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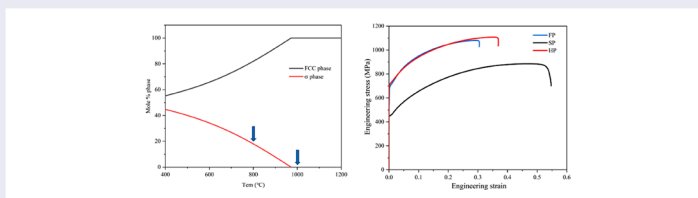
Enhanced strength and ductility in $(\text{CoCrNi})_{92}\text{Mo}_2\text{Al}_6$ medium entropy alloy via dual heterogeneous structures

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ABSTRACT

In the present $(\text{CoCrNi})_{92}\text{Mo}_2\text{Al}_6$ medium entropy alloy (MEA), a dual heterogeneous structure is achieved by regulating the degree of recrystallization of the FCC matrix and σ phase precipitation through incomplete annealing. Compared to the alloys with single-phase FCC structure or uniform precipitation structure, this dual heterostructured MEA has superior mechanical properties by possessing the hetero-deformation-induced (HDI) hardening, precipitation strengthening and twinning-induced plasticity (TWIP) mechanism. Our research contributes to the understanding of the micromechanical behavior of heterogeneous materials and also provides a solution to the problem that precipitation increases strength at the expense of plasticity.



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1. Introduction

High-/medium-entropy alloys (HEAs/MEAs), initially conceptualized by Yeh and Cantor et al., are of great interest currently in the material community due to their unique metallurgy philosophies as well as exceptional mechanical and functional attributes [1–6]. The properties of HEAs/MEAs are derived not from a dominant constituent but rather from multiple principal elements, thus presenting great potential for the unique combination of mechanical response compared with conventional alloys, such as superior ultimate strength, good ductility, damage tolerance at cryogenic temperature, fatigue properties and wear resistance [7–10]. However, due to the intrinsic character of the face-centered cubic (FCC) structure, the tensile yield strength of single-phase FCC HEAs cannot deliver desirable strength for engineering applications. Although many mechanisms [11–16] have been imposed to strengthen FCC HEAs, they usually inevitably sacrifice the plasticity of alloys.

Many studies found that the introduction of heterostructures in HEAs can provide excellent strength and plasticity balancing [17–22]. The heterostructured materials achieve unprecedented performances by controlling defect distributions, long-range internal stresses and nonlinear inter-zone interactions [23]. Benefiting from the huge composition space, multi-component HEAs get advantages in regulating the formation of heterogeneous microstructures compared with traditional alloys. There have been many reports on heterogeneous structures in HEAs, such as twinning or/and martensitic transformation, precipitation strengthening, multiphase eutectic and grain bimodal distribution [17,18,24,25]. The heterostructure obtained by regulating the inhomogeneous distribution of precipitates is an effective strategy to achieve synergistic strengthening and toughening in HEAs. For instance, Ruobin Chang et al. [26] found that the heterogeneous structure with banded precipitates caused by Mo inhomogeneity can lead to an excellent

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strength-ductility combination in the $(\text{CoCrNi})_{93}\text{Mo}_7$ alloy. Although chemical inhomogeneity is a prevalent issue in HEAs that is associated with their complex composition and the sluggish diffusion of multiple principal elements [26,27], it is not a controllable means to regulate the distribution of the precipitates.

In the present work, with the help of thermodynamic calculations, the dual heterostructure, featuring both grain size and σ phase precipitation gradient distribution, was achieved in a $(\text{CoCrNi})_{92}\text{Mo}_2\text{Al}_6$ MEA by thermomechanical treatment. Compared with $(\text{CoCrNi})_{93}\text{Mo}_7$ MEA, the reduction of Mo element and the addition of Al elements showed advantages in reducing the density and cost of the alloy. This component also lowers the precipitation temperature (1220°C – 980°C) of the σ phase which facilitates a more controlled manipulation of the alloy's microstructure. This dual heterostructure introduces the hetero-deformation-induced (HDI) hardening and other strengthening mechanisms, enabling the studied alloy to realize an excellent combination of strength and ductility.

