Microstructure, Phase Composition and Mechanical Properties of Intermetallic Material Ni-Al-Cr Produced by a Dual-Wire Electron-Beam Additive Manufacturing

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Motivation. Electron beam additive manufacturing is one of the most promising methods for creating complex metal parts and structures. Additive manufacturing has already gained wide acceptance in the creation of various constructions from aluminum, copper, titanium and their alloys, different classes of steels, and other metallic materials. However, there are still many challenges associated with the additive manufacturing and post-production processing of intermetallic alloys. Thus, it is currently an urgent task for research.

The purpose of this work is to study the microstructure, phase composition and mechanical properties of an intermetallic material obtained by two-wire electron beam additive technology using commercial NiCr and Al wires in a 3:1 ratio.

Materials and methods. A workpiece with linear dimensions of 30×35×40 mm³ was manufactured by the two-wire EBAM method using laboratory equipment and software developed at ISPMS SB RAS, Tomsk, Russia. In the process, two wires (1.2 mm diameter) NiCr and Al were used simultaneously with a wire feed rate ratio of 3:1. During the EBAM process, a sequential deposition of the six parallel tracks of materials was performed in parallel to each other in each layer (a total of 40 layers was deposited in each track and the thickness of the single layer was 0.5–0.7 mm). The additive manufacturing process was carried out in a vacuum chamber ($P = 1 \times 10^{-3}$ Pa) with the following technological parameters: accelerating voltage – 30 kV, beam current – 43 mA. An ellipsoidal beam measuring 5 × 5 mm with a scanning frequency of 100 Hz was used. Austenitic stainless steel (Fe – 18%Cr – 8%Ni – 0.1C, wt%) was chosen as the substrate material, and five intermediate layers of NiCr were deposited before the double-wire deposition mode to avoid the influence of the steel composition on the main intermetallic compound. The substrate was not cooled during the deposition of materials.

- The microstructure of the specimens was investigated using an Altami MET 1C light microscope and scanning electron microscope (SEM), equipped with an energy-gy dispersive X-ray spectroscopy (EDS) supply.
- The phase characterization was conducted by X-ray diffraction (XRD) using a DRON 7 diffractometer in the θ-2θ Bragg-Brentano geometry with a Co-Kα radiation.
- A DM8 microhardness tester was used to measure the microhardness by the Vickers method (load on the indenter – 100 gm.).
- Tension tests were carried out at initial strain rate of 5×10^{-4} s^{-1} using an LFM 125 electromechanical testing system with a high-temperature chamber covered the temperature interval 297 – 1273 K.

