Microstructure and Small-scale Size Effects in Plasticity of Individual Phases of $Al_{0.7}CoCrFeNi$ High Entropy Alloy

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Abstract

High Entropy alloys (HEAs) are solid solution alloys containing five or more principal elements in equal or near equal atomic percent (at %). We synthesized $Al_{0.7}CoCrFeNi$ HEA by vacuum arc melting and homogenized it at 1250°C for 50 hours. The microstructure shows the presence of two phases: the Body-Centered Cubic (BCC: A2+B2) and the Face-Centered Cubic (FCC). Using the Focused Ion Beam, we fabricated single-crystalline cylindrical nano-pillars from each phase within individual grains in the $Al_{0.7}CoCrFeNi$ HEA. These nano-pillars had diameters ranging from 400 nm to 2 μ m and were oriented in the [324] direction of the FCC phase and in the [001] direction of the BCC phase. Uniaxial compression experiments revealed that the yield strength is 2.2 GPa for the 400 nm diameter samples in the BCC phase and 1.2 GPa for the equivalent diameter samples in the FCC phase. We observed the presence of a size-effect in both phases, with smaller pillars having substantially greater strengths compared with bulk and with larger-sized samples. The size-effect power exponent for the BCC phase was - 0.28, which is lower than that of most pure BCC metals, and the FCC phase had the exponent of - 0.66, equivalent to most pure FCC metals. We discuss these results in the framework of nano-scale plasticity and the intrinsic lattice resistance through the interplay of the internal (microstructural) and external (dimensional) size effects.

Keywords: High Entropy Alloys, Nano pillars, $Al_{0.7}CoCrFeNi$

1. Introduction

High Entropy alloys are a relatively novel class of material defined as alloys with multiple principal elements in equimolar or near equimolar ratios (5 to 35 molar % of each components) that form stable solid solutions because of the large mixing entropies [1]. The mixing entropy is represented as;

$$\Delta S_{mix} = -R \sum c_i \ln c_i \quad (1)$$

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where R is 8.31J/K mol, and c_i is the molar concentration of the i th constituent metal. When the entropy is maximized and there is an equal molar concentration of each element, Eq. (1) becomes

$$\Delta S_{mix} = R \ln N \quad (2)$$

where N is the number of constituent metals. As the number of constituent metals increases, the entropy of mixing also increases.

Phase stability of HEA was predicted as that of a solid solution by relating the atomic size difference (δ) and the enthalpy of mixing (ΔH_{mix}) of the multi component system. It can be summarized that the quantitative criterion for the formation of a simple solid solution is $-10 \text{ KJ/mol} < \Delta H_{mix} < 5 \text{ KJ/mol}$ and $\delta < 4 \%$ [2]. In this work, we use the term “high-entropy alloy” to refer to the $Al_{0.7}CoCrFeNi$; a more accurate term for it is multi-principal element alloy because of the presence of two distinct phases; FCC and BCC [3].

Guo et al. [4] predicted the crystal structures of HEAs to be FCC and BCC based on the X-ray diffraction studies of some HEAs and suggested that the Valence Electron Concentration (VEC) can be used to predict the crystal structure, where $VEC < 6.8$ corresponds to the BCC structure and $VEC > 8.0$ correlates with the FCC structure. The VEC is defined as;

$$VEC = \sum C_i (VEC_i) \quad (3)$$

where C_i is the atomic percent of the i th element, and VEC_i is the VEC of the i th element.

HEAs have four core effects that set them apart from the conventional alloys which usually have one or two principal components. The core effects are high entropy effect, severe lattice distortion, sluggish diffusion effect and cocktail effect [1, 2]. High entropy effect stabilizes the solid solution phase by having several principal elements of different atomic sizes which occupy the lattice sites and cause the severe lattice distortions. The substantial interactions among these metals cause sluggish atomistic diffusion [2]. These phenomena lead to the so-called cocktail effect, which is as a result of inter-elemental interactions, composition change and alloying [5, 2]. Improved properties of HEAs such as high strength and toughness, as well as high resistance to temperature, wear, diffusion, radiation and chemical reactions are observed.

Aluminum-containing HEAs are known for the flexibility in adjusting their crystal structure, it is possible to vary the Al ratio in an alloy with other principal elements. Increasing the relative ratio of Al induces a larger lattice strain and the ensuing distortion leads to relaxation via phase change in the crystal structure [1, 2, 6, 7, 8].

