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Precipitation strengthening of ductile $Cr_{15}Fe_{20}Co_{35}Ni_{20}Mo_{10}$ alloys

Kaisheng Ming [a], Xiaofang Bi [a,*], Jian Wang [b,*]

[a] School of Materials Science and Engineering, Beihang University, Beijing 100191, China
[b] Mechanical and Materials Engineering, University of Nebraska-Lincoln, Lincoln, NE 68588, USA

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ABSTRACT

Non-equiatomic $Cr_{15}Fe_{20}Co_{35}Ni_{20}Mo_{10}$ (Mo_{10}) high-entropy alloys (HEAs) are produced by arc-melting and subsequent thermal-mechanical processing. The single-phase Mo_{10} HEAs with face centered cubic (FCC) structure exhibit an exceptional ductility and strain hardening capability associated with twinning. Tailoring thermal-mechanical processing condition enables the evolvement of single-phase Mo_{10} HEAs to multiphase microstructure that comprises simple FCC solid solution and nanoscale precipitates of Mo-rich μ phase. These precipitates primarily grow at grain boundaries and twin boundaries, strengthening Mo_{10} HEAs. This work sheds light on realizing a superb strength-ductility combination of HEAs by coupling solid solution hardening, nanotwinning and precipitation hardening mechanisms.

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1. Introduction

High-entropy alloys (HEAs) that are composed of five or more elements in an equiatomic or near-equiatomic composition have attracted increasing attentions because of their unique compositions, microstructures, and adjustable properties [1–6]. According to the structure (simple/complex) and ordering (ordered/disordered) of phases, HEAs are classified into three types: simple disordered phase, simple ordered phase and complex ordered phase [3]. Simple structures are the most frequently seen in as-cast HEAs, and generally refer to solid solutions of simple face-centered cubic (FCC), body-centered cubic (BCC), or hexagonal close-packed (HCP) structures [7–10]. Besides simple phases, different kinds of complex ordered phases, such as σ, μ, Laves, etc., are also observed in HEAs [11–14]. These complex ordered phases strengthen HEAs while often decrease their ductility. However, it has been demonstrated experimentally that manipulating the shape, dimension and distribution of strengthening units, e.g. hard and brittle particles, fibers, and precipitates, can optimize the strength and ductility of structural materials [15–18]. For example, nanoscale precipitate or lamina can plastically co-deform with matrix associated with dislocations transmission across the strengthening unit [17,18].

HEAs with single phase FCC structure generally exhibit low yield strength but superb ductility and strain hardening capability [1,2]. Plastic deformation of FCC HEAs (for example of Co-Cr-Fe-Ni system plus one or two other elements) is accommodated by slip of 1/2 ⟨110⟩ type dislocations on {111} planes and/or twinning associated with successive gliding of 1/6 ⟨112⟩ type dislocations on {111} planes [19,20]. Deformation twinning generates twin boundaries that block dislocation motion and further strengthen materials (referred to as twin boundary strengthening mechanism) [21,22]. Besides boundary strengthening associated with grain refinement and twinning, FCC HEAs can be strengthened through adjusting elements and their composition ratio corresponding to solid solution hardening mechanism [23,24] and precipitation hardening mechanism [25]. In this work, we aim to optimize strengthen and ductility of Cr-Fe-Co-Ni-Mo HEAs by coupling the three strengthening mechanisms.

We fabricate a series of non-equiatomic $Cr_{20-x}Fe_{20}Co_{20+3x}Ni_{20}Mo_{20-2x}$ HEAs. The Cr-Fe-Co-Ni-Mo system is chosen for this study because Cr-Fe-Co-Ni HEAs are FCC phase solid solutions [26,27]. Although the role of each element in developing microstructure and changing mechanical properties of HEAs is not understood comprehensively, previous study of Cr-Fe-Co-Ni system provides valuable guidance of designing the composition ratio of Cr-Fe-Co-Ni HEAs [28,29,30]. For example, equiatomic CoCrNi alloys [28,29] that have low stacking fault energy exhibit superior mechanical properties (good ductility and toughness) compared to equimolar CoCrFeMnNi due to the earlier onset of twinning [31,32]. Therefore, adjusting Cr concentration may achieve low stacking fault energy of {111} planes and promote twin boundary strengthening effect. To promote solid solution hardening and precipitation hardening effects, we simultaneously adjust the concentration of Mo and Co elements. Lattice distortion due to size misfit of atoms impedes dislocation movement and leads to the pronounced solid solution strengthening. For example, increasing concentration of Al (larger radius than others) in $Al_xCrFeCoNi$ HEAs significantly increases yield and flow strength of the HEAs [33]. Among the five elements in Cr-Fe-Co-Ni-Mo system, Mo has the largest metallic radius (Ni (0.124 nm), Co (0.125 nm), Fe (0.126 nm), Cr (0.128 nm), Mo

