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A precipitation-hardened high-entropy alloy with outstanding tensile properties

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ABSTRACT

Recent studies indicated that high-entropy alloys (HEAs) possess unusual structural and thermal features, which could greatly affect dislocation motion and contribute to the mechanical performance, however, a HEA matrix alone is insufficiently strong for engineering applications and other strengthening mechanisms are urgently needed to be incorporated. In this work, we demonstrate the possibility to precipitate nanosized coherent reinforcing phase, i.e., $L1_2$-Ni_3(Ti,Al), in a fcc-FeCoNiCr HEA matrix using minor additions of Ti and Al. Through thermomechanical processing and microstructure controlling, extraordinary balanced tensile properties at room temperature were achieved, which is due to a well combination of various hardening mechanisms, particularly precipitation hardening. The applicability and validity of the conventional strengthening theories are also discussed. The current work is a successful demonstration of using integrated strengthening approaches to manipulate the properties of fcc-HEA systems, and the resulting findings are important not only for understanding the strengthening mechanisms of metallic materials in general, but also for the future development of high-performance HEAs for industrial applications.

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1. Introduction

Conventional alloy design strategy, which is usually based on one principal constituent and adds other minor elements for further optimization of properties and performances, has created a variety of metallic materials for our daily life. Recently, a revolutionary alloy design concept, namely, high-entropy alloy (HEAs) concept, was proposed and the basic idea is to simultaneously alloy multiple principal elements in equimolar or near equimolar ratios to increase the configuration entropy to stabilize the structures. Since its inception, this new family of alloys has been attracted extensive attention due to their unique properties and the related scientific importance [1–5]. Due to their high mixing entropy, these alloys tend to form single-phase structures with a high symmetry, such as fcc (face-centered cubic), bcc (body-centered cubic), and hcp (hexagonal close-packed) [4,6–8]. They have been demonstrated to exhibit several special intrinsic characteristics, for example, high configuration entropy [9], sluggish atomic diffusion [10], and large lattice distortion [3,11]. These features are anticipated to enhance formation and stabilization of solid solution phases and impede dislocation motion, thereby improving the mechanical strength, particularly at high temperatures. Nevertheless, recent studies [12–14] indicate that a HEA matrix alone, especially single-phase fcc structure, is insufficiently strong for practical applications. In other words, other strengthening mechanisms are needed so that desirable mechanical properties can be obtained. While, qualitative descriptions and equations of strengthening mechanisms for traditional solid solution alloys are well established, such theories are missing for the highly concentrated HEAs because it is difficult to clearly identify their “solvent” and “solutes”. Therefore, strengthening mechanisms in these emerging metallic materials must be carefully investigated so that reliable theories can be established.

FeCoNiCrMn is generally recognized as a “model” HEA with a simple fcc structure, and exhibits both outstanding ductility and

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fracture toughness property [12], even at the liquid nitrogen temperature. However, its strength is relatively low, only around 200 MPa in the as-cast state [13,14], which is far from practical structural applications. To make it useful, additional strengthening methods, without sacrificing its ductility, must be induced in the alloy and, the underlying mechanisms need to be scrutinized so that general understanding of the strengthening of HEAs can be achieved.

Along this line, Otto et al. made attempts to enhance the strength of this fcc-FeCrNiCoMn by grain refinement and found that the room-temperature yield strength of the alloy increased from 200 to 350 MPa when the grain size was reduced from 144 to 4.4 μm [13]. This work suggests grain boundary hardening can be induced in the fcc-FeCrNiCoMn alloy, but the hardening is seemingly not extraordinary. To further improve the strength without losing plastic stability, one must, therefore, rely on other mechanisms, such as precipitation hardening, which would require a modification of the chemical composition of the alloy.

In terms of precipitation hardening in HEAs, published results appear somewhat sketchy. A few publications reported the observation of precipitation behavior in HEAs, e.g., in a mixed fcc + bcc $CuCr_2Fe_2NiMn$ [15], a mixed fcc + bcc $Al_{0.3}CrFe_{1.5}MnNi_{0.5}$ and single-phase bcc-$Al_{0.5}CrFe_{1.5}MnNi_{0.5}$ [16]. The precipitates in these HEAs were relatively bulky (generally, larger than μm in size), significantly different from the distribution of fine precipitates (~nm) commonly observed in traditional precipitation-hardened alloys. Nevertheless, Yeh et al. have reported the presence of nano-scale precipitates in some HEAs [5]. In this case, a distribution of extremely tiny precipitates (diameter ~3–7 nm) was observed in a CuCoNiCrAlFe HEA with a modulated structure. Tensile properties and possible strengthening of the alloy were unfortunately not evaluated. However, these observations are quite encouraging and suggest that proper selection of the chemical composition combined with appropriate thermomechanical treatment may offer the opportunity to manipulate precipitation strengthening of HEAs.

In the present paper, we demonstrate that minor alloy additions of Ti and Al to a single-phase fcc-FeCoNiCr HEA can induce the formation of $L1_2$ coherent nano-size precipitates in the alloy matrix. Subsequently, both yield and ultimate tensile strengths of the alloys are drastically increased. The strengthening efficacies from various strengthening mechanisms are evaluated based on the resulting microstructure in the current highly concentrated HEA matrix.

