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Effect of severe cold-rolling and annealing on microstructure and mechanical properties of AlCoCrFeNi_{2.1} eutectic high entropy alloy

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Abstract. The possibility of microstructural refinement and improvement of mechanical properties by severe cold-rolling was investigated in an AlCoCrFeNi_{2.1} lamellar eutectic high entropy alloy (EHEA). The as-cast alloy revealed fine scale eutectic mixture of L₁₂ (ordered FCC) and B2 (ordered BCC) phases. During severe cold-rolling up to 90% reduction in thickness the B2 phase maintained the ordered structure, while the L₁₂ phase showed the evolution of a nanocrystalline structure and progressive disordering. Annealing of the severely cold-rolled material resulted in the formation of duplex microstructures composed of two different phases with equiaxed morphologies and significant resistance to grain growth up to 1200°C. Annealing at 1000°C resulted in an optimum strength-ductility balance with the tensile strength of 1175 MPa and the total elongation of 23%. The present results showed that severe cold-rolling and annealing can impart very attractive mechanical properties in complex EHEAs.

1. Introduction

High entropy alloys (HEAs) are originally proposed as a novel class of multicomponent alloys containing sufficiently large number of alloying elements (≥ 5) in equiatomic or near equiatomic proportions [1]. Despite the presence of large number of constituents, the HEAs may show simple phases due to their large configurational entropy. In spite of their complex chemistry, the HEAs show several attractive and intriguing mechanical properties which have generated tremendous research interest [2].

In order to further enhance the strength-ductility combination, multiphase HEAs possessing novel microstructures have been proposed. Development of Eutectic HEAs (EHEAs) is a noteworthy approach in this regard [3]. Although the as cast microstructure of the EHEAs show promising mechanical properties, it is envisaged that suitable thermo-mechanical processing route can tailor the



microstructure and properties of these novel alloys significantly as observed in other HEAs [4] but needs to be clarified.

In this paper, the effect of severe cold-rolling and annealing on microstructure and mechanical properties of a AlCoCrFeNi_{2.1} EHEA is investigated. It is believed that clarifying the mechanisms of microstructure development and enhancement of properties in the present EHEA by severe plastic deformation should be helpful in developing appropriate severe plastic deformation based processing strategies for other HEAs.

2. Experimental procedure

The AlCoCrFeNi_{2.1} EHEA (15mm (width) x 90mm (length) x 3mm (thickness)) used in the present study was prepared by an optimized arc melting route starting from high purity elemental powders. The as-cast samples were cold-rolled up to 90% reduction in thickness (final thickness ~0.3 mm) using a laboratory scale two-high rolling machine (Fenn, USA). The cold-rolled samples were annealed at temperatures ranging between 800°C to 1200°C for 1 hour (h).

The microstructure of the different cold rolled and annealed samples were investigated using scanning electron microscope(FEG-SEM) (SUPRA 40, Carl-Zeiss, Germany) equipped with electron backscatter diffraction (EBSD) (Oxford Instruments, UK) and transmission electron microscope (TEM) (JEOL 2010) operated at 200 KV. The samples for EBSD and TEM were prepared by standard electro-polishing techniques using electrolyte content of 90% ethanol + 10% perchloric acid. The hardness of the constituent phases in the as-cast EHEA was analyzed by nanoindentation hardness mapping using a TI950 Triboindenter (Hysitron, USA) with the applied load linearly increasing from 1 μN to the maximum of 500 μN over a period of 0.25 s and unloaded to 1 μN over a period of 0.25 s before moving to the next indent position. Tensile tests were carried out at ambient temperature using a floor mounted testing machine (Shimadzu, Japan) using an initial strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$.

3. Results and discussion

Figure 1(a) shows SEM micrograph of the as-cast EHEA revealing the fine eutectic mixture. The TEM micrograph (Fig.1 (b)) shows in detail the eutectic microstructure, while the selected area diffraction patterns (SADPs) obtained from the regions marked by green and red circles in (Fig.1(b)) show that the eutectic mixture consists of L₁₂ and B2 phases having thickness of ~600 nm and ~200 nm, respectively.

