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Deformation Mechanisms of Mo alloyed FeCoCrNi High Entropy Alloy: *In Situ* Neutron Diffraction

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Abstract

A FeCoCrNiMo_{2.3} high entropy alloy was processed by powder metallurgy with two conditions: hot extruded and annealed. *In situ* neutron Diffraction, together with electron microscopy, was used to study the deformation mechanisms and concomitant microstructural evolution for both conditions. The as-extruded alloy has a single face-centered-cubic structure with a calculated stacking fault energy of ~ 19 mJ/m². When the alloy is tensile deformed, nano-twins and microbands are induced, resulting in an excellent combination of strength and ductility. Annealing at 800 °C for 72 h led to an increase of the strength of the alloy, but a decrease of the ductility. This is due to the decomposition of the alloy after annealing, causing the formation of Mo-rich intermetallic particles and a decrease of the stacking fault probability. These results highlight that combined mechanisms (i.e. solute strengthening and twin/microband induced plasticity) can effectively improve both the strength and ductility of high entropy alloys.

1. Introduction

The compelling need for high strength and ductile engineering alloys is driving the development of new concepts in alloy design. One intriguing new strategy is high-entropy alloys (HEA), which aims to maximize the configuration entropy and to form a single phase microstructure through combining multiple elements in an equimolar or near equimolar ratio

[1]. One typical HEA system is based on five transition elements (Ni, Cr, Mn, Fe and Co), denoted here as tHEA, e.g. FeCoCrNiMn [1]. This system has been developed extensively with a number of variants in compositions, showing great potential for creating exceptional engineering alloys [2–9].

Although many of tHEA variants form multi-phase microstructures with superior properties [6,10–12], single phase FeNiCrMnCo based tHEAs are still desirable in order to make use of high entropy effects, and to illustrate fundamental mechanical aspects of HEAs. Single phase tHEA can be obtained by thermo-mechanical processing of cast alloys [10] or powder metallurgy (PM) approaches [4,13,14]. Here, we have produced a face-centered cubic (FCC) single phase FeCoCrNiMo_{0.23} alloy (tHEA-Mo) through a powder metallurgy route. This powder metallurgy process includes (i) fabrication of pre-alloyed tHEA-Mo powder via gas atomization, and (ii) hot extrusion of the canned powders, as detailed in Ref. [14]. The newly-developed FeNiCrCoMo_{0.23} alloy displays an excellent strength-ductility combination (see Table 1) with an ultimate tensile strength of 784 MPa and elongation over 50%. A good combination of strength and ductility has been found in various tHEA variants, which is attributed to twinning induced plasticity (TWIP) [2,7,10,15], phase transformation induced plasticity [16] and/or micro-band formation [17]. Interestingly, all three mechanisms have also been identified in austenite steels with a low to medium stacking fault energy [18–20]. By analogy with its counterparts and austenite steels, we suspect that similar mechanisms may play an important role in the outstanding strength and ductility of the present tHEA-Mo alloy, which we aim to confirm in this study.

Transmission electron microscopy (TEM) has generally been used to study the microstructural response to the applied deformation in a wide variety of tHEAs, revealing the dislocation structure formation [17], stacking faults [21], deformation twins [2], and microbands [17]. However, only a small region is characterized by TEM, which may not

provide representative information of the bulk. Neutrons can penetrate deeply into most metallic alloys, allowing neutron diffraction to probe the average microstructural response of the bulk material, including lattice strain evolution [22,23], mechanical twinning and stress-induced phase transformation [22,24]. It has also been used in tHEAs at both room temperature [25,26] and high temperatures [25]. Here, we have performed *in situ* time-of-flight neutron diffraction studies on PM FeNiCrCoMo_{0.23} tHEA alloy during uniaxial tensile loading in conjunction with post-mortem microstructure analysis. Two specimens at as-extruded and as-annealed conditions were tested. The evolution of micro-scale lattice strain and stacking fault probability as a function of deformation was quantified. This study attempts, on one hand, to elucidate the microstructure-mechanical behaviour relationship of FeNiCrCoMo_{0.23} alloy, and on the other hand to facilitate our understanding of the deformation mechanisms activated in high entropy alloys.