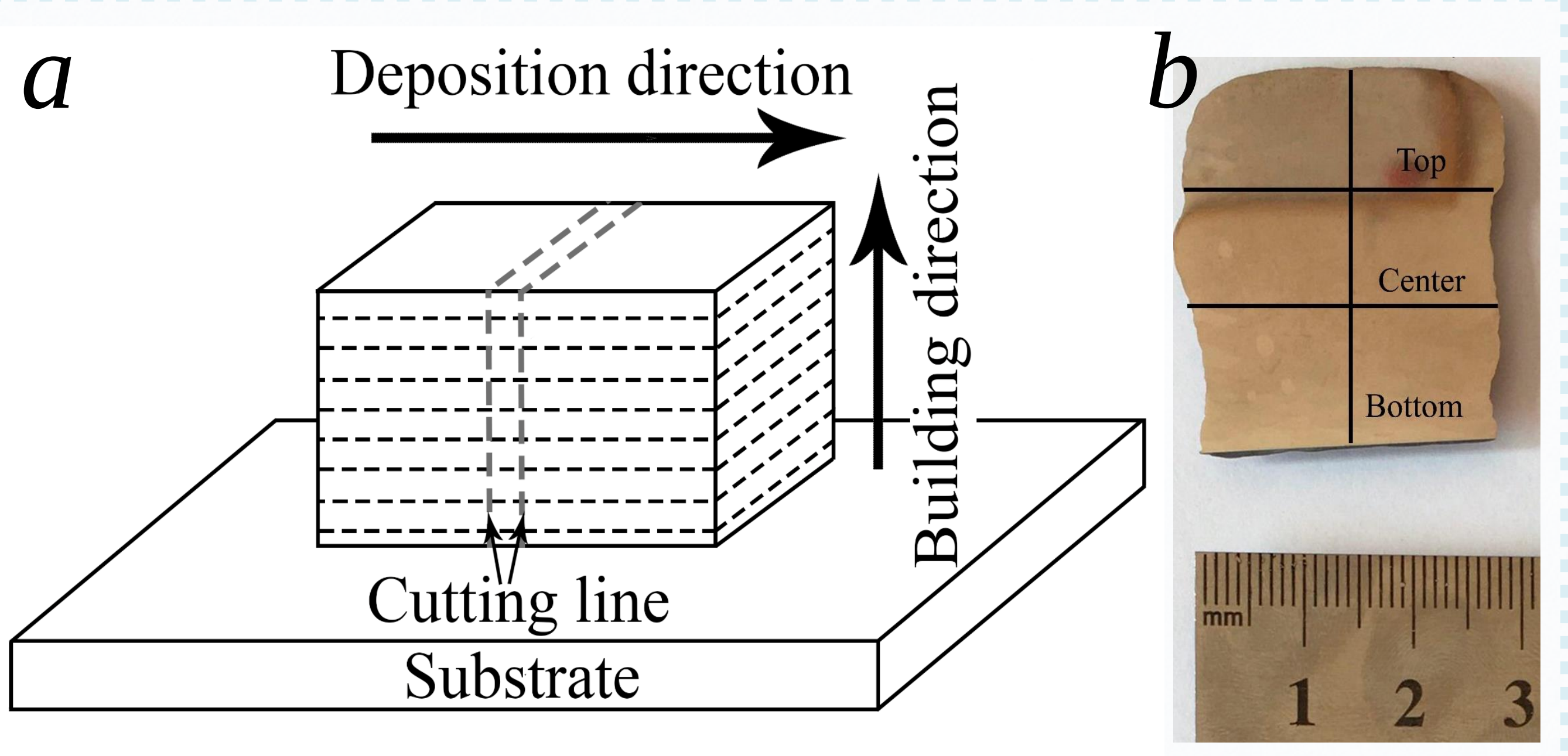

Figure 1. *Schematic representation of the intermetallic preform obtained by EBAM and the position of the sections cut for microstructural analysis (a). Metallographic image of a thin section made of the "3NiCr+Al" alloy (b).*

Results and Discussion. The 3NiCr+Al alloy is characterized by a relatively uniform distribution of the microhardness value along the entire length of the specimen (Fig. 2a). The average value for the 3NiCr+Al alloy is 4.1 GPa, but microhardness slightly grows moving to the top of the billet. X-ray diffraction pattern obtained for the top, central and bottom parts of the EBAM-fabricated specimens (the scheme of cutting is shown in Fig. 1b) are shown in Fig. 2b. The X-ray diffraction peaks with the highest intensity belong to the phases Ni3Cr and Ni3Al. Complex nature of the obtained X-ray diffraction patterns does not allow to calculate the lattice parameters and volume fractions of the identified phases with high accuracy. The $Ni_3Al(Cr)$ intermetallic phase, possessing the ordered L12 structure, is a dominating phase in the 3NiCr+Al alloy. The appearance of a superstructural reflec-tions at small XRD angles are typical of it (for example, (110) Ni_3Al in Fig. 2b). When moving from the bottom to the top of the specimen, the intensity of the su-perstructural reflections decreases and in the top part of the specimens it completely dis-appears. That is, the process of disordering occurs in the upper part of the billet, and dis-ordered $Ni_3Cr(Al)$ phase becomes the dominating one. In addition to the $Ni_3(Al,Cr)$, the equiatomic NiAl and pure Al phases are possible, but cannot be interpreted for sure. The XRD lines of these phases are seen in all three parts of the specimen, and the intensity of the single reflection at 2θ≈52° remains high.

