Structural Phase Variations in High-Entropy Alloy upon Pulsed Electron Beam Irradiation

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Abstract—In this paper, we obtain a high-entropy alloy (HEA) of the Al-Co-Cr-Fe-Ni system with a nonequiatomic composition using the technology of wire-arc additive manufacturing (WAAM) in a pure nitrogen atmosphere. Using the methods of modern physical materials science, we showed that the alloy in the initial state has a dendritic structure, which indicates a non-uniform distribution of alloying elements. The alloy is a multiphase material, and the main phases are Al₃Ni, Cr₃C₂, and (Ni,Co)₃Al₄. Cubic nanosized (Ni,Co)₃Al₄ particles are located along the interfaces of submicron Al₃Ni and Cr₃C₂ phases. Irradiation of the HEA with pulsed electron beams with an energy density of $E_s = 10-30 \text{ J/cm}^2$, a pulse duration of 50 µs, a frequency of 3 Hz, and a pulse number of 3 leads to high-speed melting and subsequent crystallization of the surface layer. At $E_{\rm s} = 10$ J/cm², no destruction of the dendritic crystallization structure occurs. The interdendritic spaces are enriched with aluminum, nickel, and iron while the dendrites themselves are enriched with chromium atoms. The most liquating element is aluminum, and the least liquating element is cobalt. At $E_s = 20 \text{ J/cm}^2$, a nanocrystalline structure is formed in the grain volume in a layer with a thickness of 15 μ m. The size of the crystallization cells is 100-200 nm, the size of inclusions are 20-25 nm at the junctions of cells and 10-15 nm along the boundaries of cells. High-speed crystallization cells are enriched with aluminum and nickel. Cobalt atoms are evenly distributed over the volume of the surface layer. The most liquating element is chromium, and the least liquating element is cobalt. An increase in the energy density of the electron beam to 30 J/cm^2 does not lead to significant (compared to 20 J/cm²) changes in the structure of the surface layer. The irradiation mode ($E_{\rm s} = 20 \text{ J/cm}^2$, 50 µs, 3 pulses, 0.3 Hz), which allows to form a surface layer with the highest homogeneity of the chemical element distribution in the alloy, is revealed.

Keywords: high-entropy alloy, wire-arc additive manufacturing, pulsed electron beam, structure, phase and elemental composition

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INTRODUCTION

In the last decade, the attention of researchers in the field of physical materials science was focused on the study of the structure, phase composition, stability, production methods and fields of application of a new class of metallic materials – high-entropy alloys (HEAs) with a whole range of properties that significantly exceed those of traditional metals and alloys [1-4]. The authors of numerous publications are developing methods and approaches for increasing the parameters and properties of HEAs [5]. Improvement of the properties of HEAs and, first of all, mechanical [6-11], can be achieved due to the creation of a bimodal grain size distribution [12], the formation of a nanostructured two-phase amorphous-crystalline state [13], the plasticity effect induced by the transformation [14], grain-boundary [15] and solid solution hardening [16], and many other methods [5, 17–23].

Improved properties can be achieved by treating the surface with concentrated energy flows. As a result, the probability of microcrack nucleation in the surface layers that lead to destruction is significantly reduced while the strength and plasticity increase [24].

One of the methods allowing homogenization of the alloy under conditions of high-speed melting and subsequent high-speed crystallization is irradiation of the sample surface with a low-energy intense pulsed electron beam [25]. Ultra-high (up to 10^9 K/s) heating rates of a relatively thin (tens to hundreds of μ m) surface layer to the melting temperature and subsequent high-speed crystallization of the melt allow the formation of a submicro- and nanocrystalline structure



Fig. 1. Electron microscopic image of the HEA: (a) initial state, (b, c) after irradiation with a pulsed electron beam with parameters of $E_s = 10 \text{ J/cm}^2$, $\tau = 50 \text{ µs}$, n = 3 pulses, and f = 0.3 Hz.

characterized by a high degree of uniformity of the chemical element distribution [26].

Numerous studies devoted to the analysis of the structure and phase composition of metals and alloys and cermet and ceramic materials processed by pulsed electron beams showed that the structural-phase state of the modified layer depends both on the thermophysical characteristics of the irradiated material and on the parameters of the electron beam [25–27].