2. Materials and Methods

The melt was prepared using a mixture of high purity (99.99%) Fe, Co, Cr, Ni and Mo in an induction vacuum furnace. The melt was then dropped through a ceramic tube, and atomized by high purity Ar with 4 MPa atomization pressure, forming droplets which rapidly solidified into powder particles. The as-prepared powder (average diameter 50 μm) was encapsulated into a can made of stainless steel with an inner diameter of 60 mm and a length of 150 mm. The encapsulated powder was pre-heated to 1200 °C for 1h, and immediately subjected to hot extrusion at 1200 °C with an extrusion ratio of 9.5:1 and a velocity of ~10 mm/s on a 2500 T hydraulic press. After hot extrusion, the bars were air cooled. The resulting bar composition was Fe_{23.2}Co_{23.9}Ni_{23.8}Cr_{23.7}Mo_{2.3} (at.%). The grain structure of the as-extruded alloy is shown in Fig. 1a. The grain size is around 35 μm , and a few twins can be observed. Part of the as-extruded samples were subjected to annealing treatment at 800 °C for 72 h. After annealing, dense intermetallic particles enriched with Mo

(Mo_{25.2}Co_{20.6}Cr_{25.4}Fe_{17.8}Ni_{11.1} in wt.%, as compared to the matrix: Mo_{8.2}Co_{24.2}Cr_{21.5}Fe_{22.4}Ni_{23.7}, measured by EDS in SEM) were formed both along grain boundaries and in the grain interior (Fig. 1b).

In situ time-of flight (TOF) neutron diffraction measurements during tensile deformation were performed on the ENGIN-X diffractometer using a stress rig (ISIS spallation neutron source, the Rutherford Appleton Laboratory, UK)[27]. Fig. 2 shows the schematic of the *in situ* tensile loading setup. The stress rig has a load capability of ± 100 kN mounted on the diffractometer horizontally. Dog-bone tensile samples (dimensions shown in Fig. 2) were used from both the as-extruded and as-annealed bars. The loading axis is parallel to the extruded direction and oriented 45° relative to the incident beam. The two $\pm 90^\circ$ detector banks (i.e., axial and radial detectors) allow simultaneous collection of diffraction patterns parallel and perpendicular to the loading direction of the specimen. The patterns recorded in the axial detector contain refractions from grain families for which the plane normals are parallel to the applied load. The neutron scattering gauge volume was $4 \times 4 \times 4$ mm³, which was defined by the 4×4 mm² incident slit, and the 4 mm wide receiving collimators. Diffraction patterns were acquired for 20 min intervals between tensile loading steps, iterating until the sample failed. A stress control mode was used until yielding, followed by displacement-control.

The diffraction spectra was analysed by the Rietveld method using the GSAS software package [28], allowing the determination of interplanar lattice spacing (d), diffraction intensity (I) and full width at half maximum (FWHM). Lattice strain was calculated using:

$$\varepsilon_{hkl}^{exp} = \frac{d_{hkl} - d_{hkl}^0}{d_{hkl}^0} \quad (1)$$

where d_{hkl} is the interplanar lattice spacing of (hkl) plane at different load steps whereas d_{hkl}^0 is the interplanar spacing at the start of deformation.

Both scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were performed to examine the microstructure of the samples. a field emission scanning electron microscope (FESEM) (FEI Nova Nano230, USA) coupled with electron backscattered diffraction (EBSD) was used. The acceleration voltage during the EBSD measurements was 30 kV, the beam current was approximately 100 mA, and the step size is 1 μ m. Electron Probe Micro Analysis (EPMA) was carried out by a JEOL-JA-8230 SEM equipped with a wavelength dispersive spectrometer at 15 kV and dwell time of 40 ms. FEI Quanta 650 SEM was used to characterize the fracture morphology and intermetallic particles. TEM samples were extracted from the necked region of the failed specimens after tensile deformation. They were ground down to $\sim$ 100 μ m foil, then disks of 3 mm diameter were punched out. Ion milling (Fischione Model 1050) at 6 kV with a final angle of 1° was used to produce electron transparent regions in the central part of the disks. TEM investigations were conducted in a JEOL-2100 microscopy operated at 200 kV. Techniques including bright field (BF) imaging, diffraction, dark field scanning-TEM imaging (DF-STEM) and energy dispersive X-ray spectroscopy (EDS) were used.