2. Materials and experimental methods

The investigated MEA with a nominal composition of $(\text{CoCrNi})_{92}\text{Mo}_2\text{Al}_6$ and approximate masses of 50 g were prepared from commercially obtained raw materials (99.9 wt.% pure) in an arc melting furnace with a water-cooled copper plate in an Ar atmosphere. The master ingots were then drop-casted into a copper mold with a dimension of $10 \times 10 \times 60 \text{ mm}^3$, and subsequently tube-sealed and homogenized at 1200°C for 48 h. Such rectangular slab ingots were then cold-rolled with a total thickness reduction of 75%. Three heat treatment processes were adopted on the rolled specimens: annealing at 800°C for 60 minutes, denoted as FP; annealing at 800°C for 10 minutes followed by annealing at 1000°C for 3 minutes, denoted as HP; annealing at 1000°C for 3 minutes, denoted as SP.

Vickers microhardness, HV, was measured on the polished cross-section surfaces under a 200 g load applied for 12 s. Tensile tests were performed using a mechanical testing machine (Instron-5966) at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ at room temperature. The flat dog-bone specimens, with gauge dimensions of $3.1 \times 1.0 \times 10.2 \text{ mm}$, were cut from the sheets with the longitudinal axes parallel to the rolling direction through electrical discharge machining. To estimate the HDI strengthening, the loading-unloading-reloading tests [20] were conducted at a constant strain rate of 10^{-3} s^{-1} and a constant unloading rate of 2000 N/min. The phases in the samples were identified using a Rigaku SmartLab

X-ray diffractometer (XRD). The crystallographic features were characterized by the electron backscattered diffraction (EBSD) system equipped with a Zeiss Super 55 scanning electron microscope (SEM). The EBSD samples were prepared by mechanical and electrolytic polishing. A Tecnai G2 F20 transmission electron microscopy (TEM) was used to observe the fine microstructural features of the studied HEAs. The TEM samples were primarily ground to about $50 \mu\text{m}$ in thickness using SiC paper, then punched to $\phi 3 \text{ mm}$ discs and finally electropolished in an electrolyte consisting of 10 vol% HClO_4 and 90 vol% CH_3OH solutions under 25 V at -30°C .

3. Results and discussion

Figure 1(a) shows the computational phase diagram of $(\text{CoCrNi})_{92}\text{Mo}_2\text{Al}_6$ MEA. The annealing temperatures of 800°C and 1000°C are selected for tuning the σ phase precipitation. Figure 1(b) shows the kernel average misorientation (KAM) map after annealing at 800°C for 10 minutes, where the blue regions indicate recrystallized areas and the green regions indicate non-recrystallized areas. The inverse pole figure (IPF) map shown in Figure 1(c) illustrates the recrystallized zones characterized by exceedingly a fine grain structure and a plethora of unidentified submicron precipitates. The TEM-EDS elemental analysis outcomes, as shown in Figure 1(d), disclose that the precipitates in Figure 1(b, c) are enriched in Cr, Co and Mo elements. Combining the TEM results and subsequent XRD characterization, it is determined that the precipitates are σ phase. The partial precipitation associated with partial recrystallization is common in incomplete annealed alloys [28,29]. During the annealing process, the development of recrystallization and precipitation is correlated with the localized dislocation density. Regions with higher dislocation density can serve as possible nucleation sites for recrystallization and precipitation [30–32]. In the studied MEA, regions with high dislocation density undergo preferential recrystallization and precipitation during incomplete annealing, forming a gradient structure with precipitation occurring only in the recrystallized areas. According to the precipitation temperature range of σ phase in Figure 1(a), the fully recrystallized and partially recrystallized matrix accompanied by different degrees of precipitation can be achieved by single or double annealing at 800°C and 1000°C for varying periods.

