

Structure and mechanical properties of B2 ordered refractory AlNbTiVZr_x (x = 0–1.5) high-entropy alloys



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ABSTRACT

Structure and mechanical properties of the AlNbTiVZr_x (x = 0; 0.1; 0.25; 0.5; 1; 1.5) refractory high-entropy alloys were investigated after arc melting and annealing at 1200 °C for 24 h. The AlNbTiV alloy had a B2 ordered single phase structure. Alloying with Zr resulted in (i) change of the degree of order of the B2 phase; and (ii) precipitation of the Zr₅Al₃ and C14 Laves ZrAlV phases. The density of the AlNbTiVZr_x alloys varied from 5590 kg m⁻³ for the AlNbTiV alloy to 5870 kg m⁻³ for the AlNbTiVZr_{1.5} alloy. The compression yield strength at 22 °C increased with an increase in the Zr content from 1000 MPa for the AlNbTiV alloy to 1535 MPa for the AlNbTiVZr_{1.5} alloy. The plasticity raised from 6% for the AlNbTiV alloy to > 50% for the AlNbTiVZr_{0.5} alloy and then dropped to 0.4% for the AlNbTiVZr_{1.5} alloy. At 600 °C, the strongest alloy was also the AlNbTiVZr_{1.5}, whereas, at 800 °C, the AlNbTiVZr_{0.1} alloy demonstrated the maximum strength. The plasticity of the AlNbTiV alloy at 600 °C increased up to 14.3%, while the Zr-containing alloys had lower plasticity. At 800 °C, all the AlNbTiVZr_x alloys could be plastically deformed up to 50% of strain without fracture. Ordering in the alloys and the reasons of a complicated dependence of mechanical properties of the AlNbTiVZr_x alloys on the Zr content and temperature were discussed.

1. Introduction

So-called high-entropy alloys (HEAs) provide a new tempting approach to creation and elaboration of materials with outstanding properties [1–3]. According to the original definition [1], HEAs composed of 5 or more principal elements taken into equiatomic or nearly equiatomic proportions. However, alloys with four constitutive elements can also be considered as HEAs. [4–9]. Conversion from a single-basic-element approach to the multi-component one allows the development of new alloys with highly promising properties for various potential applications [5,7,10,11]. For example, refractory HEAs introduced by Senkov et al. in 2010 demonstrated very high strength up to 1600 °C [5]. However, the density of these HEAs (> 10 g/cm³) is noticeably higher than that of commercial superalloys. The necessity to decrease the density of these alloys served as a trigger for the developing new light-weight refractory HEAs [6–8,12–16].

Obvious approaches to develop refractory HEAs with low weight are: (i) using elements with high melting points and relatively low density (like Cr, Nb, Ti, V, Zr) and (ii) adding light-weight metals such as Al. Stepanov et al. [8] introduced an equiatomic single phase AlNbTiV alloy with the density of 5.59 g/cm³. This alloy exhibited

surprisingly high specific strength up to 800 °C but poor plasticity at room temperature. Aluminum was thought to be responsible for the properties demonstrated by the AlNbTiV alloy [8]. Addition of Cr to the AlNbTiV alloy has resulted in the formation of Laves phase particles and further increase in the strength at room and elevated temperatures [13]. However, room temperature ductility of the AlCr_xNbTiV alloys decreased pronouncedly with an increase in the Cr content. In contrast, alloying the AlNbTiV alloy with 0.5 M fraction of Zr provided a drastic increase in the room-temperature plasticity [14]. This is an interesting finding since the alloy possesses good plasticity despite a relatively high amount (~15 vol%) of intermetallic phases. The reasons for a considerable growth in plasticity of the AlNbTiV alloy at ambient temperature in the presence of Zr were not clarified so far. In the current study an effort to elucidate this issue was made. For that purpose, structure of the AlNbTiVZr_x alloys with different Zr content was analyzed and the effect of Zr on mechanical properties of the AlNbTiV alloy was evaluated.

2. Experimental procedures

The alloys with a nominal composition of AlNbTiV, AlNbTiVZr_{0.1},

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AlNbTiVZr_{0.25}, AlNbTiVZr_{0.5}, AlNbTiVZr, and AlNbTiVZr_{1.5} were produced by arc melting of the elements in a low-pressure, high-purity argon atmosphere inside a water-cooled copper cavity. The purities of the alloying elements were no less than 99.9 at%. The produced ingots of the alloys measured $\sim 6 \times 12 \times 40$ mm³. As-cast ingots were annealed at 1200 °C for 24 h. Prior to the annealing, the samples were encapsulated in vacuumed (10^{-2} Torr) quartz tubes filled with titanium chips to prevent any oxidation.

