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ВЫСОКОЕНТРОПІЙНІ СПЛАВИ Al-Co-Cr-Fe-Mn-Ni-Si-V У ЛИТОМУ ТА РІДКОЗАГАРТОВАНОМУ СТАНІ

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Анотація. ***Мета.*** Дану роботу присвячено встановленню впливу складу та швидкості охолодження розплаву на мікротвердість, фазовий склад та параметри тонкої структури високоентропійних сплавів (ВЕС) системи Mn-Ni-Si-V в литому та рідкозагартованому стані. ***Методика.*** Литі зразки сплавів були отримані із застосуванням мідної виливниці (швидкість охолодження ~10^2 К/с). Гартування з рідкого стану (ЗРС) проводилося за відомою методикою splat-охолодження. Оцінена за товщиною фольги швидкість охолодження розплаву становила ~10^6 К/с. Рентгеноструктурний аналіз проводився із використанням діфрактометра ДРОН-2.0. Мікротвердість вимірювалася за допомогою мікротвердоміра ПМТ-3. Підбір компонентів досліджених ВЕС здійснювався, виходячи з прийнятих у літературі критеріїв компоновки та оцінки фазового складу ВЕС, основаних на обчисленні ентропії та ентальпії змішування, концентрацій валентних електронів а також різниці атомних радіусів компонентів. ***Результати.*** Встановлено, що у структурі литих сплавів присуттні невпорядковані тверді розчини типу ОЦК та впорядкований твердий розчин типу В2, в той час, як в ЗРС сплавах – лише невпорядковані тверді розчини типу ОЦК. Визначені параметри решітки дозволяють припустити, що формування твердих розчинів відбувається на основі Cr як елементу із найвищою температурою плавлення. Усі литі зразки сплавів системи Al-Co-Cr-Fe-Mn-Ni-Si-V демонструють наявність чітко вираженої дендритної структури із різними конфігураціями та об'ємами міждендритного простору. Показано позитивний вплив мікронапружень та густини дислокацій на рівень механічних характеристик досліджених сплавів. Встановлено, що ЗРС сплави системи Al-Co-Cr-Fe-Mn-Ni-Si-V характеризуються більш високими значеннями мікротвердості, ніж литі сплави. ***Наукова новизна.*** У даній роботі були вперше отримані і досліджені ВЕС системи Al-Co-Cr-Fe-Mn-Ni-Si-V у литому і рідкозагартованому стані. ***Практична значимість.*** ВЕС характеризуються унікальною структурою та цілим комплексом виняткових експлуатаційних характеристик, таких як твердість, зносостійкість, стійкість до окислення, корозії та іонизуючих випромінювань, висока термічна стабільність.. Дослідження тонких плівок ВЕС, отриманих шляхом гартування з рідкого стану, також становить великий практичний інтерес, оскільки одним з перспективних застосувань ВЕС є саме тонкоплівкові покриття.

Ключові слова: высокоентропійний сплав, гартування з рідкого стану, фазовий склад, структура, мікротвердість

ВЫСОКОЭНТРОПИЙНЫЕ СПЛАВЫ Al-Co-Cr-Fe-Mn-Ni-Si-V В ЛИТОМ И ЖИДКОЗАКАЛЕННОМ СОСТОЯНИИ

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Аннотация. ***Цель.*** Настоящая работа посвящена установлению влияния состава и скорости охлаждения расплава на микротвердость, фазовый состав и параметры тонкой структуры высокоэнтропийных сплавов (ВЭС) системы Al-Co-Cr-Fe-Mn-Ni-Si-V в литом и жидкозакаленном состоянии. ***Методика.*** Литые образцы сплавов были получены с использованием медной изложницы (скорость охлаждения ~10^2 К/с). Закалка из жидкого состояния (ЗЖС) проводилась по известной методике splat-охлаждения. Оцененная по толщине фольги скорость охлаждения расплава составляла ~10^6К/с. Рентгеноструктурный анализ проводился с использованием дифрактометра ДРОН-2.0. Микротвердость измерялась на микротвердомере ПМТ-3. Подбор компонентов исследованных ВЭС осуществлялся, исходя из принятых в литературе критериев компоновки и оценки фазового состава ВЭС, основанных на вычислении энтропии и энтальпии смешивания, концентраций валентных электронов а так же разницы атомных радиусов компонентов. ***Результаты.*** Установлено, что в структуре литых сплавов присутствуют неупорядоченные твердые растворы типа ОЦК и упорядоченный твердый раствор типа В2, в то время, как в ЗЖС сплавах – только неупорядоченные твердые растворы типа ОЦК. Оцененные параметры решетки позволяют предположить, что формирование твердых растворов происходит на базе Cr как элемента с наивысшей