Material properties can be manipulated by alloying, previous studies on HEAs show improved mechanical properties such as high strength, high temperature ductility etc. [9, 10, 11, 12]. Fewer studies have been done to understand the deformation mechanism of HEAs at the nano-scale particularly in comparing them with pure metals with a focus on yield strength, size effect and strain hardening. The choice of HEA for this

work stems from previous studies by [Manzoni et al. and Singh et al.](#), who observed that the $AlCoCrFeNi$ had no single element rich phases unlike its counterpart $AlCoCrCuFeNi$ [13, 14], thereby obscuring the possibility of the nanomechanical properties being studied as a function of a single element. Wang et al.'s high temperature XRD study of $Al_xCoCrFeNi$ HEA ($x = 0.1$ to 2.0) concludes that when $x = 0.7$, i.e., HEA with the composition $Al_{0.7}CoCrFeNi$ has a mixture of both the FCC and BCC phases and the crystal structure does not change with increasing temperature [15, 16]. This renders the $Al_{0.7}CoCrFeNi$ a useful candidate to study the nanomechanical properties of each phase as a function of its composition. In this work, we probed the compressive strength of the single-crystalline small-scale samples fabricated from each individual phases in the $Al_{0.7}CoCrFeNi$ HEA and compared the results with those of pure FCC and BCC metals of equivalent dimensions.

2. Materials and Methods

The $Al_{0.7}CoCrFeNi$ HEA was fabricated using the direct current (DC) powdered button melter with the copper mold in the argon gas. The raw materials - (Al, Co, Cr, Fe, and Ni powders) of 99.9% purity in weight percent were triple melted and formed into ~ 300 g button with a thickness of 10 mm. The as-cast buttons were heated in a vacuum furnace to $1250^\circ C$ for 2 hours and subsequently annealed at the same temperature for 50 hours to ensure homogenization. The samples were then cooled in the furnace down to room temperature. The homogenized buttons were embedded in a carbon steel to avoid surface oxidation. The carbon steel was a 76 mm diameter Iron Pipe Size (IPS) tubing with a 3 mm thick wall. The tubing was flattened and then welded shut with the button inside. Then, the whole button was upset-forged without restriction to the four sides to achieve a 50% reduction in thickness at $1250^\circ C$. The final forged portion was ~ 5 mm thick.

The as-forged $Al_{0.7}CoCrFeNi$ HEA was electropolished by Able Electropolishing Co. The morphology was analysed using the Scanning Electron Microscope (SEM) and the elemental composition analysis was carried out using the Oxford INCA 300 X-ray Energy Dispersive Spectrometer (EDS) system. The microstructure, grain size and grain orientation were determined by Orientation Imaging Microscopy (OIM) using the HKL Electron Back Scatter Diffraction (EBSD) system. Thinned HEA samples were also analyzed via Transmission Electron Microscopy (TEM FEI Tecnai F30) with 300 keV accelerating voltage. Uniaxial compression samples with diameters of 400 nm to $2\ \mu m$ were fabricated using the Focused Ion Beam (FIB) following the [procedures](#) of Greer et al. [17]. This methodology involves concentric circular milling patterns in the FIB until a cylindrical pillar is formed with an aspect ratio of 3:1 i.e., height : diameter. Final pillar dimensions were measured based on SEM images corrected for the tilt angle. The compression tests were carried out in a nanoindenter (Triboscope, Hysitron Inc) with an $8\ \mu m$ diameter diamond flat punch. Experiments were conducted in a displacement-rate-controlled mode at a constant prescribed strain rate of

$10^{-3}S^{-1}$ to a final total strain of 15% of the pillar height. Constant strain rate was maintained by the feedback-loop algorithm in the nanoindenter software.

3. Results

3.1. Microstructural analysis of the bulk HEA

Figure 1 shows the microstructure of the as-forged electropolished $Al_{0.7}CoCrFeNi$ HEA. This image clearly reveals the dendrite and the interdendrite features typical for aluminum containing HEAs [18]. The back scattered [Figure 1(b)] and the secondary [Figure 1(c)] electron images show the precise layout of the microstructure and reveal the interdendrites region as the matrix into which the dendrites are embedded. Each dendrite appears to be 10 - 15 μm wide, on further magnification, we found the dendrites to contain nanoparticles that range from about 7 nm to 100 nm in diameter (Figure 1(d)).