Microstructure and phase composition of the alloys after annealing at 1200 °C were studied using X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The XRD analysis was performed using RIGAKU diffractometer and CuK α radiation. The obtained XRD data were also used for calculation of a long-range order parameter (LROP) of the AlNbTiVZr_x alloys in accordance with the following equation:

$$LROP = (I_{sl}/I_{fund})(Lp_{fund}/Lp_{sl})(F_{fund}/F_{sl}), \quad (1)$$

where I_{sl} and I_{fund} are the intensities of the superlattice reflection and the fundamental reflection peaks from the alloy, respectively; Lp_{sl} and Lp_{fund} are parameters which include the Lorentz and the polarization factors and the geometry of the X-ray diffraction recording:

$$Lp_{fund} = (1 - \cos^2 2\theta_{fund}) / (\sin 2\theta_{fund} \sin \theta_{fund});$$

$$Lp_{sl} = (1 - \cos^2 2\theta_{sl}) / (\sin 2\theta_{sl} \sin \theta_{sl}), \quad (2)$$

where θ_{sl} and θ_{fund} are the Bragg angles for the fundamental and superlattice reflections, respectively; and F_{fund} and F_{sl} are the structure factors for the fundamental and superlattice lines, respectively. For the stoichiometric (50:50) composition of a B2 phase, the structure factors for the fundamental and superlattice lines are calculated as follows:

$$F_{fund} = f_{A,fund} + f_{B,fund}; F_{sl} = f_{A,sl} - f_{B,sl}, \quad (3)$$

where f_A and f_B are the atomic scattering factors for A and B elements, respectively. In the case of AlNbTiVZr_x alloys, it is impossible to define a stoichiometric composition of the B2 phase due to the simultaneous presence of a variety of constitutive elements. Therefore, based on a number of earlier studies [17–25] about the site occupations of alloying elements in ordered Ti alloys, the Ti sublattice (i.e. A sites) was assumed to be occupied with Ti, Nb and Zr, while Al and V enter into the Al sublattice (i.e. B sites). The final formula for the calculation of the LROP of the B2 phase in the AlNbTiVZr_x alloys can be written as:

$$LROP = (I_{sl}/I_{fund})(Lp_{fund}/Lp_{sl}) \left(\frac{(f_{A,fund}^* + f_{B,fund}^*)^2}{(f_{A,sl}^* - f_{B,sl}^*)^2} \right), \quad (4)$$

Where $f_A^* = f_{Ti}(c_{Ti}/(c_{Ti} + c_{Nb})) + f_{Nb}(c_{Nb}/(c_{Ti} + c_{Nb})) + f_{Zr}(c_{Zr}/(c_{Ti} + c_{Zr}))$; $f_B^* = f_{Al}(c_{Al}/(c_{Al} + c_V)) + f_V(c_V/(c_{Al} + c_V))$; c_{Ti} , c_{Nb} , c_{Zr} , c_{Al} and c_V are the concentrations of the constitutive elements in the matrix phase, taken from Table 2.

Samples for SEM observations were prepared by mechanical polishing. SEM investigations were carried out using either FEI Quanta 600 FEG or Nova NanoSEM microscopes; both instruments were equipped with energy-dispersive (EDS) and electron backscattered diffraction (EBSD) detectors. The volume fraction of different phases was measured by a Digimizer Image Analysis Software using SEM-BSE images. Mechanically pre-thinned to 100 μ m foils were prepared for TEM analysis by conventional twinjet electro-polishing at temperature of -35 °C and an applied voltage of 29.5 V in a mixture of 600 ml of methanol, 360 ml of butanol and 60 ml of perchloric acid. TEM investigations were performed using a JEOL JEM-2100 apparatus equipped with an EDS detector at an accelerating voltage of 200 kV.

The density of the annealed alloys was measured by hydrostatic weighting method. The density was measured on compressive specimens (see below), 3 samples of each alloy were measured.

