies, which have TWIP/TRIP effects and demonstrate high mechanical characteristics. At the same time, the direct laser deposition (DLD) method is an effective and promising method of additive production of gradient material (GM), due to the possibility of layer-by-layer composition change by varying the feed rate of different powders. Meanwhile, the relationship between the composition, structure and properties in promising gradient materials based on medium-entropy alloys, especially those obtained by additive technologies, is practically unexplored.

This paper discusses the structure and properties of a GM with Fe variation across the crosssection (from 45 at.% to 80 at.% with a step of 5 at.%) based on the Fe-Cr-Co-Ni-C system with a metastable structure obtained by the DLD method. The structure and mechanical behavior of layers/alloys under tension at room and cryogenic temperatures were studied. The GM has a coarse-grained, non-uniform microstructure consisting of complex and small cells in the form of colonies growing in different directions. It was established that at a Fe concentration of up to 55 at.%, the layers in the GM have a single-phase fcc structure, and at a concentration of 60 at.% and higher, they consist of fcc and bcc phases. It is shown that an increase in strength and a decrease in the plastic properties of layers/alloys are observed at an Fe concentration of 70 at.% and higher. Thus, the maximum strength values of 1190 MPa are demonstrated by the Fe80 (80 at.% - Fe) layer with the plasticity of 3%. The mechanical properties of the GM layers are increased at cryogenic temperatures of testing. Laver Fe65 demonstrates noticeable strain hardening, which is associated with the development of the effect of plasticity induced by transformation (TRIP). Whereas, layers/alloys of Fe70, Fe75 and Fe80 were destroyed in the elastic region in the stress range of 1101-1257 MPa. The relationship between technology, structure and mechanical properties of the alloy is discussed.

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THE EFFECT OF CELLULAR REACTION ON MECHANICAL BEHAVIOR AND MICROSTRUCTURE EVOLUTION OF β -SOLIDIFIED γ -TIAL BASED ALLOY DURING HOT DEFORMATION

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In the last few decades, there has been an active study of β - solidified γ -TiAl based alloys [1]. Such alloys have a high possibility for use as gas turbine engine blades in high-pressure compressors and low-pressure turbines [1]. The low ductility in a wide temperature range significantly complicates processing [2]. An improvement in mechanical properties, in particular strength and ductility, can be achieved with a decrease in the size of lamellar colonies [3]. Hot deformation makes it possible to significant refine the structure. However, hot deformation accompanied by high flow stresses and localization of deformation, which does not allow for a high volume fraction of recrystallized/spheroidized structure. One of the ways to reduce stresses and increase the proportion of volume fraction of recrystallized/spheroidized structure is modification of initial structure. Using heat treatment states with thin-lamellar (HT1) and metastable α_2 -grain structures (HT2) were obtained. A study of the microstructure of the alloy after heating to the deformation temperature showed that in the HT1 state the thin-lamellar structure was stable, while heating the α_2 -grain structure leads to the formation of $(\alpha_2 + \gamma)$ nanolamellar colonies and the development of a cellular reaction with the formation of coarselamellar colonies. It has been established that in the HT2 state, compared to HT1, the flow stresses are significantly reduced and the nature of the mechanical behavior changes. It was shown that this behavior was caused by the localization of plastic flow in coarse-lamellar colonies, the formation of which continues during deformation, and the development of recrystallization and spheroidization processes in them. It was revealed that by the final stage of deformation, the proportion of recrystallized/spheroidized structure increases in the HT2 state to 87%, while in HT1 it is only 50%. Mechanical behavior, structural evolution, and plastic deformation mechanisms are discussed.

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DESIGN OF STRUCTURAL METALLIC MATERIALS FOR ADDITIVE MANUFACTURING

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Additive technologies provide unique capabilities for the efficient production of complex shaped parts and are increasingly used in various industrial applications. But in products produced by additive technologies, a specific structure is formed, determined by a thermal cycle, which includes repeated cycles of heating and cooling. This thermal cycle is radically different from the conventional thermal or thermomechanical processing regimes recommended for commercial alloys. As a result, a coarse-grained structure, elongated in the growth direction, is often formed, caused by epitaxial growth of grains. The formation of defects (cracks) along the boundaries of epitaxial grains and/or the formation of undesirable phases, also leading to embrittlement or cracking, is possible, as a result of which printing of products becomes impossible. Similar problems occur with many alloys commonly used in the aerospace industry, such as high-gammaphase high-temperature nickel alloys or high-strength aluminum alloys.

This report provides a brief overview of (i) possible causes of crack formation when printing nickel alloy products and (ii) possible strategies for modifying alloy compositions, with a particular focus on the use of high-performance CALPHAD computing. The results of a study of the causes and mechanisms of crack formation in nickel alloys ZhS6K and Rene 80 are presented. Next, the results of experimental studies on direct laser growth of modified alloys based on ZhS6K, proposed on the basis of CALPHAD calculations, are presented. The influence of the growth regime and the chemical composition of the alloys on the processes of crack formation and the mechanical properties of the resulting products is analyzed in detail.