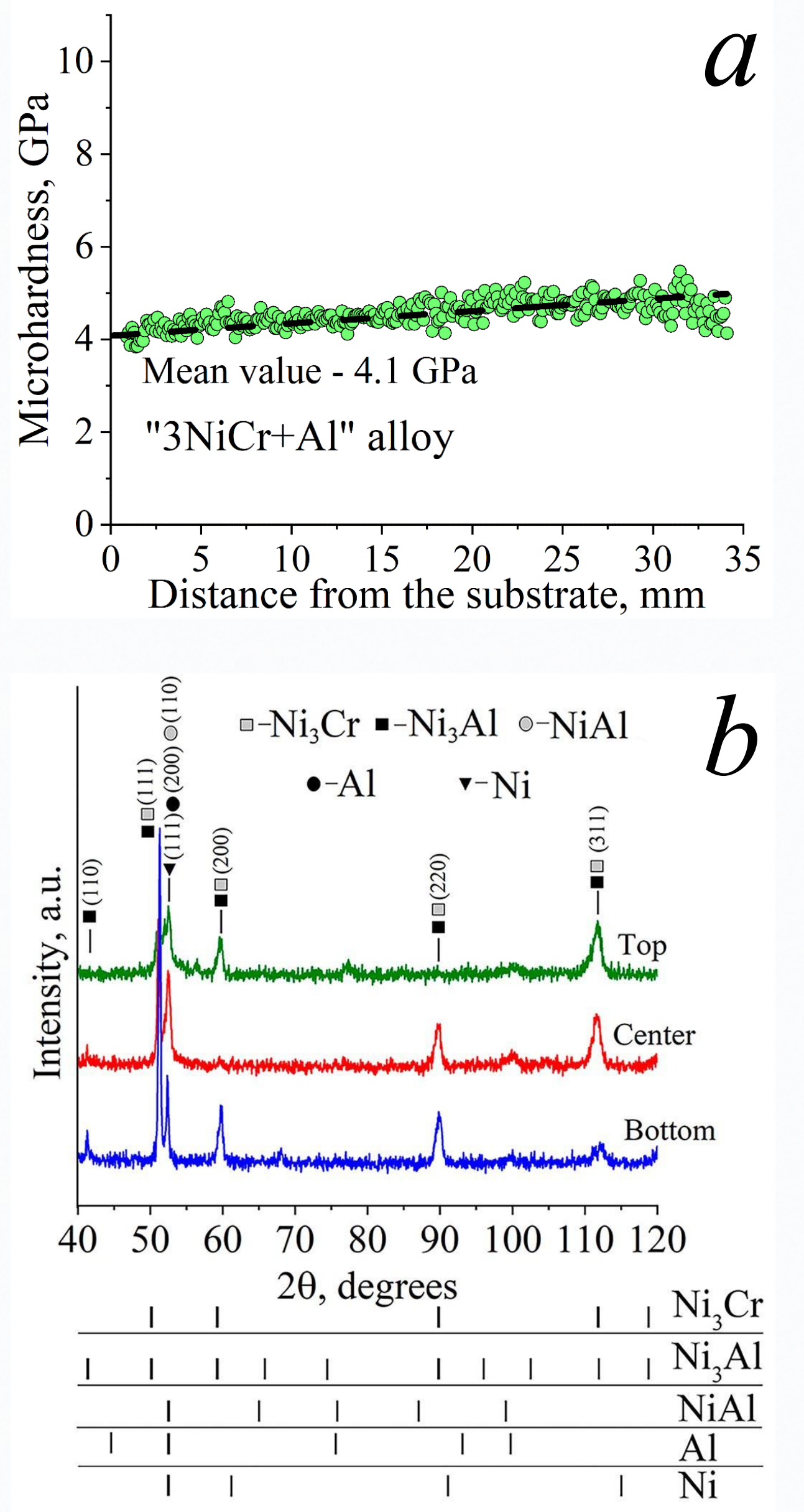

Figure 2. *Microhardness profile (a) and X-ray diffraction pattern (b) of the 3NiCr+Al specimen of the EBAM-fabricated intermetallic materials.*

