СТРУКТУРОУТВОРЕННЯ. ОПІР РУЙНУВАННЮ ТА ФІЗИКО-МЕХАНІЧНІ ВЛАСТИВОСТІ

STRUCTURE FORMATION. RESISTANCE TO DESTRUCTION AND PHYSICAL-MECHANICAL PROPERTIES

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STRUCTURAL AND PHASE FORMATION IN A MEDIUM-ENTROPY, HIGHALLOYED COMPOSITION OF THE AL-MG-SI-V-CR-MN-FE-NI-CU SYSTEM

Purpose. To obtain, in the simplest possible way, an alloy with a relatively low melting temperature for HEAs and MEAs, containing non-deficit components capable of dissolving in aluminum and exhibiting mutual solubility. The concentration of most components did not exceed 2.5–5 at.%, therefore the main was to increasing the mixing entropy was the number of elements included in the alloy composition.

Research methods. The melt was prepared using a laboratory resistance furnace. Pure components and concentrated master alloys were used as charge materials, added gradually in small amounts to prevent the formation of refractory intermetallics. Melting was carried out in an alumina crucible at 1000 °C, which ensured dissolution and assimilation of all components.

Results. Structural and phase characteristics of the experimental alloy were compared depending on the cristalization rate. When cooled with the furnace at a rate of 0.5 °C/s, a heterogeneous structure formed, represented mainly by three phases: an intermetallic of the Al_6Me type, based on Al_6Mn with dissolved Fe, Ni, Cr, and V; an intermetallic based on the Al_6Cu_2Ni-type phase, which by stoichiometry could be expressed as $(Al,Ni)_2Cu$; and the Mg_2Si phase. Rapid solidification at $5 \cdot 10^2$ °C/s resulted in some refinement of the structure and increased its homogeneity, but did not significantly change the phase composition. Notably, in the Al_6Me intermetallic, the higher-temperature compound $Al_{23}V_4$ became predominant. The formation of an intermetallic in the Al-Ni-Cu system under these conditions could correspond to a compound formation with the formula $Al_2(Ni,Cu)_3$. The Mg_2Si phase was observed as part of the eutectic (Mg_2Si+Si).

Scientific novelty. A new approach to producing medium-entropy multicomponent alloys by resistance furnace melting has been demonstrated. The influence of cooling rate on phase formation in the Al-Mg-Si-V-Cr-Mn-Fe-Ni-Cu alloy has been revealed.

Practical value. An approach has been developed for obtaining medium-entropy alloys from non-deficit components with a reduced melting temperature, which simplifies their synthesis. The results can be used for further studies of high-entropy and medium-entropy alloys.

Key words: Al-Mg-Si-V-Cr-Mn-Fe-Ni-Cu system, HEA, MEA, highly alloyed compositions, new materials, structure, phase formation.

Introduction

In recent decades, particular attention of materials scientists has been drawn to the development of high-entropy alloys (HEAs), medium-entropy alloys (MEAs), and multi-principal element alloys (MPEAs), which are distinguished by a set of extraordinary properties determined by their specific structural–phase state. HEAs were first mentioned in 1995, which subsequently stimulated the advancement of scientific research in this field, and since 2003 they have been regarded as one of the latest breakthroughs in the development of a new class of materials [1, 2].

Since the first publications in 2004 in independent studies by Jien-Wei Yeh and Brian Cantor, high-entropy alloys have opened new opportunities for the development of a wide range of novel materials [3, 4]. By the general definition, such alloys contain at least five principal elements, each with a concentration ranging from 5 to 35 at. % and may also include minor elements with concentrations below 5 at.%.

Over the past decade, the evolution of materials research has led to a modification of the definition of HEAs. The compositional constraints of 5–35 at. % for principal elements are now applied less frequently, thereby enabling the formation not only of single-phase solid-

solution structures but also dual-phase, eutectic, and multiphase structures, intermetallic compounds, as well as their combinations, which may exhibit either crystalline or amorphous configurations [5–7]. The microstructure and specific crystallographic features of HEAs account for their high strength and hardness, wear resistance, resistance to high-temperature oxidation, enhanced corrosion resistance, fatigue strength, and fracture toughness.

