



# Effect of annealing heat treatment on microstructural evolution and tensile behavior of Al<sub>0.5</sub>CoCrFeMnNi high-entropy alloy

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## ABSTRACT

In this work, the mechanical characteristics and microstructural evolution of Al<sub>0.5</sub>CoCrFeMnNi high-entropy alloy (HEA) were studied after annealing at various temperatures (1000, 1100, and 1200 °C). X-ray diffraction, scanning electron microscopy, and energy dispersive spectroscopy analyses were performed to reveal the phase and microstructural variations. The mechanical properties related to different microstructures of the alloy were characterized using tensile testing with digital image correlation. Annealing at lower temperatures led to a higher fraction of B2 phase and finer grain size of FCC (face-centered cubic) phase. A good combination of strength and ductility in this alloy was attributed to the ductile FCC matrix and hard secondary B2 phase. The alloy showed the active evolution of deformation twinning due to the low stacking fault energy when Al was added to CoCrFeMnNi to make the HEA. However, for alloy annealed at lower temperatures, twinning activity was suppressed by the smaller size of grains and depletion of Al content in the FCC matrix. The correlation between the microstructure and mechanical properties was also explored using a simple composite model.

## 1. Introduction

High-entropy alloys (HEAs) are defined as multi-component alloys of which most contain five or more principal elements in equiatomic or near-equiatomic composition with high configurational entropy. In contrast, most conventional (low-entropy) metallic alloys are composed of one or two principal elements and some minor elements [1,2]. The high configurational entropy of HEAs promotes the formation of simple single-phase crystalline materials of face-centered cubic (FCC) [3], body-centered cubic (BCC) [4,5], or sometimes hexagonal close-packed (HCP) structures [6]. These multicomponent alloys have been attracting considerable attention all over the world [2–6]. This is because many HEAs reportedly exhibit good mechanical properties, excellent corrosion resistance, high thermal stability, and resistance to fatigue [7–10].

In general, the FCC-HEAs are ductile and soft, while the BCC-HEAs have high strength but relatively low tensile ductility [11]. From this point of view, the FCC-HEAs need improved yield strength and the BCC-HEAs need more plasticity. Therefore, there have been several attempts to enhance the mechanical properties of HEAs using secondary phases to reach a reasonable balance between strength and ductility to broaden the applications for HEAs [12–15].

Recent studies showed that addition of Al to FCC-HEAs could have strong effects on the formation of secondary phases resulting in the variation of mechanical properties [16,17]. He et al. [18] studied the alloying effect of Al on the structure and tensile properties with respect to an FCC CoCrFeMnNi HEA. They found that the crystalline structure changed from the initial single FCC structure to an FCC + BCC structure in the range 8–11 at% of Al. This dual phase alloy showed composite behavior with sharp increases in strength and hardness due to the hard BCC phase. However, a certain problem arose as the Al content was increased in the alloy: a large fraction of disordered and ordered (A2 and B2, respectively) BCC phases could lead to poor ductility. Moon et al. [19] investigated cracking phenomenon in Al<sub>0.5</sub>CoCrFeMnNi HEA during cold-rolling. They reported, from the results of microstructural analysis and thermodynamic calculation, that the cracking behavior was induced by the formation of an AlNi-rich B2 phase. They proposed that homogenization heat treatment at high temperature might minimize the poor workability of this alloy by reducing the B2 phase which is known to be brittle in the ingot [11]. However, no studies on the microstructural and mechanical properties of the Al<sub>0.5</sub>CoCrFeMnNi alloy after annealing heat treatment and recrystallization have been reported.

In this study, we explored the microstructural evolution of the

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Al<sub>0.5</sub>CoCrFeMnNi HEA after cold rolling followed by annealing heat treatment. The characterization of tensile behaviors of the annealed alloy was also investigated in parallel. In addition, the strengthening behavior associated with the microstructure was described using a simple composite model to quantify the effect of the annealing heat treatment in the present alloys.

## 2. Experimental procedure

### 2.1. Sample preparation

Al<sub>0.5</sub>CoCrFeMnNi HEA ingots were fabricated using vacuum induction melting (VIM) of the pure elements (purity above 99.9%). The homogenization heat treatment for the cast ingots was carried out at 1200 °C for 6 h to reduce the inhomogeneity of the chemical compositions. This heat treatment also contributed to dissolving the secondary phase and improving their workability for cold rolling in accordance with recent publication [19]. After the homogenization treatment, the ingot was cold rolled for thickness reduction from 7.0 to 1.5 mm (reduction ratio: 78.6%) to break up the initial coarse-grained microstructure.

To control the phase fraction of the Al<sub>0.5</sub>CoCrFeMnNi, annealing heat treatments were performed under various conditions. Annealing heat treatments for the as-rolled samples were conducted at 1000 °C for 15 min, 1100 °C for 10 min, and 1200 °C for 5 min with subsequent water quenching. An equilibrium phase diagram of the present alloy can be utilized to determine annealing temperatures for controlling the BCC phase fraction. This was calculated using Thermo-Calc software, the thermodynamic database TCFe2000, and its upgraded version [19].

The annealing times were varied to ensure sufficient nucleation and growth processes of the secondary phase because lower temperature led to slower diffusion of the elements [20]. In this paper, the alloys annealed at 1000 °C, 1100 °C, and 1200 °C are labelled H10, H11, and H12.

### 2.2. Phase identification of the Al<sub>0.5</sub>CoCrFeMnNi HEA

Phase identification of the Al<sub>0.5</sub>CoCrFeMnNi HEA was performed by X-ray diffraction (XRD) using a RIGAKU D/MAX-2500 X-ray diffractometer with an incident beam of Cu K $\alpha$  radiation (wavelength  $\lambda = 1.5418 \text{ \AA}$ ). All samples were polished with silicon carbide papers (400–1200 grit) and 1  $\mu\text{m}$  diamond powder for the XRD analysis. The scans were performed from 30 to 100° of 2 $\theta$  with a step size of 0.02° and scan speed of 2°/min.