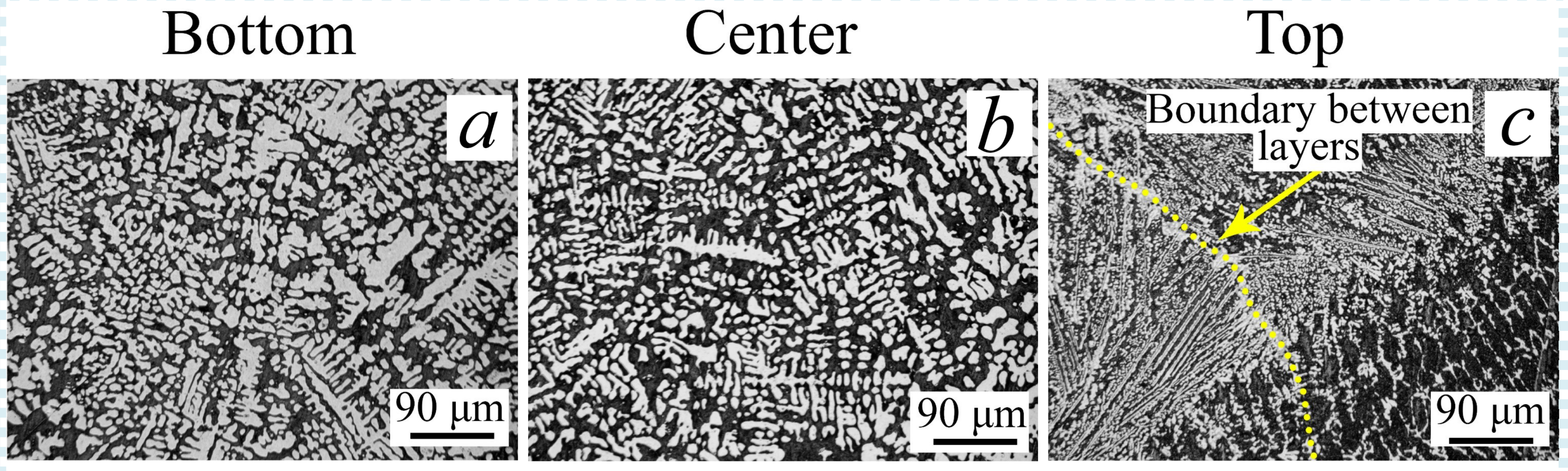

Figure 3. *Microstructure of the 3NiCr+Al specimen in top (a), center (b), and bottom (c) parts of the billet (metallography).*

In the 3NiCr + Al alloy, the microstructure under study is rather homogeneous with dendritic segregations (Fig. 3). The morphology of dendrites is similar for the top, central, and upper parts of the specimen, but the dendritic arms are thinner and longer in the lower part of the billet due to the higher cooling rate at the beginning of the EBAM process.

