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Microstructural features and tensile behaviors of a novel FeMnCoCr high entropy alloys

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ABSTRACT

C and Si-doped FeMnCoCr high entropy alloys (HEAs) with grain size of approximate 200 μm were prepared. Quasi-in-situ EBSD was used to characterize the microstructural evolution under different tensile strains. The tensile strength of the HEAs is about 670 MPa and the fracture elongation is 47%. The single-phase FCC structure was maintained during the entire tensile procedure, whereas the deformation mechanism was found to change from dislocation slip to twinning under higher strains. Moreover, it was observed that twins are inclined to form within grains with ⟨1 1 1⟩ texture.

1. Introduction

The design of high entropy alloys (HEAs) with high strength and plasticity combination is one of key focuses in the research of structural materials [1]. Among high-entropy alloys reported so far, FeMnCoCr alloys possess excellent mechanical properties [2]. The investigation of $Fe_{80-X}Mn_XCo_{10}Cr_{10}$ HEAs showed that stacking fault energy (SFE) could be tailored by adjusting the Mn content, so as to trigger transformation-induced plasticity (TRIP) or twin-induced plasticity (TWIP) in the process of deformation to improve toughness [3]. On the other hand, the incorporation of C, Si or Al in Fe-Mn TWIP steel can bring about interstitial solution and nano-twinning simultaneously, leading to remarkable strain-hardening [4]. Such design strategy was recently adopted, for example, by micro-alloying, to improve the mechanical performances of FeMnCoCr HEAs [5]. In contrast to single-element solid solution approach, little attention has been paid to dual-elements solid solution high-entropy alloy (De-HEA) [6]. Moreover, the addition of distinct non-metallic elements can not only ameliorate the strength, but also modify the SFE, stabilize the alloy structure and hinder the phase transformation [5]. Therefore, it is important to understand the active deformation mechanisms and their interactions in the quest of high performance De-HEA.

To address these issues, C and Si containing FeMnCoCr high entropy alloy was designed and prepared, according to semi-empirical criterion used for the prediction of single phase HEAs. Quasi-in-situ EBSD was utilized to investigate the tensile deformation behaviors of the new alloy, through which the relationship between deformation twinning and grain orientation was established.

2. Experimental

C and Si containing FeMnCoCr high-entropy alloy ingots were manufactured by vacuum induction melting technique. The purity of raw materials used are higher than 99.9%. Based on previous research results [7], intermetallic compounds would form if the carbon content is more than 1%. Therefore, the combined addition of C and Si was controlled at less than 1%. The alloy composition was determined to be C 0.46%, Si 0.44%, Mn 28.1%, Co 8.8%, Cr 8.6%, and Fe 53.6%. For crystal structure identification, X-ray diffractometer (XRD, Bruker D8) was employed in the range of 40-100°, with Cu Kα radiation. The microstructure and distribution of chemical elements and grain orientation were analyzed by field emission scanning electron microscopy (FE-SEM, FEI Sirion), equipped with energy dispersive spectroscopy (EDS). The quasi-

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in-situ electron backscatter diffraction (EBSD) observation is based on special tensile mold and precise selective positioning method. Microstructure characterization was also performed by transmission electron microscopy (TEM, FEI Tecnai G2), operating at 200 kV accelerating voltage. The hardness of the new alloy was examined with nano-indentation (Agilent Nano indenter, G200). Dog bone-shaped tensile specimens were prepared with a gauge geometry of 20 mm × 2 mm × 1.5 mm. Tensile tests were carried out at room temperature [8], with the stain rate kept at 1×10^{-3} s^{-1}.

3. Results and discussion

As-cast De-HEA shows a dendrite structure with visible directionality (Fig. 1a). After homogenization treatment, single phase FCC solid solution structure was identified (Fig. 1b), the lattice constant is about 3.615 Å. C and Si are thus completely dissolved in the matrix. EDS mapping indicates that all constituting elements are uniformly distributed without segregation (Fig. 1c). Therefore, the new alloy is different from DP-TRIP-HEA ($Fe_{50}Mn_{30}Co_{10}Cr_{10}$) alloy, whose structure is composed of FCC and HCP [3].