For quantitative identification of the FCC and BCC phases, the volume fractions of each phase were determined using the relative intensities of the diffraction peaks. The integrated intensity ( $I$ ) of a diffraction line can be expressed as [19,21]:

$$I = KRV, \quad (1)$$

where  $K$  is a constant for the given experimental conditions,  $R$  is the material scattering factor, and  $V$  is the volume of a phase exposed to the X-ray beam [22]. The scattering factor  $R$  value depends on the diffraction angles, diffraction planes, and materials, as follows [19]:

$$R = \frac{m}{v^2}(LP)e^{-2mF^2}, \quad (2)$$

where  $m$  is the multiplicity,  $v$  is the volume of the unit cell,  $LP$  is the Lorentz polarization factor,  $e^{-2m}$  is the Debye-Waller temperature factor, and  $F$  is the structure factor. The  $R$  values for each peak position were from Ref. [19]. Using the relationship of intensities of the diffraction peaks ( $I$ ) and exposed volume fraction ( $V$ ) from the Eq. (1), the relative volume fractions of FCC ( $V_{\text{FCC}}$ ) and BCC ( $V_{\text{BCC}}$ ) phases can be estimated using the following equations:

$$V_{\text{FCC}} = \frac{I_{\text{FCC}}/R_{\text{FCC}}}{[(I_{\text{FCC}}/R_{\text{FCC}}) + (I_{\text{BCC}}/R_{\text{BCC}})]}, \quad (3)$$

$$V_{\text{BCC}} = \frac{I_{\text{BCC}}/R_{\text{BCC}}}{[(I_{\text{FCC}}/R_{\text{FCC}}) + (I_{\text{BCC}}/R_{\text{BCC}})]}. \quad (4)$$

From the XRD data, the diffraction peak positions used for determining the volume fractions of each phase are:

FCC phase: {111}<sub>FCC</sub>, {200}<sub>FCC</sub>, {220}<sub>FCC</sub>, {311}<sub>FCC</sub>, and {222}<sub>FCC</sub>.

BCC phase: {110}<sub>BCC</sub>, {200}<sub>BCC</sub>, {211}<sub>BCC</sub>, and {220}<sub>BCC</sub>.

Also, based on the Bragg's law ( $n\lambda = 2d_{hkl}\sin\theta$ ), the interplanar spacing ( $d_{hkl}$ ) of each plane was obtained from the diffraction angles ( $\theta$ ) of each XRD peak position of FCC and BCC phases. Using these values, the lattice parameters ( $a$ ) of the FCC and BCC phases could be estimated using the following relationship;  $d_{hkl} = a/(h^2 + k^2 + l^2)^{1/2}$  for cubic systems. The estimated lattice parameters were  $\sim 3.61$  and  $\sim 2.89 \text{ \AA}$  for the FCC and BCC phases, respectively. The XRD analysis was repeated three times to ensure the reliability of the data for calculating the phase volume fractions of all the samples.

### 2.3. Microstructural analysis

Microstructural analysis was performed using scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD). All the specimens were finely polished to a surface roughness of 1  $\mu\text{m}$  and then polished more using a colloidal silica suspension. SEM analysis was conducted using a high-resolution field emission SEM (FE-SEM, JSM-7800F PRIME, JEOL Ltd., Japan) equipped with back-scattered electron (BSE) and dual energy-dispersive X-ray spectroscopy (Dual EDS) detectors.

After the SEM analysis, EBSD analysis was performed using FE-SEM (XL-30S FEG, FEI Company, USA). The EBSD data were then interpreted using orientation imaging microscopy (OIM) analysis software (TSL OIM Analysis 5.2). The inverse pole figure (IPF), grain boundary (GB), and phase maps were extracted from the EBSD data.

Furthermore, the transmission electron microscope (TEM, JEM-2200FS, JEOL Ltd., Japan) analysis was carried out to clarify the secondary phase. The TEM specimen was also prepared by fine mechanical polishing (1  $\mu\text{m}$ ) followed by colloidal silica polishing. The site-specific specimen involving secondary phase was extracted using a focused ion beam (FIB, FEI Company, USA) technique [23].

### 2.4. Mechanical testing and characterization

The tensile tests were performed at a strain rate of  $10^{-3} \text{ s}^{-1}$  using a universal testing machine (Instron 1361, USA) with the digital image correlation (DIC) technique using an optical 3-D deformation analysis system (ARAMIS 5 M, GOM Co., Germany) to measure precise strains. Dog-bone shaped specimens with a gauge length of 5.0 mm, a width of 2.5 mm, and a thickness of 1.5 mm were used in the tensile tests. All the tests were performed at room temperature (RT) and repeated at least three times. After the tensile tests, the EBSD analysis was carried out on the planes with normal directions of the specimens for interpretation of the microstructural behavior of the alloys using FE-SEM (XL-30S FEG, FEI Company, USA).

## 3. Results and discussion

### 3.1. Phase evolution behaviors of the Al<sub>0.5</sub>CoCrFeMnNi HEAs after annealing

Fig. 1(a) indicates the XRD patterns for the samples, which reveal the presence of two phases designated as FCC and BCC in the H10 and H11 alloys, while the H12 alloy indicates a single FCC crystal structure. The measured volume fraction of the BCC phase (shown in Fig. 1b) was compared with the volume fraction calculated using the thermodynamic method [19]. In the H10 and H11 alloys, the volume fraction of the BCC phase measured, was  $\sim 10.56$  and  $\sim 4.07\%$ , respectively.

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