The XRD patterns and SEM images of the SP, HP and FP MEAs are shown in Figure 2. The XRD analysis (Figure 2(a)) reveals that both the HP and FP alloys comprise the FCC phase and σ phase, whereas the SP alloy exclusively contains the FCC phase. The intensity of the σ phase's diffraction peaks indicates the absence

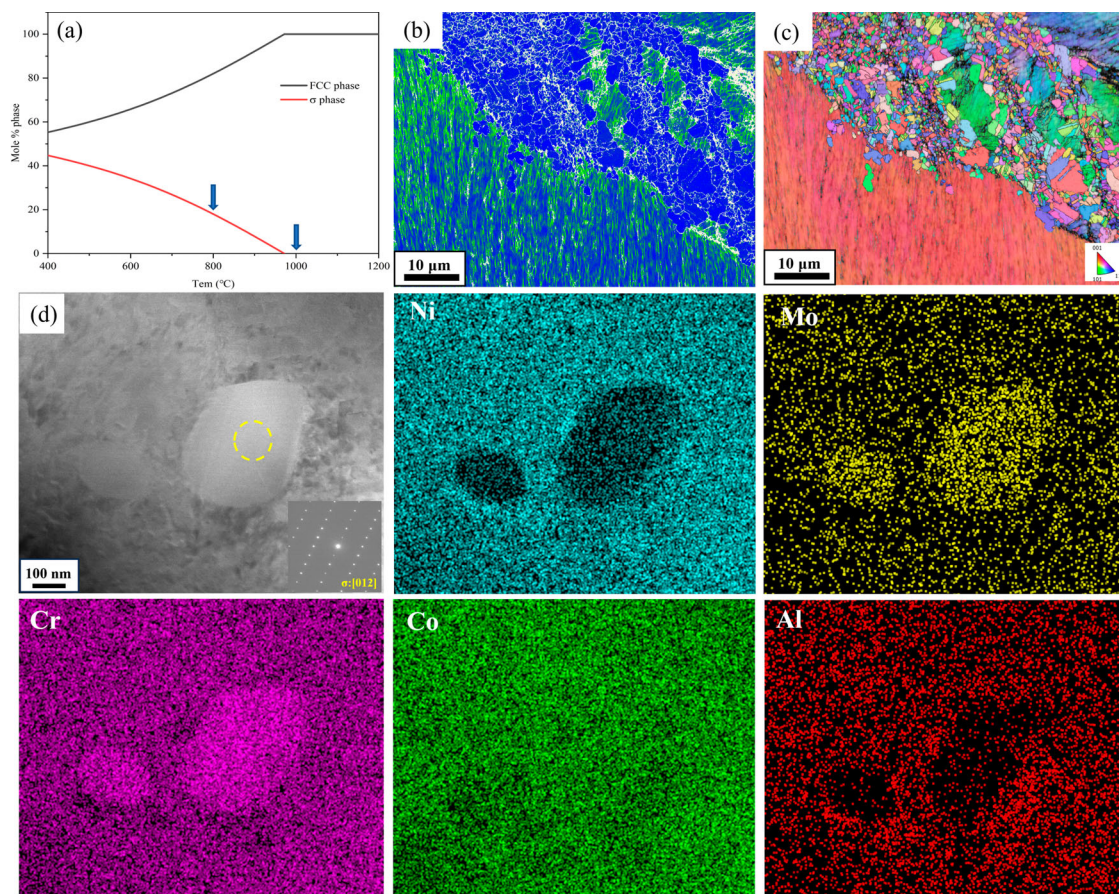


Figure 1. Construction of the dual heterostructure. (a) The computational phase diagram of $(\text{CoCrNi})_{92}\text{Mo}_2\text{Al}_6$ MEA; The KAM (b) and IPF (c) image of the partially recrystallized MEA showing the heterogeneous grain size; (d) TEM characterization and corresponding EDS mapping of the σ phase in the partially recrystallized MEA.