This study is aimed at obtaining new knowledge about the structure and phase composition of a HEA with a composition of Al–Co–Cr–Fe–Ni that is irradiated by a pulsed electron beam.

MATERIAL AND RESEARCH METHOD

The HEA samples were prepared by layer-by-layer deposition on a stainless-steel substrate using the wire-arc additive manufacturing technology in an inert atmosphere (99.99% Ar). As a initial material, we used a three-core cable consisting of an aluminum wire (99.95% Al, the diameter is 0.5 mm), chromium-nickel wire Kh20N80 (20% Cr, 80% Ni, the diameter is 0.4 mm), and 29NK alloy wire (17% Co, 54% Fe, 29% Ni, diameter 0.4 mm). The following layer deposition mode was used: the cable feed speed is 8 m/min, voltage is 17 V, torch speed is 0.3 m/min, and substrate temperature is 523 K. The resulting ingot in the form of a parallelepiped with a size of $60 \times 140 \times 20$ mm consisted of 20 deposited layers in height and 4 layers in width. The ingot was cut into samples with a size of

 $15 \times 15 \times 5$ mm. The samples were irradiated with a pulsed electron beam using a SOLO setup [25, 26]. Irradiation mode: accelerated electron energy is 18 keV, the electron beam energy density is 10–30 J/cm², the beam pulse duration is 50 µs, the pulse repetition rate is 0.3 Hz, and the number of irradiation pulses is 3. Irradiation was performed at a residual argon pressure of 2×10^{-2} Pa in the operating chamber of the setup.

The structure and elemental composition of the samples were studied by scanning electron microscopy (LEO EVO 50 and TESCAN VEGA instruments equipped with an INCA Energy energy-dispersive analyzer). The defect substructure and distribution of chemical elements were studied by transmission electron microscopy (JEOL JEM-2100, Japan) [28–30]. The studied objects (foils with a thickness of 150–200 nm) for a transmission electron microscope were prepared by ion etching (an Ion Slicer (EM-09100IS) device, argon) of plates cut from a HEA bulk ingot.

RESULTS AND DISCUSSION

The structure of the AlCoCrFeNi HEA manufactured by the methods of wire-arc additive production in an inert-gas atmosphere is considered in detail in [1]. X-ray microanalysis showed that the alloy contains 36.5% Al, 33.7% Ni, 16.4% Fe, 8.6% Cr, and 4.9% Co (at.). The resulting material should be attributed to the HEA with a nonequiatomic composition. The alloy has a pronounced dendritic structure, which indicates an inhomogeneous distribution of



Fig. 2. Electron microscopic image of the foil section obtained by the STEM method (in panel (b), the sections of the micro X-ray analysis of the foil are indicated).

alloying elements in the ingot bulk (Fig. 1a). It was established by mapping that the boundaries and border regions of grains are enriched with chromium and iron atoms, the volume of grains is enriched with nickel and aluminum atoms, and cobalt is quasi-uniformly distributed in the alloy. Transmission electron diffraction microscopy showed that the HEA is a multiphase material, the main phases of which are Al₃Ni, Cr_3C_2 , and $(Ni,Co)_3Al_4$. The Al₃Ni phase has a cubic form, the Cr_3C_2 phase has a ribbon form, and the $(Ni,Co)_3Al_4$ phase is a spherical form. 7–10 nm $(Ni,Co)_3Al_4$ particles are located along the separation

 Table 1. Results of the elemental analysis of foil sections

Content at %

Spectrum	Content, ut /c				
	Al	Cr	Fe	Со	Ni
Spectrum 1	3.01	59.28	13.05	2.82	13.43
Spectrum 2	2.08	48.27	17.36	2.96	13.56
Spectrum 3	2.42	31.90	40.50	5.50	13.82
Spectrum 4	2.84	28.51	38.35	5.17	13.68
Spectrum 5	18.15	11.50	21.49	5.00	27.29
Spectrum 6	9.79	19.31	30.09	5.35	22.17
Spectrum 7	4.15	24.16	34.36	5.46	17.49
Spectrum 8	3.11	35.56	25.35	4.24	14.51
Spectrum 9	1.97	59.66	15.11	3.02	13.50
Spectrum 10	16.26	13.70	23.19	5.84	29.63
Average	6.38	33.18	25.88	4.54	17.91
Maximal value	18.15	59.66	40.50	5.84	29.63
Minimal value	1.97	11.50	13.05	2.82	13.43
δ (max/min)	9.20	5.20	3.10	2.10	2.20

boundaries of the Al_3Ni and Cr_3C_2 phases, which have submicron sizes [1].

