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X-ray Diffraction and EBSD Study of Al–Ti–Co–Ni–Fe High-Entropy Alloy

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$Al_{15}Ti_{20}Co_{20}Ni_{25}Fe_{20}$ alloy, synthesized in an arc furnace, was characterized by microscopic investigations, X-ray diffraction and electron backscatter diffraction techniques. Theoretical assumptions indicate a dual-phase crystal structure fcc+bcc. It was shown that dendrites and eutectic are bcc phase, and the interdendritic region is fcc phase.

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1. Introduction

High entropy alloys are described in the literature as alloys which are composed of at least five elements (5 to 35 atomic percent each) [1]. These alloys, in contrast to the conventional, have properties that do not result directly from the synergistic interaction of individual elements [2]. Each element can be located in any lattice position with equal probability, furthermore the simple crystallographic structures like fcc, bcc, or duplex of these are created instead of complex structures [3, 4]. The elements Al, Ti, Co, Ni, and Fe were chosen because of their ability of mutual substitution. Metallic diameters of cobalt, iron and nickel are similar and close to 2.5 Å, while for titanium and aluminium they are close to 2.8 Å [5]. Elements of similar metallic diameters can create solid solutions. Differences in metallic diameter and the ability of mutual substitution indicate a lattice distortion effect which is one of four core effects characteristic of high entropy alloys [6]. Crystallographic structure can be simply predicted by valence electron concentration (VEC), which can be calculated using Eq. (1) [7, 8]. Values lower than 6.87 mean that the alloy has the bcc structure, values higher than 8.00 mean that alloy has the fcc structure. Between the aforementioned values there is a duplex range [9]:

$$VEC = \sum c_i (VEC_i), \qquad (1)$$

c_i — atomic fraction.

2. Experimental procedure

Investigated alloy was synthesized in an arc furnace (Arc Melter AM Edmund Bühler GmbH) in a Ti-gettered high-purity (purity >99 wt%) argon atmosphere from metals into an ingot on a water cooled copper mold. Nominal chemical composition is presented in Table I. The ingot was at least 5 times re-melted to obtain uniform chemical composition. Part of the ingot (centre) was selected to make a cross-section. Specimen was etched in aqua regia in alcohol.

TABLE I

Nominal chemical composition of investigated alloy [at.%].

Al	Ti	Co	Ni	Fe
15	20	20	25	20

Obtained sample was observed using scanning electron microscopy (SEM) (FEI Versa 3D; accelerating voltage 20.00 kV) and analysed by energy-dispersive X-ray spectroscopy (EDS; accelerating voltage 20.00 kV) to investigate distribution of components in the alloy and electron backscatter diffraction (EBSD) point analysis to identify crystal structure of components of the microstructure. Material was also analysed using X-ray diffraction (XRD) (Panalytical Empyrean; Co K_α 1.7903 Å) to identify alloy phases. Using Eq. (1), VEC value was calculated: 7.15. This implies that the alloy could be in duplex phase fcc + bcc.

3. Results and discussion

Figure 1 presents microstructure of investigated alloy with marked dendrites, interdendritic regions and eutectic. Interdendritic regions constitute the large volume fraction of the microstructure, while the dendrites do not form a continuous structure. Individual parts of the dendrites are separated from each other by interdendritic regions and the eutectic. Presence of eutectic phase suggests near eutectic chemical composition of the alloy.

The distribution of individual elements was examined by EDS analysis (Fig. 2). Obtained pictures show that

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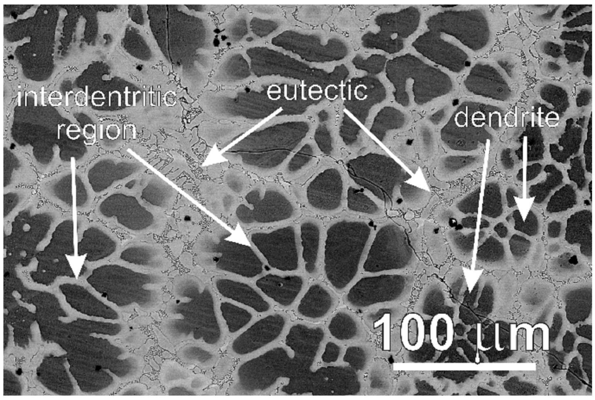

Fig. 1. SEM image of investigated alloy in as-cast state with marked dendrites, interdendritic regions and eutectic.

