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Strengthening in $Al_{0.25}CoCrFeNi$ high-entropy alloys by cold rolling

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ABSTRACT

The effects of cold rolling on the evolution of microstructure and the mechanical behavior of $Al_{0.25}CoCrFeNi$ high-entropy alloys were investigated. Cold rolling resulted in extensive grain elongation, formation of deformation bands within the grains, and development of crystallographic textures that depended on the rolling reduction. The textures were investigated by electron backscattered diffraction after cold rolling. The present cold-rolled alloy indicates a strong brass-type texture. Cold rolling results in a substantial strengthening of the alloy; its ultimate tensile strength approaches 1479 MPa, which was 2.8 times of that in the as-cast condition, but at the expense of low ductility ($\varepsilon \sim 2.3\%$).

1. Introduction

High-entropy alloys (HEAs) or multiprincipal component alloys are defined as solid solution alloys that contain five or more principal elements in equal or near equal atomic percent (at%) [1,2]. Despite the presence of a large number of components, HEAs often show rather simple crystal structures such as face centered cubic (FCC e.g. equiatomic CoCrFeMnNi) [1], body centered cubic (BCC) and FCC + BCC [2], and hexagonal close packed (HCP e.g. equiatomic HoDyYGdTb) [3,4], instead of many intermetallic phases or other complex phases. The distinctive characteristic of HEAs has been originally attributed to the high configurational entropy associated with the mixing of a large number of constituents stabilizing simple solid solution phases [1].

Recent studies have shown that HEAs possess adjustable and attractive properties compared to the conventional alloys [5,6]. HEAs often exhibit unusual properties: such as high strength/hardness, exceptional high/low temperature strength [7–9], excellent thermal stability [10], and good corrosion [11] and oxidation resistance [12]. The origin of these unique properties in HEAs is explained on the basis of the core effects of multicomponent solid solution formation, including distorted lattice structure [3], cocktail effect [3,5], sluggish diffusion [3,6], and extensive formation of deformation nano-twins [6].

Since high strength and ductility are important for structural materials, research efforts have tended to be application driven and directed towards finding strengthening methods with promising mechanical properties. The strategies of interstitial strengthening [13–15], phase-transformation strengthening [16], precipitation hardening [17,18], and ultrafine-grained strengthening [19,20] were applied to achieve high mechanical performances in HEAs. Furthermore, major research in widening the potential applications of HEAs is using the thermo-mechanical processing (TMP) involving heavy deformation and annealing to refine the microstructure and crystallographic of HEAs [21–23]. Tsai et al. [24] reported the deformed and annealing $Al_{0.5}CoCrCuFeNi$ HEAs and found that nanocrystallines can be obtained by simple rolling to work hardening and strengthening the materials. Cold rolling (CR) is a main technology in improving the strength of HEAs. Wang et al. [25] reported that $Al_{0.25}CoCrFe_{1.25}Ni_{1.25}$ HEAs exhibit {111} type texture after cold rolling, and after CR 80% reduction, the tensile strength was 702 MPa, 1.62 times that in the as cast condition. Senkov [26] firstly reported a BCC type HfNbTaTiZr HEA after cold rolling and annealing, and the tensile stress and ductility of the sheet after CR 86.4% were 1295 MPa and 4.7%, respectively. Wu et al. [20] found that a family of FCC-structured HEAs show a Hall-Petch dependency of microhardness by CR and subsequent annealing. In spite of this, it is noted that most reports on the mechanical properties of HEAs are still focused on the as-cast condition [12]. As commonly known that cast defects, such as pores, are not avoided, which may evolve into the preferred nucleation sites for cracks. Severe plastic deformation (SPD) and/or heat treatment is expected to alleviating these defects to improve the mechanical properties. For the SPD method, the formation of nanograins mainly depends on the accumulation and evolution of lattice defects, which is expected to strengthen

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the alloys. Although the significant role of the CR in strengthening the mechanical properties of HEAs is determined, a systematic investigation of the effect of CR reductions on the microstructures and mechanical properties of HEAs is lacking. It should be noted that CR without subsequent annealing results in very pronounced strengthening at the expense of dramatically reduced ductility.

