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Hierarchical microstructure for improved fatigue properties in a eutectic high entropy alloy

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ABSTRACT

Microstructural hierarchy can enable enhanced properties in high entropy alloys. A unique microstructure was observed in 50% cold-rolled and heat-treated eutectic high-entropy alloy (EHEA). Formation of recrystallized FCC grains and B2 precipitates in FCC lamellae with different microstructural length scale constitute the hierarchy in cold-rolled and heat-treated EHEA. Monotonic loading and high cycle fatigue properties of both microstructures were compared. In as-cast microstructure, the fatigue crack originated in FCC lamellae with the formation of persistent slip bands. The B2 precipitates in cold-rolled and heat-treated alloy delayed the crack initiation and improved fatigue properties.

Since the discovery of copper in 9000 BCE, humankind has tried to improve the strength of metals in numerous ways. Improvement in strength has been largely due to grain refinement, work-hardening and alloying or any other combinations of these mechanism [1,2]. A new class of material, known as high entropy alloys (HEAs), has drawn significant attraction in recent years. Some of the initial literatures have defined HEAs as “single phase solid solutions composed of five or more principal elements, generally in equimolar quantities” [3,4]. Although the definition emphasized “single phase”, the ever-expanding research on this topic has led to various multi-phase HEAs and hence other terms like multi-principal elemental alloys (MPEAs) and complex concentrated alloys (CCAs) have been coined [5]. With their vast range of compositions, HEAs possess many remarkable properties such as excellent tensile properties and hardness [6,7], good high-temperature mechanical as well as refractory properties [7,8], and excellent cryogenic fracture resistance [9,10].

Cumulative damage of materials due to cyclic loading is known as fatigue, which is a crucial property for any structural component as in-service alloys are generally subjected to a non-monotonic loading. Only a few studies have focused on the fatigue behavior of HEAs. Hemphill et al. [11] investigated the fatigue behavior of $Al_{0.5}$CoCrFeCuNi in cold-rolled and annealed condition which exhibited high fatigue endurance limit. A large variance in the fatigue limit and scatter was attributed to the aluminum oxide particles and cracks during casting and rolling. Also, in another study on the same alloy, Tang et al. [12] reported on the effect of impurities and processing route on the fatigue properties. They also investigated the effect of nano-twins on fatigue properties, concluding that nano-twinning behavior resulted in a better fatigue life and high fatigue strength of about 383 MPa. A recent study by Niendorf et al. [13] focused on the low cycle fatigue properties of $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ HEA alloy in rolled and annealed condition with two different grain sizes. They reported a completely different deformation mechanism in $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ HEA when compared with high-Mn twinning-induced plasticity (TWIP)/transformed-induced plasticity (TRIP) steel. Apart from the above-mentioned studies on two-phase HEAs, some single phase HEAs were also subjected to fatigue testing. Thurston et al. [14] studied the effect of temperature on the fatigue crack growth of single phase CrMnFeCoNi HEA in rotary swaged and recrystallized condition.

Microstructure containing hierarchical features can be engineered to obtain favorable properties [15]. In past, such a process has been utilized to enhance superplastic properties of titanium alloys [16] and aluminum alloys [17]. Also, in Ni based superalloys, hierarchical microstructure develops during heat-treatment [18] and provide key benefits to balance of properties. Given the complexity of physical metallurgy in HEAs, they provide excellent opportunity for building complex microstructure to get desired combination of properties. Studies of many multiphase HEAs have established that an excellent combination of strength and ductility can be achieved by blending relatively harder and softer phases. Eutectic HEAs (EHEA), with a combination of hard BCC phase and soft FCC phase in lamellar morphology, have excellent castability in addition to good mechanical properties in as-cast condition [19]. Furthermore, 90% cold-rolled and annealed microstructure has an excellent combination of strength and ductility [20,21]. A recent study on the thermomechanical processing of EHEA shows that lamellar

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microstructure can be retained [22]. Although high-density dislocation network and disordering of L1_2 phase have been observed, B2 phase ordering was still present in the 50% rolled microstructure. Furthermore, Cr-rich disordered BCC nano-precipitates have been observed in the BCC(B2) phase. These observations point towards three distinct features of EHEA. (i) lamellar morphology, (ii) semi-coherent interface between BCC (B2) and FCC (L1_2) phases and (iii) intra-lamellar features within the BCC and FCC phases. Hence, it provides an opportunity to understand the effect of all these features on fatigue behavior of EHEA.