Irradiation of the HEA by a pulsed electron beam with an electron beam energy density of $E_s = 10 \text{ J/cm}^2$ does not lead to the destruction of the structure of dendritic crystallization (Figs. 1b and 1c), which is characteristic of the alloy in the initial state (Fig. 1a). This fact indicates the absence of surface layer melting of the irradiated samples. The liquid-phase transformation of the structure of the HEA surface layer is found only in the border regions of the grain (dendritic) structure. The high-speed transformation of the structure is accompanied by the formation of interlayers with a submicron-nanocrystalline structure with a crystallite size of 100–200 nm (Fig. 1c). Structure studies of fractures very often reveals the transformation of the surface layer of a material subjected to processing by concentrated energy flows. However, in the considered case, the fused layers are very thin and brittle fracture is not detected by scanning electron microscopy.

An analysis of images of the surface layer structure of the material that were obtained in the characteristic X-ray radiation of various elements confirms the structure specific to dendritic crystallization of the melt.

The elemental composition of dendrites and interdendrite spaces of the surface layer of a HEA irradiated by a pulsed electron beam with an electron beam energy density of $E_s = 10 \text{ J/cm}^2$ was studied using the methods of X-ray spectral analysis of foil "by points." Figure 2 shows STEM images of the foil structure, which indicate the places of the analysis of the elemental composition of the alloy. The quantitative results of this analysis are shown in Table 1. The presented results (Fig. 2, Table 1) give grounds to conclude that interdendritic spaces (analyzed areas 5–7,



Fig. 3. Electron microscopic image of the surface of the HEA fracture irradiated by a pulsed electron beam ($E_s = 20 \text{ J/cm}^2$, $\tau = 50 \text{ µs}$, f = 3 pulses, n = 0.3 Hz) (in panel (a), the irradiation surface is indicated with the arrow).

10) are enriched with aluminum, nickel, and iron. Dendrites (analyzed areas 1, 2, 9) are enriched mainly in chromium atoms. Cobalt atoms are evenly distributed throughout the ingot volume. The most liquating element of the alloy is aluminum (the liquation coefficient is $\delta = 9.2$), and the least liquating element of the alloy is cobalt ($\delta = 2.1$).

Irradiation of the HEA by a pulsed electron beam with an electron-beam energy density of $E_s = 20 \text{ J/cm}^2$ leads to partial destruction of the structure of dendritic crystallization characteristic of the alloy in the initial state. This fact indicates the melting of the surface layer of the irradiated samples. The liquid-phase transformation of the structure of the HEA surface layer is accompanied by the formation of a nanocrystalline structure in the grain bulk.

The determination of the thickness of the molten layer and the study of its structure were performed by analyzing brittle fractures (Fig. 3). An analysis of micrographs indicates that irradiation of the HEA with a pulsed electron beam ($E_s = 20 \text{ J/cm}^2$, $\tau = 50 \text{ µs}$, n = 3 pulses, f = 0.3 Hz) leads to the formation of a surface layer with a submicro-nanocrystalline structure (Fig. 3b). The thickness of this layer can be up to 15 µm.

STEM studies of foils showed that irradiation of the HEA by a pulsed electron beam with an electron beam energy density of 20 J/cm² is accompanied by the formation of a high-speed cellular crystallization structure in the surface layer (Fig. 4a). The volume of crystallization cells is enriched with atoms of aluminum, nickel, and iron. Inclusions of the second phase, which are enriched mainly in chromium atoms, are located at the junctions and along the boundaries of the cells. The crystallization cell size is 100-200 nm while the size of the inclusions is 20-25 nm for located at the junctions of the cells and 10-15 nm for located along the cell boundaries.