Figure 1: (a) SEM image showing the interdendrite and dendrite in the $Al_{0.7}CoCrFeNi$ HEA (b) Using the back scatter detector (c) Using the secondary electron detector (d) Showing the varying sizes of nanoparticles contained in the dendrite of the HEA.

The elemental composition of $Al_{0.7}CoCrFeNi$ was analyzed employing the EDS with line scans in the two regions. Figure 2(a) presents the line scan of the interdendrite region and it reveals an average composition of 23.2 ± 2.1 atomic percent (at.%) Co, 25.7 ± 2.4 at.% Cr, 27.0 ± 2.7 at.% Fe. The average content of Al and Ni in this region is less than 15.0 at.% each. Hereafter, we refer to the interdendrite region as the “Co-Cr-Fe rich region”. Figure 2(b) shows the line scan of the dendrite region whose average composition was determined to contain 30.3 ± 3.1 at.% Al, 25.6 ± 3.3 at.% Ni the remaining elements (Co, Cr and Fe) have less than 16.0 at.% each. Hereafter, we refer to the dendrite region as the “Al-Ni rich region”. This classification of both regions based on specific elements rich regions is consistent with those in [14, 13, 15, 5, 19].

The Electron Back Scatter Diffraction maps shown in Figure 3 convey the phase contrast [Fig. 3(b)] and the grain structure, as well as the crystallographic orientation of each grain [Fig. 3(c)] of the Co-Cr-Fe rich region (interdendrite) and the Al-Ni rich region (dendrite). We used the lattice parameters of 0.3572

Figure 2: EDS showing the elemental composition of the (a) Interdendrite region and (b) Dendrite region. The purple and yellow lines of (a) and (b) show the region where the line scans were taken.

nm for the FCC phase and 0.2868 nm for the BCC (A2 + B2) phase obtained via the X-ray diffraction spectrum of $Al_xCoCrFeNi$ ($x = 0.1 - 2.0$) from Ref: [16]. OIM results reveal that the Co-Cr-Fe rich region (interdendrite) is the FCC phase and the Al-Ni rich region (dendrite) is the BCC (A2+B2) phase. We found the grain orientation to be random in the two phases with an average grain size distribution of 2.85 μm in diameter on the orientation map shown.

Figure 3: (a) Electron Image (b) Phase information; the blue is the FCC phase and the red is the BCC (A2 + B2) phase (c) Crystallographic orientation of the phases present (d) Pole figure labeling the orientations

Figure 4 exhibits the Transmission Electron Microscopy analysis of the HEA which further demonstrates the two distinct phases present in this alloy. Figure 4(a) presents the bright field image of the interdendrite region with the diffraction pattern in the inset, indicative of the FCC phase. Figure 4(b) provides the TEM image of the dendrite region and the inset gives the diffraction pattern which indicates the presence of two phases from the “satellite-like” diffraction spots next to the main diffraction spots. The diffuse spots suggests the presence of the super-lattice diffraction which likely corresponds to the ordered B2 phase. Tung et al. [20] suggests that it was the spinodal decomposition in the Al-Ni rich region that occurred during cooling of the HEA that led to the transformation from A2 to A2+B2.

Figure 4: (a) Bright Field (BF) image of the interdendrite region with the diffraction pattern FCC: ZA $[\bar{1}23]$ at the top left (b) TEM image of the dendrite region with the diffraction pattern BCC (A2+B2): ZA $[001]$ at the top left, where ZA is the zone axis

3.2. Uniaxial Compression experiments

Figures 5(a) and (b) show the SEM images of the representative pre and post compressed samples of the $[324]$ oriented nanopillars fabricated from the FCC phase of the HEA. Crystallographic slip lines appear to be evenly distributed along multiple parallel slip planes which is typical of the FCC crystals in low symmetry operations [21]. Figure 5(c) shows the representative stress vs strain data of the FCC phase samples, showing the elastic loading region, the steep post-yield strain hardening to a strain of $\sim 2\%$ and the steady-state flow starting at the strain of $\sim 2\%$. The smaller pillars exhibit higher yield strength and flow stress typical of pure FCC metals. The slip angle after compression is measured to be 32.4° with respect to the loading axis. We observe multiple displacement bursts in pillars with diameters of 400 nm and smaller likely due to the inability of the instrument to catch the very fast burst events [22, 23].