2. Experimental

Two HEA compositions were prepared by vacuum arc melting: the base alloy, FeCoNiCr (in equimolar ratio) for comparison, and another with the nominal composition of $(FeCoNiCr)_{94}Ti_2Al_4$ (at.%). The alloy ingots were prepared by arc-melting a mixture of pure metals (purity larger than 99.9%), and re-melted at least four times to ensure homogeneity. The master ingots were then drop-casted into a copper mold with a dimension of 10 × 10 × 60 mm^3, and subsequently tube-sealed and homogenized at 1473 K for 4 h.

Two thermomechanical procedures were conducted on the alloyed HEA to obtain fine structures. The first treatment, P1, includes an initial cold rolling of 30%, subsequent annealing at 1273 K for 2 h, aging at 1073 K for 18 h, and followed by water quenching. The second process, P2, includes an initial cold rolling of 70%, and then aging at 923 K for 4 h, followed by water quenching. Therefore, four different HEA samples were prepared, i.e., the as-homogenized FeCoNiCr (alloy A), as-homogenized $(FeCoNiCr)_{94}Ti_2Al_4$ (alloy B), P1 and P2.

A CMT4105 universal electronic tensile testing machine was employed for tensile tests at room temperature with a nominal strain rate of 1×10^{-3} s^{-1}. The dog bone-shaped tensile samples had a gauge length of 20 mm, a width of 5 mm and a thickness of 1.3 mm. The surface of test samples was polished down to a 2000-grit SiC paper to eliminate scratches.

Considering the relative low accuracy of X-ray diffraction (XRD) in nano-precipitates detection, phase identification in this study was conducted by neutron diffraction, at the VULCAN instrument, beam line 7, Oak Ridge National Laboratory Spallation Neutron Source (ORNL, SNS), USA, at room temperature. The microstructure was characterized by a Zeiss Supra55 scanning electron microscope (SEM) and a JEOL ARM200 transmission electron microscope (TEM) equipped with an objective lens corrector and a thermal field-emission gun (FEG). SEM specimens were initially polished to 2000-grit SiC paper and, subsequently, electrochemically polished for the final surface clarification using a $HClO_4$:C_2H_6O = 1:9 solution with a direct voltage of 30 V at room temperature. TEM samples were primarily punched to Φ3 mm circle sheets and then ground to about 50 μm, followed by twin-jet electro-polishing using a mixed solution of HNO_3:CH_4O = 1:4 with a direct voltage of 28 V and a current of 60 mA at a temperature around 233 K.

Sharp tip specimens for atom probe tomography (APT) were made by a two-step electrochemical polishing. The 0.3 mm × 0.3 mm blanks were cut by low speed diamond saw, followed by electrochemical polishing using 10% perchloric acid at a direct voltage of 15 V, the tips were then polished by using weaker electrolyte (2%) of perchloric acid at a direct voltage of 8 V. Data acquisition was performed by using a local electrode atom probe (LEAP 4000HR) equipped with an energy-compensated reflectron by which the mass resolution can be greatly improved. The APT acquisition temperature was set at ~60 K and the pulse frequency and pulse fractions were 200 kHz and 20%, respectively. CAMECA Integrated Visualization and Analysis Software (IVAS 3.6.8) package was used for the data processing and three-dimensional (3D) atomic reconstruction.

Shear modulus and Poisson's ratio of polished specimens with a dimension of 5.5 × 5.5 × 2.5 mm^3 were measured using RUSpec resonant ultrasound spectrometer, Teclab, USA. To determine the dislocation density using the Williamson-Hall method, XRD tests were conducted using CuKα radiation (Rigaku Dmax 2500 V) with a scanning 2θ range of 40°–100° and a step of 0.02°. Annealed single crystal Si powder was also tested to define the instrument peak broadening in this method.

3. Results

3.1. Neutron diffraction and SEM results

Neutron diffraction patterns of the four HEA specimens, i.e., the homogenized FeCoNiCr (Alloy A) and $(FeCoNiCr)_{94}Ti_2Al_4$ (Alloy B), and two thermomechanically processed $(FeCoNiCr)_{94}Ti_2Al_4$ alloys P1 and P2, respectively, are presented in Fig. 1. A single family of fcc peaks are clearly observed for alloy A and B. By contrast, extra series of minor peaks identified as $L1_2$-Ni_3(Ti, Al) are detected in P1 and P2 samples, indicating the precipitation of secondary phases. It should also be noticed that, there are still two weak peaks at ~1.4 Å in alloy A and B and another one at ~1.1 Å in alloy P1 and P2 being unknown. Due to the relative low intensity and limited amount of peaks, one can hardly determine the exact phase structure they belong to. Nevertheless, the matrix of the four alloys is mainly composed of an fcc structure.

Fig. 2 shows the corresponding SEM micrographs of the four kinds of HEA samples. As can be seen, both A and B alloys appear to be single-phase structure in Fig. 2a and b, respectively, with only few dirt on the surface probably introduced during the electro-polishing process. In accordance with the neutron diffraction

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