in SP alloy, with the highest σ phase content in the FP alloy and the intermediate amount in the HP alloy. The SEM images corroborate the different σ phase contents in these alloys. The FP alloy is characterized by abundant σ phase precipitation (Figure 2(b)) with uniformly dispersed distribution. The volume fraction of the σ phases in FP is 10%. Uniform precipitation in Figure 2(b) indicates that the gradient precipitation in Figure 1 may be independent of the uneven distribution of elements. In contrast to the FP alloy, the SP alloy presents a single-phase structure (Figure 2(c)) with an equiaxed grain size of $16 \pm 1.6 \mu\text{m}$. The structure of the SP alloy shows that σ precipitation can be completely inhibited when annealing at 1000°C . The σ phase in the HP alloy displays uneven precipitation, as evidenced in Figure 2(d) and Figure 2(e). The content of the σ phases in HP is 5%. The regions with precipitates achieve significantly smaller grain sizes. This attribute to the freshly precipitated σ phases hinders grain growth during recrystallization [33]. Figure 2(f) shows the EBSD orientation map of HP alloy, where the region 'A' with σ phase has the mean grain size of $3.9 \pm 3.3 \mu\text{m}$, while the average grain size of the region 'B'

without σ phase is $17.8 \pm 2.3 \mu\text{m}$. The above results show that the HP alloy contains a dual heterostructure of precipitation and grain size. The HP alloy can be divided into 'hard region' and 'soft region' and their microhardness is 573HV and 367HV, respectively.

Representative tensile engineering stress-strain curves for the SP, HP and FP MEAs are presented in Figure 3(a). The SP alloy without σ phase displays characteristics of typical single-phase FCC H/MEAs, with high ductility (55%) and strain hardening capability but lower yield strength (450 MPa). The FP alloy strengthened by σ precipitation exhibits a yield strength of 690 MPa and an ultimate tensile strength of 1080 MPa with ductility of 30%. Compared to the FP alloy, the HP alloy with fewer σ phases displays comprehensive superior tensile properties with a yield strength of 707 MPa and ultimate tensile strength of 1112 MPa, while maintaining a ductility of 37%.

The strain hardening rate curves of the studied MEAs are illustrated in Figure 3(b). At the initial stage of deformation, the strain hardening rate of FP alloy is the highest. This is due to the highest content of the σ phase