Figure 6(a) shows the SEM image of the representative compression $[001]$ -oriented nanopillar carved from the BCC phase of the HEA. This image reveals wavy slip lines typical of BCC crystals. Figure 6(b) shows the stress vs strain data for the BCC phase pillars with diameters ranging from 400 nm to 2 μm and illustrates that the smaller pillars have a higher yield strength and flow stress in a similar fashion to the pure BCC pillars under compression Ref: [24]. We also observed multiple displacement bursts in pillars with diameters of 400 nm and smaller likely due to the inability of the instrument to catch the very fast burst events [22, 23]. We observed a significant amount of strain hardening in this phase, which also appears to increase with reduced pillar size.

4. Discussion

4.1. Strength

Compressive strengths in the samples fabricated from both phases present in the HEA exhibited the “smaller is stronger” size effect, with the smallest; 400 nm-diameter nanopillar having the highest strength of ~ 2.2 GPa in the BCC phase and ~ 1.2 GPa in the FCC phase. In bulk form, HEAs have been reported

Figure 5: Representative (a) Pre-compression image (b) Post-compression image (c) Stress vs strain curve of the $[324]$ -oriented nanopillar in the FCC phase

Figure 6: Representative (a) Post-compression image (b) Stress vs strain curve of the $[001]$ -oriented nanopillar in the BCC(A2+B2) phase

to have strengths higher than those of the conventional metals and alloys because of the heterogeneities in the atomic sizes leading to significant lattice distortion and higher intrinsic lattice resistance [2]. In the HEA studied here, Al has a larger atomic size of 1.43 Å compared with the other components: (Co: 1.25 Å, Cr: 1.25 Å, Fe: 1.24 Å, Ni: 1.24 Å). The presence of Al in the lattice likely increases the lattice distortion energy and leads to significant solid solution strengthening of the alloy [25]. A simple solid solution strengthening model [26] allows for the calculation of τ_{max} , the resolved shear stress required to tear the dislocation from the solute atom, Al in this case, as $\tau_{max} = F_{max}/bL'$, b is the magnitude of the Burgers vector, and L' is the mean free path between the solute atoms. The mean spacing between the solute atoms in a crystallographic slip plane can be approximated as $b/\sqrt{(2c)}$, c represents the concentration of the solute atoms using the EDX data obtained experimentally to calculate the relative atomic fraction of Al for a typical 400 nm-sized pillar, we find that the τ_{max} , or the shear stress required to push a dislocation through an array of solute atoms is 0.68 MPa. This value is approximately an order of magnitude less than the resolved shear stress of 0.9 GPa, obtained from the data for the compression of a 400nm-diameter nanopillar calculated from the axial applied stress. These results imply that solid-solution strengthening cannot be the sole or dominant cause of the observed high strength, although it likely elevates the background stress of the BCC phase of the HEA. (See supplementary material for calculation).

In pure metals, size effects have been usually attributed to the distribution and motion of dislocations. For the same dislocation density in the bulk, smaller-diameter samples have fewer dislocations. The “smaller is stronger” size effect observed in the nanopillars fabricated from pure FCC metals and some alloys is thought to arise from the annihilation of some of the pre-existing mobile dislocations at the available free surfaces, and the subsequent operation of harder and stronger dislocation surfaces to maintain the dislocation density necessary to accommodate the applied force and the ensuing strain [27, 28]. In pure FCC metal nanopillars, dislocations present at the onset of plastic deformation leave the specimen before they multiply, forming the state of “dislocation starvation” which requires the nucleation of new dislocations to accommodate plastic deformation [17, 29]. FCC metals undergo this type of deformation due to the glide dislocation motion during deformation. It is likely that the nano-pillars fabricated from the FCC phase of the HEA studied here (interdendrite; Co-Cr-Fe rich region) experience the same plasticity mechanism. The distinct displacement bursts in the stress-strain data and multiple slip lines present in all deformed pillars suggest that this mechanism is retained even in these more complex alloys.

BCC nanopillars also exhibit the smaller is stronger size effect, which has been shown to arise from a more complex nanoplasticity mechanisms in pure BCC metals. In this type of crystal structure, individual dislocations can form kinks, with each component interacting with one another, forming junctions, all of which lead to greater dislocation densities, sessile junctions and forest-like hardening upon the application of force [29, 30]. Similar arguments can be made for the size effect that arises in the BCC phase of the

$Al_{0.7}CoCrFeNi$ HEA. Wavy slip lines are present after compression due to the cross slips of dislocations in the $\langle 111 \rangle$ direction. This phenomenon was observed to be in W nanopillars [24].