температурой плавления. Все литые образцы сплавов системы Al-Co-Cr-Fe-Mn-Ni-Si-V демонстрируют наличие ярко выраженной дендритной структуры с различными конфигурациями и объемами междендритного пространства. Показано положительное влияние микронапряжений и плотности дислокаций на уровень механических характеристик исследованных сплавов. Установлено, что ЗЖС сплавы системы Al-Cu-Fe-Ni-Si характеризуются более высокими значениями микротвердости, чем литые сплавы. ***Научная новизна.*** В данной работе были впервые получены и исследованы ВЭС системы Al-Co-Cr-Fe-Mn-Ni-Si-V в литом и жидкозакаленном состоянии. ***Практическая значимость.*** ВЭС характеризуются уникальной структурой и целым комплексом замечательных эксплуатационных характеристик, таких как твердость, износостойкость, устойчивость к окислению, коррозии и ионизирующим излучениям, высокая термическая стабильность. Исследование тонких пленок ВЭС, полученных при помощи ЗЖС, также представляет большой практический интерес, поскольку одним из перспективных применений ВЭС являются именно тонкопленочные покрытия.

Ключевые слова: высокоэнтропийный сплав, закалка из жидкого состояния, фазовый состав, структура, микротвердость

HIGH-ENTROPY ALLOYS Al-Co-Cr-Fe-Mn-Ni-Si-V IN AS-CAST AND SPLAT-QUENCHED STATES

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Abstract. ***Purpose.*** This work is dedicated to establish the effects of the composition and the melt cooling rate on microhardness, phase composition and parameters of the fine structure of high-entropy alloys (HEA) of Al-Co-Cr-Fe-Mn-Ni-Si-V system in the as-cast and rapid quenched state. ***Metodology.*** As-cast alloy samples were obtained using a copper mold (cooling rate ~ 10^2 K/s). Quenching from a liquid state was carried out by a known technique of splat-quenching (SQ). Cooling rate estimated by foil thickness was ~ 10^6 K/s. The X-ray diffraction analysis was carried out with use of the DRON-2.0 diffractometer. Microhardness was measured on the PMT-3 microhardnessmeter. Selection of components of the studied HEAs was carried out on the basis of the criteria adopted in the literature for the HEA composition based on calculation of the entropy and enthalpy of mixing, valence electron concentrations as well as the difference between the atomic radii of the components. ***Findings.*** It was found that the as-cast alloys show a multiphase BCC+B2 structure, while the SQ alloys - fully disordered BCC crystal structure only. The value of lattice parameters of the investigated alloys suggests that the solid solutions are form on the base of Cr lattice, in view of its higher melting temperature. All of the as-cast alloys display a typical cast dendritic structure with various configurations and volumes of the interdendritic space. The positive influence of microstrains level and dislocation density on the microhardness values of splat-quenched Al-Co-Cr-Fe-Mn-Ni-Si-V alloys has been established. Improved mechanical characteristics are ensured by the strong distortion of the crystal lattice due to the differences in atomic radii of the elements. It was found that the splat-quenching HEAs of Al-Co-Cr-Fe-Mn-Ni-Si-V system are characterized by higher values of microhardness than as-cast alloys. ***Originality.*** At present work were first obtained and studied HEAs of Al-Co-Cr-Fe-Mn-Ni-Si-V system in the as-cast and splat-quenched state. ***Practical value.*** The HEAs possess many attractive properties, such as high hardness, outstanding wear resistance, irradiation resistance, excellent high-temperature strength, good thermal stability and corrosion resistance. The study of thin films obtained by splat-quenching from the liquid state is also of great practical interest, since one of the promising applications of HEAs are thin film coatings.

