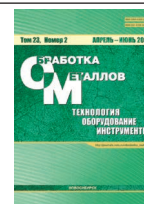




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Review of alloys developed using the entropy approach

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ABSTRACT

This paper provides a review of studies on the development and characterization of high-entropy alloys (HEAs). It is structured in the following way. **Alloys' design strategy based on entropy approach. Expectations and modern perceptions.** This section describes the initial principles of multicomponent alloys design which provide stable structure and mechanical properties. It is noted that the role of high mixing entropy in the formation of disordered solid solutions and the suppression of the brittle intermetallic phases formation have been significantly reconsidered over time. Currently, obtaining a single-phase solid solution structure is not the main requirement for HEAs. **The composition of HEAs.** This section describes some typical multicomponent alloys having different elemental compositions. It is shown, that at present time the most studied alloys are based on 3-d transition elements. Using alloys of this group the possibility of providing both high and low values of strength and ductility is shown. **Fabrication methods of HEAs.** This section describes the methods for the fabrication of high-entropy alloys. It is noted that the most commonly used methods are based on the melting of the initial materials and its subsequent crystallization. Such methods of HEAs fabrication as powder metallurgy, magnetron sputtering, self-propagating high-temperature synthesis, melt spinning, and diffusion welding are also discussed. **Structure of HEAs.** This section provides the data on HEAs possessing multiphase structure and containing fine nanosized precipitates. Besides, the studies in which HEAs have been obtained in the form of metallic glasses, carbides, oxides, and borides are reviewed. The factors that can affect the structural state of the multicomponent alloys are discussed. The ambiguity of opinions of different research groups is noted. **Properties of HEAs.** This section mainly concentrates on the mechanical properties of HEAs. However, some other promising properties of HEAs like high wear resistance and reduced diffusivity are also discussed. **Plastic deformation of HEAs.** This section describes the evolution of the structure and properties of HEAs caused by thermal and mechanical processing. **Characterization methods of HEAs.** This section lists the characterization techniques, which are most frequently used to study HEAs. The structure of these alloys is mainly studied by scanning electron microscopy, transmission electron microscopy, energy-dispersive X-ray spectroscopy, and optical microscopy. The methods for properties measurements are also briefly reviewed. **Application of HEAs.** This section describes the promising fields of HEAs application. It can be utilized in the aerospace, aircraft, and nuclear industries as well as for car manufacturing, acoustoelectronics, and in the design of microwave devices. **Russian-language publications on HEAs.** This section lists the studies, published in the Russian language as well as the thesis, done in Russian universities.

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Development of alloys using the entropy approach. Expectations and modern ideas.

The first studies related to the fabrication and characterization of high-entropy alloys (HEAs) were carried out at the end of the 20th century. An US patent for this type of materials was registered in 2002 by Taiwanese scientist J.-W. Yeh [1]. In 2004, the first studies of J.W. Yeh et al. [2] and B. Cantor et al. [3] were published, which are now highly cited. Thus, about 20 years ago, a new class of materials appeared, called “high-entropy alloys” [2, 3-6]. These alloys contain from 5 to 13 elements in an approximately equiatomic or equimolar ratio [2, 7]. The content of each element in the HEA is usually in the range from 5% to 35%. The high interest to HEAs is evidenced by the fact that in such a short period of time more than 5000 studies devoted to the analysis of these alloys were published. Among them, several reviews on the synthesis, characterization of structure and properties of HEAs can be recommended [8-17]. The interest to high-entropy alloys was due to its attractive properties, including high strength, ductility, wear resistance, and corrosion resistance [2, 18, 19].

Unlike traditional alloys, for example, steels, brass, bronze, aluminum or titanium alloys, high-entropy alloys do not have a “principal” or “matrix” component. All the elements present in the equiatomic ratios and thus can be considered as a principal, since in a disordered solid solution, each of the elements of the system has the same probability of being found in any of the available atomic positions of the crystal [20]. Therefore, in the case of high-entropy alloys with a solid solution structure, it is impossible to categorize the components as principal and minor (or alloying) ones. In fact, equiatomic alloys are located in the central regions of multicomponent state diagrams. Since none of components presented in significant quantities can be distinguished as a principal, sometimes such multicomponent alloys are called baseless (without a base) or compositionally complex [21].

Insufficient attention devoted in previous years to equiatomic alloys containing five or more components, and the dominance of materials based on one major element, was explained by the expectation that multicomponent mixtures will consist of brittle intermetallic compounds or complex phases. For this reason, the “entropy” approach to the alloys design began to be applied only in the last two decades [22]. One of the main ideas behind the development of the HEAs was to obtain a single-phase structure in the form of a disordered substitutional solid solution. It was assumed that the formation of phases with an ordered structure, including various intermetallics, would lead to embrittlement of the material consisting of multiple components.

An obvious feature of HEAs is the high entropy of mixing, which reduces the tendency to form intermetallic compounds in alloys and promotes the formation of single-phase substitutional solutions with a BCC or FCC structures. The high entropy of mixing is considered as a measure of the probability of preserving the structure and phase composition of alloys, ensuring its thermal stability, and maintaining high mechanical, physical, and chemical properties [23, 24]. The entropy of an alloy is determined by the value of four components – the configurational entropy of mixing (ΔS_{conf}), the entropy of atomic vibration (ΔS_v), the entropy of electron motion (ΔS_e), and the entropy of magnetic moments (ΔS_m) [11].

The concept of HEAs development is based on the fact that in multicomponent alloys, the configurational entropy is of a high level, which is not typical for traditional materials [2, 25, 26]. In comparison with the configurational entropy, the contribution of the ΔS_v , ΔS_m and ΔS_e components in the HEAs is small. Thus, the name “high-entropy alloys” is associated with an increased configurational component of entropy.

The transition of the system from an unstable state to a more stable one is accompanied by a decrease in the Gibbs energy. An increase in the number of elements of the system, leading to an increase in the configurational entropy, is a factor contributing to a decrease in the Gibbs energy. This reduces the tendency of the system to form ordered solid solutions and intermetallics and increases the probability of disordered solid solutions formation [26].