The influence of the different types of dislocation motion in both phases makes the BCC phase have higher strengths than those of the FCC phase [31]. The strength of the bulk sample (σ_{bulk}) was estimated to be $H/3$ where H represents indentation hardness. Following this methodology, we found the strength of the BCC phase as ~ 1.40 GPa and that of the FCC phase to be ~ 1.01 GPa. The yield stress of the 400 nm samples in the FCC phase of the HEA is 1.2 GPa, a factor of 1.5 greater than that of pure Ni nanopillars of equivalent dimensions, 0.8 GPa [21], Ni being the strongest of the commonly studied pure FCC metals (Cu, Al, and Au). The yield stress of the 400 nm-diameter nano-pillars fabricated from the BCC phase of the HEA studied here is 2.2 GPa, which is nearly equivalent to 2.0 GPa [24], the strength of 243 nm-diameter W, which is the strongest of the commonly studied BCC (Mo, Ta, Nb and V) metals.

4.2. Size effect

In most single crystalline metals at the micron and sub-micron scale, size dependence in strength can be described using the power law relation $\sigma = A(D)^m$, where A is a constant and D is the pillar diameter. The exponent m is the main figure of merit for how much size-induced strengthening a particular family of materials exhibit. For example, Kim et al. studied the size dependence of several pure BCC metals and found the power law exponent of the compressed W nanopillars to be - 0.43 [24]. To compare the size effect across different materials classes, it is useful to resolve the axial stress, which is what is typically measured in an experiment for samples in different orientations to the shear stress that operates on a relevant slip system [32]. When normalized by the shear modulus (G) and the Burgers vector (b), the size dependence relation between the resolved shear stress τ and the pillar size D becomes $\tau/G = A(D/b)^m$.

4.2.1. FCC phase

We used the $\{111\} \langle 110 \rangle$ family of slip systems to calculate the resolved shear stress (τ) for the nanopillars fabricated from the FCC phase (Co-Cr-Fe rich region). These samples were oriented in the $[324]$ direction with the Schmid factor of 0.634. We used the shear modulus of 75 GPa [33] reported for FeCr alloys to normalize the shear stress. Figure 7(a) shows the plot of axial yield stress and the flow stress at a 10 % strain on the stress-strain curve shown in Figure 5(c) as a function of pillar diameter D . We found that the slope m values are - 0.70 for the yield strength and - 0.63 for the 10 % flow stress.

Figure 7(b) shows the dependence of the resolved shear stress normalized by the shear modulus G as a function of pillar diameter normalized by the Burgers vector b on a log-log plot. A straight line that represents size effects in pure FCC metals is also provided for comparison (Data obtained from [27]) and this reveals a similar size dependence, with the ubiquitously reported m value of - 0.66 for the yield strength and - 0.59 for the 10 % flow stress. The plasticity mechanism in nano-sized FCC metals has been explained in

the framework of dislocation starvation and dislocation source nucleation theory [27, 28]. A unified power law exponent m of - 0.6 has been observed in nearly all studied FCC metals (Ni, Cu, Al, Au) [27] with non-zero dislocation densities. The FCC phase of our HEA can likely be explained by similar arguments because of the observed similarities in the morphology of the deformed samples, the stress vs strain data, and the power law strengthening exponent of - 0.6. The similarity in strengthening slope can be explained by the lower lattice distortion present in the FCC phase of the $Al_{0.7}CoCrFeNi$ compared with the BCC phase because the elements within the FCC phase; Co, Cr, Fe are similar in atomic size. The significantly higher strengths in the HEA likely stem from solution strengthening and high background stress [1, 2, 7, 8], and the multiple bursts observed in the stress strain signature in Figure 5(b) is an indication that there is ease of dislocation movement typical of the FCC metal.