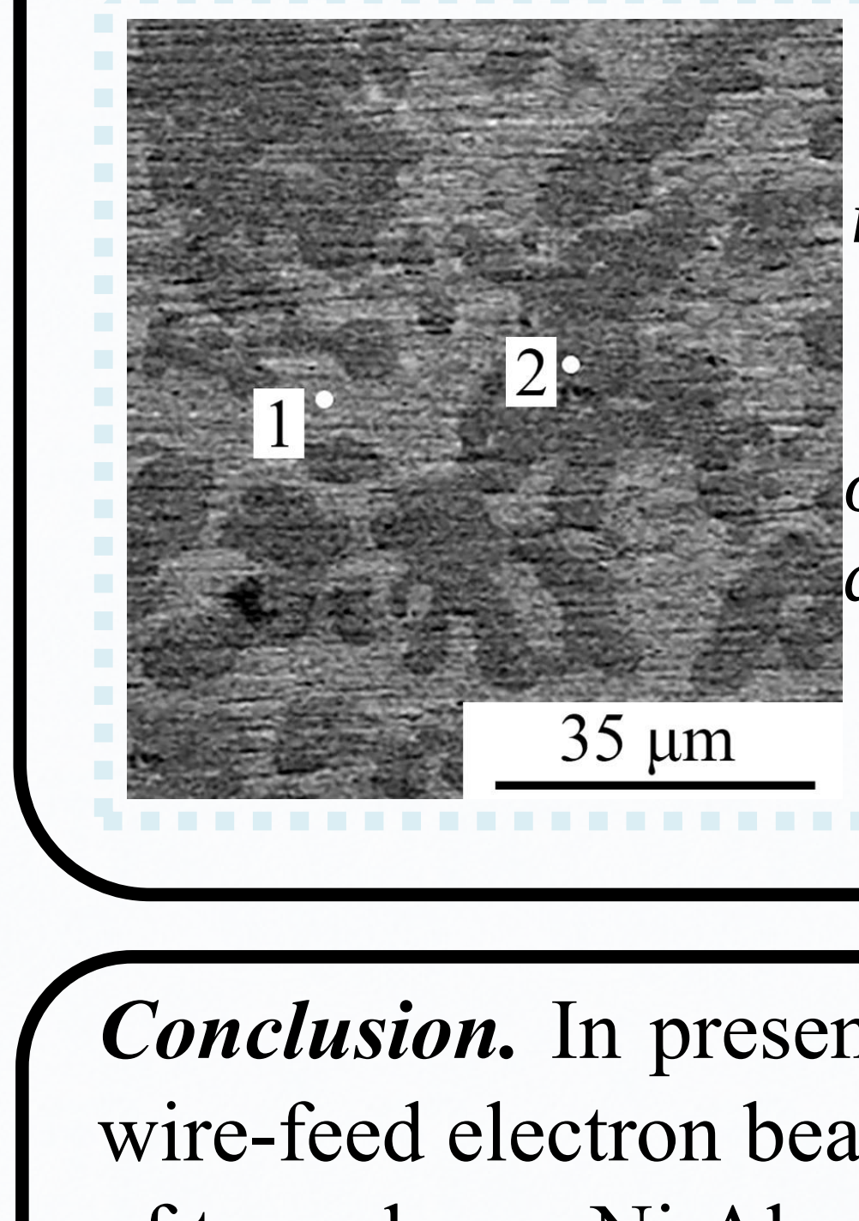

Figure 4. *SEM image of the microstructure of the 3NiCr+Al alloy in the central part of the EBAM-specimen. The numbers on figure indicate points of EDS analysis (elemental composition is presented in Table 1).*

According to the EDS analysis, the dendritic phase is enriched by Ni and Al (Fig. 4, Tab. 1) and can be in both the $Ni_3(Al,Cr)$ or NiAl phases assuming the Al content is overestimated in the EDS analysis. The detected phases in the interdendritic regions are most likely intermetallic Ni_3Cr-based phases or mixtures of $Ni(Cr,Al) + Ni_3(Cr,Al)$. Some of the Al and Cr atoms are substituted by Cr and Al, respectively, in the Ni_3Cr-based phases. The SEM EDS data correlate well with the results of the X-ray diffraction analysis of the alloy (Fig. 2b and Fig. 4, Tab. 1). So, the 3NiCr + Al specimen is an intermetallic alloy with a complex composition based on the Ni_3Al phase. The microhardness of the 3NiCr + Al alloy is slightly above 4GPa, which is very close to the values reported previously for the bulk Ni_3Al and NiAl alloys: about 450 $HV_{0.3}$ for the NiAl + Ni_3Al alloy and about 300 $HV_{0.3}$ for the γ-Ni+ γ'-Ni_3Al one.

Table 1. *EDS data on the elemental composition of the EBAM-fabricated intermetallic alloys and phase composition of the 3NiCr + Al alloys according to the EDS and X-ray analysis.*

Spectrum Number	Ni, at. %	Cr, at. %	Al, at. %	Predicted Phase (EDS)	Predicted Phase (XRD)
Spectrum 1	61.4	25.9	12.7	$Ni_3(Cr, Al)$ or $Ni(Cr,Al)+Ni_3(Cr, Al)$*	Ni_3Al, Ni_3Cr (Ni, NiAl-?)
Spectrum 2	60.5	10.4	29.1	$Ni_3(Al,Cr)$ or NiAl*	

*Assuming that the aluminum content is overestimated and moving to the right in the binary phase diagram Ni-Al

As the alloy is macroscopically homogeneous, the microhardness changes insignificantly along the as-fabricated billet, and the scattering of the values is low. As microstructure changes insignificantly along the AM-fabricated billet, this change in microhardness is associated with the variation in the elemental composition of the main phase. Moving to the top of the 3NiCr + Al specimen, the fraction of the ordered phase $Ni_3(Al,Cr)$ decreases, and contrarily, the fraction of the disordered $Ni_3Cr(Al)$ phase increases. Two phases, $Ni_3(Al,Cr)$ and $Ni_3Cr(Al)$, are Ni_3Al-based phases, which are slightly different in elemental composition (in one of them the concentration of chromium prevails and the concentration of aluminum is higher in the other), but the $Ni_3(Al,Cr)$ is the ordered phase. Due to the similar nature of both phases, the scattering of the microhardness is low, and the microhardness variation along the billet height is insignificant but visible.