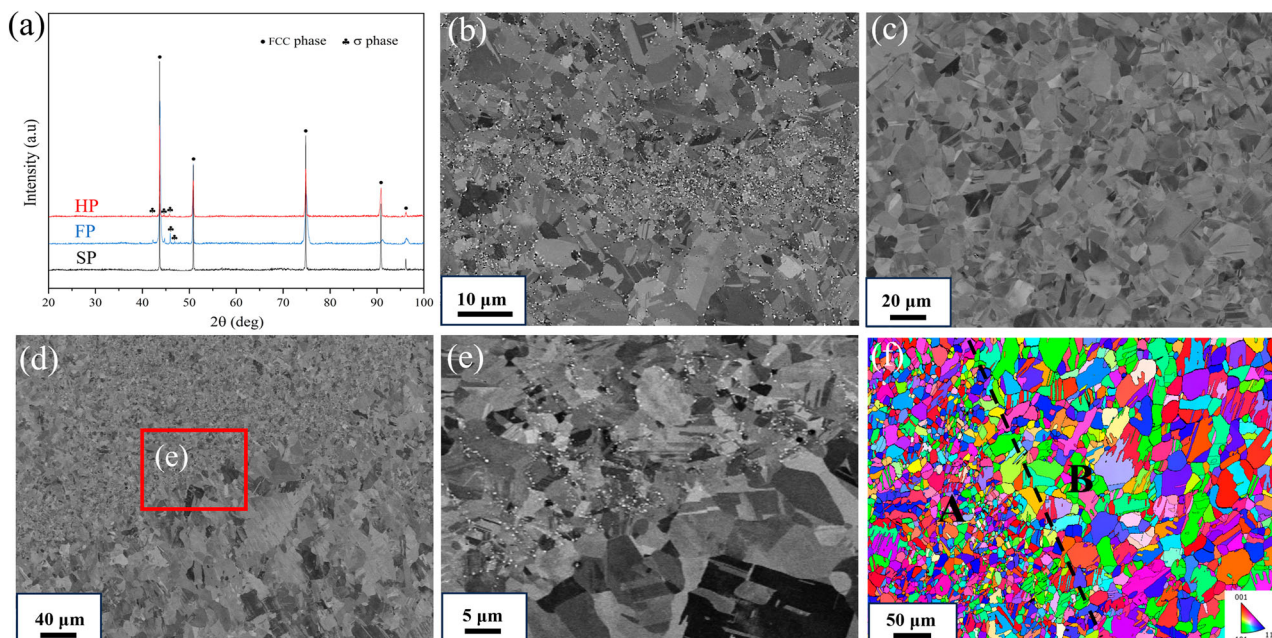


Figure 2. (a) XRD profile of the SP, HP and FP MEAs. SEM images of FP (b) SP (c) and HP (d, e). (f) EBSD orientation map of HP.