As SFE has a strong effect on the deformation mechanism of the alloy, the intrinsic stacking fault energy (γ_{int}) of De-HEA was calculated as follows [9]:

$$\gamma_{int} = 2\rho\Delta G^{\gamma\to\varepsilon} + 2\Delta\sigma^{\gamma\to\varepsilon}$$

where $\Delta G^{\gamma\to\varepsilon}$ is the molar free energy difference between the γ-FCC and ε-HCP phase (-201.5 mJ/m^2) [9], $\Delta\sigma^{\gamma\to\varepsilon}$ is the coherent interfacial energy between these two phases (15 mJ/m^2) [10], $\rho = \frac{4}{\sqrt{3}\alpha^2}\frac{1}{N}$ is the planar packing density of the close-packed γ-{1 1 1} plane based on the lattice constant of γ-FCC phase (α=3.615 Å) and N represents Avogadro's number (N = 6.02 × 10^{23})-. The intrinsic stacking fault energy of the FeMnCoCr was calculated and found to be 18.17 mJ/m^2, slightly above the upper limit of SFE required for the activation of phase transformation (10–18 mJ/m^2) [11]. The addition of C and Si appears to be responsible for the increases of the SFE, which helped to stabilize the structure of the as-cast HEA. Consequently, the TRIP would be suppressed, while TWIP is embraced during deformation.

Quasi-in-situ EBSD was utilized to analyze the deformation mechanism under different tensile strains. When the tensile strain was 5% (Fig. 2a), there was almost no change to the microstructure. A few twins were detected locally, as the strain reached to 15% (Fig. 2b). With the increase of tensile strain, the grains were stretched, refined and rotated, and the number of twins continued to increase. Besides, it can be found that the twins preferentially generate in the grains with ⟨1 1 1⟩ and ⟨1 1 0⟩ orientation, and gradually expands to equiaxed grain boundaries with the increase of deformation, as shown in Fig. 2c (white arrow indicates the direction of expansion). As the stretching continues, the grains break and rotate. From the white frame in Fig. 2d, it can be demonstrated that the grains with ⟨1 1 1⟩ orientation obtain higher density of twins, indicating that the twins have obvious preferred orientation [12,13]. Bruno et al. [5] reached similar conclusions and believed that the preferred orientation of twinning was influenced by Schmid factors. In the grain with ⟨1 0 0⟩ orientation