3. Results and Discussion

3.1. Mechanical properties

Fig. 3a shows the macroscopic engineering stress/strain (blue line) behaviour of the as-extruded PM tHEA-Mo alloy together with its true stress-true strain curve (pink line). The points at which neutron diffraction patterns were taken are clearly visible due to the load relaxation that occurred during the 20 min holding period. The alloy exhibits an excellent combination of high strength and ductility, with a yield stress (YS) of 328 MPa, an ultimate tensile strength (UTS) of 784 MPa, together with 53 % total elongation. The Young's modulus E of the alloy is 287 GPa. A very high work hardening rate can be observed from the true stress-strain curve.

The stress-strain curve of the annealed tHEA-Mo alloy is shown in Fig. b. The UTS of tHEA-Mo after annealing at 800 °C for 72 h increases to 942 MPa, but the yield stress slight decreases to 352 MPa, and the elongation lowers to 18%. Its Young's Modulus is about 274 GPa. The increase of strength might be due to the formation of hard intermetallic particles during heat treatment (Fig. 1b) [29].

Ludwik's equation [30] is used to fit the measured true stress-strain curves. Equ. 2 and 3 are the resulting equations for the as-extruded and as annealed alloys, respectively. The work hardening exponent (n) of the HEA-Mo alloy at the extruded and annealed conditions are 0.83 and 0.51, respectively. The exponent value of the as-extruded tHEA-Mo alloy is very close to the measurement by Liu *et al* on an as-cast FeCoCrNi alloy (e.g. 0.75) [29]. Annealed tHEA-Mo has a lower hardening exponent compared with the as-extruded alloy. Higher exponent value is often related to enhanced uniform elongation and more homogeneous deformation, resulting in superior formability [29,31].

$$\sigma = 378 + 40\varepsilon^{0.83} \quad (2)$$

$$\sigma = 350 + 149\varepsilon^{0.51} \quad (3)$$

The mechanical properties of tHEA-Mo alloy processed by both routes are listed in Table 1, together with a few other HEA alloys. A few points can be drawn.

Firstly, PM processed tHEA alloys (both FeCoCrNi and FeCoCrNiMo_{0.23}) have higher YS and UTS than those alloys fabricated by casting and thermo-mechanical processing with similar compositions. Casting of HEA alloys tends to form segregation during solidification. Although subsequently thermo-mechanical processes can reduce the segregation, it may still lead to heterogeneity in both the composition and microstructures of the final alloy, hence deteriorating the mechanical performance. The PM approach in this study used fine powders made by gas atomization, minimizing segregation and alleviating the undesirable effects.

Secondly, as-extruded PM FeCoCrNiMo HEA exhibits over 110 MPa increase in UTS compared to PM FeCoCrNi alloy. The alloying of Mo might cause local distortion in the FCC structure [29], inducing the solution strengthening effect. This demonstrates that micro-alloying could be used to further strengthen single phase HEAs, opening a potentially new prospective for HEA alloy design. Thirdly, exceptional elongation (around 50%) with good strength has been achieved in most of the FeNiCoCrMn based HEAs with single FCC structure (see Table 1).

Table 1 Yield strength (YS), ultimate tensile strength (UTS) and elongation of tHEAs at room temperature in the present study and selected prior studies. Values in brackets are from *in situ* neutron tensile tests.

Materials	Processing methods	YS (MPa)	UTS (MPa)	Elongation (%)
FeCoCrNiMo _{0.23}	PM-extruded	378 (369)	784 (829)	53 (51)
FeCoCrNiMo _{0.23}	PM-annealed	(350)	(942)	(18)
FeCoCrNi [14]	PM-extruded	359	712.5	56
FeCoCrNiMo _{0.2} [29]	As-cast	254.7	589.6	55.1
FeNiCoCrMn [32]	Cast+cold rolled	165	520	65
FeCoCrNi [29]	As-cast	155	472.4	58.9
FeCoCrMnFeNi[2]	Cast+Rotary swage+Anneal	265	600	45

3.2. Neutron diffraction

Fig. 4a and 4b show the neutron spectra of the as-extruded FeCoCrNiMo_{0.23} alloy recorded on the axial and radial detectors, respectively. Three different stress levels (0, 418 and 755 MPa) are shown. General observations are listed below, followed by detailed quantification:

- The diffraction pattern acquired before deformation shows that the alloy is an FCC single phase structure. No new peaks appear during deformation.