A lot of studies were related to the search of criteria that determine the formation of high-entropy alloys and predict their structure and phase state. Gorban et al. analyzed more than 200 high-entropy alloys and derived the relationship between the electron concentration, phase composition, lattice parameters, and



properties of solid solutions with BCC and FCC lattices [27]. The averaged electron concentration (e/at.) was found to be the main factor determining the phase formation in equiatomic high-entropy alloys. The authors of the study formulated the conditions for the formation of high-entropy chemical compounds (Laves phase, σ -phase, μ -phase). It was noted that a 100% σ -phase forms in those alloys in which this phase is formed for all pairs of its constituent elements. The second prerequisite is the value of the electron concentration in the range from 6.7 to 7.3 e/at. The 100% high-entropy Laves phase, according to the aforementioned study, arises when the total negative enthalpy of mixing of the alloys is equal to or less than -7 kJ/mol. In this case, the alloy should contain two elements with the enthalpy of mixing less than -30 kJ/mol, and the average electron concentration should be in the range from 6 to 7 e/at. It is noted that the nuclei of a solid phase in a high-entropy alloy are formed on the basis of the most refractory element [27].

In study of Firstov et al., the relationship between the electron concentration and the type of crystal lattice of a solid solution was analyzed for several HEAs [23]. It was noted that at a concentration of up to 4 e/at. a ductile HCP-based solid solution is formed. The concentration range from 4.25 to 7.2 e/at. corresponds to one or several types of BCC-based solid solutions. Two-phase solid solutions with BCC and FCC structures are formed in the electron concentration range of 7.2-8.3 e/at. Alloys with an FCC lattice correspond to an electron concentration above 8.4 e/at. Such alloys possess high ductility. The results of Firstov et al. indicate that brittle HEAs have mainly a BCC structure and correspond to the electronic concentration range of ca. 5.5-7.5 e/at. [23].

There exist ambiguity of opinions about the role of various factors on the formation of high-entropy alloys and their structural state. Based on the analysis of atomic radii, valence values, electronegativity, types of crystal structures of single-element metal components, and enthalpy, Rogachev concluded that the main criterion for the formation of high-entropy alloys is the proximity of the sizes of its constituent atoms [17]. The role of other factors, in his opinion, is less significant, which is consistent with the conclusions made earlier in a number of other works.

Thereby, numerous attempts to identify the factors that determine the phase composition and the structure of high-entropy alloys have not led to the formulation of reliable, well-founded conclusions. Using the criteria discussed in the literature, it is not possible to accurately predict the structure of newly developed HEAs and the degree of their stability under thermal and thermoplastic impact. Most of the conclusions on the HEAs structure are based on the experimental studies. For instance, using the CoCrFeNi system it was shown that the structure of alloys obtained by adding manganese, aluminum, or vanadium to this alloy is significantly different. For example, the addition of aluminum into this alloy leads to the formation of a multiphase structure [28]. Under certain conditions, the CoCrFeNiV system is also characterized by the presence of several phases [29]. It should be emphasized that the information about the structure of the currently developed HEAs is constantly updated and supplemented with new data.

In review [17], Rogachev notes that bulk amorphous alloys (bulk amorphous alloys, metallic glasses) were predecessor of high-entropy alloys, because they also contain several components. Both these materials have fundamental difference in the degree of their structural stability. Metallic glass is a metastable phase, the atoms of which do not have enough time to rearrange and form a crystalline structure during solidification of melt. The metastability of the amorphous phase becomes obvious when the material is heated. At certain temperature and holding time the atoms are rearranged and form a crystalline structure. In single-phase HEAs, dissimilar atoms occupy random positions in the crystal lattice and thus form a disordered solid substitution solution (HCP, BCC, or FCC). Considering the degree of stability, the HEAs having significantly distorted lattices due to the proximity of atoms of different sizes, occupy an intermediate position between metallic glasses and stable phases, which have a low density of defects [17].

Currently, the multicomponent high-entropy alloys having a complex multiphase structure are actively studied [30]. It has been experimentally established that more than six phases can be formed in the CrFeNiCoAlCu alloy [31], some of which are nanoscale. These group of HEAs also includes alloys containing an amorphous phase [32], as well as mixtures of intermetallic phases [33].



Composition of high-entropy alloys

There are ca. 40 elements in various families of high-entropy alloys. The combination of five to twenty components in alloys makes it possible to obtain a huge number of high-entropy alloys [10, 34]. It is noted that only about five hundred materials related to the HEA family have actually been studied [11, 17]. The strategy for selection of HEA's composition depends on many factors, the most important of which are related to the operating conditions of the material [26].

Alloys based on 3-d transition elements are the most studied HEAs. Alloys of this group contain at least 4 of the following 9 elements: Fe, Co, Cr, Ni, Mn, Al, Ti, Cu, V [2, 3, 15, 35]. Interest in the materials of this family was due to its promising properties such as high hardness, positive temperature hardening coefficient, high wear and corrosion resistance [2, 5, 18, 30, 36, 37]. A well-known and frequently discussed five-component HEA is the CoCrFeMnNi alloy, which was proposed to the scientific community in 2004 [3]. In the literature, this single-phase material with the structure of a disordered substitution solid solution is sometimes referred to as a "Cantor alloy". One of the most studied materials belonging to the class of high-entropy alloys is AlCoCrCuFeNi [2, 7, 30, 38, 39]. In addition to the 3-d transition elements, the composition of polymetallic HEAs includes other elements, for example, Zr: AlTiVFeNiZr, AlTiVFeNiZrCoCr, CuTiVFeNiZrCoCr, MoTiVFeNiZrCo, MoTiVFeNiZrCoCr, CuTiVFeNiZrCo, etc. [1].

A large amount of research is related to alloys based on refractory metals (Hf, Ta, Mo, Nb, V, W, Cr, Zr, Ti). The third group of HEAs, which is given special attention by specialists, includes light alloys (Al, Sn, Be, Li, Mg, Ti, Sc, Si, Zn). All these multicomponent materials include aluminum and titanium [17]. The fourth HEAs family is based on 4f transition metals (rare earth elements Gd, Dy, Lu, Tm, Tb, Y). There are also the fifth (Cu, Al, Mn, Ni, Zn, Sn) and sixth (Au, Ag, Cu, Co, Cr, Ni, Pt, Pd, Ru, Rh) families of HEAs. It can also include such elements as Fe, Co, Ni, Si, B, Zr, Hf, Cu, Be, Mo, Cr. There is no doubt that the HEAs nomenclature will expand.