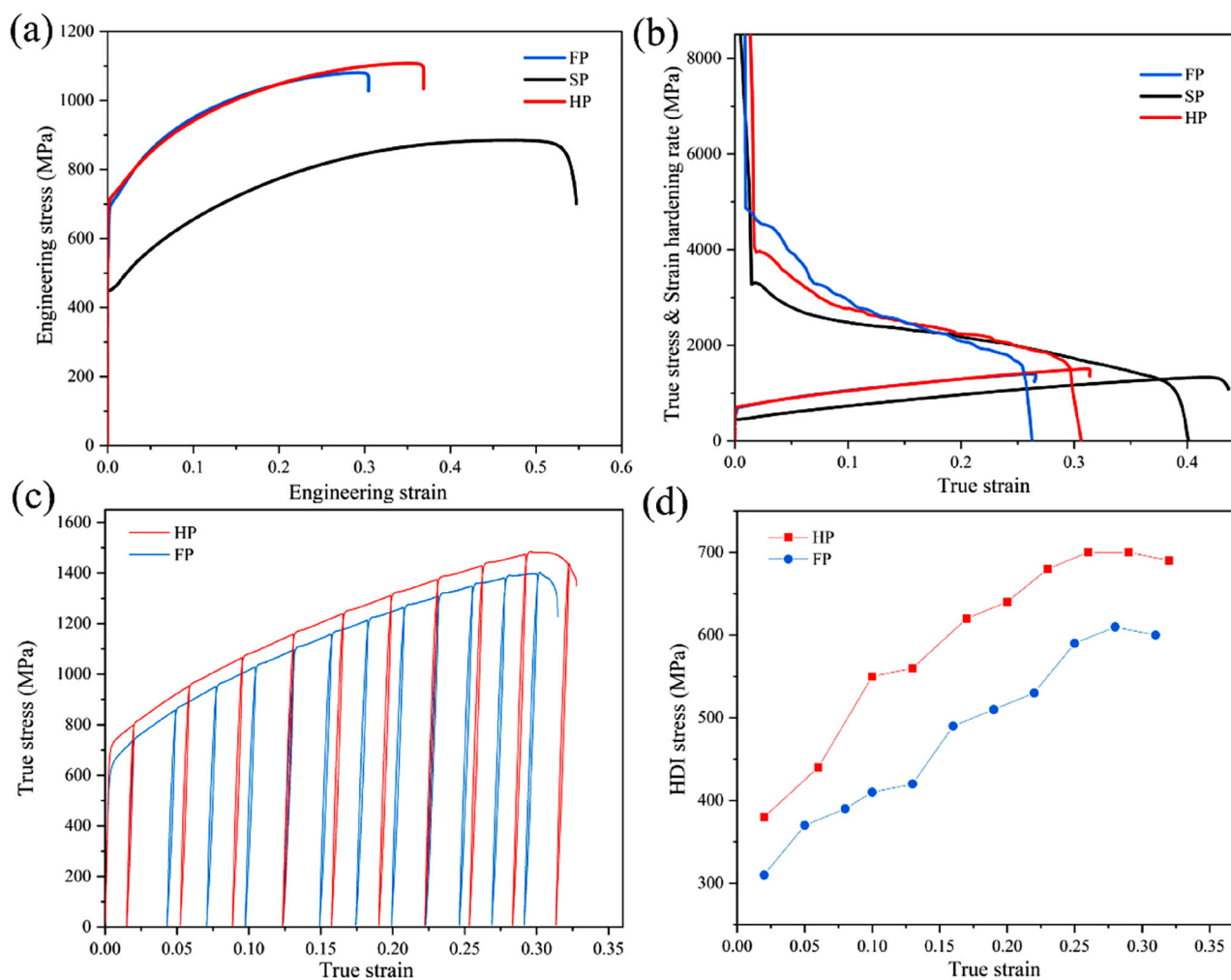


Figure 3. The engineering stress-strain curves (a) and work hardening rate curves (b) of FP, SP and HP MEAs. The LUR stress-strain curves (c) and evolutions of HDI stress with true strain (d) of FP and HP MEAs.