Analysis of research and publications

In the design of metallic alloys, the conventional approach typically involves a base metal with minor additions of one to four soluble elements, which act through mechanisms of solid-solution or dispersion strengthening. With the advent of high-entropy alloys, the paradigm of alloy design has shifted: all constituent elements, present in approximately equiatomic concentrations, exhibit such a high degree of energetic uncertainty regarding the formation of individual phases that this leads to the development of complex and distinctive crystalline structures. The key contributing factors include the high degree of configurational entropy, sluggish diffusion, lattice distortion, and the so-called «cocktail effect», which together give rise to the unique physical and mechanical properties of these materials [3].

The synthesis of high-entropy alloys is feasible only within a rather limited range of technologies. In many cases, these processes require a protective atmosphere and are associated with powder metallurgy methods, as well as several metallurgical and casting techniques. The latter two categories reveal significant limitations when the alloys contain low-melting elements, metals with high vapor pressure, or components with substantial differences in density.

Density is a critical characteristic of most materials; therefore, the development of HEAs and MEAs incorporating lightweight elements represents an important task. The most suitable constituents in this context are aluminum, magnesium, and silicon. To preserve technological feasibility, particularly castability, the concentration of these elements in the alloy should be predominant. For effective incorporation, it is desirable that the components exhibit high mutual solubility, a condition that is satisfied by the aforementioned three elements. To enhance the configurational entropy of mixing, while adhering to the criteria outlined above, it is necessary to increase the number of alloying components.

For improved processability, reduced melting temperatures, and enhanced solubility of the constituents, the aluminum content was raised to as much as 60 at.%. The concentrations of the other elements remained relatively low; however, both their presence and number contributed to achieving a mixing entropy level approaching that of conventional HEAs.

As a result, a high-alloy composition of 60Al-10Mg-10Si-2.5V-2.5Cr-2.5Mn-2.5Fe-5Ni-5Cu (at. %) was selected. Within this system, certain elements and groups of elements possess similar atomic radii, which is one of

the essential criteria for the formation of homogeneous solid solutions in HEAs. Copper and magnesium exhibit high solubility in aluminum, whereas silicon and manganese are less soluble. Copper can also dissolve nickel, which in turn, along with manganese, facilitates the dissolution of otherwise insoluble or poorly soluble elements such as iron, chromium, and vanadium.

It was initially assumed that the high configurational entropy of mixing (ΔSmix) in HEAs would inherently promote the formation of homogeneous solid solutions based on simple crystal lattices. However, subsequent studies have demonstrated that other thermodynamic and geometric factors—such as the enthalpy of mixing (ΔHmix), atomic size mismatches among the constituent elements, interaction parameters, and valence electron concentration—also play a decisive role in governing the phase stability of HEA solid solutions [8–11].

Atomic size mismatch (δ_r): The percentage deviation in atomic radii of the constituent elements within the system. Values within the range of 0–6.6% favor the formation of stable solid solutions, whereas larger deviations may lead to the formation of intermetallic phases. The atomic size mismatch is calculated according to the following equation:

$$\delta_r = \sqrt{\sum_{i=1}^{n} c_i \left(1 - \frac{r_i}{\underline{r}}\right)^2} \times 100\%, \qquad (1)$$

where c_i is the atomic fraction of the *i*-th element; r_i is the atomic radius of the *i*-th element; and $\bar{r}$ is the average atomic radius of the system, defined as:

$$\underline{r} = \sum_{i=1}^{n} c_i \cdot r_i . \qquad (2)$$

Table 1 – Atomic radii of elements

Chemical element	Al	M	Si	V	Cr	M	Fe	Ni	Cu
Atomic radius, (pm)	143	160	111	134	128	127	126	124	128

The average atomic radius (r̄) was calculated as $\bar{r} = 138.375$ pm for the charge and $\bar{r} = 138.043$ pm for the final alloy. Substituting the atomic radii of the elements, the average atomic radius, and their atomic fractions into the formula yielded $\delta_r = 9.15\%$. This value is relatively high, since for the stability of solid solutions in high-entropy alloys it is generally desirable that $\delta_r < 6.6\%$. A deviation of 9.15% may cause lattice distortion and promote the formation of intermetallic phases.

The concentration of valence electrons (VEC) provides a useful criterion for predicting the type of crystal lattice that a system may form. A value of VEC > 8 indicates the formation of an FCC structure, whereas VEC < 6.87 points to the preferential formation of a BCC structure. For 6.87 < VEC < 8, the formation of a mixed

FCC + BCC structure can be expected. The corresponding data for the selected system are presented in Table 2.