High-entropy metal glasses are considered as an independent family of HEAs. Besides, in recent years, the attention of specialists has been paid to the development of high-entropy metal-like compounds [11, 17]. These include ceramic materials in the form of carbides [40], oxides [41], borides [42], and nitrides [43]. Examples of compounds of this kind are $(\text{TiZrNbHfTa})\text{C}$, $(\text{AlCrTaTiZr})\text{O}_x$, $(\text{Ti}_{10.2}\text{Hf}_{0.2}\text{Zr}_{0.2}\text{Nb}_{0.2}\text{Ta}_{0.2})\text{B}_2$, $(\text{AlCrTaTiZr})\text{N}_x$. Speaking about oxide materials [44], we note simple oxide systems formed by divalent and trivalent metals, as well as high-entropy systems with the structure of perovskite [45] and spinel [46].

The literature provides examples of compositions in which the matrix materials are HEAs obtained by mechanical fusion of pure components (CoCrFeNiMn, CoCrFeNiV, CoCrFeNiCu, CoCrFeNiAl), and the hardening is provided by ceramic particles (WC, TiB_2 , Ti(C, N)) [17, 47–49].

Considering the main components, HEAs are sometimes conditionally divided into several groups [17]. For example, there are high-entropy steels, heat-resistant alloys, light alloys, rare-earth alloys, bronzes and brasses, and catalysts. It is emphasized, however, that this type of classification is not generally accepted.

Methods for producing high-entropy alloys

Various technical solutions were proposed to obtain alloys having high entropy of mixing, namely, melting, powder metallurgy (mechanical alloying of powders), welding, melt spinning, splat-cooling, self-propagating high-temperature synthesis, magnetron sputtering, cladding of powder mixtures on a metal base.

In most cases, high-entropy alloys are obtained by melting the materials followed by crystallization [17]. Methods of electric arc melting in vacuum [2, 5, 6, 50–52], argon-arc [53] and induction [3, 54] melting, melting in resistance furnaces can also be used for HEAs fabrication. One of the most common methods for producing HEAs is vacuum arc melting [55, 56]. The initial material is a mixture of metals with a purity of more than 99 %. The alloys are smelted in an inert gas atmosphere, which prevents the material's oxidation. In order to increase the homogeneity of ingots, it is remelted several times. The melting point of the material



and its components is an important factor determining the choice of the method for HEA fabrication. For the production of refractory HEAs, it is rational to use the technology of electric arc melting.

In order to form an optimal structure and improve the mechanical properties, high-entropy alloys obtained by casting are usually subjected to thermoplastic processing – cold and hot rolling [54, 57, 58], forging [7], and drawing [59]. As a final technological operation, homogenizing annealing or other methods of heat treatment are usually used to promote the formation of an equilibrium structure [54, 59, 60].

Mechanical alloying of pure powder components in planetary ball mills is also one of the commonly used methods for fabrication of HEAs [26, 61-67]. The mixtures of elementary powders are deformed in high-energy ball mills, which provides their interaction. The processing time is ca. 10 - 20 hours. For example, CoFeNiXY alloys (where X is Cr and Si, and Y is Mn, Al, Ti or Zn) were obtained by mechanical alloying. Hot isostatic pressing is a method of compaction that improves the quality of the HEAs structure [68].

Wear debris left by steel balls during mechanical alloying of powder mixtures containing iron do not affect the quality of the resulting HEAs. In other cases, one can expect that iron will appear in the composition of HEA when it is produced by ball milling. A typical peculiarity of mechanical alloying of highly ductile material is its sticking to the surface of deforming balls, which ultimately affects the quality of the HEA. The powder materials obtained by mechanical alloying require subsequent consolidation. One of the most effective approaches to address this issue is spark plasma sintering [64-66, 69]. In a number of studies, multicomponent high-entropy alloys were obtained using self-propagating high-temperature synthesis (SHS) technologies [70, 71].

High-entropy alloys in the form of thin films and multilayer coatings are obtained by magnetron sputtering [9, 11, 17, 43, 72]. A thin-layer HEAs can be produced by spraying a single target, which includes several components. The second approach is based on the simultaneous magnetron sputtering of several targets [73]. High-entropy alloys in the form of nitrides [74], carbides [40] and other compounds are obtained by magnetron sputtering. Alternating targets of different compositions, nanostructures consisting of layers of HEAs and pure metals were also formed by magnetron sputtering [75].

In recent studies the HEAs in the form of metallic glasses attract a lot of attention [17, 76, 77]. The fabrication of glassy HEAs is done by melt spinning. The metal glass thus obtained as an amorphous strip, which is cooled at high rate on a rotating copper cylinder. One of the technologies of rapid solidification of HEAs is based on splat cooling, which consists in the collision of a drop of melt with the inner surface of a copper cylinder rotating at high speed [78].

In [79], the possibility of obtaining heat-resistant composites based on FeCoNiMnCr and FeCoNi₂MnCrCu high-entropy alloys was studied. Layered composites were formed by diffusion welding of aluminum foils and thin layers of high-entropy alloys. The HEAs ingots obtained in a vacuum arc furnace were deformed by rolling to a thickness of 0.4 mm on two-roll mills at room temperature with a degree of ~ 15 % in one pass [79, 80]. Layered billets were welded by the diffusion method in two stages. At the first stage with a duration of 2 hours, the temperature was 600 °C, at the second stage the temperature was 950 °C.

Structure of high-entropy alloys

The most important parts of studies on HEAs are related to characterization of their structure, since it determines the properties and the possible applications of the materials. The structure of high-entropy alloys obtained by melt crystallization is largely determined by the cooling rate. According to the data presented in [17], the crystallization processes of HEAs and traditional alloys are identical. Under conditions of rapid cooling of materials, a fine-grained structure is formed. In cases where the melt cools at a low rate of tens of degrees per second, a dendritic-type structure is formed [81]. With rapid cooling of the melt, the tendency to form dendrites disappears.