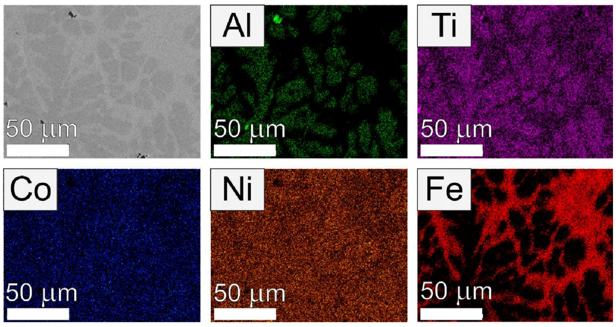

Fig. 2. EDS map of element distribution in the alloy.

Al and Ti enriched the dendrites while Fe enriched the interdendritic regions. Cobalt and nickel are uniformly distributed in the alloy. Aluminum and titanium function as bcc formers, while cobalt, nickel and iron as fcc formers. According to EDS analysis dendrites should have the bcc structure, while interdendritic regions the fcc structure. In comparison with EBSD and XRD analysis the conclusions could be proved.

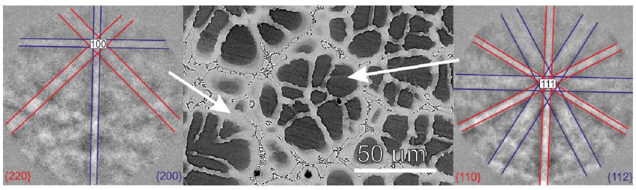

Fig. 3. EBSD point analysis of investigated alloy.

EBSD point analysis for dendrites and interdendritic regions are presented in Fig. 3. Point analysis revealed that dendrites have the bcc structure, while interdendritic regions have the fcc structure. Complementary to EBSD studies, XRD (Fig. 4) shows that in microstructure two bcc-like phases $Al_{0.5}NiTi_{0.5}$ and AlFeNi exist. According to EDS and XRD studies it could be concluded that dendrites are represented by bcc-like phases and the interdendritic regions are solid solution of Fe, Ni, and Co. Both of bcc-like phases were assigned to the dendrites because of the similar crystal structures and possibility of mutual substitution of the elements in such complex materials like HEA. Eutectic phase is a AlFeNi-like phase. Also by XRD analysis Al–Ni martensite was identified, which was not observed by SEM analysis, therefore it can be suggested that its volume fraction was minor. High intensity of eutectic phase (XRD) suggests its high volume fraction in the microstructure.

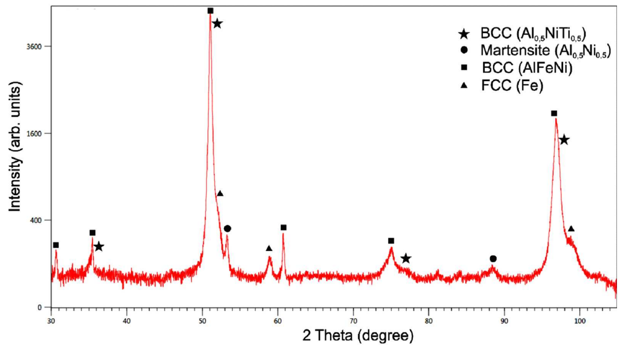

Fig. 4. XRD pattern of the investigated alloy.

4. Conclusion

According to theoretical assumptions (VEC parameter) the synthesized alloy was supposed to be dual phase structure fcc and bcc. By X-ray diffraction and EBSD point analysis this conjecture was confirmed.

EDS analysis shows that the dendritic regions are mainly composed by Al and Ti, the interdendritic regions of Fe while Co and Ni are uniformly distributed in the alloy. From X-ray diffraction and EBDS point analysis it can be concluded that the dendritic regions are bcc-like phases, interdendritic regions (fcc) are solid solution of cobalt and nickel in iron while eutectic (bcc) is AlFeNi-like phase. X-ray diffraction reveals Al–Ni martensite phase in the investigated alloy, however presence of that phase was not confirmed by microscopic observation.

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