Commercially pure elements of Al (99.9 wt%), Co (99.9 wt%), Ni (99.9 wt%), Cr and Fe (99.5–99.6 wt%) were used in the casting of $AlCoCrFeNi_{2.1}$ (in atomic ratio). Two conditions were chosen for the present study – (i) EHEA in as-cast condition, hereafter named EHEA$_c$ and (ii) as-cast EHEA rolled to 50% of initial thickness and heat-treated at 700 °C for 12 h, hereafter named EHEA$_W$. The mini-tensile and mini-fatigue samples were prepared via CNC machine. Samples were mechanically polished to a surface roughness of 0.05 μm using colloidal silica to minimize any surface flaw. Gage length of the mini-tensile samples was ~5 mm with width of ~1 mm, and thickness of ~1.1 mm. Mini-tensile testing was carried out on a custom build computer-controlled mini-tensile tester at an initial strain rate of 10^{-3} s^{-1}. Bending fatigue testing was performed using a custom-made tabletop mini-fatigue testing machine. Thickness of mini-fatigue samples was ~1 mm with an effective gage length of ~3.05 mm and a taper angle of 28°. The details regarding the geometry of fatigue samples and the description of the bending fatigue setup can be found in [23]. All samples were tested at 20 Hz frequency and stress ratio (R) of −1. Backscattered electron (BSE) imaging was done on FEI Nova NanoSEM 230, with an 18 kV accelerating voltage. The EBSD analysis was executed using SL Digiview III electron backscatter diffraction (EBSD) detector on FEI Nova NanoSEM 230, with a 20 kV accelerating voltage and a step size of 40 nm. For electron microscopy, samples were polished down to a surface finish of 0.02 μm.

Fig. 1(a–d) show the microstructure of the as-cast $AlCoCrFeNi_{2.1}$ (EHEA$_c$). The initial microstructure is composed of lamellae of FCC (L1_2) and BCC(B2). The EBSD analysis (Fig. 1(b, c)) reveals the FCC (L1_2) fraction to be around 71% while BCC(B2) to be around 29%. Spacing between each FCC lamellae is around 0.70 μm and spacing between BCC lamellae is around 1.3 μm. Cr-rich precipitates have been marked with an arrow in high-resolution BSE image (Fig. 1(d)). Microstructure and EBSD images of EHEA$_W$ (after cold rolling to 50% and heat treatment at 700 °C for 12 h) are shown in Fig. 1(e–h). For EHEA$_W$ the fraction of FCC (L1_2) is around 68% and BCC(B2) around 32% respectively. The hierarchy in EHEA$_W$ is characterized by four distinct microstructural features - (i) lamellar FCC region (ii) deformed and subsequent recrystallized FCC phase (Fig. 1(f–g)) with various BCC/B2 phases having different morphologies (Fig. 1(h)), (iii) lamellar BCC(B2) with Cr rich precipitates and (iv) recrystallized BCC phase. Details regarding the precipitation mechanism of B2 phase inside FCC matrix via cold rolling and heat-treatment can be found elsewhere [24]. The recrystallized FCC phase and BCC phase within the FCC lamellae can be attributed to the cold-rolling and subsequent heat treatment process.