Studies of the elemental composition of high-speed crystallization cells and second-phase particles by X-ray micro-spectral analysis of foils "point by point" showed that high-speed crystallization cells are enriched with aluminum and nickel. Particles of the second phase located at the boundaries and at the junctions of crystallization cells are enriched mainly in chromium and iron atoms. Cobalt atoms are evenly distributed over the volume of the surface laver. The most liquating element of the surface layer of the alloy treated with a pulsed electron beam ($E_s = 20 \text{ J/cm}^2$, $\tau = 50 \text{ } \mu\text{s}, n = 3 \text{ } \text{pulses, and } f = 0.3 \text{ } \text{Hz}$) is chromium (the liquation coefficient is $\delta = 5.4$), and the least liquating element is cobalt ($\delta = 1.9$). Based on the comparison of the results of X-ray spectral microanalvsis of the HEA after irradiation by a pulsed electron beam with different energy densities, it can be noted that high-speed melting and subsequent high-speed crystallization led to an increase in the homogeneity degree of the distribution of chemical elements that form the HEA.

An increase in the energy density of the electron beam to 30 J/cm^2 does not lead to significant changes in the structure of the surface layer. The thickness of this layer reaches ~20 µm. In this case, the surface layer also has a cellular-type high-speed crystallization structure. Crystallization cells are round. The cell size varies from 100 to 150 nm. The cells are bordered by interlayers of the second phase. The thickness of the interlayers varies from 15 to 30 nm. The cell volume is enriched with aluminum and nickel. Chromium and iron atoms predominantly form second-phase particles. Cobalt atoms are evenly distributed over the volume of the modified layer.

The most liquating element of the surface layer of the alloy treated with a pulsed electron beam ($E_s =$ 30 J/cm², $\tau = 50 \mu s$, n = 3 pulses, f = 0.3 Hz) is chromium (the liquation coefficient is $\delta = 10.5$), the least liquating element is cobalt ($\delta = 1.6$). The generalizing



Fig. 4. (a) Electron microscopic image of the foil section obtained by the STEM method and (b–f) images of this foil section obtained in the characteristic X-ray radiation of Al, Cr, Fe, Co, and Ni atoms.

results obtained by the X-ray spectral microanalysis of the HEA in the initial state and after irradiation with a pulsed electron beam and characterizing the heterogeneity degree of the distribution of alloying elements in



Fig. 5. Dependence of the distribution heterogeneity coefficient of chemical elements forming the HEA on the electron-beam energy density: (1) Cr, (2) Al, (3) Ni, (4) Fe, and (5) Co.

the surface layer are shown in Fig. 5. It can be noted that the Al–Co–Cr–Fe–Ni HEA obtained using the wire-arc additive production technology is characterized by a high degree of heterogeneity in the distribution of chemical elements of the alloy. The most liquating alloy elements are chrome and aluminum. Irradiation of the HEA with a pulsed electron beam promotes the homogenization of the alloy. The highest level of homogeneity in the distribution of chemical elements in the alloy is achieved upon irradiation by a pulsed electron beam with an electron beam energy density of 20 J/cm².

CONCLUSIONS

The performed studies show that irradiation of the HES with a pulsed electron beam (18 keV, 10– 30 J/cm^2 , 50 µs, 3 pulses, 0.3 Hz) leads to the formation of a structure of high-speed cellular crystallization. The cell size varies from 100 to 200 nm. Along the boundaries of the cells, there are nanoscale (15–30 nm) second-phase inclusions enriched with chromium and iron atoms. The HEA obtained using the technology of wire-arc additive production is shown to be characterized by a high degree of heterogeneity in the distribution of chemical elements that form the alloy. The most liquating elements of the alloy are chromium and aluminum. Irradiation of the HES with a pulsed electron beam promotes the homogeni-

zation of the alloy. The irradiation mode (20 J/cm^2 , 50μ s, 3 pulses, 0.3 Hz) that allows the formation of a surface layer, which is characterized by the highest level of homogeneity of the distribution of chemical elements in the alloy, was revealed.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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