* Corresponding authors.
E-mail addresses: bixf@buaa.edu.cn (X. Bi), jianwang@unl.edu (J. Wang).

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(0.139 nm)), and thus potentially maximizes solid solution strengthening. In addition, Mo has been demonstrated to facilitate formation of hard and brittle intermetallic phases in AlCoCrFeNiMo and AlCrFeNiMo alloys [34,35]. Interestingly, increasing the concentration of Co may inhibit the formation of intermetallic phases in $(FeNiCrMn)_{(100-x)}Co_x$ HEAs [36]. Therefore, we hypothesize that optimizing the composition ratio of Cr, Co and Mo simultaneously may develop $Cr_{20-x}Fe_{20}Co_{20+3x}Ni_{20}Mo_{20-2x}$ HEAs with superb mechanical properties.

2. Experimental methods

Alloy ingots with nominal compositions of $Cr_{20-x}Fe_{20}Co_{20+3x}Ni_{20}Mo_{20-2x}$ (x = 0, 2.5, 5, 7.5 at.%) are prepared by arc-melting under an argon atmosphere, subsequently homogenized in vacuum at 1200 °C for 48 h and then cooled in the furnace. Purity of the raw materials is greater than 99.9%. The synthesized HEAs are referred to as Homogenized-HEAs. The homogenized HEA ingots are further hot-rolled at 1100 °C to the sheet with a thickness of 8 mm, then cold-rolled to 2.4 mm with a thickness reduction of 70%, followed by annealing at various conditions, 800 °C for 5 min then water quenching (WQ), 800 °C for 1 h then air cooling (AC), 850 °C for 5 min then water quenching, 900 °C and 1000 °C for 5 min then water quenching, 1000 °C and 1150 °C for 1 h then air cooling. The HEAs that are treated through such thermal-mechanical processing and annealing are referred to as Recrystallized-HEAs. Tension tests are conducted using an INSTRON-8801 instrument at room temperature with an engineering strain rate of 10^{-3} s^{-1}. Dog-bone-shaped tensile specimens with a gauge length of 18 mm and width of 3 mm are machined from homogenized and recrystallized sheets by electric discharge machining.

The phase constitution of the alloys is examined by X-ray diffraction (XRD). Microstructure characterization is performed by optical microscope (OM) and JEM 2100F transmission electron microscopy (TEM) coupled with energy dispersive spectroscopy (EDS). OM specimens are initially polished to 5000-grit SiC paper and, subsequently, carefully polished down to 1.5 μm diamond polishing compound, and etched with a solution that is composing of supersaturated copper sulfate, hydrochloric acid and alcohol. Two surfaces of tensile specimens are carefully polished with SiC paper, leading to a final specimen thickness of 1.5 mm. Specimens for TEM observation are thinned using mechanical grinding followed by double-jet electropolishing in a solution of 6% perchloric acid, 35% n-butyl alcohol and 59% alcohol at −30 °C and an applied voltage of 30 V.

3. Results

3.1. Mechanical properties

Tension testing shows that homogenized $Cr_{20-x}Fe_{20}Co_{20+3x}Ni_{20}Mo_{20-2x}$ alloys as x = 0 at.% and 2.5 at.% (referred to as Mo_{20} and Mo_{15} HEAs, respectively) are extremely brittle without apparent plasticity. The XRD patterns and optical micrographs reveal that Mo_{20} and Mo_{15} HEAs contain σ phase networks in FCC matrix (Fig. S1 in Supplementary information), which is consistent with previous work [37,38]. With decreasing the concentration of Mo, homogenized Mo_{10} and Mo_5 HEAs (corresponding to x = 5 at.% and 7.5 at.%) display good ductility. Fracture occurs at tension strain of 21.9% for Mo_{10} and 55% for Mo_5. However, homogenized Mo_{10} and Mo_5 HEAs display a low yield and ultimate strength, as shown in Fig. 1a and b. The XRD patterns

