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Microstructural origins of high strength and high ductility in an $AlCoCrFeNi_{2.1}$ eutectic high-entropy alloy

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ABSTRACT

Recent studies indicate that eutectic high-entropy alloys can simultaneously possess high strength and high ductility, which have potential applications in industrial fields. Nevertheless, microstructural origins of the excellent strength–ductility combination remain unclear. In this study, an $AlCoCrFeNi_{2.1}$ eutectic high-entropy alloy was prepared with face-centered cubic (FCC)($L1_2$)/body-centered-cubic (BCC)(B2) modulated lamellar structures and a remarkable combination of ultimate tensile strength (1351 MPa) and ductility (15.4%) using the classical casting technique. Post-deformation transmission electron microscopy revealed that the FCC($L1_2$) phase was deformed in a matter of planar dislocation slip, with a slip system of {111} <110>, and stacking faults due to low stacking fault energy. Due to extreme solute drag, high densities of dislocations are distributed homogeneously at {111} slip plane. In the BCC(B2) phase, some dislocations exist on two {110} slip bands. The atom probe tomography analysis revealed a high density of Cr-enriched nano-precipitates, which strengthened the BCC(B2) phase by Orowan mechanisms. Fracture surface observation revealed a ductile fracture in the FCC($L1_2$) phase and a brittle-like fracture in the BCC(B2) lamella. The underlying mechanism for the high strength and high ductility of $AlCoCrFeNi_{2.1}$ eutectic high-entropy alloy was finally analyzed based on the coupling between the ductile FCC($L1_2$) and brittle BCC(B2) phases.

1. Introduction

High-entropy alloys (HEAs), which emerged in 2004, are solid solution multicomponent alloys that contain more than four principal elements in equal or non-equal atomic percentage [1–5]. HEAs are a new kind of alloys because they are different from conventional alloys that have one or two principal elements as main components. HEAs have attracted increasing attention recently due to their unique atomic structure and properties, such as high strength of body-centered-cubic (BCC) AlCoCrFeNi alloy at room temperature and TaNbHfZrTi alloy at elevated temperatures, high ductility of face-centered cubic (FCC) CrMnFeCoNi alloy and transformation-induced plasticity-assisted meta-stable dual-phase $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ alloy, sluggish diffusion, and high thermal stability [6–17].

The review of literature indicates that two kinds of HEAs have been widely investigated in the last 10 years: single-phase FCC HEAs and BCC HEAs. Single-phase FCC HEAs generally have high tensile ductility but low yield strength. Examples are CrMnFeCoNi alloy with an elongation to failure of ~50% and yield strength of ~410 MPa [7], $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ alloy with a tensile ductility of ~58% and low yield strength of ~240 MPa [17], and $Fe_{40}Mn_{26}Ni_{27}Co_5Cr_2$ alloy with a total elongation to failure of ~58% and low yield strength of ~95 MPa at room temperature [18]. However, BCC HEAs have high yield strength but low ductility. For example, [316]-oriented and [001]-oriented $Nb_{25}Mo_{25}Ta_{25}W_{25}$ single crystalline pillars have an extraordinarily high strength level (about 4–4.5 GPa) but low plasticity (less than 0.2%) [19]. Therefore, for the single-phase HEAs, it seems difficult to achieve a balance between high strength and high ductility. In addition, the industrial applications of HEAs are restricted certainly by poor castability,

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liquidity, and composition segregation.

Recently Lu et al. [20] proposed a designation concept of eutectic high-entropy alloys (EHEAs) to combine the high strength of BCC HEAs and high ductility of FCC HEAs and produced an $AlCoCrFeNi_{2.1}$ EHEA with regular FCC($L1_2$)/BCC(B2) lamellar structures and an excellent combination of high strength and high ductility. More recently, the mechanical properties of $AlCoCrFeNi_{2.1}$ EHEA were optimized by thermal mechanical processes, that is, multi-pass cold rolling to a 90% reduction in thickness and subsequently annealed at 800–1200 °C for 1 h [21,22]. As a result, the processed HEAs had tensile ductility more than 10% and high tensile strength greater than 1 GPa. This indicated that the properties of the EHEA could be successfully tailored using thermo-mechanical processing for a wide range of engineering applications. However, the microstructural origins of excellent strength–ductility combination and underlying deformation mechanisms of EHEAs still need systematic investigations.

In this study, transmission electron microscopy (TEM) and atom probe tomography (APT) were used to systematically characterize the microstructural characteristics of post-deformed $AlCoCrFeNi_{2.1}$ EHEA to find answers to the aforementioned problems.

2. Experiments

The master alloy of the eutectic $AlCoCrFeNi_{2.1}$ (elements in atomic ratios) was prepared from commercially pure elements (99.9 wt% for Al, Co, and Ni; 99.5–99.6 wt.% for Cr and Fe) in a ZrO_2 crucible of a vacuum induction melting furnace. The ZrO_2 crucible was first heated to 600 °C and held for 1 h to remove the water vapor. After the elements (approximately 2.5 kg) were put into the furnace, the furnace chamber was evacuated to 0.06 Pa and back-filled with high-purity argon gas to reach 0.06 MPa. The elements were finally melted, superheated, and poured into a high-purity graphite crucible with an inner length of 220 mm, upper inner diameter of 62 mm, and bottom inner diameter of 50 mm. The pouring temperature was set to be 1500 °C. A TRTM-2CK infrared pyrometer was used to monitor the temperature with an absolute accuracy of ± 2 °C. A Walter + bai LFM 20 kN tensile testing machine was used for tensile testing at room temperature with a normal strain rate of 1×10^{-3} s^{-1}. The flat dog bone-shaped tensile samples had a gauge dimension of $20 \times 3 \times 2$ mm^3. The strain was measured using a standard non-contacting video extensometer. Three tensile specimens were measured to obtain reliable results.