- The diffraction peak positions change at different stress levels. As stress increases, the peaks in Fig. 4a (axial detector) shift to higher d-spacing, while those in Fig. 4b (radial detector) shift to lower d-spacing. This is due to the Poisson's ratio contraction.
- As applied stress increases, the intensity of diffraction peaks varies significantly. For example, the intensity of (111), (200) peaks collected in the axial detector increases, whereas they decrease in the radial detector. Interestingly, the intensity of (220) peak decreases, and finally disappears in the axial direction, while it increases in the radial direction. On the other hand, it seems that (311) peak intensity (at least its height) decreases in both detectors.

The neutron spectra of the annealed FeCoCrNiMo_{0.23} (not shown here) shows a similar trend. With the help of the Rietveld refinement method, the lattice parameter a_0 of both alloys are derived as listed in Table 2. Annealing slightly decreases the lattice parameter.

Fig. 5a and 5b shows the evolution of measured axial lattice strains ε_{hkl}^{exp} parallel to the loading direction for different grain families up to a stress level close to the failure of the as-extruded and as-annealed tHEA-Mo alloy, respectively. Note that there are two possible sources that can contribute to peak shifts: (1) (hkl) dependent macro-strain referring to $\varepsilon_{hkl}^{strain}$, and (2) stacking faults referring to ε_{hkl}^{sf} . The stacking fault and (hkl) dependent macro-strains can be correlated with the experimentally measured lattice strain via [33,34]:

$$\varepsilon_{hkl}^{exp} = \varepsilon_{hkl}^{strain} - \varepsilon_{hkl}^{sf} = \varepsilon_{hkl}^{strain} - \frac{\sqrt{3}}{4\pi} \frac{\sum_b \pm(h+k+l)}{(u+b)(h^2+k^2+l^2)} SFP \quad (4)$$

where u and b are the numbers of non-broadened and broadened components due to stacking faults, respectively, and SFP is the stacking fault probability.

In alloys where the stacking fault energy (SFE) is low, a significant amount of stacking fault can be introduced when the sample is strained, which will contribute to the peak shifts. This has been shown to occur in austenitic steels with low SFE [33]. Single phase

FCC tHEAs is considered to be similar to austenitic steels, hence the change of the inter-planar spacing (ε_{hkl}^{exp}) might be a result of both the occurrence of stacking fault and (hkl) -plane dependent macro-strain. Here we first discuss the evolution of ε_{hkl}^{exp} upon deformation, then we adopt the method used by Jeong *et al.* [33,34] to measure the stacking fault probability (SFP).

Two deformation stages with varying slopes are visible in Fig. 5a: (I) linear elastic loading and (II) a linear plastic stage after a short transition from elastic to plastic deformation. In stage I (elastic regime), before the yield point, all the lattice strains respond proportionately to the applied stress. We have calculated the elastic modulus of various grain orientations, shown in Table 2. The (200) grains exhibit the lowest elastic modulus, followed by (311), (111), and then (110), consistent with the cubic elastic anisotropy factor [35]. After stage I, a clear transition point/short period can be found where the stress-lattice strain relationship becomes nonlinear, indicating the onset of yielding as loads are redistributed. In this period, the load transfers from ‘softer’ grain families, e.g. (220), to ‘harder’ grain families, e.g. (200). Afterwards, stage II is reached, during which the lattice strains respond linearly to the applied stress again. In this stage almost all grain families except (200) show the same slope as that in the elastic regime. The fact that the slope of (200) grain drops from stage I to stage II indicates that (200) orientated grains have a higher directional strength-to-stiffness ratio among those orientations [36], hence accommodating more load when the sample is further tensile plastically strained.