At the beginning of HEAs development it was believed that the high entropy of mixing, being a factor preventing the formation of ordered phases and intermetallic compounds, promotes the formation of disordered substitutional solid solutions. From this point of view, one could expect that the structure of



HEAs is quite obvious. However, characterization of structure of multicomponent equiatomic alloys is as difficult tasks as characterization of structure of convenient metallic alloys. To understand the structure of HEAs it is also necessary to use a wide variety of characterization techniques. The most important are the methods of X-ray diffraction analysis, transmission and scanning electron microscopy. The characterization methods involved in a particular study depends on its goals, the composition of the material, the geometric parameters of the samples and other features of the analyzed HEAs.

It is generally believed, that the properties of “classical” single-phase HEAs are related to random location of dissimilar atoms in the crystalline lattice. It is believed that the atoms located in a multicomponent system, which differs in size, electronic structure, and thermodynamic properties, lead to a significant distortion of the crystal lattice of the solid solution, an increased efficiency of the solid-solution hardening and stabilization of the material properties [23]. This feature significantly distinguishes HEAs from convenient alloys [20]. In [17], using the example of a five-component equiatomic ABCDE alloy, it is graphically shown that in elementary cells of the BCC and FCC types, the long-range order for each type of atom is absent and the resulting phase is a completely disordered substitution solid solution. An equiatomic alloy of the ABCDE type can only be constructed from a set of elementary cells that differ in composition.

CoCrFeNi and CoCrFeNiMn alloys are typical representatives of single-phase high-entropy alloys with the structure of a disordered substitution solid solution with an FCC structure. In the thesis of Shaisultanov [82], it was shown that when V or V and Mn are added to the CoCrFeNi system, a tetragonal σ -phase is formed in the alloy structure along with the FCC phase. An even more complex structure is formed in an alloy containing, in addition to these four components, Al and Cu (CoCrFeNiAlCu). Four phases are observed in the structure of this alloy, including the disordered BCC phase (which predominant consists of chromium and iron), the ordered B2 phase ((which predominant consists of aluminum and nickel), the ordered $L1_2$ phase (enriched in copper), and the ordered $L1_2$ phase (enriched in cobalt, chromium, and iron).

The most important characteristic that determines the interest of many specialists in high-entropy alloys is the stability of their structure, and hence stability of their properties. The statement that the structural stability of HEA's is explained only by high values of the configurational entropy has now lost its relevance. In many works, it has been experimentally shown that in alloys with high values of the mixing entropy, other phases, including intermetallics can appear along with the solid solution.

Using the method of anomalous X-ray scattering and neutron diffraction, it was shown in [83] that a two-week exposure at 753 K of a four-component FeCoCrNi alloy obtained by arc melting did not lead to the appearance of the solid solution ordering effect and the formation of a long-range order in it. This stability of the analyzed alloy is associated with its high configuration entropy.

The question of the HEAs stability during thermal and thermoplastic impact remains open. A detailed analysis of this issue is presented in the work of Rogachev [17]. A large amount of research is devoted to the stability of the five-component CoCrFeNiMn alloy (“Cantor alloy”). The diameter of the manganese atoms (0.274 nm) is significantly larger compared to the atoms included in the four-component CoCrFeNi system. For this reason, the maximum distortion of the crystal lattice, localized near the manganese atoms, in the five-component system is significantly higher than in the CoCrFeNi alloy. The analysis of the behavior of the Cantor alloy under various conditions of thermal and thermoplastic action does not give grounds to formulate unambiguous conclusions about its stability. In the literature, there are data on the long-term preservation of the single-phase structure of the material in a wide temperature range (1273-1473 K), which indicates its high stability [29, 84-86]. At the same time, based on the results of experimental studies, it is concluded that plastic deformation and high-temperature exposure are factors leading to precipitation of secondary phases from the CoCrFeNiMn alloy [17, 50, 85, 87, 88], including nanoscale intermetallics such as NiMn, FeCo.

An increase in the chromium and manganese content accelerates the formation of secondary phases. At the same time, it is noted [17] that when a secondary phase precipitates from a high-entropy CoCrFeNiMn alloy, its matrix phase remains a solid solution with an FCC structure. The CoCrFeNiAl system, like CoCrFeNiMn, is characterized by metastability. As a result of short annealing of this HEA, several types of structural components appear in it [89].

The analysis of the structure of heat treated and deformed alloys consisting of refractory components (TiZrTaHf, TiZrTaNb, TiZrHfNbV) indicates that, like many other HEAs, they can be considered as meta-stable materials [90-93]. Rogachev concluded that most high-entropy alloys consist of several phases, the number of which increases as a result of annealing [17]. CoFeNiMnCu [94], AlNbTiVZrx [95], CoCrFeNiMnTi0.1 [96], ZrTiHfCuNiFe [97] and some other alloys are classified as stable or limited stable HEAs. At the same time, it should be kept in mind that the assessment of the degree of the HEAs stability, in many cases, is a methodically complex task. This is especially relevant, for example, in the case of observation of particularly fine precipitations of phases, the volume fraction of which is small. It is likely that during characterization of structure, some of these phases may be overlooked.

It should be noted that the concept of “metastability”, which contradicts the initial concept of stable high-entropy alloys, currently is not considered as a fundamental disadvantage of real multicomponent systems. The positive effect caused by the precipitation of several phases may lead to effect of dispersion hardening of materials [17]. At the same time, in order to avoid embrittlement of HEAs it is suggested to prevent the formation of σ -phases.

The analysis of structural transformations HEAs presented in [17] allowed us to conclude that it is difficult to interpret the phenomena associated with the stability of multicomponent systems. It is believed that the relationship between the stability of the HEAs exclusively with the level of configurational entropy is oversimplified. A more reasonable approach to solving this problem is associated with the development of semi-empirical criteria for the stability of HEAs, the quantum mechanical calculations [98-100], and the thermodynamic analysis of multicomponent systems.