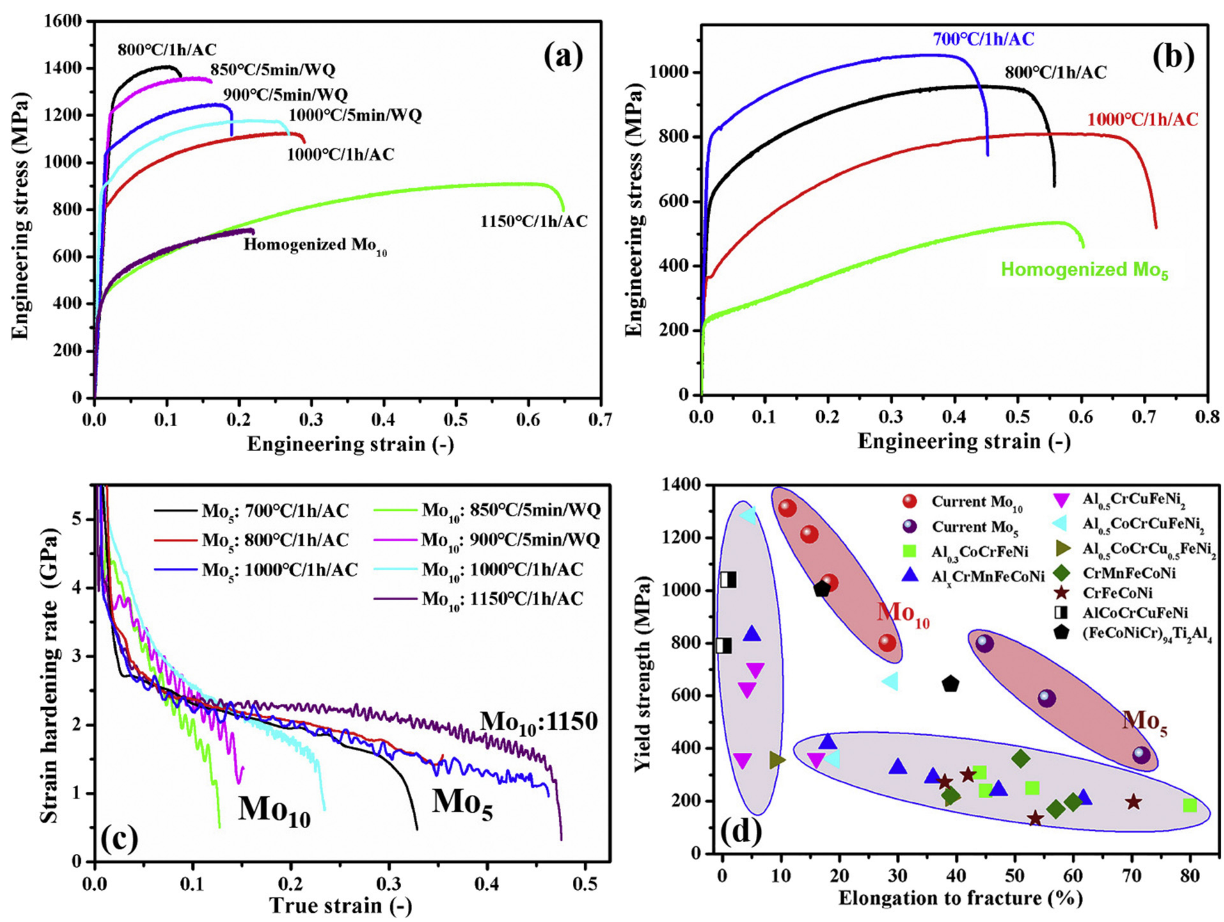

Fig. 1. Representative room-temperature engineering stress-strain curves for the (a) homogenized and recrystallized Mo_{10} alloys and (b) Mo_5 alloys. (c) Variation of strain hardening rates of Mo_{10} and Mo_5 alloys with plastic strain. (d) Yield strength versus elongation to fracture of current Mo_{10} and Mo_5 alloys compared with various HEAs.

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