Fig. 5b shows the development trend of lattice strain in the annealed tHEA-Mo sample. Similar to that of the as-extruded one (Fig. 5a), two distinctive stages can be observed. The slope of (200) grain family in the annealed sample becomes smaller than that in the as-extruded sample in the transition period, which suggests the (200) grain family becomes ‘harder’ after annealing.

The elastic modulus of various grain orientations are shown in Table 2 for both conditions. The annealed alloy has a higher elastic modulus in all grain families than the as-extruded sample. The Young's Modulus anisotropy ($=E_{111}/E_{200}$) is about 1.95 and 1.86 for the two alloys, which is consistent with the work of Wu *et al.*[26], (i.e. 1.98 for $\text{Fe}_{20}\text{Co}_{20}\text{Ni}_{20}\text{Cr}_{20}\text{Mn}_{20}$, at.%).

Table 2. Lattice parameter, elastic modulus (for different grain orientations) and stacking fault energy of tHEA-Mo alloys in as-extruded and annealed conditions.

	a_0	E_{200}	E_{311}	E_{111}	E_{220}	Modulus	SFE
HEA-Mo	(nm)	(GPa)	(GPa)	(GPa)	(GPa)	anisotropy	(mJ/m ²)
As-extruded	0.3604	145	207	283	299	1.95	19
Annealed	0.3575	166	233	308	311	1.86	~

Fig. 5c plots the (111) and (222) lattice strain as a function of true strain of the as-extruded tHEA-Mo alloy. Increasing differences between the two were observed when true strain exceeded 10%. When considering only peak shifts induced by (hkl)-plane dependent macro-strain, the measured lattice strain of the (111) and (222) planes should be exactly the same since they are equivalent crystallographic orientations [33]. However, the occurrence of stacking faults can lead to differences in lattice strain in the (111) and (222) reflections [33,36].

Using equation (2), the stacking fault probability (SFP) was determined (Fig. 5c). It is noticeable that under 10% true strain, the SFP fluctuates with values less than 0. After 10% true strains, the SFP becomes positive, and continuously increases with applied strain in an almost linear manner, reaching to about 21×10^{-3} at the end of deformation. This clearly demonstrates the increasing fraction of stacking faults as deformation proceeds in the as-extruded alloy. In addition, it seems that a critical amount of strain (about 10% true strain and

645 MPa true stress) is needed to promote stacking fault formation in the as-extruded tHEA-Mo alloy. For the annealed sample (Fig. 5d), the SFP varies with negative values below 7.6% true strain, and increases to only about 2.5×10^{-3} before fracture, which is half of the value of the as-extruded sample at a similar strain level, and one magnitude lower than that of the as-extruded alloy before fracture. This indicates that the amount of stacking faults formed in the annealed sample is insignificant. The particles formed during annealing will strongly influence the evolution of dislocations in the matrix during deformation hence the formation capacity of stacking faults. Specifically, the intermetallic particles can block the motion of dislocations (strengthen the alloy) and prevent the formation of stacking faults in the matrix which means a reduction of stacking fault probability. In both cases, at lower strain levels, the stacking fault probability stays negative, which indicates that the effect of stacking faults on peak shift is not distinct at low strains [36].

Stacking fault energy (SFE, γ_{SF}) represents the ease of dissociation of a perfect dislocation into two partial dislocations and the propensity for the formation of SFs. It can be evaluated by Reed and Schramm's equation [37]:

$$\gamma_{SF} = \frac{6.6a_0}{\pi\sqrt{3}} \left(\frac{2c_{44}}{c_{11} - c_{12}} \right)^{-0.37} \frac{\langle \varepsilon_{50}^2 \rangle_{111}}{SFP} \left(\frac{c_{44} + c_{11} - c_{12}}{3} \right) \quad (5)$$

where a_0 is the lattice parameter, $\langle \varepsilon_{50}^2 \rangle_{111}$ is the mean square strain, c_{11} , c_{12} , and c_{44} are the single crystal elastic constants.