The severely deformed alloys, as well as alloys with a crushed grain structure are expectedly less stable [101]. The problems solved by Ivchenko in his thesis were related to the structure and properties of the AlCrFeCoNiCu high-entropy alloy [102]. Of particular interest were the experimental data on the structural-phase transformations occurring in the alloys fabricated by melt spinning and splatting, and on the effect of severe plastic deformation and heat treatment on the structure and phase composition of the AlCrFeCoNiCu alloy.

When cooled at a rate of 10 K/s, a complex dendritic structure is formed in the AlCrFeCoNiCu alloy, each of the phases of which consists of six components. The nanoscale phases isolated within the dendrites and in the inter-dendritic space are uniformly distributed over the volume of the ingot. They are characterized by equiaxed and lamellar morphology, and have ordered (structural types B2 and L1₂) and disordered (A1, A2) structures [102]. Rapid solidification by the melt splatting ($\sim 10^6$ K/s), as well as by melt spinning ($\sim 10^5$ K/s), leads to the formation of an ultra-fine-grained (560 nm) dendritic structure, which consists of nanoscale six-component phases.

One of the features of six-component AlCrFeCoNiCu alloys after rapid quenching and severe plastic deformation is the formation of local nanoscale precipitations. The corresponding rearrangement of the alloy components results in dimensional-spatial periodicity of the elemental and phase composition in the volume of the ingot [102]. Concentration fluctuations in the form of clusters in the size range from one to several tens of nanometers were observed in the AlCrFeCoNiCu alloy by 3D-AP tomography.

Dislocation hardening mechanisms of high-entropy alloys are much less studied than those in classical alloyed steels and alloys. Nevertheless, many works pay special attention to this problem. The solid solution hardening mechanism, hardening by grain boundaries, dislocation pileups, and dispersion hardening are considered as the main mechanisms which increase the strength of HEAs.

One of the problems discussed in [21] was related to the study of the structure and properties of the CoCrFeMnNi alloy alloyed with carbon and aluminum. It was found that the introduction of 0.7 at. % C and 3.4 at. % Al is accompanied by an increase in stacking fault energy and a suppression of the twinning process at the initial stages of deformation. The higher strength of CoCrFeMnN(Al,C) alloy after reduction to 80% compared to the equiatomic five-component CoCrFeMnNi alloy is due to the large contribution of solid-solution hardening caused by carbon and aluminum. It is established that grain boundaries have the most significant effect to hardening of the CoCrFeMnN(Al,C) alloy annealed after cold rolling ($\varepsilon = 80\%$). The effect on strengthening of nanoscale carbides formed during annealing at 700 - 900 °C of a cold-rolled alloy is comparable with that of the grain boundaries.

Properties of high-entropy alloys

The main factor determining the interest of specialists in the developed materials is the nature of its behavior in various environmental conditions. The set of parameters recorded at the initial stages of HEAs development, as well as expectations based on theoretical ideas about the possible manifestations of multi-component systems, allow us to conclude that its use in various industries is promising. Given that the high-entropy alloys analyzed in the literature are a family of materials that differ significantly in composition, it is necessary to evaluate the complex of properties corresponding to its purpose and operating conditions in each specific case. The properties of HEAs are determined by its components, structural condition and production technology.

The literature contains a wide range of ideas (sometimes contradictory) about the properties of high-entropy alloys. As important indicators of HEAs, its hardness, strength, and wear resistance [2, 7, 102, 103], increased ductility at low temperatures, corrosion resistance, thermal stability [17], resistance to ionizing radiation [22, 104] are noted. It should be emphasized that this type of characteristic is generalized and cannot be applied to all types of HEAs. For structural materials, the combination of strength properties, ductility, crack resistance, and a number of other characteristics that determine the behavior of alloys under severe external loading conditions is of particular importance.

The unique properties of multicomponent HEAs are due to the manifestation of four effects [17, 9-13]. One of it is associated with the high entropy of the alloy, the second – with distortions of the crystal lattice, the third – with the delayed diffusion of the alloy components. The fourth was called the “cocktail effect”.

The effect of high entropy, from which the name of the alloys analyzed in the work is derived, is determined by the level of configuration entropy. In accordance with one of the classifications of the analyzed alloys, low-entropy alloys include those with $\Delta S_{mix} \leq 0.69R$, where R is the universal gas constant. Medium entropy alloys are described by the ratio $0.69R \leq \Delta S_{mix} < 1.61R$. The high-entropy alloys include ones with $\Delta S_{mix} \geq 1.61R$ [4]. The search for compositions of stable high-entropy alloys is complicated by a number of circumstances [17]. One of it is that the applicability of the dependences that reliably describe the stability conditions of ideal solutions in relation to real solid solutions is not obvious [11].

The second of the main effects of high-entropy alloys is the effect of crystal lattice distortions. It is caused by the difference in the size of the atoms that make up the multicomponent system. The degree of distortion is minimal in the neighborhood of atoms that are close in size. In alloys consisting of atoms that differ significantly in size, larger voids – internodes – are formed. Embedded atoms can be located in these voids, forming a region of local stresses [17]. The sources of distortion are also large atoms located in the nodes of the crystal lattice and surrounded by smaller atoms, which position also corresponds to the nodes of the lattice. Distortions of the crystal lattice largely determine the level of the HEAs strength properties.

Delayed diffusion is the third effect that determines the stability of the structure and the complex properties of high-entropy alloys. The favorable effect of the low diffusion rate is reflected in the increase in the thermal and chemical stability of the HEAs [105, 106]. Distortions of the crystal lattice and other features characteristic of HEAs are noted as factors that explain the inhibition of diffusion processes [4, 6, 37, 107]. The contradictions revealed in a number of studies allow us to conclude that the confirmation or refutation of the delayed diffusion effect in HEAs requires additional research [17].

The term “cocktail effect” implies that a complex system manifests a mixing effect that is not available to each of its components individually. In fact, this term is synonymous with such a concept as a synergistic effect.