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CREEP BEHAVIOR AND MICROSTRUCTURAL CHANGES IN HIGH-CHROMIUM MARTENSITIC STEELS SUBJECTED TO THERMO-MECHANICAL TREATMENT

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High chromium martensitic steels with a carbon content of 0.1 wt. % and alloyed with Mo, W, V, Nb, N and other elements are the main structural material used in the steam circuit of modern thermal power plants operating on gas and coal. Despite the high creep strength of modern martensitic steels during short-term tests (up to 10,000 hours at 650C), the long-term creep resistance of these steels is not very high, which is associated with degradation of the microstructure under the deformation and elevated temperature [1,2]. The most suitable solution for increasing the heat resistance of martensitic steels for long-term operation without the use of additional expensive alloying is thermo-mechanical treatment, which consists of controlled rolling in the austenite region. Two advanced martensitic steels with a chromium content of 9% and a balanced content of nitrogen and boron: Fe-0.1C-9Cr-1.8W-0.6Mo-3Co-0.05Nb-0.2V-0.012B-0.007N; Fe-0.1C-9Cr-1.8W-0.6Mo-3Co-0.05Nb-0.2V-0.013B-0.007N-0.085Ta were investigated. The steel blanks were heated to 1000°C with subsequent hot rolling with the total reduction of 40%. Next, the steel samples were tempered at 700 and 780°C. Creep tests were carried out at 650°C at an applied stresses of 180, 160, 140, and 120 MPa. The microstructures of the steels were studied by transmission electron microscopy (TEM) using a JEOL JEM-2100 microscope. It was found that hot rolling followed by water quenching provides efficient refinement of the subgrain structure compared to hot rolling with subsequent air cooling. The typical morphology of lath martensite consisting of packets and blocks was found in steel samples after hot rolling and quenching. A coarse subgrain structure with an average subgrain size of more than 1 µm is formed in the steel samples subjected to hot rolling followed by air cooling. In contrast, hot rolling with water quenching and tempering allows to retain a fine laths/subgrains with relatively high dislocation density of about 10¹⁴ m⁻². The use of hot rolling followed by water quenching and tempering increasing the creep strength of the studied steels compared to conventional heat treatment.

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RECENT PROGRESS IN DEVELOPMENT OF PROMISING HIGH-ENTROPY MATERIALS

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This report is devoted to the current state of theoretical and experimental research in the field of development and application of high-entropy phases as the basis for various groups of materials.

The authors present and analyze their own and literature data on the methods of synthesis, structure, properties, and possibilities of using high-entropy phases of various nature. An especial attention is paid to the new and most promising research directions in the field of high-entropy materials.

In particular, recent achievements in the field of creation and research of high-entropy nonmetallic materials with the aim of obtaining new structural and functional materials, as well as highly efficient catalysts are considered. Particular attention is paid to oxide materials that are promising candidates for the ceramic layer of thermal barrier coatings.

The results of the preparation and study of the properties of composite materials with the participation of high-entropy phases, as well as the preparation and study of coatings based on such materials, are presented. It is shown how research in this area can become the basis for the creation of new, relatively inexpensive coating materials used for surface modification.

The possibilities of using severe plastic deformation for modification of the structure and properties of high-entropy alloys are demonstrated. Also, the development of welding processes for high-entropy alloys and the use of high-entropy alloys in welding processes are considered.

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STRENGTH AND OXIDATION RESISTANCE OF LAVES PHASE-CONTAINING REFRACTORY Nb-Ti-Zr-Cr ALLOYS: EFFECT OF CHEMICAL COMPLEXITY

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² Saint-Petersburg State Marine Technical University, 190121 Saint Petersburg, Russia *tuchina_k@bsu.edu.ru Refractory complex concentrated alloys (RCCAs) are considered as promising materials to replace existing high-temperature alloys. It is generally assumed that chemical complexity is the source of unique properties of RCCAs, yet the systematic studies, which confirm this fact, are still lacking.

For the exemplar Nb-Ti-Zr-Cr alloy system, we showed that the effect of increasing chemical complexity on the strength and oxidation resistance depends on the elements added. A transition from Nb to NbTi and then to NbTiZr alloys, in which a single-phase body-centred cubic (bcc) structure is retained, enhanced the ambient-to-high-temperature strength and oxidation resistance at 1000 °C. However, when Cr was added, which induced the Laves phase formation, the trend was broken. Among the bcc + Laves phase compositions, binary Nb_{100-x}Cr_x alloys were much stronger at T > 600 °C than microstructurally similar multicomponent RCCAs. Meanwhile, ternary (NbTi)_{100-x}Cr_x alloys were the sole alloys that could form a protective Cr₂O₃ layer, which provided the lowest mass gain.

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REFRACTORY HIGH-ENTROPY SUPERALLOYS: CURRENT STATUS AND FUTURE TRENDS

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The development of advanced technology is impossible without new structural materials, especially those that can operate at extremely high temperatures. In this regard, over the past 15 years, the so-called refractory high-entropy alloys, which are capable of outperforming industrial high-temperature alloys, have attracted an interest from the materials science community. Although refractory alloys were already considered as a replacement for nickel superalloys about 60 years ago, their main drawbacks, such as low technological plasticity or non-competitive high-temperature strength, have not been resolved. Meanwhile, refractory high-entropy alloys, due to their extensive compositional space, offer more flexible approaches to "tuning" their properties. One such approach is to emulate the microstructural design of nickel superalloys, in which balanced mechanical characteristics are provided by a plastic γ matrix and reinforcing coherent γ ' particles embedded in it.