Eqn. 3 shows that the stacking fault energy is inversely proportional to the stacking fault probability. The single crystal elastic constants c_{11} =216 GPa, c_{12} =175 GPa, and c_{44} =189 GPa used in this study are from *ab initio* atomistic simulation on an FCC Fe₂₅Co₂₅Ni₂₅Gr₂₅ alloy [38]. $\langle \varepsilon_{50}^2 \rangle_{111}$ was estimated by an integral breadth method with a pseudo-Voigt convolution [39]. We then estimated the SFE of the as-extruded and annealed tHEA-Mo alloys at room temperature to be 19 mJ/m². The SFE of the as-extruded tHEA-Mo alloy correlates well with

the range determined by Zaddach *et al.*, e.g. 17.4-31.7 mJ/m² in an FCC Fe₂₅Co₂₅Ni₂₅Gr₂₅ alloy [40].

In prior studies Eqn. 5 has primarily been used to measure the SFE of pure metals [37] or single phase alloys [33]. It has not been widely applied to multi-phase materials, although recently it was used to calculate the SFE of the austenite phase in a duplex steel [41]. Although it may only be indicative, here we apply Eqn. 5 to the annealed multiphase tHEA-Mo sample, (which contains intermetallic precipitates, see Fig. 1b) to determine an estimate of the SFE, obtaining ~135 mJ/m². Although more indicative of an upper bound (especially as the stacking fault probability is very low adding error to the calculation), the result suggests that the stacking fault energy of the annealed alloy is high enough that the formation of deformation twins is unfavourable.

The overlapping SF and partial dislocation bounding them can be considered as twinning constituents or twinning embryos [42]. In other words, the increasing width of SFs favours the formation of mechanical twinning. A critical stress is required for a mechanical twin to form [43]. This stress in the uniaxial form (σ_T) can be described by [43]:

$$\sigma_T = \frac{2}{m \times b_p} \gamma_{SF} \quad (6)$$

where m is the Schmid factor ($m=0.471$ for loading along {220} orientation[43]), b_p is the burger vector of a Shockley partial dislocation. With $\gamma_{SF}=19$ mJ/m², the critical stress for twinning to occur in the as-extruded tHEA-Mo alloy is ~805 MPa (corresponding to the true strain of about 12). Laplanche *et al.* [2] found that nano-twins in FeNiCoCrMn tHEA started to form after the tensile stress reached 720±30 MPa at about 25% true strain at room temperature. The slight difference in the value of the critical stress might lie in the difference in alloy composition and strain rate applied. However, note that as deformation proceeds, local stress concentration may exceed the critical stress for twinning and might induce

twinning those grains which are unfavourably in the as-extruded alloy. Also this stress concentration effect might induce deformation twinning in the annealed alloy.

Fig. 5e shows the intensity changes of several peaks on the axial detector as a function of applied true stress of the as-extruded sample. Before macroscopic yielding, the peak intensity remains almost constant. After macroscopic yielding, significant changes in intensity are observed. The intensity of (111) grain family rises by a factor of about 11, and (200) grain family also increases by a factor of 4. The (311) grain family increases slightly, then it lowers and broadens. On the other hand, the (220) peak intensity drops to zero when the true stress reaches 900 MPa. Fig. 5f shows the peak intensity on the axial detector as a function of applied true stress of the annealed sample. The trend is very similar to the as-extruded sample, although the magnitude of intensity change of the annealed sample is much smaller (e.g. 4 times increase of (111) peak and 2 times increase of (200) peak). Interestingly, the (220) peak disappears when the sample is strained close to failure in both samples.

The change of peak intensity is attributed to the re-orientation of grains during the tensile deformation, which could be due to slip/rotation of grains and/or formation of mechanical twins. Grain slip/rotation has been well documented in the literature to account for peak intensity changes [25]. Deformation twinning can also cause a change in peak intensity. Firstly, when grains which are initially oriented to satisfy the diffraction condition, were twinning, the twinned portion can leave the diffraction condition. This reduces the diffraction intensity. Secondly, grains oriented such that when they twin, the twinned portion meets the diffraction condition, which can increase the diffraction intensity [44]. This concept has been used extensively in HCP structured alloys, especially Mg alloys [45], because the dominant twinning mode ($\{10\bar{1}2\}$ twinning) in Mg can result in a nearly 90° change in crystal orientation, which can cause a significant change in texture and hence peak intensity. However, there are rather limited applications of this concept in FCC structures as