Isothermal compression of rectangular specimens measured $6 \times 4 \times 4$ mm³ was carried out at 22 °C, 600 °C or 800 °C using an Instron

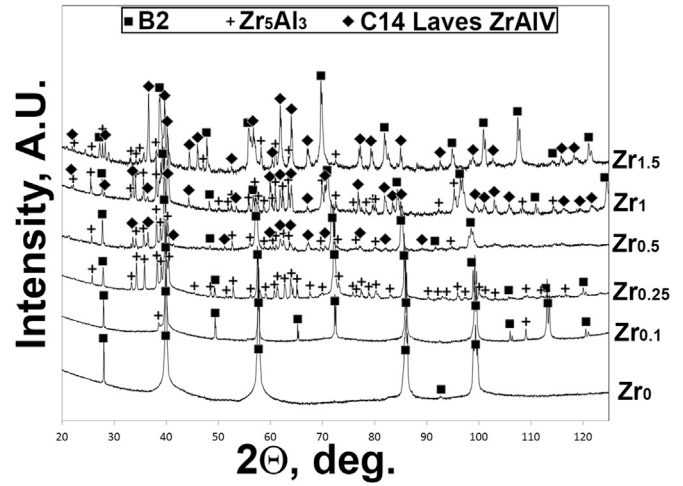


Fig. 1. XRD patterns of the AlNbTiVZr_x ($x = 0; 0.1; 0.25; 0.5; 1; 1.5$) alloys after annealing at 1200 °C for 24 h.

300LX test machine equipped with a radial furnace. The specimens were placed into the preheated to testing temperatures furnace and held for ≈ 10 min to equilibrate the temperature prior to testing. The temperature of the specimen was controlled by a thermocouple attached to a side surface of the specimen. The initial strain rate was 10^{-4} s⁻¹. Three samples of each alloy were tested at each temperature to obtain representative stress-strain curves.

3. Results

3.1. Structure of the AlNbTiVZr_x ($x = 0; 0.1; 0.25; 0.5; 1; 1.5$) alloys

Fig. 1 illustrates the XRD patterns of the AlNbTiVZr_x alloys after annealing at 1200 °C for 24 h. Table 1 summarizes data on some crystal lattice parameters of different phases. The microstructure of the AlNbTiV alloy composed of a single phase ordered B2 phase with the lattice parameters of $a_{B2} = 0.3186 \pm 0.0001$ nm (Fig. 1, Table 1). The AlNbTiVZr_{0.1} and AlNbTiVZr_{0.25} alloys consisted of the B2 and hexagonal Zr₅Al₃-type [26] phases (Fig. 1). The alloys with higher Zr content, namely the AlNbTiVZr_{0.5}, AlNbTiVZr and AlNbTiVZr_{1.5} alloys, were composed of the B2 phase, Zr₅Al₃-type phase and hexagonal C14 Laves phase of ZrAlV-type [27] (Fig. 1). The intensity of the Bragg peaks associated with the Laves phase of ZrAlV-type gradually increased with an increase in the Zr content (Fig. 1). The lattice parameter of the B2 phase demonstrated a gradual increase with an increasing in the Zr content (Table 1).

Values of the LROP as a function of the Zr content obtained for the matrix B2 phase of the AlNbTiVZr_x alloys are shown in Fig. 2. With an increase in the Zr percentage the LROP decreased pronouncedly from 0.71 for the Zr-free AlNbTiV alloy to 0.39 for the AlNbTiVZr_{0.5} alloy. Further increase in the Zr content resulted in rather weak reduction of the LROP to 0.38 and 0.37 for the AlNbTiVZr and AlNbTiVZr_{1.5} alloys, respectively.

Fig. 3 presents microstructures of the AlNbTiVZr_x ($x = 0; 0.1; 0.25; 0.5; 1; 1.5$) alloys after annealing at 1200 °C for 24 h; data on the chemical compositions, volume fractions and dimensions of the structural constituents are tabulated in Table 2. The AlNbTiV alloy had a single phase coarse-grained structure with the average size of ~ 300 μ m and the nominal chemical composition (Fig. 3a and Table 2). TEM investigation (Fig. 3b) has revealed the presence of thermal antiphase domains (APD). A selected area electron diffraction (SAED) pattern had shown the presence of (100) superlattice maxima which suggested the ordering of the B2 phase; this finding was consistent with the XRD data (Fig. 1).

The AlNbTiVZr_{0.1} alloy (Fig. 3c) had a coarse granular structure of

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