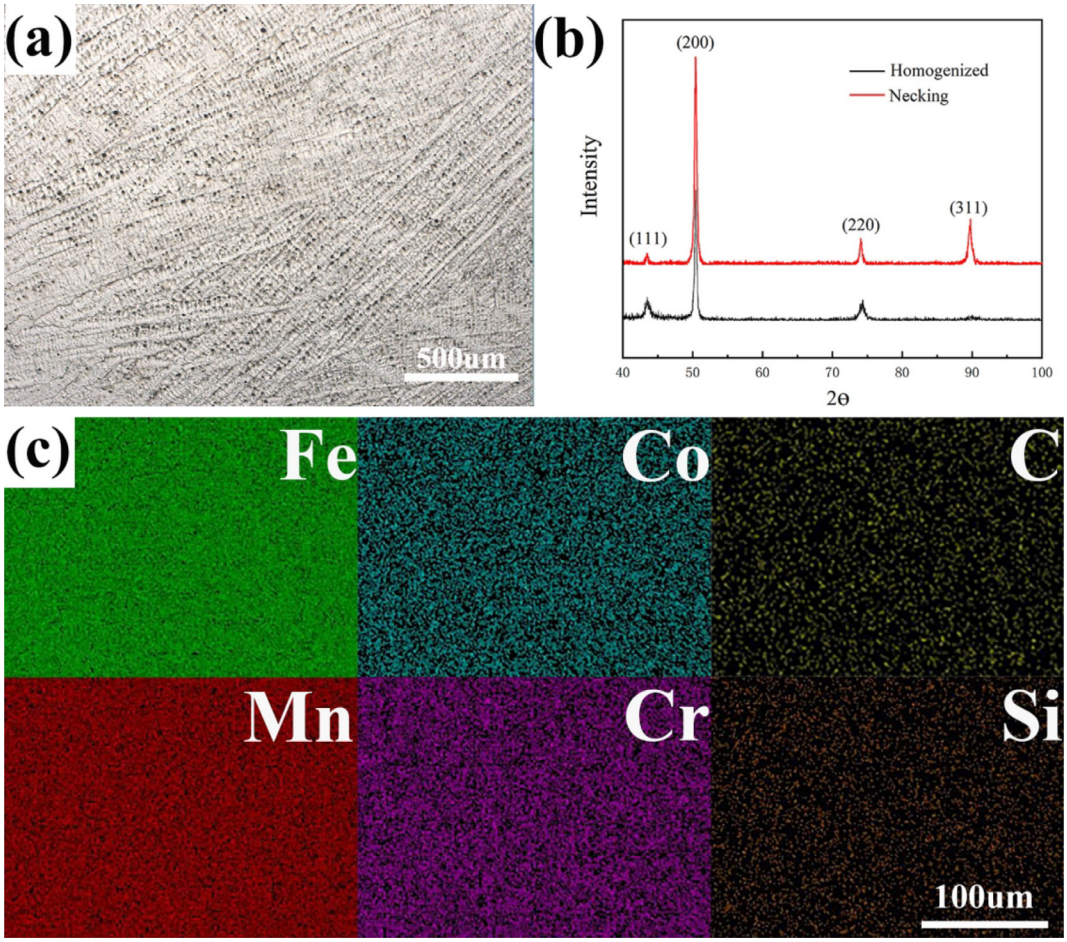

Fig.1. (a) The as-cast alloy microstructure, (b) XRD pattern of homogenized and fractured HEA, (c) EDS mapping of elements in the alloy.