Table 1. Detailed chemical composition of the phases present in the as-cast EHEA (at %)

Phases	Elements				
	Al	Co	Cr	Fe	Ni
Nominal	16.39	16.39	16.39	16.39	34.43
L ₁ ₂	8.66±0.2	18.47±0.25	20.53±0.05	19.48±0.25	32.85±0.36
B2	24.30±1.3	13.00±0.14	6.50 ±0.84	10.55±0.77	45.70±2.83

The chemical analysis of the as-cast alloy done by energy dispersive spectroscopy (EDS) shows the difference in elemental distribution in the two phase as summarized in Table 1. The B2 phase is enriched with Ni and Al but depleted in Cr. The B2 phase also show the presence of disordered Cr-rich BCC nano precipitates [5]. Figure 2 shows the nanoindentation mapping of an area of interest in the EHEA. It is clearly observed that the B2 is much harder than the L₁₂ phase.

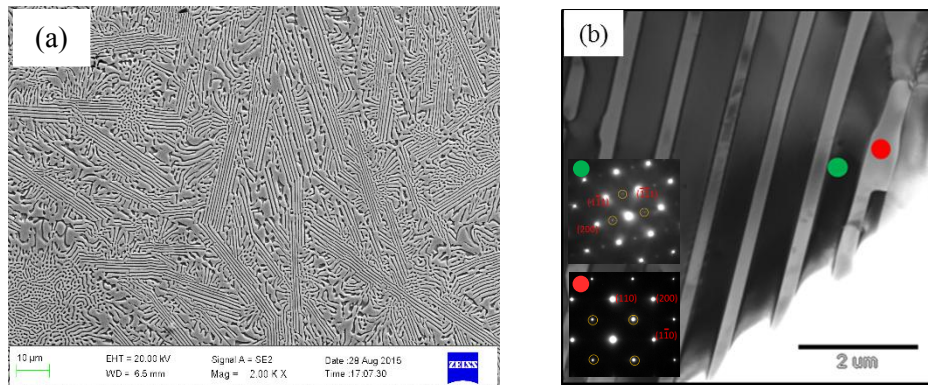


Figure 1. (a) SEM and (b) TEM micrographs of the as-cast EHEA showing the lamellar microstructure. The inset SADPs (marked by green and red spots) obtained from green and red spots in (b) correspond to the $L1_2$ with zone axis $[011]$ and B2 phase with zone axis $[001]$, respectively.

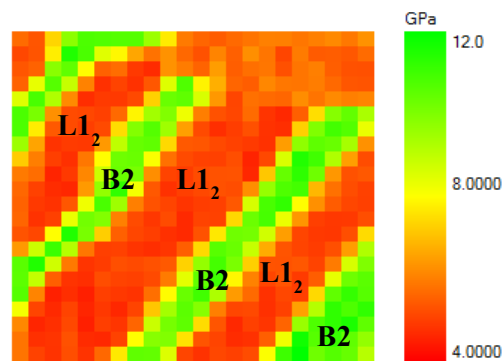


Figure 2. Nano-indentation map of the as-cast EHEA.

Figure 3 shows the TEM micrograph of the 90% cold-rolled material. The SADP in (Fig.3 (b)) (obtained from the region marked by green circle in Fig.3(a)) undergoes progressive disordering accompanied by the evolution of a nanocrystalline structure. The deformation induced disordering has been reported in other $L1_2$ alloys [6] and attributed to formation of deformation heterogeneities and the uncoupling of partial dislocations [7]. However, the B2 phase has retained its ordered structure after heavy deformation as confirmed by the SADP (obtained from the region shown in red circle in Fig.3(c)).