Keywords: high-entropy alloy, splat-quenching, phase composition, structure, microhardness

Introduction

Traditionally, the development of new metallic alloys has been carried out by selecting one or two basic elements with subsequent alloying using optimum concentrations of useful impurities. However in the last time, increasing industrial demands for various structural and functional metallic materials have stimulated the advent of new technologies and the development of multicomponent alloys. Recently a new class of materials known in the literature as multicomponent high-entropy alloys (HEA) was obtained [10]. High-entropy alloys are defined as solid solution alloys that contain more than five principal elements (usually from five to thirteen) in equal or near equal atomic percent. The basic principle of HEAs is the stabilization of solution phase by the significantly higher configurational entropy of mixing ΔS_{mix} compared to conventional alloys. The configurational entropy of mixing during the formation of regular solution alloy can be determined as

$$\Delta S_{mix} = -R\sum_{i=1}^{n} c_i \ln c_i \ , \qquad (1)$$

c_i - atomic fraction of the *i*-th component, *R* - universal gas constant. Increasing of mixing entropy reduces the Gibbs free energy of the alloy and improves the stability of the solid solution. For the alloy where *n* is the number of components maximum mixing entropy is when they are mixed in equal atomic fractions.

Usually in HEAs value of ΔS_{mix} is in the range of 12 - 19 J/(mol·K). Due to the high mixing entropy HEAs are solid solutions typically having simple crystal structures (FCC or BCC), but to avoid the appearance of brittle intermetallic compounds, complex microstructures and amorphous phases in the structure of alloys, some phase formation criteria are required to be completed. According to [20, 21], the Ω parameter can be used to estimate the phase composition of HEA.

$$\Omega = \frac{T_m \Delta S_{mix}}{|\Delta H_{mix}|}, \quad (2)$$

where T_m is the average melting temperature of alloy, ΔH_{mix} - the mixing enthalpy

$$T_m = \sum_{i=1}^{n} c_i (T_m)_i , \quad (3)$$

$$\Delta H_{mix} = \sum_{i=1, i\neq j}^{n} \Omega_{ij} c_i c_j , \quad (4)$$

where the regular melt-interaction parameter between *i*-th and *j*-th elements $\Omega_{ij} = 4\Delta H_{mix}^{AB}$, and ΔH_{mix}^{AB} - mixing enthalpy of binary liquid AB alloy. Alloy components should not have large atomic-size difference, which is described by the parameter

$$\delta = 100\sqrt{\sum_{i=1}^{n} c_i \left(1 - r_i / \bar{r}\right)^2} , \quad (5)$$

where $\bar{r} = \sum_{i=1}^{n} c_i r_i$, r_i - the atomic radius of the *i*-th element.

According to [21] the high-entropy alloys for which Ω≥1.1 and δ≤6.6 can form the solid solutions without intermetallic compounds and amorphous phases. However, simple (not ordered) solid solutions form if -15 kJ/mol < ΔH_{mix} < 5 kJ/mol and δ≤4.6.

The other useful parameter is the valence electron concentration, *VEC*, which has been proven useful in determining the phase stability of high- entropy alloys [4,5]. VEC is defined by:

$$VEC = \sum_{i=1}^{n} c_i (VEC)_i , \quad (6)$$

where $(VEC)_i$ - valence electron concentration (including the *d*-electrons) of the *i*-th element. As pointed in [5] at *VEC* ≥8.0, sole FCC phase exists in alloy; at 6.87 ≤ *VEC* < 8.0, mixed FCC and BCC phases will co-exist and sole BCC phase exists at *VEC* < 6.87.

In some studies also proposed structure prediction method, based on an evaluation of the difference of electronegativity values and concentrations of valence electrons for the alloy components. This method is based on the Hume-Rothery rules, according to which for the formation of a binary substitutional solid solution the solvent and solute elements must be characterized by similar values of electronegativity and valence, as well as the minimum difference in the atomic sizes. However, based on the results of [2,9,11], one may say that, in general, for HEAs this method cannot reliably predict the phase composition of the alloys.