Microstructure and composition analyses were carried out by means of x-ray diffraction (XRD), electron back-scattered diffraction (EBSD), TEM, and APT. Specifically, the XRD analyses of crystalline structures were performed on a Bruker D8 with Cu radiation target scanning 2θ from 20 to 80°. EBSD of as-cast specimen was conducted using a high-resolution field emission Carl Zeiss-Auriga-45-66 scanning electron microscope (SEM) equipped with a fully automatic Oxford Instruments Aztec 2.0 EBSD system (channel 5 software). Before EBSD, the specimens were mechanically polished and then electro-polished in an electrolyte containing 90 vol.% acetic acid and 10 vol.% perchloric acid using a voltage of 50 V and polishing time of 45 s in Buehler electromet-4. TEM observations were conducted in a FEI-Tecnai G^2 20 S-TWIN microscope operated at 200 kV, and the high-resolution TEM (HRTEM) was conducted on a Titan G2 60-300. TEM specimens were prepared as follows: the tensile deformed gauge parts were carefully ground to foils with a thickness of about 50 μm, punched into disks with a diameter of 3 mm, and finally electro-polished to an electron-transparent thickness in an aqueous electrolyte containing 10% perchloric acid and 90% ethanol at −25 °C using a twin-jet polishing system. The HRTEM specimen was prepared by means of ion milling on a GATAN 691 to avoid the different electro-polishing rates of the two eutectic phases.

The APT specimen was prepared by electro-polishing combined with a focus iron beam (FIB) cutting along the phase interface. A low-energy (5 keV) Ga beam was used for final ion milling to minimize beam damage. The APT experiment was performed with a local electrode atom probe (LEAP 4000X Si) under ultraviolet laser pulsing at a laser energy of 40 pJ, a pulse repetition rate of 200 kHz, and a target evaporation rate of 0.5% per pulse at 25 K. APT data reconstruction and quantitative analysis were performed using a CAMECA visualization and analysis software (IVAS) 3.6.8.

3. Results

3.1. Initial structures and tensile testing results

Fig. 1a shows the XRD pattern of the as-cast $AlCoCrFeNi_{2.1}$ alloy. The as-cast $AlCoCrFeNi_{2.1}$ alloy consisted of FCC($L1_2$) and BCC(B2) dual-phase. The $L1_2$ and B2 structures, which were further confirmed by subsequent TEM-SAEDs (select area electron-beam diffraction patterns), were consistent with the results reported in the literature [21,22]. Fig. 1b shows the large-area EBSD phase mapping of typical FCC($L1_2$)/BCC(B2) lamellar microstructures. Fine BCC(B2) lamellae (about 2 μm thick, yellow color) were parallel to each other and distributed in the FCC($L1_2$) phase (cyan color). In addition, some coarse BCC(B2) lamellae and islands also appeared. The volume fraction of FCC($L1_2$) and BCC(B2) phases was about 66.3% and 31.2%, respectively. Moreover, some amounts of high-angle grain boundaries (marked in red) were formed during casting.

Tensile testing showed that the as-cast $AlCoCrFeNi_{2.1}$ alloy possessed an excellent combination of high strength and high ductility owing to the uniform FCC($L1_2$)/BCC(B2) lamellar microstructures. Fig. 2 shows the tensile engineering and true stress–-strain curves of the as-cast $AlCoCrFeNi_{2.1}$ alloy. It is evident that the ultimate tensile stress was 1100 ± 50 MPa and the ductility was 18 ± 2%. When the engineering stress–strain curve (blue line) was converted into a true stress–strain curve (red line), the ultimate tensile stress and ductility were 1351 MPa and 15.4%, respectively.

3.2. Deformation mechanisms via TEM characterizations

3.2.1. Deformation of FCC($L1_2$) phase

TEM observations were performed to reveal the deformation mechanisms and understand the mechanical properties. Fig. 3a shows a representative bright-field TEM micrograph of the tensile tested $AlCoCrFeNi_{2.1}$ EHEA. Distinct modulated FCC($L1_2$) and BCC(B2) lamellar structures were obviously observed based on different contrasts (bright and dark, respectively) in the TEM image. This was because the FCC($L1_2$) phase was thinner than the BCC(B2) phase after twin-jet polishing. This chemical polishing result also suggested that the corrosion resistance of BCC(B2) phase was better than that of FCC($L1_2$) phase. Fig. 3b and c show the TEM-SAEDs of FCC($L1_2$) and BCC(B2) phases, respectively. The presence of superlattice spots obtained from the two phases (marked by cyan and yellow circles, respectively) revealed both ordered $L1_2$ (Fig. 3b) and B2 (Fig. 3c) phases. The formation of ordered $L1_2$ structure is rarely reported in eutectic alloys in the literature, whereas the formation of ordered B2 phase is frequently reported owing to the strong negative enthalpy in the Al–Ni system [23–25]. The compositions of the present B2 phase were revealed as ~38 at.% Ni and ~37 at.% Al by the following APT analysis (see Table 1), which hinted that Ni and Al atoms alternately occupied the lattice sites in the B2 unit cell, and other element atoms were substitutional solution atoms [26–28].

The deformation mechanisms of FCC($L1_2$) and BCC(B2) phases