Here, the objective of this study is to investigate the effect of CR reductions on the microstructures and mechanical properties of $Al_{0.25}$CoCrFeNi HEAs. Additionally, the fracture feature, texture evolution, strengthening mechanisms of the present HEAs are discussed in detail.

2. Materials and methods

Rectangular ingots (85 × 10 × 2 mm^3) with a nominal mole composition of $Al_{0.25}$CoCrFeNi were produced by arc melting and drop casting under the pure Ar atmosphere. The purity of each raw material was at least 99.9 wt%. The ingots were remelted four times in a water-cooled Copper crucible to improve the chemical homogeneity, flipping them over between each melt, and then, the liquid alloys were suctioned into a copper mold.

The plate (sheet) ingots were subsequently cold rolled along the longitudinal direction at room temperature using a rolling equipment having a roll diameter of 140 mm. The thickness reductions of CR were 0% (as-cast), 30%, 50%, 70%, and 90%. Each reduction in thickness was carried out using small incremental deformation in each pass. About 10–20 passes were required to achieve the target reduction in thickness. The hardness values were measured with a Vickers hardness tester under a load 500-g load holding for 15 s. The phase structure and lattice parameters were characterized by X-ray diffraction (XRD) using a PHILIPS APD-10D diffractometer with Cu Kα radiation. The position of each peak was measured on the diffractogram from which the lattice parameter was calculated through Nelson-Riley extrapolation method [27]. Microstructure observations of the surface were carried out by XJP optical microscope (OM) and scanning electron microscope (ZEISS SUPRA55 SEM) equipped with an energy-dispersive spectrometer (EDS). Transmission electron microscopy (TEM) foils were prepared from the surface layer of slices in rolling planes, TEM observations were performed using a Tecnai G20 microscope operating at 200 kV. Electron backscattered diffraction (EBSD) was carried out using a JEOL JSM-7100F scanning electron microscope (SEM) operated at 30 kV and a sample tilt of 70°. The EBSD patterns obtained were indexed, using *fcc* Copper as the base crystal structure, and were analyzed using the commercially available *HKL* Channel 5 software. The samples for EBSD investigations were prepared using careful mechanical polishing followed by electro-polishing using a mixture of acetic acid and perchloric acid (8:2 by volume). Then, the tensile samples are artificially machined into a dog-bone shape, which have a nominal gauge length and width of the tensile specimens were 10 and 2 mm, respectively, by electrical discharge machining. Tensile tests were carried out on samples using Instron5969 materials testing machine at room temperature at a strain rate of 5×10^{-4} s^{-1} along the rolling direction. There are more than 5 samples used for the experiments.