It has been reported that HEAs possess many attractive properties, such as high hardness, outstanding wear resistance, irradiation resistance, excellent high-temperature strength, good thermal stability and corrosion resistance, magnetic and thermoelectric propetries [1, 6-8,12-14,16-19,22]. Improved mechanical characteristics are ensured by strong distortion of the crystal lattice due to the differences in atomic radii of the elements.

In this work effect of the value of mixing entropy and composition on the microhardness, phase composition and parameters of the fine structure of HEAs of Al-Co-Cr-Fe-Mn-Ni-Si-V alloy system is discussed (Mn and Si are added as minor elements to improve mechanical properties and corrosion resistance).

Experimental Details

The samples of Al-Co-Cr-Fe-Mn-Ni-Si-V high-entropy alloys were taken from the as-cast (cooling rate of ~ 10^2 K/s) ingots, polished and etched for examining the microstructure under an optical microscope Neophot-21 and scanning electron microscope (SEM, JSM-6490LV) with energy dispersive spectrometry (EDS). The quenching from the molten state (splat-quenching, SQ) was performed using the well-known technique of melt spinning, i.e., spreading of melt droplets on the internal surface of a rapidly rotating copper cylinder. The rate of cooling as estimated from the thickness of the obtained foils was ~10^6 K/s. The XRD studies were carried out using a DRON-2.0 X-ray diffractometer operating with Cu *Kα* radiation. The microhardness was measured on a PMT-3 microhardness-meter at a load of 200g.

Results and Discussion

Using the data from [4,15], the following quantities are calculated for the investigated HEAs (Tab.1): ΔS_{mix} , ΔH_{mix} , δ, Ω and *VEC*.

The phase composition of investigated alloys, crystal lattice parameters and fine structure parameters (size of coherently scattering domains and microstrains) (Tab.2) were determined from the XRD patterns (Fig.1, 2). The microhardness values of HEAs are also given in Tab. 2.

The analysis of the XRD patterns allowed us to establish that the investigated HEAs in the as-cast state have two-phase body-centered cubic (BCC) + B2 (caesium chloride) structure. Indeed, from the analyses of Tab. 1 it is seen that ΔH_{mix} has a large negative value favoring the formation of a compound. The low value of VEC favours the formation of a BCC phase. These factors put together leads to the formation of a mixture of BCC and B2 phases (the ordered version of BCC). Exception is the $AlCoCrFe_{1.87}Mn_{0.03}NiSi_{0.1}V$ alloy, for which the value of VEC lies in the range, where a face-centered cubic (FCC) + BCC mixture is favoured. But, as pointed in [11], if the value of VEC is close to the boundary values, predictions of the phase compositions sometimes not work.

Meanwhile the XRD patterns of SQ alloys do not have a (100) B2 superlattice reflection and consequently SQ HEAs contains only disordered BCC phase. In our opinion the high cooling rate during the formation of thin SQ film should prevent it from possible separation and hinder the appearance of structures and phases typical for equilibrium as-cast states.

Table 1

Values of ΔHmix, ΔSmix, δ, VEC и Ω for HEAs of Al-Co-Cr-Fe-Mn-Ni-Si-V alloy system

Alloy	ΔHmix, kJ/mol	ΔSmix, J/(mol·K)	δ	VEC	Ω
$AlCoCrFe_{0.87}Mn_{0.03}NiSi_{0.1}V$	-17.04	15.52	5.18	6.76	1.6
$AlCoCrFe_{1.87}Mn_{0.03}NiSi_{0.1}V$	-14.6	15.18	4.96	6.94	1.84
$Al_2CoCrFe_{0.87}Mn_{0.03}NiSi_{0.1}V$	-18.98	15.07	5.78	6.22	1.3
$Al_2CoCrFe_{1.87}Mn_{0.03}NiSi_{0.1}V$	-16.81	14.97	5.66	6.45	1.48

Table 2

Phase composition, size of coherently scattering domains (L), degree of distortion of the crystal lattice (Δa/a) and microhardness (Hμ) of investigated alloys

