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Cold-rolling and recrystallization textures of a nano-lamellar $AlCoCrFeNi_{2.1}$ eutectic high entropy alloy

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The development of texture during cold-rolling and recrystallization was investigated in a $AlCoCrFeNi_{2.1}$ eutectic high entropy alloy (EHEA). For this purpose, the as-cast alloy was cold-rolled to 90% reduction in thickness and annealed at temperatures ranging from 800 °C to 1200 °C. The microstructure of the as-cast EHEA showed a nano-lamellar mixture of $L1_2$ and B2 phases. The B2 phase was significantly harder than the $L1_2$ phase. Development of an ultrafine microstructure was observed after 90% cold-rolling. During cold-rolling, progressive disordering of the $L1_2$ phase was observed while the B2 phase maintained the ordered structure. The progressive disordering and development of a predominantly brass type texture in the $L1_2$ phase correlated well with profuse shear band formation during cold-rolling. The B2 phase showed the presence of the {111}<110> component which was typical for cold-rolled B2 alloys. An ultrafine duplex structure of equiaxed $L1_2$ and B2 phases developed after complete recrystallization that showed significant resistance to grain growth up to very high annealing temperatures (~1300 °C). The remarkable resistance to grain growth compared to conventional or even other single or dual phase HEAs was due to the formation of a homogeneous duplex structure where growth of one phase was effectively retarded by the other phase. The strong presence of the α-fiber components, but weak BR ({236}<385>) and D ({113}<332>) components in the recrystallization texture of the $L1_2$/FCC phase was due to the absence of strong preferential nucleation or growth. Presence of ND-fiber (ND//<111>) with strong {111}<011> component in the recrystallization texture of the B2 phase indicated ease of nucleation from similarly oriented regions in the deformed microstructure.
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1. Introduction

High entropy alloys (HEAs) have been classified as a novel class of multicomponent alloys containing five or more elements in equiatomic or near equiatomic proportions [1]. Despite having the presence of a rather large number of components, HEAs can exist with simple phases, such as FCC, BCC and FCC + BCC [1,2]. The original explanation offered for this unique structure is that high configurational entropy due to the presence of a large number components in equal atomic fraction in HEAs can sufficiently decrease the free energy to stabilize simple phases. However, recent investigations have indicated that high configurational entropy is not sufficient to completely explain the phase formation in HEAs. This can be followed from the fact that equiatomic HEAs with fixed number but different combination of constituents can show mixture of more complex phases [3], whereas non-equiatomic alloys can surprisingly form simple solid solution phases [4]. The HEAs have triggered remarkable research interest in recent years [2,5–8]. As a consequence, many unique and intriguing properties of HEAs have been discovered [5,6,9–11].

In order to develop HEAs with significantly enhanced strength-ductility combination, as desired in many engineering applications, dual phase HEAs having a mixture of soft and hard phases have been suggested. Development of a non-equiatomic $AlCoCrFeNi_{2.1}$

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HEA having lamellar eutectic structure of two phases is an outcome of this alloy design philosophy [12]. Subsequent to that report, a series of dual phase lamellar HEAs have been reported [13,14].

Although the eutectic high entropy alloys (EHEAs) show significant potential [12], the properties of these alloys need to be further improved. Thermo-mechanical processing comprising of plastic deformation and heat treatments can be particularly useful in this regard, as observed in the case of conventional metallic alloys as well as single phase HEAs, such as CoCrFeMnNi [15]. Thermo-mechanical processing can significantly influence the development of microstructure and texture, which in turn affect the properties. Effect of thermo-mechanical processing on texture development has been intensely investigated in single [16–22] and dual phase HEAs [23], but a very limited studies have only been carried out in EHEAs [24,13].

Wani et al. [24,25] have reported the development of ultrafine microstructure and considerable improvement in tensile properties in $AlCoCrFeNi_{2.1}$ eutectic HEAs subjected to thermo-mechanical processing. However, the development of texture has not been reported so far. In comparison to single phase materials, the development in texture in the dual phase materials could be more complex, as it is affected by the interaction and presence of different phases. For EHEAs with fine intimate mixture of two phases, the behavior may be particularly intriguing.

In the present work, the development of texture in $AlCoCrFeNi_{2.1}$ eutectic HEA after heavy cold-rolling and annealing at different temperatures has been investigated for the first time. The major interest has been focused on understanding the development of texture in the individual phases and to clarifying the relevant mechanisms. It is quite envisaged that the present results should be useful for understanding the texture of other lamellar EHEAs.