Table 2 – The concentration of valence electrons

Chemical element	Al	Mg	Si	V	Cr	Mn	Fe	Ni	Cu
VEC	3	2	4	2	1	2	2	2	1

The concentration of valence electrons determines the average number of valence electrons in an alloy and is calculated using the formula:

$$VEC = \sum_{i=1}^{n} c_i \times VEC_i, \qquad (3)$$

where VEC_i is the number of valence electrons of the i-th element.

By substituting the data from Table 2 into equation (3), the valence electron concentration was calculated as $VEC = 2{,}7$, which indicates the predominant formation of a BCC structure.

Electronegativity difference ($\Delta\chi$): A small electronegativity difference ($\Delta\chi \leq 0{,}12$) promotes the formation of solid solutions, whereas larger differences may stimulate the formation of intermetallic (IM) phases. The electronegativity difference is calculated according to the following equation:

$$\Delta\chi = \sqrt{\sum_{i=1}^{n} c_i(\chi_i - \underline{\chi})^2}, \qquad (4)$$

where χ_i is the electronegativity of the i-th element, and $\underline{\chi}$ is the average electronegativity of the alloy, calculated as:

$$\underline{\chi} = \sum_{i=1}^{n} c_i\chi_i, \qquad (5)$$

The relevant data for the selected system are given in Table 3.

Table 3 – Electronegativity of components

Chemical element	Al	Mg	Si	V	Cr	Mn	Fe	Ni	Cu
Electronegativity, χ_i	1.61	1.31	1.9	1.63	1.66	1.55	1.83	1.91	1.9

By substituting the electronegativity values of the constituent elements and their atomic concentrations into equation (4), the electronegativity difference was calculated as $\Delta\chi = 0{,}162$. This value exceeds the optimal range (≤ 0.12), which may indicate structural instability.

The design and application of alloys based on simple solid solutions are sometimes justified by their superior processability, strength, and retention of ductility. At the same time, intermetallic phases are often regarded as detrimental, since they tend to embrittle alloys and complicate their processing. However, many structural alloys with advanced properties, including superalloys – where IM phases frequently constitute the dominant volumetric fraction – can exhibit both high strength and ductility. These characteristics depend strongly on the type of intermetallic lattice, their interaction with other phases, as well as the size, morphology, and distribution of the intermetallic [12].

The formation of intermetallic in amounts exceeding several volume percent is characteristic of alloys which, similar to HEAs, contain significant concentrations of multiple elements. However, their composition is designed in such a way that the configurational entropy of mixing is noticeably lower, thereby favoring a partial manifestation of the "cocktail effect." Such alloys are generally referred to as medium-entropy or high-alloy systems. Alloys developed through partial implementation of HEA design principles, but incorporating intermetallic phases, are now considered one of the key strategies for the development of new materials with tailored properties [13–15].

Purpose

The objective of the present study was to ascertain the particularities inherent in the process of obtaining a high-alloyed multicomponent alloy based on aluminum, in which the conditions for obtaining HEA and MEA could be realized.

Materials and methods

The alloy was prepared under conditions closely resembling simple industrial processes. Although this approach could complicate the achievement of the desired outcome, it allowed for the evaluation of component assimilation, their mutual interactions, and the overall potential of such a processing route. The alloy of the Al-Mg-Si-V-Cr-Mn-Fe-Ni-Cu system was produced in a resistance furnace using an alumina crucible. Initially, concentrated ligatures of Al-20Mg, Al-20Si, Al-16Mn-4Fe, Al-50Cu, Al-15V, Al-10Cr, and Al-8Ni were remelted in small quantities at a temperature of 800 °C. To obtain the planned chemical composition of the alloy, the lacking components were subsequently added into the melt in small portions. During this process, the melt was stirred and its temperature was gradually increased to 950–980 °C to ensure maximum assimilation of all components. The melt was held at the maximum temperature for 15 minutes.