In this study, we will review the current status, issues, and future trends of the development refractory high-entropy superalloys, which consisted of bcc and B2 phases. The design principles and mechanical performance of existing refractory high-entropy superalloys will be extensively discussed.

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HYDROGEN INTERACTION FEATURES OF THE MULTI-PRINCIPAL-COMPONENT HYDRIDE-FORMING ALLOYS

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Synthesis of the multi-principal-component alloys, both amorphous and crystalline, includes the so-called "high-entropy" ones, with an opportunity to provide good hydrogen sorption properties due to the features of their crystalline structure attract a big interest of the scientists all over the world. In this regard, in the present work, the hydrogen interaction of the multi-principal-component hydride-forming alloys was investigated. In frame of this investigation a thermodynamic model to assess the feasibility of the synthesis of single-phase multi-principal-component alloy was proposed. Based on this model, single-phase TiVZrNbTa equiatomic alloys with body centered cubic (BCC) structure were obtained by arc melting (AM), electron beam melting with pendant drop melt extraction (EBM-PDME) and mechanical alloying (MA). The alloys were characterized by powder X-ray diffraction, scanning and transmission electron microscopy, thermal analysis and mechanical testing. The hydrogenation behavior of the synthesized materials was studied by a volumetric method. It was found that for AM and EBM-PDME alloys a complete BCC-to-FCC structure transformation occurs upon hydrogenation, and hydrogen concentration in the hydrogen absorption capacity of 0.9 wt% [1].

Other types of multi-principal-component alloys, with a hexagonal Laves phase (type C14) and amorphous structure have been also investigated [3-4].

It was suggested that the differences hydrogenation behavior depending on the preparation technique is due to the degree of equilibrium of the initial alloy. High-temperature synthesis methods (AM and EBM-PDME) allow metal atoms to take stable positions in course of the transition from liquid phase to solid solution. In contrast, low-temperature mechanical alloying results in the formation of highly disordered imperfect crystal structure that makes it more prone to amorphization.

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MODELS OF MICROSTRUCTURE EVOLUTION DURING DEFORMATION AND HEAT TREATMENT OF TWO-PHASE TITANIUM ALLOYS

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The most typical structure in two-phase titanium alloys is an α -lamellar microstructure, but a globular morphology of the phases can result in a better balance of technological and operational characteristics. In practice, the globular microstructure can be obtained during deformation in the two-phase region; in this case, a decrease in the deformation temperature can ensure refinement of the microstructure even to the UFG state.

In this paper, we consider models describing microstructure evolution during thermomechanical treatment of two-phase titanium alloys both in hot and warm deformation regions. Particular attention was paid to spheroidization of the lamellar microstructure, coarsening of alpha particles and final heat treatment in the $\alpha+\beta$ region. The mechanisms and kinetics of fragmentation/spheroidization of α lamellae by forming transverse boundaries, creating grooves at the points where intraphase boundaries crossed the interphase surface, and activating the processes of spheroidization and particle coarsening by the mechanism of dissolution and precipitation due to this were discussed with respect to Ti-6Al-4V, Ti-6Al-3.2Mo and Ti-6242 alloys. The application of the Livshits-Slezov-Wagner model for describing the static and dynamic coarsening of the α particles was shown.

The influence of temperature, degree and deformation scheme on the processes of fragmentation and spheroidization was discussed. The relationship between the kinetics of spheroidization during deformation and the coherence of interphase α/β boundaries was shown.

The approaches and modes for obtaining massive blanks with a homogeneous structure of the required grain size in two-phase titanium alloys were analyzed.

INVESTIGATION OF THE STRUCTURE, MECHANICAL PROPERTIES, AND OXIDATION RESISTANCE OF REFRACTORY MEDIUM-ENTROPY ALLOYS Alx(NbTiZr)100-x (X= 10; 25; 40 at%)

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Alloys used in the aviation and space industries must have balanced mechanical properties and oxidation resistance to operate at high temperatures.

Recently, a new class of multicomponent alloys has attracted quite a lot of attention – refractory high/medium-entropy alloys (RH/MEAs), showing promising mechanical characteristics. Among different RH/MEAs, an equiatomic NbTiZr MEA stands out favourably. Due to a single-phase bcc structure and good mechanical performance, this alloy appears as a suitable model alloy for further modifications of its properties, namely oxidation resistance. It is known that alloying with aluminum can improve not only the mechanical properties but also to increase the oxidation resistance of RH/MEAs. Also, the addition of aluminum affects phase

composition, leading to either B2 ordering or formation of secondary phases. However, no systematic studies have been conducted to date to elucidate the effect of Al.

In this work, we found that with an increase in the Al content led to improvement of strength and oxidation resistance, and the reasons for such behaviour were discussed.

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