it becomes difficult to qualitatively separate the contributions of twinning and grain rotation to the measured peak intensity change. In our case, we would not expect such a significant change of peak intensity (e.g. 11 times increase of (111) peak intensity) by grain rotation alone. Therefore, it is suggested that both rotation and twinning lead to re-orientation of grains, which accounts for the significant intensity increase in the as-extruded tHEA-Mo alloy. If we assume that the annealed tHEA-Mo alloy is unlikely to form twins during room temperature deformation due to its high stacking fault energy, its intensity change can only be a result of grain rotation, which is consistent to the fact that the intensity change of the annealed sample is much smaller than that of the as-extruded sample. However, further work is needed to distinguish the contribution of slip and twinning to the overall intensity change.

3.3 *Ex situ* microscopic analysis

In addition to the *in situ* diffraction quantification, we performed correlated TEM and SEM analysis on the failed specimens after *in situ* neutron measurement. Fig. 6 show TEM images taken from the fractured specimen of the as-extruded tHEA-Mo alloy. Lamellar nano twins (Fig. 6a, recorded from [110] zone), confirmed by the inset selected-area diffraction patterns (SADP, Fig. 6b), are clearly visible. High densities of dislocation structures are trapped within and intersecting with the twin bundles. Two intermetallic particles (red arrows) can also be found. It seems the twins are curved, in particularly around the particle (the longer red arrow). Fig.6c shows intersections of twins, forming rhombic blocks. Fig. 6d shows the formation of plate-like microbands in another grain. These TEM images confirm that both twins and microbands have formed during room temperature tensile deformation to accommodate strain.

The deformation twins forms as Shockley partial dislocations passes on every {111} plane [46]. The resulting nano-sized twins can act as strong obstacles to dislocation glide, attributing to the high strain hardening of the as-extruded alloy. The microbands (nano-sized

banded structure with a small difference in grain orientation) form when perfect dislocations become hard to move at a higher strain, hence they move into localized bands [21]. Microbands can also inhibit dislocation motion and strengthen the alloy, which was illustrated in high Mn steels [47]. These results show that both twin induced plasticity and microband induced plasticity can be active in this FCC single phase tHEA. This is consistent with the work of Zhang *et al.* [21], which found that stacking faults, microbands and twins were formed simultaneously in a FeCoNiCrMn HEA alloy that was *in situ* loaded under tension in a TEM. Normally, deformation twinning occurs in alloys with a low SFE [48,49] while microbands are formed in alloys with a medium to high SFE [47,50,51]. Although this is the dominate mechanism, in addition to the aforementioned work of Zhang *et al.* [21], the simultaneous observation of twinning and microband has been reported in several other materials including austenite Fe-Mn-Al-C steels [19,52] and austenite–ferrite Fe–Mn–Al–C steel [50]. This has been attributed to the role of grain orientation on both deformation twinning and microband formation [19,53]; i.e. a grain will form deformation twins or microbands depending on its orientation relative to the loading direction. This is indicated by that fact that the Schmid factors for slipping and twinning in FCC alloys for the same grain orientation are different [43]. For this tHEA alloy with SFE as low as 19 mJ/m², deformation twinning is the primary mechanism; however, grains with an unfavourable orientation for twinning may, alternatively, form microbands to accommodate the strain.

Fig. 7a and 7b show the bright field TEM images of the annealed sample after failure. Dense dislocation structures and microbands were observed. The microbands form in one grain but are absent in its neighbour (Fig. 7b), indicating their dependence on grain orientation. The formation of microbands The SADP shows that no twinning was found in this region. We did not find obvious twins in the TEM sample; confirming the neutron diffraction suggested hypothesis that the annealed tHEA-Mo sample is inhibited from

forming deformation twins due to its low stacking fault probability and high stacking fault energy. Fig. 7c shows the dark field STEM image of the annealed sample. Quite a few intermetallic particles are found which are enriched in Mo and deficit in Ni and Fe (Fig. 7d). These submicron intermetallic particles inhibit the motion of dislocations, enhancing the strength after the annealing treatment.