Upon completion, a small volume of the molten alloy was withdrawn from the crucible at 950 °C using a ceramic sampler (2 cm³ volume) and immediately transferred into a copper cylindrical mold in order to achieve the highest possible cooling rate. The remaining portion of the melt was left in the furnace, solidifying as the furnace cooled down. The cooling rate of the melt was approximately 0.5 °C/s in one case, and about 5·10² °C/s in the other. The appearance of the charge and the obtained samples is presented in Figure 1.
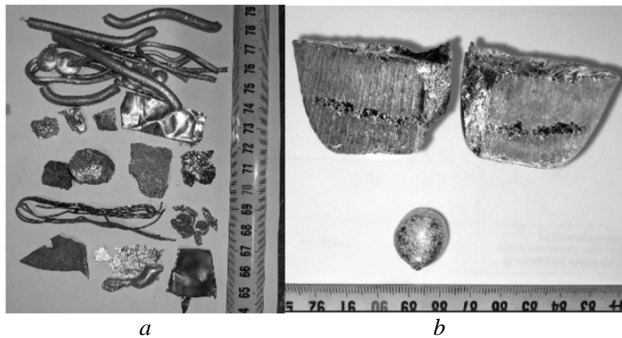
Figure 1. Appearance of charge materials (*a*) and obtained samples of experimental alloy (*b*)

Results and discussion

During alloy preparation, complete assimilation of all components was not achieved, as evidenced by the data in Table 4. Some constituents remained undissolved, while others were most likely lost due to evaporation or oxidation. The microstructural and phase analyses of the obtained samples revealed the presence of multiphase structures in both cases (Fig. 2). The corresponding local chemical compositions, in accordance with the designations in the figure, are summarized in Table 5.

In the case of slow solidification, a heterogeneous structure was formed, consisting predominantly of three phases. Based on the chemical composition of point 1, the formation of an intermetallic compound of the Al_6Me type, derived from Al_6Mn, can be assumed, in which Fe and Ni, as well as Cr and V, are dissolved.

Table 4 – Chemical composition of the experimental alloy charge and mixing entropy (ΔS) values

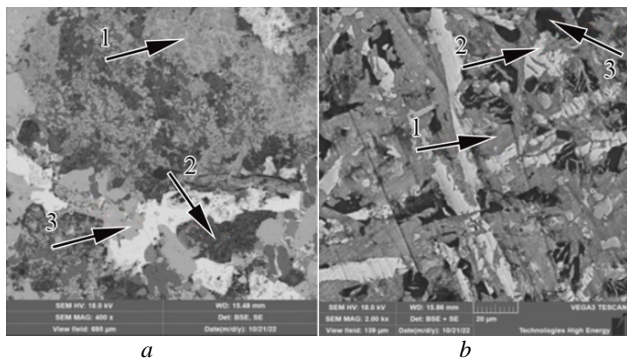| Chemical composition		Al	Mg	Si	V	Cr	Mn	Fe	Ni	Cu	ΔS
Alloy charge	at. %	60	10	10	2.5	2.5	2.5	2.5	5	5	1.44R
	wt.%	49.23	7.39	8.54	3.87	3.95	4.18	4.25	8.92	9.66	
Alloy in cast form	at. %	61.16	8.95	10.92	2.22	0.61	2.03	2.24	5.17	6.70	1.37R
	wt.%	49.97	6.59	9.29	3.42	0.96	3.37	3.79	9.18	12.9	

Figure 2. Microstructure of the experimental Al-Mg-Si-V-Cr-Mn-Fe-Ni-Cu alloy under slow (*a*) and rapid (*b*) cooling conditions

The second phase is also an intermetallic, but based on an Al_6Cu_2Ni-type structure, although its stoichiometry could alternatively be represented as $(Al,Ni)_2Cu$. The composition of point 2 clearly corresponds to the Mg_2Si compound.

Rapid solidification promoted partial refinement of the microstructure and an increase in its homogeneity, yet it did not lead to significant changes in the phase composition. It is noteworthy that in the Al_6Me-type intermetallic, a higher-temperature phase, likely $Al_{23}V_4$, may have become the dominant constituent. The formation of the Al-Ni-Cu intermetallic under these conditions could correspond to a compound approximating the formula $Al_2(Ni,Cu)_3$. The Mg_2Si phase in this sample was observed as part of a eutectic mixture (Mg_2Si + Si).