Fig. 2(a) depicts a tensile plot for both EHEA$_c$ and EHEA$_W$ tested at an initial strain rate of 10^{-3} s^{-1}. EHEA$_c$ exhibits a yield strength (YS) of ~746 MPa and ultimate tensile strength (UTS) of ~1057 MPa with a uniform elongation of ~8%. Except for the % elongation value, all the other values are in accordance with the earlier studies [19,20,21]. The properties of as-cast materials are highly dependent on the the casting conditions and hence the low ductilty. The variance of the quasi-static properties of EHEA$_c$ from literature can be attributed to the size of ingots, cooling rate, etc. Besides, the as-cast microstructure features (i.e. lamellae size, spacing and fraction of BCC to FCC phases etc.) are also responcible for the resultant mechanical properties. In comparison, the YS and UTS of EHEA$_W$ are substantially enhanced to a value of ~1110 MPa and ~1340 MPa with a total elongation of ~10%. Fig. 2 (b) illustrates the work hardening response of both microstructures as a function of the strain. Clearly evident is the difference in stage B (highlighted by red and blue shaded regions Fig. 2(b)) of both microstructures. Since stage B is generally associated with twinning mediated plasticity, it is apparent that EHEA$_{W,}$ with a combination of recrystallized FCC grains and higher fraction of annealing twin boundaries (Fig. 1(f–h)), is expected to exhibit an almost zero slope in stage B causing sustained strain-hardening rate. An earlier investigation by Wani et al. [20] on 90% cold-rolled and heat treated EHEA reported the formation of ultrafine grained (UFG) microstructure with YS and UTS values of 1100 MPa and 1200 MPa, respectively. The current study achieves even higher strength and improved work-hardening without the loss of ductility. The microstructural hierarchy is responsible for enhanced properties of EHEA$_W$ when compared with UFG EHEA as different length scale microstructural features such as B2 precipitates, with the

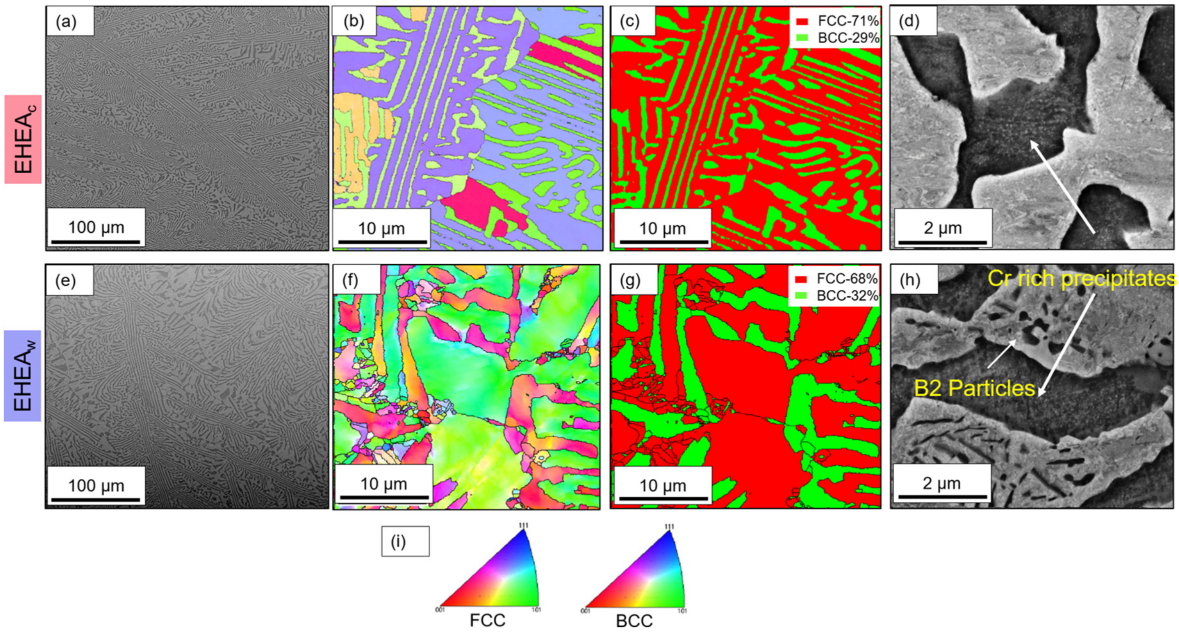

Fig. 1. Initial microstructure of EHEA with (a) BSE image of EHEAc, (b) and (c) EBSD and phase map of EHEA$_c$, (d) high magnification BSE image of EHEA$_c$, (e) BSE image of EHEA$_{W,}$ (f) and (g) EBSD and phase map of EHEA$_W$, (h) high magnification BSE of EHEA$_W$, and (i) inverse pole figure legend for FCC and BCC phases.