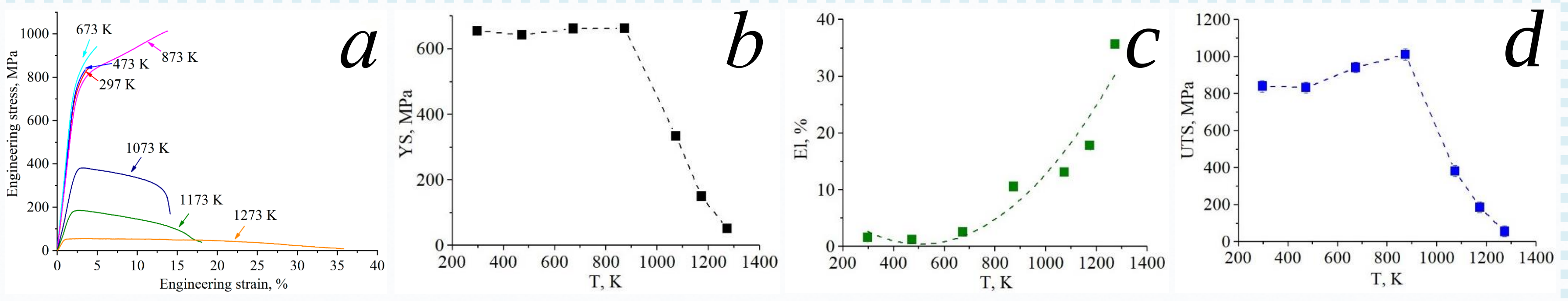

Figure 5. *Tensile "engineering stress-engineering strain" diagrams (a), temperature dependence of the yield strength (YS) (b), elongation-to-failure (EL) (c) and ultimate tensile stress (UTS) (d) of the 3NiCr+Al EBAM-fabricated alloy.*

Fig. 5 show the tensile diagrams in engineering coordinates and the tem-perature dependencies of the main mechanical properties of the 3NiCr+Al alloy. The plasticity of the alloy is limited at low temperature deformation regime, but at temperatures higher 673K specimens show good elongations (Fig. 5b). The best combination of strength and plasticity is achieved at the temperature 873K (Fig. 5). The value of the yield strength (YS) changes slightly (about 650 MPa) in the temperature range from 297 K to 873 K (Fig. 5b). High mechanical properties at T ≤ 873 K is associated with a solid-solution strengthening of the γ'-Ni_3Al phase by chromium.

The mechanisms for the anomalous plastic behavior of Ll_2 compounds like Ni_3Al is based on the role of dislocation core configurations (superdislocations in ordered crystal structure) and operating slip systems. Further increase in test temperature to 1273 K is accompanied with a normal temperature dependence of the YS, so YS is just 52 MPa at T = 1273 K (Fig. 5b). The temperature dependence of the ultimate tensile stress (UTS) demonstrates a similar to the YS behavior: the 3NiCr+Al alloy is characterized by high strength properties in the temperature range from 297 K to 873 K (Fig. 5d). Therefore, additively-fabricated intermetallic compound shows perspective mechanical properties for the high-temperature applications.

Conclusion. In present work we investigated the microstructure, phase composition, microhard-ness, and mechanical properties of the intermetallic material fabricated using the wire-feed electron beam additive manufacturing with a deposition of industrial NiCr and Al wires 3:1. 3NiCr+Al alloy possesses homogeneous dendritic microstructure on the base of two phases Ni_3Al and Ni_3Cr. The microhardness of the 3NiCr+Al alloy is slightly above 4 GPa, which is two times lower than that for the NiCr+3Al aluminum matrix composite material. Despite this, the 3NiCr+Al alloy show good combination of the tensile properties, with the best combination of strength and plasticity at temperature 873 K (650 MPa, more than 10% elongation). The anomalous temperature dependence of the yield strength was observed for this alloy in the temperature interval (300-873) K, which is the significant property of the plastic deformation of intermetallic compounds.

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