Table 5 – Local chemical composition of the alloy phases corresponding to the designations in Figure 2

Chemical composition, at. %		Al	Mg	Si	V	Cr	Mn	Fe	Ni	Cu
Slow cooling	point 1	56.98	5.65	2.29	1.7	1.72	9.25	7.98	9.42	4.86
	point 2	3.44	59.91	31.77	0.05	0.06	0.01	0.07	0.65	3.78
	point 3	42.41	0.45	0.27	0.06	0.18	0.14	0.47	36.21	19.68
Rapid cooling	point 1	60.57	0.97	3.38	7.47	1.22	6.72	5.83	9.84	3.96
	point 2	41.8	0.71	0.17	0.07	0.04	0.44	1.06	24.99	30.65
	point 3	11.8	37.66	47.86	0.16	0.04	0.23	0.38	0.36	0.51

Conclusions

Analyzing the obtained data, it can be concluded that the experimental Al-Mg-Si-V-Cr-Mn-Fe-Ni-Cu alloy, which falls into the category of medium-entropy alloys based on its mixing entropy, does not form solely solid solutions, regardless of the cooling rate of the melt. The phase composition of the obtained samples is characterized by the presence of three types of intermetallic compounds. In the rapidly solidified sample, a high-temperature eutectic reaction, L → Mg$_2$Si + Si, is observed. The simultaneous presence of magnesium and silicon in the alloy and the formation of an intermetallic phase between them may reduce the likelihood of forming an aluminum-based solid solution.

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СТРУКТУРО- ТА ФАЗОУТВОРЕННЯ В СЕРЕДНЬОЕНТРОПІЙНОМУ ВИСОКОЛЕГОВАНОМУ СПЛАВІ СИСТЕМИ AL-MG-SI-V-CR-MN-FE-NI-CU

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Мета роботи. *Одержати найбільш простим можливим способом сплав з відносно низькою для високоентропійного сплаву (ВЕС) та середньоентропійного сплаву (СЕС) температурою плавлення, який би містив недефіцитні компоненти, здатні розчинятися в алюмінії та мати взаємну розчинність. Концентрація більшості компонентів не могла перевищувати значення 2,5–5 % ат., тому основним засобом підвищення ентропії змішування слугувала кількість елементів, що входили до складу сплаву.*

Методи дослідження. *Для приготування розплаву було використано лабораторну піч опору. В якості шихтових матеріалів використовували чисті компоненти і концентровані лігатурні добавки, які додавалися почергово і поступово у невеликих кількостях, щоб запобігти утворенню тугоплавких інтерметалідів. Плавлення відбувалось в алундовому тиглі при температурі 1000 °C, що дало можливість розплавитись і засвоїтись всім компонентам.*

Отримані результати. *Було проведено порівняння структурних та фазових характеристик експериментального сплаву в залежності від швидкості кристалізації. При охолодженні розплаву з піччю зі швидкістю 0,5 °C/c формується неоднорідна структура, представлена переважно трьома фазами – інтерметалідом типу Al_6Me, на основі Al_6Mn, в якій розчинені залізо, нікель, хром та ванадій, інтерметалідом на основі фази типу Al_6Cu_2Ni, яка за стехіометрією могла б бути виражена формулою $(Al,Ni)_2Cu$, та фазою Mg_2Si. Швидка кристалізація $5\cdot10^2$ °C/c сприяла певному подрібненню структури та збільшенню її однорідності, проте не призвела до помітних змін фазового складу. При цьому, варто відзначити, що в інтерметаліді типу Al_6Me основою стала виступати більш високотемпературна сполука $Al_{23}V_4$. Формування інтерметаліду системи Al-Ni-Cu за даних умов могло б відповідати утворенню сполуки з формулою $Al_2(Ni,Cu)_3$. Фаза Mg_2Si для даного зразку виділилась у складі евтектики (Mg_2Si+Si).*

Наукова новизна. *Показано підхід до виготовлення середньоентропійних багатокомпонентних сплавів методом плавки в печі опору. Показано вплив швидкості охолодження на фазоутворення в сплаві Al-Mg-Si-V-Cr-Mn-Fe-Ni-Cu.*

Практична цінність. *Розроблено підхід до отримання середньоентропійних сплавів із недефіцитних компонентів та зниженою температурою плавлення, що спрощує їх синтез. Отримані результати можуть бути використані для подальших досліджень високоентропійних та середньоентропійних сплавів.*

Ключові слова: *Al-Mg-Si-V-Cr-Mn-Fe-Ni-Cu, ВЕС, СЕС, високолеговані сплави, нові матеріали, структура, фазоутворення.*

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