Out of the whole set of mechanical properties of HEAs, the specialists paid more attention to the strength indicators. At the same time, the question of the brittleness of high-entropy alloys, the degree of which is determined by the ductility, impact strength and crack resistance of materials, is of fundamental importance. It should be emphasized that the absence of embrittling phases is one of the distinctive requirements for HEAs, which determine its technological properties, in particular, deformability during cold pressure treatment. Taking into account these ideas, it is believed that the high entropy of mixing, preventing the formation of intermetallic phases in the HEAs and contributing to the formation of mainly disordered

substitution solid solutions, is a factor that favorably affects the combination of strength properties and its tendency to deformation [26, 7, 108-110]. The combination of strength and ductility indicators allows us to assess the prospects of the developed materials as structural materials in the first approximation.

The analysis of the HEAs properties in various conditions of external influence is described in many works [8, 9, 11, 13, 15, 35, 111]. The largest amount of information is related to systems based on 3d-transition metals. The level of strength properties of some of it can be defined as high [17]. According to the data presented in [112], the value of this indicator for the alloy Co30Cr10Fe50V10 reaches 2000 MPa. At the same time, the level of deformation ε corresponding to the destruction of the material reaches 71 %.

Castings of the high-entropy CoCrFeNiMn alloy having the structure of FCC solid solution are highly plastic ($\delta = 68$ %) and at the same time have a low level of strength properties ($\sigma_{0.2} = 140$ MPa, $\sigma_B = 443$ MPa) [82]. The author made similar conclusions about the four-component CoCrFeNi alloy. In contrast to manganese, which addition does not significantly affect the strength properties and ductility of the four-component system, the role of vanadium, which leads to the formation of a brittle σ -phase, in reducing the ductility of CoCrFeNiV and CoCrFeNiMnV alloys is very noticeable. Vanadium-alloyed alloys exhibit noticeable ductility only when subjected to compressive stresses. A low level of ductility (0.2 %) is also characteristic of CoCrFeNiAlCu alloys with a multiphase structure at room temperature.

The thermal stability of the HEAs assumes the preservation of the structure of a disordered solid solution when the material is heated and kept in a high-temperature state. The result of the ordering of the solid solution during the annealing process, which consists in the diffusion redistribution of atoms with its fixation in certain thermodynamically favorable positions of the unit cell, can be the formation of a superstructure [17]. Attempts to study the rearrangement of the structure of a solid solution during its heating and control by X-ray diffraction methods were discussed. The solution of this problem, which is important from an applied and fundamental point of view, is methodically problematic.

High-entropy alloys are considered as promising heat-resistant materials [26, 113, 114]. In [24], we studied the thermal stability of superhard nitride coatings based on a five-component high-entropy alloy containing Ti, V, Zr, Nb, and Hf. The objects of the study were thin coatings obtained by vacuum-arc evaporation of a pre-prepared multicomponent cathode. The substrates were plates made of chromium-nickel austenitic steel. Nitride coatings (TiVZrNbHf)N obtained in the presence of nitrogen (0.27–0.66 Pa) when a constant negative potential is applied to the steel plate are characterized by high hardness values (50–60 GPa). This level of material properties is explained by the formation of a polycrystalline structure with a grain size of ~ 30 -50 nm and significant distortions of the crystal lattice. The studies carried out in this work indicate a high thermal stability of single-phase nitride coatings. Its structure is preserved during annealing up to 1100 °C [24]. Conclusions about a sufficiently high thermal stability in the temperature range from 20 °C to 1000 °C were made by S.A. Firstov and co-authors on the basis of experimental studies of nine high-entropy alloys [115].

In [82], the possibility of creating new composite materials by diffusion welding of plates made of FeCoNiMnCr and FeCoNi2MnCrCu HEAs and an aluminum alloy of the Al-Si system was studied. It is established that the diffusion processes that occurred during the heating of layered packages led to the formation of heterogeneous materials with the formation of transition zones, dendritic structures, and intermetallic phases. The ultimate strength of the composite obtained by pressure diffusion welding is stable at ~ 615 MPa in the temperature range of 20–850 °C. At the same time, the ultimate strength of the HEAs plates at 750 °C outside the composite did not exceed 375 MPa.

The five-component amorphous alloy TiZrHfCuNi, obtained by pouring the melt into a cooled copper mold, acquiring a high level of ultimate strength (1,930 MPa), has a ductility close to zero at room temperature [116]. A thin film made of the six-component alloy AlCrMoTaTiZr, characterized by high values of the elastic modulus (11.2 GPa) and hardness (193 GPa), was obtained by magnetron sputtering [117].

In the work of M.V. Karpets and co-authors [102], the results of evaluating the behavior of a high-entropy alloy VCrMnFeCoNi $_x$ (where $x = 1.0; 1.5; 2.0$) under friction against non-rigid abrasive particles are presented. After argon-arc melting, a solid solution with a FCC-type lattice and a σ -phase similar to the tetragonal σ -phase of the binary Fe-Cr alloy were fixed in the alloy structure. As the nickel content

increased, the volume fraction of the σ -phase decreased. Of the three high-entropy materials studied, the VCrMnFeCoNi1 alloy has the highest level of wear resistance. Its coefficient of relative wear resistance ($K = 3.03$), measured in accordance with GOST 3647-80, is close to the value of the material deposited with the T-590 electrode ($K = 3.09$), which is used for surface hardening of products operated under conditions of abrasive wear. Thus, it can be concluded that the high-entropy VCrMnFeCoNi1 alloy proposed in [103] has a high abrasive wear resistance.

In [23], using the example of a high-entropy VNbTaCrMoW alloy, it is concluded that the material acquires averaged values of most physical characteristics. The only exceptions include strength properties, which are significantly higher in HEAs due to the anomalous values of solid solution hardening [118, 119].