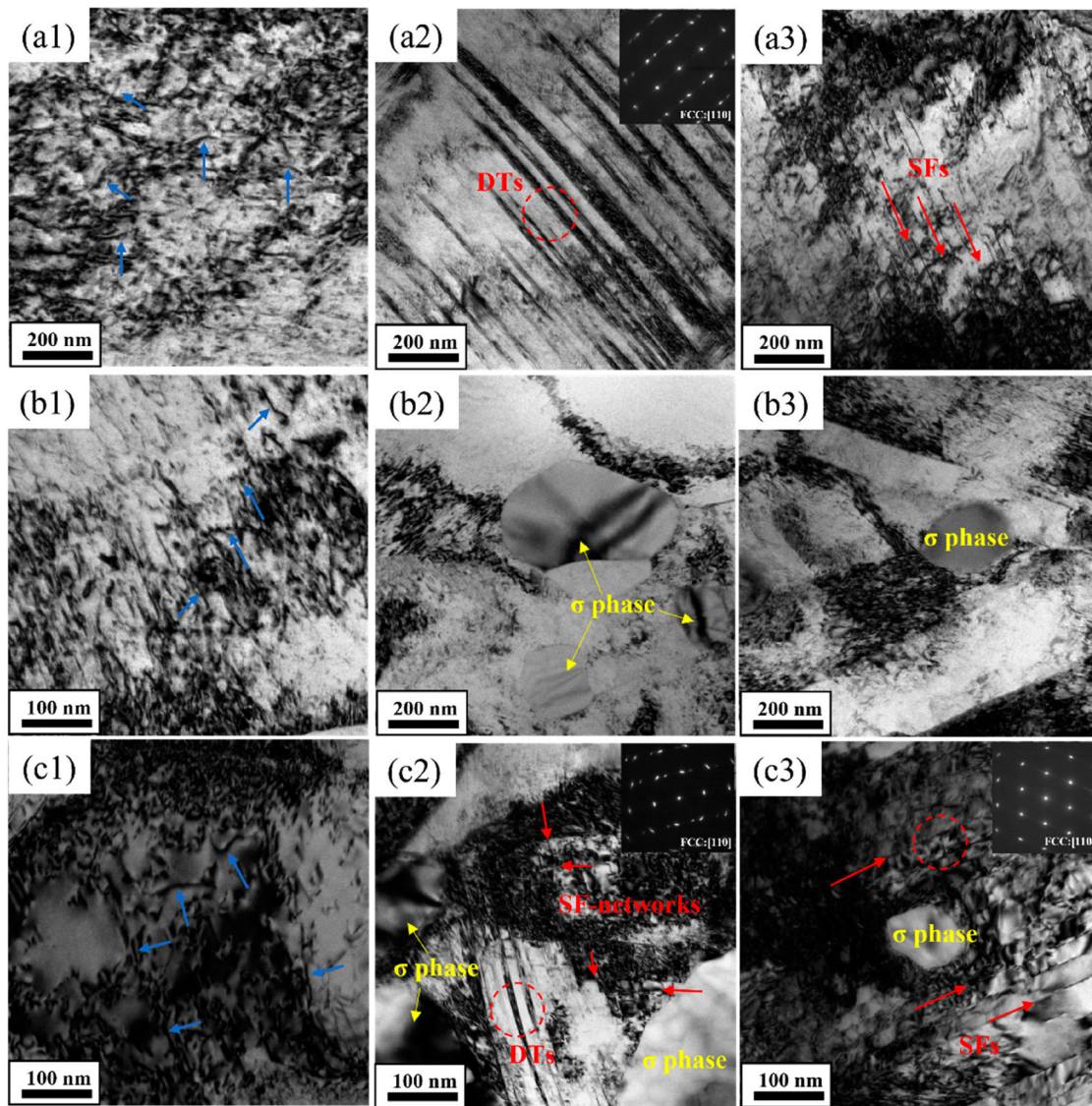


Figure 4. TEM image of typical microstructures of SP (a1–a3), FP (b1–b3) and HP (c1–c3) MEAs after tensile deformation.

in FP, where obstacles to dislocation glide from precipitates lead to a rapid increase in work hardening rate. In the later stages of deformation, the HP alloy has the highest work hardening rate, which may be attributed to deformation twinning (DT) contributions after reaching a certain degree of deformation [34,35]. Additionally, back stress strengthening from heterogeneous structures [23,36] is also a crucial factor for maintaining a high work hardening rate in HP alloys. The SP alloy with a single-phase FCC structure consistently displays lower strain hardening rates, while the HP alloy maintains a continuous high strain hardening rate and achieves the highest strength. The ultimate true stress of FP, HP and SP alloys is 1382, 1517 and 1340 MPa, respectively.

The distinctive dual heterostructure is expected to generate potent HDI strengthening and hardening, thereby facilitating the attainment of an outstanding

amalgamation of mechanical properties [20]. Loading-unloading-reloading (LUR) tensile tests are conducted to explore the underlying strain hardening mechanisms, as shown in Figure 3(c). The LUR curves reveal a hysteresis loop, indicating the presence of the Bauschinger effect [37]. The Bauschinger effect that occurs during cyclic tensile tests commonly originates from the back stress attributed to multiphases or multidomains with inhomogeneous plastic deformation [28]. The back stress developed in the ductile zone, in turn, generates a forward stress in the hard zone and the collective action of the back stress and forward stress culminates in the HDI strengthening and hardening [38]. As shown in Figure 3(d), the HDI stress can be calculated as $\sigma_b = (\sigma_u + \sigma_r)/2$, where σ_u and σ_r are the unloading yield stress and reloading yield stress, respectively [20]. During the yield stage, the back stress strengthening in