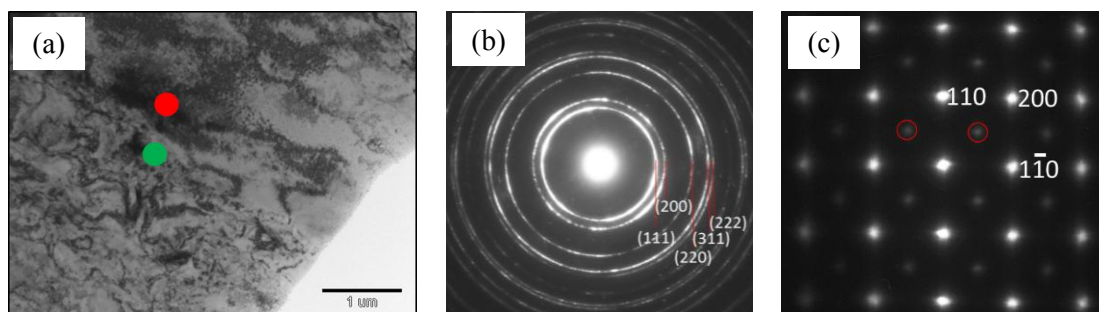


Figure 3. (a) TEM micrograph of the 90% cold-rolled EHEA; (b) and (c) are the SADPs of disordered FCC and B2 phase respectively.

Figure 4 shows the EBSD phase map of 90% cold-rolled material after annealing at 800°C (Fig.4(a)) and 1200°C (Fig.4(b)), respectively. The phase maps show the development of microduplex structure consisting of disordered FCC and B2 phases [8]. Table 2 shows the grain size of the two phases after different annealing temperature. The EHEA shows significant resistance to the grain growth with average grain size of $\sim 2 \mu\text{m}$ at 1200°C.

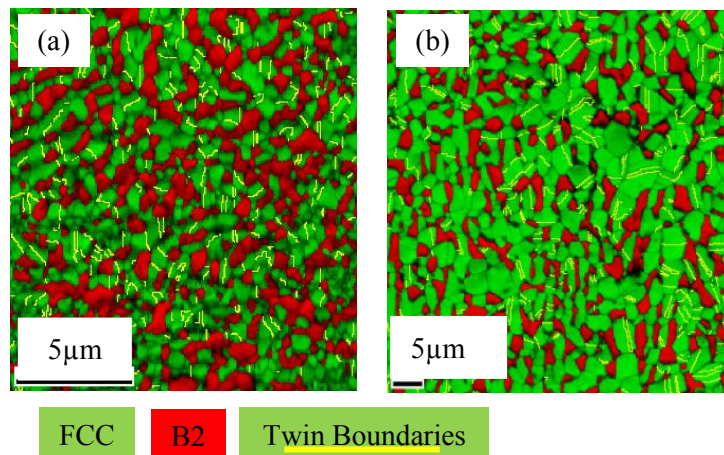


Figure 4. Phase maps of the 90% cold-rolled sample after annealing at (a) 800°C and (b) 1200°C respectively

Table 2: Grain size of the two phases in different annealed materials

90% Cold rolled	Average grain size (μm)
Annealed at 800°C	0.5 ± 0.01
Annealed at 1000°C	0.7 ± 0.05
Annealed at 1200°C	2.0 ± 0.44

The tensile properties of the EHEA in different conditions are summarized in Table 3. The improvement in the mechanical properties of EHEA after severe cold-rolling and annealing is remarkable as compared to other two phase HEAs which undergo structural coarsening leading to significant decrease in strength [9]. It can be concluded that the severe cold-rolling and annealing can impart novel properties in EHEAs and render them suitable for a wide range of industrial applications.

Table 3: Summary of mechanical properties of the EHEA in various conditions

Material Condition	YS(MPa)	UTS(MPa)	ϵ_f %
As-received	620	1050	17
90% Cold-rolled	1625	1800	6
Annealed at 800°C	1108	1200	12
Annealed at 1000°C	844	1175	23
Annealed at 1200°C	648	1075	27

4. Conclusion

The following conclusions may be drawn from the present work:

- The AlCoCrFeNi_{2.1} EHEA consists of L1₂ (FCC ordered) and B2 (BCC ordered phase) in the as cast state.
- Severe cold-rolling results in progressive disordering of the L1₂ and evolution of a nanocrystalline structure.
- Development of duplex microstructure is observed after annealing at different temperatures.
- Severe cold-rolling and annealing can significantly enhance the strength-ductility combination in The EHEA. Annealing at 1000°C results in an optimum strength-ductility balance as evidenced by a high tensile strength of 1175 MPa and total elongation of 23%.

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