Alloy	Phase composition	L, nm	$\Delta a/a$	H_μ, MPa
As-cast $AlCoCrFe_{0.87}Mn_{0.03}NiSi_{0.1}V$	BCC + B2 (a=0.2888 nm)	20±2	$3.2\cdot10^{-3}$	6800 ±300
SQ film $AlCoCrFe_{0.87}Mn_{0.03}NiSi_{0.1}V$	BCC (a=0.2882 nm)	34±2	$3.8\cdot10^{-3}$	6900 ±300
As-cast $AlCoCrFe_{1.87}Mn_{0.03}NiSi_{0.1}V$	BCC + B2 (a=0.2882 nm)	30±2	$2.5\cdot10^{-3}$	4800 ±200
SQ film $AlCoCrFe_{1.87}Mn_{0.03}NiSi_{0.1}V$	BCC (a=0.2879 nm)	25±2	$2.8\cdot10^{-3}$	6200 ±300
As-cast $Al_2CoCrFe_{0.87}Mn_{0.03}NiSi_{0.1}V$	BCC + B2 (a=0.2888 nm)	35±2	$1.6\cdot10^{-3}$	6500 ±300
SQ film $Al_2CoCrFe_{0.87}Mn_{0.03}NiSi_{0.1}V$	BCC (a=0.2887 nm)	33±2	$1.8\cdot10^{-3}$	7500 ±300
As-cast $Al_2CoCrFe_{1.87}Mn_{0.03}NiSi_{0.1}V$	BCC + B2 (a=0.2886 nm)	37±2	$1.5\cdot10^{-3}$	4600 ±200
SQ film $Al_2CoCrFe_{1.87}Mn_{0.03}NiSi_{0.1}V$	BCC (a=0.2881 nm)	33±2	$1.7\cdot10^{-3}$	5600 ±200

The values of lattice parameters of the investigated alloys suggests that the solid solutions are form on the base of Cr lattice (a =0.2884 nm), in view of its higher melting temperature.

High microhardness values of Al-Co-Cr-Fe-Mn-Ni-Si-V HEAs (Tab.2) can be explained by the presence of the dissimilar atoms of elements with different size, electronic structure and thermodynamic properties in the crystal lattice. This leads to significant distortion ($\Delta a/a$) of the crystal lattice. Consequently the hardness of the alloys increases. As seen from Tab. 2, microhardness of the SQ alloys is higher than that of the as-cast alloys. This result is not unexpected, since the microstructure and the phase composition of the as-cast alloy after decomposition is in a more equilibrium multiphase state, while SQ alloys yields higher level of microstrains and dislocation density.

Fig. 3,4 are SEM micrographs of the as-cast HEAs of Al-Co-Cr-Fe-Mn-Ni-Si-V system. All of the alloys display a typical cast dendritic structure with various configurations and volumes of the interdendritic space. The EDS analysis made it possible to reveal the dendritic segregation in alloys. Tab. 3 shows the chemical composition of different areas analyzed by EDS. According to Tab. 3 The bright dendritic area has a higher amount of Al and Ni than dark interdendritic area, but the dark section of interconnected microstructure enriched with Fe, V and Cr elements. In contrast of the previous research [3] the addition of V not reduce segregation of Al, Ni, Fe, Cr elements. Obviously this is influenced by Mn and Si.