the HP alloy is approximately 80 MPa, which is higher than that in the FP alloy. According to the precipitation strengthening calculation method [39] ($X\sigma - YS = YS\sigma / YS = C \bullet f\sigma \bullet H\sigma / (C \bullet H0) = f\sigma \bullet H\sigma / H0$, where C is a constrained factor, $f\sigma$ is the measured fraction of the σ -phase, $H\sigma$ is the macroscopic hardness of the σ -phase (~ 8.125 GPa)), the precipitation strengthening in the HP alloy is about 70 MPa, lower than in the FP alloy. Therefore, with similar grain size and assuming consistent lattice friction stress and solid solution strengthening, the HP and FP alloys exhibit nearly the same yield strength. As strain increases, the stress contribution from the HDI strengthening rises progressively, reaching a peak of 710 MPa, significantly enhancing the strength of the HP alloy.

Figure 4 shows the TEM characterization images of MEAs after deformation. Massive cross-slip dislocations were observed in the SP MEA. The presence of Mo and Al elements induced significant lattice distortion is considered to lead to the transition from planar slip to cross-slip [12]. Substantial DTs and stacking faults (SFs) are also observed in the SP alloy (Figure 4(a2–a3)). DTs and SFs are effective mechanisms to improve the mechanical properties of alloys during deformation. The generation of a substantial number of DTs and SFs is attributed to the low stacking fault energy in CoCrNi-based H/MEAs [2,40,41]. These DTs and SFs facilitated the maintenance of a high strain hardening rate (Figure 3(b)) during the deformation process in the SP alloy, thereby enhancing plasticity.

Cross-slip also drives as the main slip mode of dislocations in the FP (Figure 4(b1)) and HP (Figure 4(c1)) MEAs. A large number of accumulated dislocations around the σ phase were also observed in both FP (Figure 4(b2–b3)) and HP (Figure 4(c2–c3)) MEAs. For the alloy with incoherent precipitates, dislocations typically have to adopt bypass mechanisms, such as the Orowan mechanism [42,43]. However, since this bypassing mechanism needs to overcome greater slip resistance, dislocations are easy to accumulate near the σ phase as the strain increases [42]. The primary distinction in deformation mechanisms between FP and HP MEAs lies in the notable DTs and SFs in the HP alloy, as shown in Figure 4(c2–c3). The stress concentration near the grain boundary and the precipitated phase interface is expected to promote the generation of DTs [44,45]. Rahman et al. [46] discussed the effect of the slip length scale on the critical twinning stress, revealing a Halle-Petch-type relationship to determine the grain size dependence on the critical twinning stress. In the FP MEA, the densely dispersed precipitates reduced the slip length and thereby increasing the critical twinning stress [47]. The heterogeneous distribution of the σ phase in the HP alloy can largely avoid this

inhibition effect. The ‘soft’ zones possess the same conditions for generating a large number of DTs as the SP alloy. Furthermore, due to the higher critical shear stress of DTs [48], the presence of non-uniform deformation-induced back stress in the ‘soft’ region can promote the generation of DTs. The generation of newly formed nanoparticles made the structure more inhomogeneous and generated high HDI stress during further deformation [28]. The synergistic effect of these mechanisms leads to the maintenance of a high strain hardening rate of the HP MEA during deformation.

4. Conclusions

By manipulating the annealing conditions, we have tuned a dual heterogeneous structure with inhomogeneous grain size and gradient σ phase precipitation in the (CoCrNi)₉₂Mo₂Al₆ MEA. The mechanical properties of this dual heterostructured MEA are superior to its counterparts with the same composition having a single-phase structure or uniform precipitation structure. A tensile yield strength of 720 MPa and ultimate tensile strength of 1120 MPa with a notable failure strain of about 37% is achieved by the synergistic effect of HDI strengthening and deformation twinning strengthening mechanism in the (CoCrNi)₉₂Mo₂Al₆ MEA. This dual heterostructure design provides a solution for the problem that the precipitation phase in the alloy usually improves the strength but sacrifices the plasticity.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability statement

Raw data were generated at Li. Derived data supporting the findings of this study are available from the corresponding author Tiancheng on request.

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