2. Experimental

2.1. Processing

The EHEA $AlCoCrFeNi_{2.1}$ was prepared using arc-melting in a Ti-gettered high-purity argon atmosphere starting with high purity metals of component elements (≥99.9%). In order to ensure high level of chemical homogeneity of the melt, the melting process was repeated for at least five times. The melt was suction-cast into a copper mold with dimensions 15 mm (width) x 90 mm (length) x 3 mm (thickness). Specimens for cold-rolling having dimensions 20 mm (long) x 15 mm (width) x 3 mm (thickness)) were obtained from the as-cast material and the surfaces were polished to remove contaminations. The cold-rolling was carried out at room temperature up to ~90% reduction in thickness (final thickness ~300 μm) following a multipass rolling schedule. A laboratory scale two-high rolling equipment (SPX Precision Instruments, Fenn Division, USA) having 140 mm diameter rolls was used. The 90% cold-rolled samples were annealed for 1 hour (h) at temperatures ranging from 800 °C to 1300 °C. The annealed samples were immediately water quenched.

2.2. Characterization

The thermal analysis of the as cast EHEA for determining the eutectic melting point was carried out using differential scanning calorimeter (DSC) technique (NETZSCH, DSC404 F3). The sample weight and heating rate for the DSC experiments were 24 mg and 20 K/min, respectively. The mechanical properties of the constituent phases in the as-cast EHEA were investigated by nano-indentation hardness mapping using a TI950 Triboindenter (Hysitron, USA). An indent-indent spacing of 100 nm was deemed to be the largest spacing adequate to map the lamellar structure of the EHEA sample; Indents were obtained from a mapping area of 2.1 × 2.1 μm^2 with an indent spacing of 100 nm (22 × 22 grid, corresponding to 484 points). To prevent the residual impression of one indent from affecting the results of the next, a sharp cube-corner probe with an apex radius of curvature of approximately 40 nm (Hysitron, USA) was used, and indent depth was minimized by setting the maximum load as low as possible; 500 μN was found to be the minimum necessary load to induce plastic deformation in the harder B2 phase, and was used for all indent tests. During each test, load was increased linearly from 1 μN to 500 μN over a period of 0.25 s, held constant for 0.25 s, and unloaded to 1 μN over a period of 0.25 s before moving to the next indent position. Since shallow indentation tests are known to be strongly influenced by surface roughness [26], the sample's surface topography was first measured using the TriboIndenter's *in-situ* imaging function, and regions sufficiently flat for hardness mapping were chosen based on the topographic data. All nanoindentation data were analyzed using the standard Oliver-Pharr method [27].

The microstructural and textural characterization of the cold-rolled and annealed samples were carried out using electron backscatter diffraction (EBSD) system (Oxford Instruments, UK) attached to a scanning electron microscope (SEM) (Carl-Zeiss, Germany; Model: SUPRA 40) and by transmission electron microscope (TEM) (JEOL 2010 operated at 200 kV) techniques. The samples for EBSD and TEM investigations were prepared using mechanical polishing followed by electropolishing (electrolyte: 90% ethanol + 10% perchloric acid).

The EBSD scans were acquired using the AztecHKL software (Oxford Instruments, UK). For the purpose of further analysis, the acquired EBSD dataset were exported to the TSL-OIM™ software (EDAX Inc., USA). Several EBSD scans were obtained from each specimen and stitched together. This ensured high degree of statistical reliability of the microstructural and textural parameters. The orientation distribution functions (ODFs) were calculated from these merged dataset using the harmonic series expansion method (series rank = 22). Orthotropic sample symmetry was imposed for obtaining the ODFs. The volume fractions of different texture components were determined using a cut-off angle of 15°.

3. Results

Fig. 1 shows the microstructure obtained by the EBSD orientation mapping of the as-cast $AlCoCrFeNi_{2.1}$ EHEA. The phase map of the as-cast material (Fig. 1(a)) clearly reveals a lamellar eutectic morphology. In the phase map (Fig. 1 (a)), the FCC phase and BCC phase are highlighted in green and red, respectively. The eutectic lamellar structure composed of mutual alignment of the FCC and BCC phases is clearly seen in Fig. 1(a). The volume fraction of the FCC and BCC phases are ~65% and ~35%, respectively. The EBSD analysis could not confirm whether the phases have ordered structures or not. The as-cast microstructure is further analyzed also by TEM. The TEM analysis shows the presence of ordered FCC ($L1_2$; corresponding to green in Fig. 1(a)) and ordered BCC (B2; corresponding to red in Fig. 1(a)) phases, as has been reported in our previous work [24]. The average thickness of the $L1_2$ and B2 phases is 0.57 μm and 0.20 μm, respectively. The chemical compositions of the two phases expressed in at.% and wt% are shown in Table 1. It is clearly observed from Table 1 that the B2 phase is depleted in Cr but enriched in Al and Ni. The converse is found true for the $L1_2$ phase. The inverse pole figure (IPF) map of the FCC (Fig. 1(b)) phase clearly indicates that within a single colony the $L1_2$ lamellae possess quite similar orientation, while the orientation of the $L1_2$ lamellae within a colony differs with those in the neighboring colonies, which is typical for eutectic alloys. The IPF map of

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