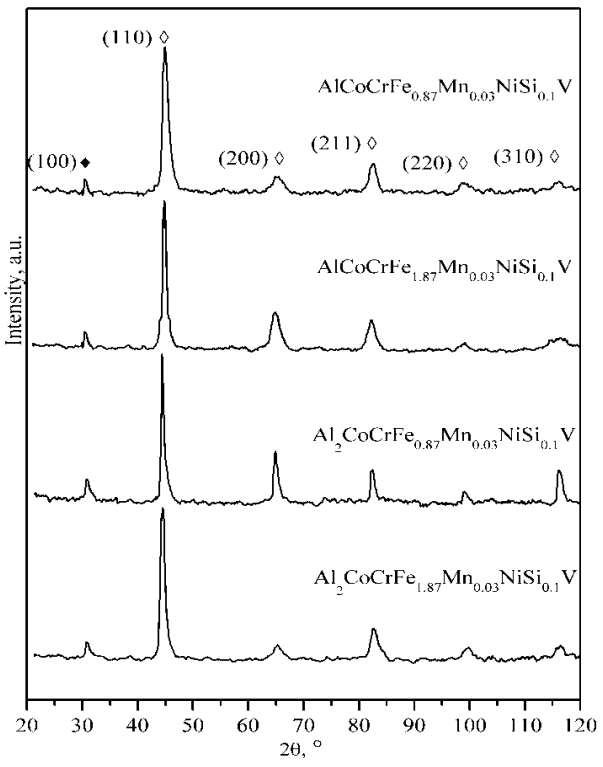

Fig.1. XRD patterns of as-cast HEAs of Al-Co-Cr-Fe-Mn-Ni-Si-V alloy system: ◊-BCC, ♦-B2.

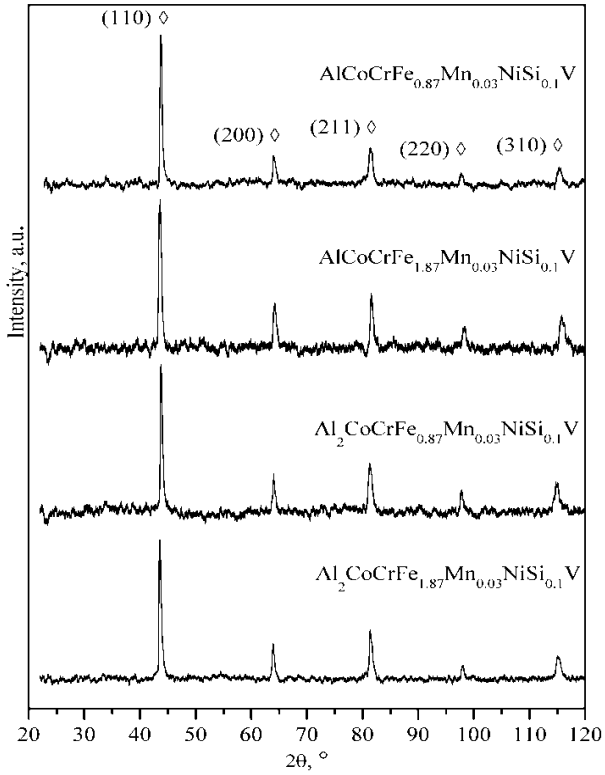

Fig.2. XRD patterns of splat-quenched HEAs of Al-Co-Cr-Fe-Mn-Ni-Si-V alloy system: ◊-BCC.

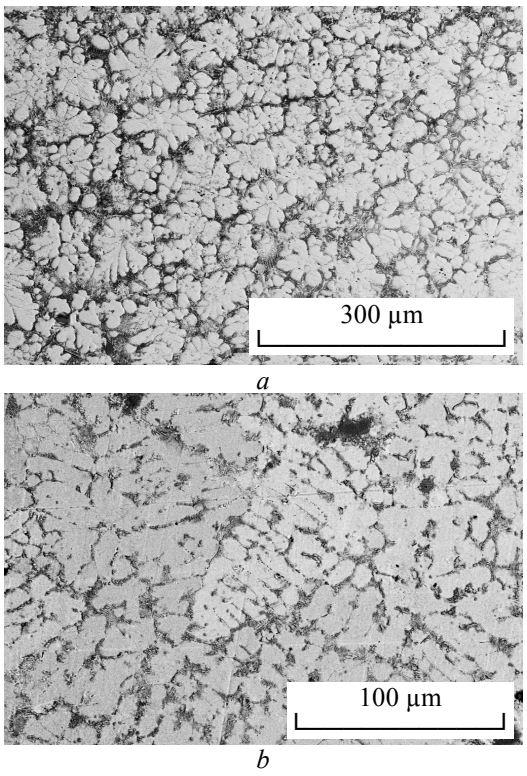

Fig.3. SEM images of as-casted HEAs specimens:
a - $AlCoCrFe_{0.87}Mn_{0.03}NiSi_{0.1}$;
b - $AlCoCrFe_{1.87}Mn_{0.03}NiSi_{0.1}V$