Fig. 8a and 8b shows the longitudinal cross-section of the fractured tHEA-Mo sample in the as-extruded and annealed condition, respectively. Elongated micro-cavities are found in both samples. In the extruded sample, the elongated cavities are shown to decorate along the grain boundaries. In the annealed sample, cavities can be seen to locate in conjunction with the particles, indicating that the stress are concentrated around the particles, and the interfacial bonding of the two is weak. In addition, the interface might be a 'sink' for vacancies, resulting in the formation of cavities around the particle/matrix interface and the early fracture of the sample.

Fig. 8c and 8d show the fracture surface of the as-extruded and annealed specimen, respectively. Ductile dimples can be found in both samples, and it seems that the as-extruded sample has slightly finer dimples than the annealed one. Intermetallic particles are present inside the dimples in both samples. Note submicron intermetallic particles can be occasionally observed by TEM in the as-extruded alloy, as shown in Fig.8c inset, although we do not observe intermetallic particles by SEM (Fig. 8a).

4. Conclusions

A single phase FCC structured FeCoCrNiMo_{0.23} high entropy alloy (HEA) was prepared by a powder metallurgy route. The as-extruded alloy from HEA powder shows a superb combination of ductility (51 % elongation) and strength (830 MPa UTS). Annealing of the as-extruded alloy at 800 °C for 72 h increases the strength to 942 MPa but sacrifices its elongation (18 %). *In situ* neutron diffraction was used to evaluate the evolution of lattice

strain and stacking fault probability in both conditions, and correlated to electron microscopy. This correlative investigation revealed the microstructural changes due to tensile deformation, leading to the following conclusions:

1. Micro alloying of Mo was found to increase the strength of the FeCoCrNi based HEA alloy, demonstrating the potential of utilizing the solute solution strengthening effect in HEAs. The as-extruded alloy has a low stacking fault energy ($\sim 19 \text{ mJ/m}^2$), which can induce the formation of stacking faults, deformation twinning and microbands, accounting for its high ductility and strength.
2. Annealing causes decomposition of the alloy, forming Mo-rich intermediate particles. The annealed alloy is strengthened by those intermetallic particles. However, its ductility decreases due to (1) the weak interfacial bonding between the particles and the matrix, and (2) the decrease of the stacking fault probability inhibiting the formation of deformation twins.

Data statement

A representative sample of research data from the experiments along with the plot data for the graphs in this manuscript is provided in supplementary material [xxx]. The underlying data is not provided online due to its very large size.

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Figures

Fig. 1. (a) EBSD of the as-extruded tHEA–Mo alloy; (b) back scattering SEM image of the as-annealed tHEA–Mo alloy (Inset, mapping of Mo distribution).

Fig. 2. Schematic illustration of in situ neutron diffraction measurement and the tensile specimen dimensions (mm).

Fig. 3. The stress-strain curve of tHEA–Mo_{0.2} alloy in (a) as-extruded and (b) annealed state

Fig. 4. Diffraction patterns at stress levels of 0, 418 and 755 MPa of the as-extruded tHEA–Mo alloy: (a) axial data; (b) radial data.

Fig. 5. (a, b) Lattice strain in the axial direction; (c, d) axial lattice strain evolution of the (111) first order and (222) second order reflections together with the stacking fault probability as a function of true strain; (e, f) normalized intensity as a function of true stress. Note that (a, c and e) are as-extruded alloy, (b, d and f) are annealed.

Fig.6. TEM images of the strained to failure tHEA-Mo alloy at the as-extruded condition: (a) bright field image showing nano-twins; (b) diffraction pattern of the area in (a); (c) BF showing nano-twin intersections; (d) bright field image showing microbands.

Fig.7. TEM images of the strained to failure tHEA-Mo alloy at the as-annealed condition: (a) and (b) bright field TEM; (c) STEM-DF image of intermetallic particles; (d) EDS mapping of the intermetallic particles within the white square box in (e).

Fig.8. (a, b), The longitudinal cross-section; (c, d): the fractograph: (a, c) as-extruded, (b, d) annealed. Inset in c: TEM image of an intermetallic particle.

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Fig.8. (a, b), the longitudinal cross-section; (c, d): the fractograph of the fractured samples: (a, c) as-extruded, (b, d) annealed. Inset in c: TEM image of an intermetallic particle