3. Results and discussions

Fig. 1a exhibits the XRD patterns of the alloys in the as-cast and various CR reduction conditions. Only a simple FCC phase was detected in all statuses, suggesting the alloys are mainly composed of a FCC phase regardless of severe plastic deformation. The lattice parameter, **a**, determined from the XRD patterns, increases linearly with increasing CR reduction (see Fig. 1b), and the values are 3.5830, 3.5857, 3.5876, 3.5883, and 3.5899 Å, indicating that the interplanar distance gradually decreasing after CR. According to the linear fit in Fig. 1b, the increase in lattice parameter (Δ**a**) per change in CR reduction Δ**c**, Δ**a**/Δ**c**, is 0.752 pm/1%, which produces a lattice strain or fractional increase in lattice parameter per 1% reduction of CR, Δ**a**/(Δ**c** × **a**) of 0.209/1%. The increase in lattice parameter (Δ**a**) per change in at% carbon (Δ**C**) and Al (Δ**A**), Δ**a**/Δ**C** and Δ**a**/Δ**A**, are 2.74 pm/at% and 0.103 pm/at% in carbon-doped $Fe_{40.4}Ni_{11.3}Mn_{34.8}Al_{7.5}Cr_6$ HEAs [15] and Al_xCoCrFeNi (x = 0.1–0.5) HEAs [28], respectively. The value of Δ**a**/Δ**c** is larger than Δ**a**/Δ**A** but smaller than Δ**a**/Δ**C**, which indicates a larger lattice distortion after CR in current HEAs than substitutional Al strengthening in Al_xCoCrFeNi (x = 0.1–0.5) HEAs. The full width at half maximum (FWHM) of the Bragg diffraction peaks show the grains have been refined. The peak intensity ratio I_{111}/I_{200} varies remarkably during CR, hinting a possible texture formation. Apparently this ratio is much higher in the cold-rolled states than the as-cast case, indicating a possible formation of {111} type texture during CR [18]. The exact texture types of the deformed HEAs were characterized, using an electron backscatter diffraction system.

Fig. 2 shows the microstructural features of the as-cast and cold-rolled statuses $Al_{0.25}$CoCrFeNi HEAs. Fig. 2a exhibits a typical as-cast dendritic structure. The gray region refers to the dendrite matrix, and the black region denotes the remaining interdendrites. The as-cast microstructure consisted of equiaxed dendritic grains and the average size of dendritic grains is about 60 μm (determined by EBSD images in Fig. 7). The chemical compositions of the dendrite, interdendrite and dendritic grain are listed in Table 1. Compared to the nominal composition of the present alloy, elemental segregation in as-cast alloys is very small. The as-cast CoCrFeMnNi HEAs show obvious compositional segregation, i.e., the dendrites are enriched in Cr, Fe and Co, and the interdendrites are enriched in Mn and Ni [29]. The present alloys show very weakly Al-enriched interdendritic regions compared with dendrite regions, which is due to the low melting point of Al, such as the segregation of Mn and Ni in the CoCrFeMnNi HEAs. And the contents of Ni

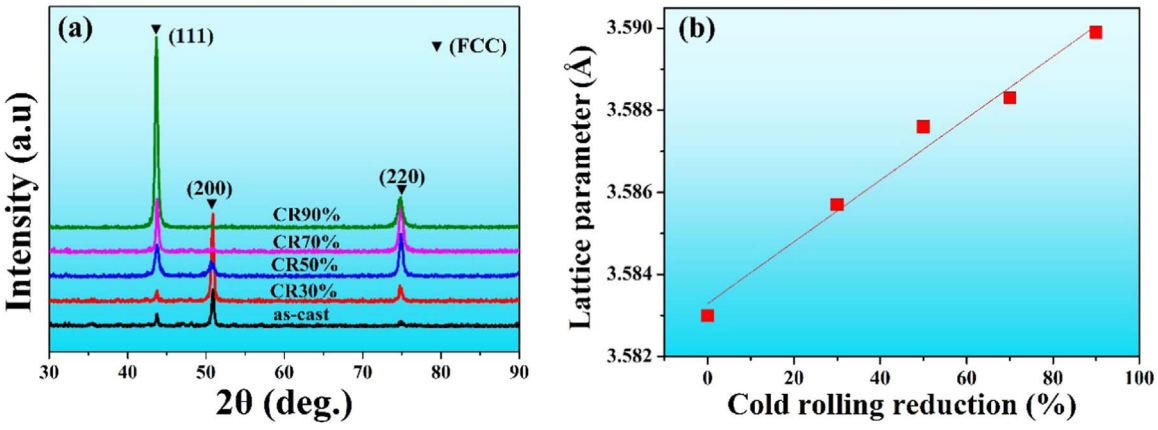

Fig. 1. XRD patterns (a) and lattice parameter (b) versus CR reductions for $Al_{0.25}$CoCrFeNi HEAs.