Conclusions

Based on the study of the Al-Co-Cr-Fe-Mn-Ni-Si-V HEAs, produced in the as-cast and splat quenched state, the following conclusions can be drawn:

1. As-cast alloys have multiphase BCC + B2 structure while SQ alloys exhibit only disordered BCC solid solution structure.
2. With the increase of cooling rate the level of microstrains, dislocation density and microhardness of Al-Co-Cr-Fe-Mn-Ni-Si-V HEAs increases.
3. The obtained results confirm the leading role of the element with higher melting temperature as the basis for the formation of solid solutions in the studied alloys.
4. The splat-quenched HEAs of Al-Co-Cr-Fe-Mn-Ni-Si-V system characterized by higher values of microhardness than as-cast alloys, which is obviously caused by higher level of microstrains and dislocation density.

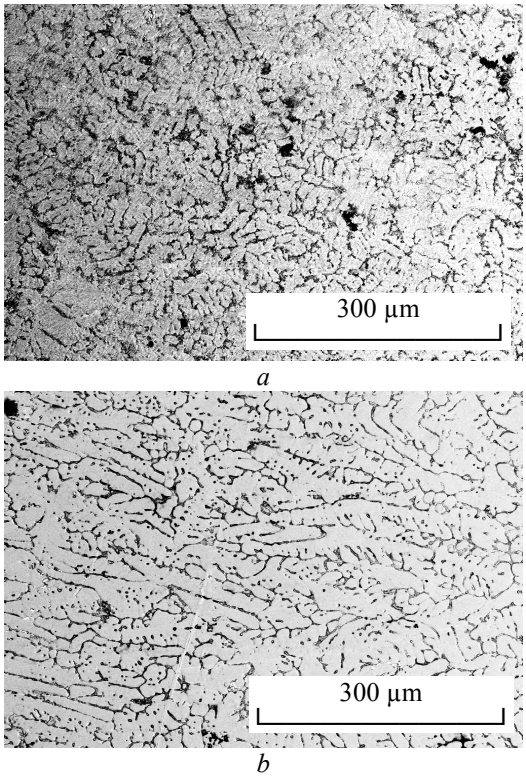

Fig.4. SEM images of as-casted HEAs specimens:
a - $Al_2CoCrFe_{0.87}Mn_{0.03}NiSi_{0.1}$;
b - $Al_2CoCrFe_{1.87}Mn_{0.03}NiSi_{0.1}V$

Table 3

Chemical composition of as-cast HEAs of Al-Co-Cr-Fe-Mn-Ni-Si-V alloy system

Alloy		Elements, at. %							
		Al	Co	Cr	Fe	Mn	Ni	Si	V
$AlCoCrFe_{0.87}Mn_{0.03}NiSi_{0.1}V$	Dendrite	20.13	17.25	14.77	14.03	0.29	19.64	1.07	12.82
	Interdendrite	10.30	16.14	20.95	17.34	0.71	14.05	2.47	18.04
	Nominal	16.67	16.67	16.67	14.49	0.5	16.67	1.66	16.67
$AlCoCrFe_{1.87}Mn_{0.03}NiSi_{0.1}V$	Dendrite	15.71	14.08	13.91	26.23	0.42	15.48	1.19	15.38
	Interdendrite	11.46	14.37	16.71	27.85	0.47	13.81	1.61	13.72
	Nominal	14.28	14.28	14.28	26.74	0.43	14.28	1.43	14.28
$Al_2CoCrFe_{0.87}Mn_{0.03}NiSi_{0.1}V$	Dendrite	30.35	16.25	11.10	11.31	0.28	18.21	1.10	11.40
	Interdendrite	9.28	10.10	21.90	18.85	0.70	8.10	1.57	29.50
	Nominal	28.58	14.28	14.28	12.44	0.43	14.28	1.43	14.28
$Al_2CoCrFe_{1.87}Mn_{0.03}NiSi_{0.1}V$	Dendrite	30.03	11.8	11.25	21.04	0.27	13.47	0.96	11.08
	Interdendrite	9.97	15.8	15.32	25.02	0.57	10.63	1.49	21.11
	Nominal	25	12.5	12.5	23.37	0.38	12.5	1.25	12.5

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