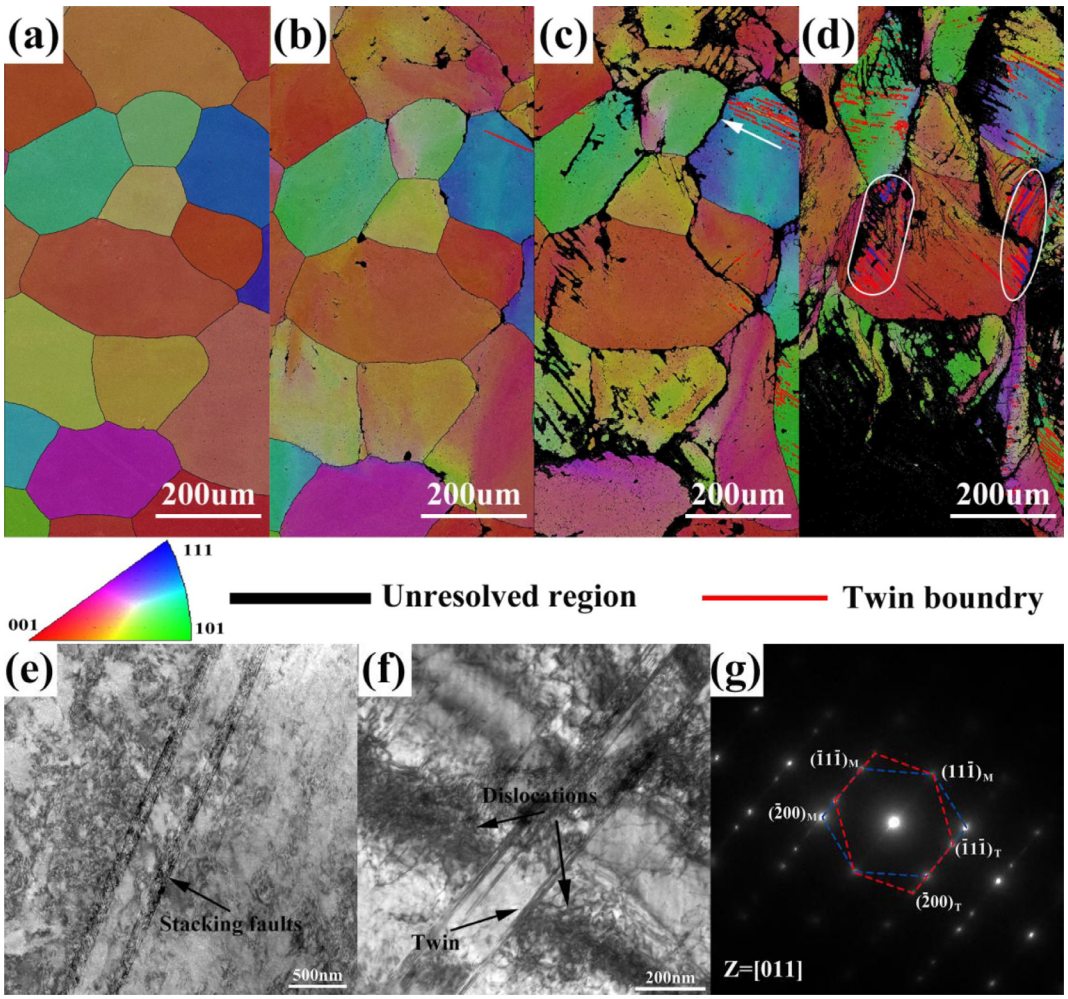

Fig. 2. Grain orientation and twin evolution of (a) tensile strain of 5%, (b) strain of 15%, (c) strain of 30%, (d) necking; and TEM of (e) stacking fault under 5% strain, (f) twinning under 15% strain, and (g) selected electron diffraction of twins.

dislocation glide is favored relative to twinning, as the Schmid factors for twinning and glide are 0.23 and 0.41, respectively. The black area in the figure represents the unresolved region, and the decrease in resolution may result from the gradual accumulation of dislocations at the grain boundaries and surface embossing.

The microstructural evolution of alloys was characterized by TEM. As shown in Fig. 2e, a small amount of stacking faults formed at 5% strain, and these stacking faults are supposed to act as precursors for the formation of deformation twins. Twins were not found until 15% of the tensile strain (Fig. 2f), revealed by select area electron diffraction (SAED). Critically, twins were observed to block dislocation slips (signaled by black arrows), thus improving the work hardening.

Fig. 3a shows the mechanical behavior of the De-HEA. The ultimate tensile strength of the alloy is 670 MPa, the fracture elongation is close to 50% and the elastic modulus is 208 GPa. Nano-indentation test (Fig. 3b) shows that the hardness of the alloy reaches 4.6 ± 0.1 GPa. The strain-hardening rate curve derived from the true stress-strain plot is shown in Fig. 3c. The strain-hardening rate is defined as the ratio of true stress-strain at a constant strain rate [14]. Accordingly, the deformation process can be divided into three regimes. In the first stage(Ⅰ), the strain-hardening decreases monotonously, and the deformation is believed to be controlled mainly by dislocation motion and entanglement; Moving into the second stage(Ⅱ), the strain-hardening begins to rise, in which the deformation process may change from dislocation slip to twinning, and twin interfaces play a large role in the hardening of the materials [15]; advancing into the last stage (Ⅲ), the hardening rate decreases rapidly again, wherein the twin growth saturates, and the dislocation motion again controls the deformation until necking occurs. Even after large tensile strain, the alloy structure still displays a single-phase FCC structure, as shown in XRD of Fig. 1b.

The tensile strength versus the elongation of various alloys obtained at room temperature is shown in Fig. 3d. Due to the additive strengthening effect of solid solution and TWIP, the De-HEA, even in coarse-grained state, achieve remarkable mechanical properties. Therefore, the newly developed De-HEA has potential for load-carrying applications where safety is a critical concern.