Plastic deformation of the HEAs

Along with heat treatment, plastic deformation is considered as one of the most effective technological factors that allow changing the structure, strength, ductility, and other properties of high-entropy alloys. At present, there is no reason to say that the potential possibilities of such an approach are obvious and can be applied to most of the analyzed materials. At the same time, when studying a number of polymetallic alloys, convincing evidence of the effectiveness of methods for processing metals by pressure was obtained. For example, after cold rolling with a degree of 80%, the CoCrFeMnN(Al, C) alloy has a high complex of mechanical properties: $\sigma_{0.2} = 870$ MPa, $\sigma_u = 1,060$ MPa, $\delta = 25\%$ [21]. The result of hot plastic deformation of the CoCrFeNiMnV alloy is a change in its phase composition and the transformation of the original lamellar structure into an ultra-fine-grained one, which has a favorable effect on the properties of the material, in particular, leads to a decrease in the temperature of the visco-brittle transition [82].

Research focused on filling the gaps in the field of plastic and thermoplastic impact is successfully carried out at the Belgorod State National Research University under the leadership of G.A. Salishchev. One of the tasks solved in D.G. Shaisultanov's thesis work was related to the development of deformation modes that provide an increase in the complex of mechanical properties of CoCrFeNiMn and CoCrFeNiAlCu alloys [82]. It has been experimentally established that at room temperature, CoCrFeNiMn alloy blanks can be formed by uniaxial rolling without losing the continuity of the material by tens of percent. As a result of this impact, the yield strength of the alloy increased by 8 times (from 140 to 1120 MPa), and the ultimate strength – by 2.7 times (from 443 to 1175 MPa). As expected, the level of relative elongation significantly decreased (from 68 to 14%).

An analysis of the effect of cold rolling on the structure and properties of the $\text{Al}_x\text{CoCrFeNi}$ alloy was performed in [120]. The experimentally recorded increase in the hardness of alloys in comparison with the cast state is due to the manifestation of strain hardening mechanisms. In particular, based on the results obtained by transmission electron microscopy, it has been established that an increase in the strength properties of materials is associated with the formation of numerous structures in the form of nanotwins. With an increase in the degree of plastic deformation, the volume fraction of these defects in the crystal structure increases. In [82], the role of dislocation sliding and twinning processes in the formation of strength properties was shown using the example of the cold rolled CoCrFeNiMn alloy [82].

M.V. Klimova's thesis work is associated with the study of the effect of deformation-heat treatment on the structure and mechanical properties of high-entropy alloys of the Co-Cr-Fe-Mn-Ni (Al, C) system [21]. The experimentally revealed microstructure of the CoCrFeMnNi alloy during rolling at room temperature deserves attention. The author of the work identifies three stages of structural transformations associated with the degree of plastic deformation of the material: an increase in the dislocations' density ($\varepsilon = 5\text{--}20\%$); intense deformational twinning ($\varepsilon = 20\text{--}60\%$); formation of shear bands ($\varepsilon = 60\text{--}80\%$). In the cryogenic temperature range (-196°C), the twinning stage shifts to lower values of the degree of deformation.

After megaplastic deformation according to the high-pressure torsion scheme (6 GPa), the microhardness of the AlCrFeCoNiCu alloy reaches 12 GPa [102]. Under these conditions, all the excess phases are dissolved and a mechano-induced BCC $\rightarrow$ FCC transformation develops. The subsequent annealing of the alloy leads to the reverse FCC $\rightarrow$ BCC structure transformation.



Poor deformability at room temperature is a factor limiting the use of some high-entropy alloys [2, 7, 121]. For example, in [7], a low set of mechanical properties of castings made of the AlCoCrCuFeNi alloy is noted. As a solution to this problem, it was proposed to use the method of comprehensive hot forging (a-b-c forging) at a temperature of 950 °C. The alloy was obtained by induction melting, followed by electroslag remelting and casting into a copper cooled mold. Forging was carried out in an isothermal die block on a hydraulic press at a traverse movement speed of 1 mm/s. The total degree of deformation was ~ 1,000 %. In the process of comprehensive forging, the dendritic structure of the cast alloy is eliminated, the structure of the HEAs becomes finer (2.1 microns) and more homogeneous. The result of structural transformations accompanying the hot plastic deformation of the alloy is an increase in the yield strength from 790 MPa to 1170 MPa. The increase in the relative elongation observed under these conditions (from 0.2 % to 1 %) does not allow us to speak of a significant improvement in the plasticity indicators [122]. One of the features of the behavior of the forged AlCoCrCuFeNi alloy, recorded by the authors of the work [7], is the manifestation of the superplasticity effect in the high temperature range (800–1,000 °C). When deformed at a rate of 10^{-2} s^{-1} , the relative elongation of the samples subjected to stretching is 1,240 % [82].

Methods for studying high-entropy alloys

The choice of methods for studying the structure and properties of high-entropy alloys is determined by various factors, including the structural features of the materials, its operating conditions, and the size of the samples. One of the most important research methods is related to the X-ray diffraction analysis of alloys, the identification of the phases present, and the determination of the parameters of its crystal lattices. In many works in the field of HEAs, structural studies are carried out using the methods of transmission and scanning electron microscopy, X-ray spectral analysis, and light microscopy.

The methods of mechanical and other tests are determined by the purpose of the developed high-entropy alloys. For structural alloys, information about strength properties under uniaxial tension and compression is of the utmost importance. The level of reliability and durability of products made from HEAs is related to such properties of materials as impact strength, static and fatigue crack resistance. In some cases, the corrosion resistance characteristics are important.

The tasks associated with the study of nanoscale particles released in HEAs involve the use of high-resolution transmission electron microscopy methods [123]. In [124], the method of small-angle scattering of synchrotron radiation was used to solve such problems in the study of a deformed CoCrFeNiMn alloy.

In [125], a method based on neutron diffraction was used to study the structural-phase state of a three-component CoCrFeNi alloy. The experimental results obtained in this way allowed us to make a conclusion about the features of the fine structure of the CoCrFeNiMn alloy [84]. Using the method of anomalous X-ray scattering and neutron diffraction, we studied the processes of structural transformations during heating of a four-component FeCoCrNi alloy obtained by arc melting. It was shown [83] that the two-week exposure of the alloy at 753 K did not lead to the manifestation of the ordering effect of the solid solution and the formation of a long-range order in it.