4. Conclusion

The addition of C and Si can enhance the structural stability of FeMnCoCr HEA by increasing the SFE. No phase transformation was observed during the tensile loading. The deformation mechanism was changed from the dislocation slip to twinning under increasing strain. Twins have a preferred orientation and are inclined to form in grains with ⟨1 1 1⟩ texture. The combination of high strength and plasticity is the result of the synergy of solid solution strengthening and twinning induced plasticity.

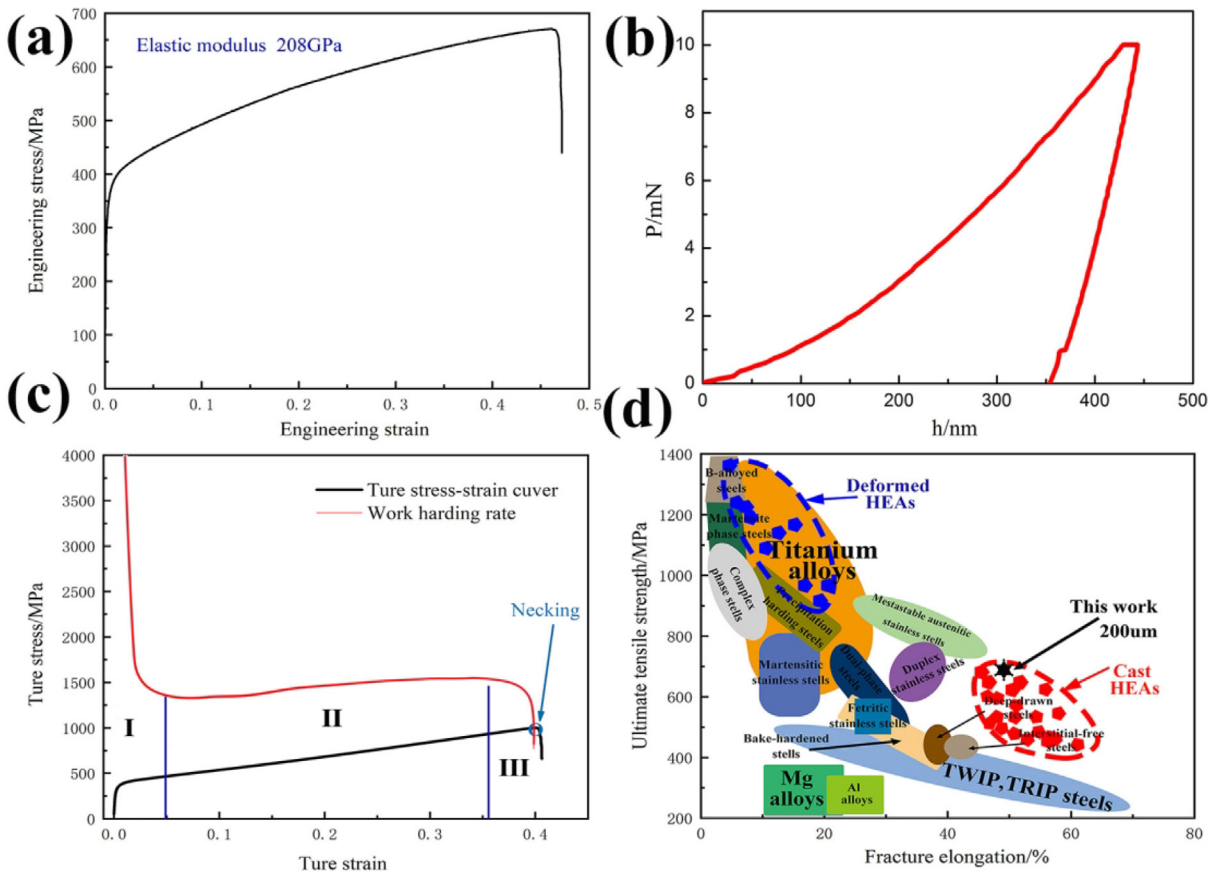

Fig. 3. (a) Engineering stress-strain curve, (b) Nano-indentation load-displacement curve, (c) Work hardening rate and true stress-strain curve, (d) Comparison of the strength versus elongation of various alloys at room temperature.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matlet.2020.128154.

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