One of the most important characteristics of multicomponent alloys is the degree of structure ordering. In the work of M.V. Ivchenko, an optical tomographic atomic probe “Cameca atom probe” (3D-AP) was used for a precision study of the local atomic composition of the six-component AlCoCrCuFeNi alloy [18, 102]. The same method was used to study the structure and properties of the six-component alloy AlCrFeCoNiCu after casting and rapid quenching from the melt [36].

The traditional approach to substantiating the compositions of multicomponent systems and analyzing its properties is associated with a high labor intensity of research, conducting many experiments. One of the tasks typical for the analyzed materials is associated with the need to model phase diagrams of the state. Examples of its solution by the CALPHAD method (CALculations of PHase Diagrams) are given in [126, 127]. The development of computer technologies and special software in many cases can significantly reduce the cost of developing high-entropy alloys. By methods of mathematical simulation,



using thermodynamic calculations, studies were conducted on the choice of HEAs rational compositions [128, 129]. There is no doubt that in the coming years this approach will be one of the most promising when justifying HEAs for various purposes.

Purpose of high-entropy alloys

The properties, characteristic of various types of HEAs, give grounds to consider these materials promising for use in the rocket and space industry, aircraft, mechanical engineering, and nuclear power [9, 14, 36, 78, 104]. Attempts are being made to develop HEAs characterized by a high level of heat resistance [26]. According to the results of the work [130], some HEAs can perform the function of radiation-resistant coatings applied to the shells of fuel elements. One of the applications of amorphous HEAs is associated with the formation of high-temperature diffusion barriers between copper and silicon [131]. The possibilities of using high-entropy oxide systems in electronics, magneto-optics, microwave devices, and acousto-electronics are discussed [44]. Some of the developed HEAs are characterized by high corrosion resistance and can be used as functional coatings.

One of the directions of HEAs development is associated with the development of alloys that can be operated under high loads in a high-temperature state [2, 26]. We are talking primarily about materials for the modern aviation industry. In the temperature range of 800-1600 °C, the yield strength of the high-entropy VNbMoWTa alloy is higher than that of the Haynes 230 and Inconel 718 superalloys [19]. The possibility of using HEAs as high-temperature materials was discussed in the works [19, 23, 26, 130, 132-135]. One of the main disadvantages of HEAs based on refractory metals is the high density, which limits its practical use as heat-resistant materials. At the same time, VIAM specialists believe that there are grounds for increasing the ductility of heat-resistant materials by expanding the range of elements and forming strengthening phases in the materials.

Among the positive qualities, characteristic for the equiatomic alloy CoCrFeMnNi, its high level of fracture toughness, which is $200 \text{ MPa} \times \text{m}^{1/2}$, deserves attention [136]. The increase in the strength properties and ductility of the specified material when cooled to cryogenic temperature [58, 136] makes it attractive for the manufacture of equipment for responsible purposes intended for operation in the Far North.

In the works of D.A. Vinnik et al. [44, 137, 138] multicomponent oxide phases with high values of the configurational entropy of mixing are analyzed. The related hexaferrites with the magnetoplumbite structure are considered as materials for the manufacture of permanent magnets, as well as devices for storing and rewriting high-density information. The main factors explaining the possibility of widespread use of hexaferrites in magneto-optics, microwave devices, acoustoelectronics are high values of its hardness, Curie temperature, coercive force, and indices of chemical inertness [44]. It was shown in [139, 140] that hexaferrites containing more than one element replacing iron can differ in the ferromagnetic resonance frequency and transmission capacity. Thus, by varying the composition of the material, one can smoothly change the level of the properties noted above.

Russian-language publications in the field of HEAs

There are relatively few studies in the field of high-entropy alloys published in Russian. The most detailed review of the work in the field of HEAs, published in the Russian Federation, was carried out by Rogachev and presented in 2020 in the journal "Physics of Metals and Metallurgy" [17]. It reflects the most important primary sources related to the development of multicomponent equiatomic alloys, as well as analyzes the research carried out in 2017-2020. One of the tasks set by the author was to identify the degree of correlation between the trend chasing of the HEAs concept and the prospects of these alloys as objects corresponding to the "new paradigm of materials science".

The works of Bashev and Kushnerev [22, 78], Firstov, Karpov, Gorban, Krapivka, etc. [23, 24, 27, 103, 115], Nadutov, Makarenko, Volosevich [51], Pushin, Ivchenko, Kourov et al. [31, 36, 141], Chikov, Vyukhin et al. [142-145], Salishchev, Shaisultanov, Kuznetsov et al. [60, 122], Kochetov, Rogachev et al.



[65], Sanin. Yukhvid et al. [71], Gorbachev, Popov et al. [146], Trofimenko et al. [26], Vinnik et al. [44, 138] deserve attention.

Russian-speaking specialists will also benefit from reviews presented in thesis research carried out at the Belgorod State National Research University by Shaisultanov [82], Klimova [21], Yurchenko [147], as well as at the Institute of Metal Physics of the Ural Branch of the Russian Academy of Sciences by Ivchenko [102].

Conclusion

The branch of materials science focused on the study of the structure and properties of multicomponent systems, the elements of which are present in equiatomic or close to it ratios, can be defined as a new, intensively developing one. The principles underlying the formation of the structure of multicomponent equiatomic alloys are radically different from the approaches that were previously used in the development of other types of metal materials.

The initially formulated concept of creating high-entropy alloys, according to which the high entropy of mixing ensures the formation of disordered substitution solid solutions and suppresses the formation of intermetallics and ordered phases, has undergone fundamental changes. Later experimental studies have shown that a high level of entropy of mixing is not the determining criterion for structure formation in multicomponent alloys. At the same time, obtaining a single-phase structure of a solid solution is currently not a mandatory requirement for the HEAs being developed.

The data presented in the literature on the structure of multicomponent materials, the criteria for its stability, and the influence of processing parameters on the structure and complex of properties are ambiguous.

Based on numerous studies of multicomponent alloys, no universal parameter has been proposed and no combination of several parameters has been identified, taking into account which it would be possible to predict the structure, and, consequently, the properties of materials, with a high degree of reliability. Accurate prediction of the phase composition of high-entropy alloys is currently not achieved by any of the approaches or methods available to specialists.

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Conflicts of Interest

The authors declare no conflict of interest.

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