Figure 7: (a) Un-normalized size dependence plot of the FCC phase of the $Al_{0.7}CoCrFeNi$ HEA, where m_y is the exponent calculated from the yield strength and $m_{0.10}$ is calculated from 10 % flow stress (b) Normalized size dependent plot of the FCC phase of the $Al_{0.7}CoCrFeNi$ HEA compared with studied FCC metals (Au, Cu, Ni and Al: Data adapted from [27])

4.2.2. BCC phase

We used the $\{110\} \langle 111 \rangle$ family of slip systems to calculate the resolved shear stress (τ) for the nanopillars fabricated from the BCC phase (Al-Ni rich region) These nanopillars were oriented in $[001]$ with a Schmid factor of 0.408. We used the shear modulus G of 70 GPa [34] which is consistent with 65 ± 4 GPa that Frommeyer et al. reported in the study on Ni-Al alloys [35]. Figure 8(a) shows the axial as-measured yield stress; 2.25 GPa for the 400 nm sized pillar, as well as the flow stress at a 10 % strain; 3.5 GPa for equivalent size pillar as a function of nanopillar diameter, obtained from the stress vs strain data shown in Figure 6(b). Fitting a power law functional form to each of these data sets reveals the slope m of - 0.28 for the yield strength and - 0.27 for the flow stress. Figure 8(b) gives the normalized size dependence for the BCC phase of the HEA and compares with the normalized size dependence of reported pure BCC metals (V, Nb, Mo, Ta and W). The BCC phase of the HEA appears to exhibit higher strengths and a reduced size dependence ($m = - 0.28$) compared to the pure counterparts. This value is slightly less than the lowest

exponent of all pure BCC metals of - 0.32, reported for [24, 27] ; although power law exponents as low as - 0.21 have also been reported for W [36], - 0.22 for [001] Mo and - 0.34 for [235] Mo [37]. Zou et al. studied a BCC refractory HEA ($Nb_{25}Mo_{25}Ta_{25}W_{25}$) and found that the strengthening size effect slope was $m = - 0.33$.

Figure 8: (a) Un-normalized size dependence plot of the BCC phase of the $Al_{0.7}CoCrFeNi$ HEA, where m_y is the exponent calculated from the yield strength and $m_{0.10}$ is calculated from 10 % flow stress. (b) Normalized size dependent plot of the BCC phase of the $Al_{0.7}CoCrFeNi$ HEA compared with studied BCC metals (V, Nb, Mo, Ta, W: Data adapted from [27])

The difference in size dependence amongst the BCC metals has been attributed to the difference in their residual Peierls barrier at room temperature, with the highest Peierls barrier having the least pronounced size dependence [27]. In addition to the Peierls barrier, HEAs have the lattice distortion effect [1, 2, 6, 7, 8] which is even more prominent in the BCC phase of the $Al_{0.7}CoCrFeNi$ HEA. Wang and Zou reported that higher lattice distortion leads to greater lattice resistance, which amplifies the effective Peierls barrier in the system [22, 31, 38]. Using the temperature-dependent relation of the sum of lattice friction [39] as a contributon to the resolved shear stress, $\tau^* = (1 - T_t / T_c) \tau_0^*$, where τ^* is the stress required to overcome the Peierls barrier, T_t is the test temperature, T_c is the critical temperature and τ_0^* is the Peierls stress at 0 K, Zou et al. reported that materials with higher T_t / T_c are expected to have a reduced size effect i.e. a lower slope “ m ” [22]. More work needs to be done to study the critical temperature and the Peierls barrier of HEAs experimentally and theoretically.

4.3. Strain Hardening

The stress vs strain data of the FCC phase shown in figure 5(c) indicates the presence of multiple, densely populated discrete strain bursts. We applied the hardening angle method used by Kunz et al. [40] and the Holloman relationship in an attempt to quantitatively describe the strain hardening behaviour in the FCC phase. Hardening angles were measured from the region of the stress-strain data where contact had been established in the steady-state flow region to the onset of unloading, using 0.04 and 0.15 strains to define the end points. We observed that the measured angles fall within the range of 2 to 11 degree. This trend suggests

that some marginal strain hardening is present in this region and does not appear to be size dependent, as shown in Figure 10(a). The Holloman formula, $\sigma = K\epsilon^n$ defines the relationship between the engineering stress and engineering strain, with K being the strength coefficient, the measured parameter, n, is the strain hardening exponent. 4 % and 15 % strains were also used as end points in this calculation. We calculated n to vary between 0.06 and 0.15; as shown in Figure 10(b). This shows that no apparent size effect or exponent trend in strain hardening were present in the FCC phase of the HEA. The discrete strain bursts observed in the stress vs strain curve of Figure 5(c) suggests irregular hardening angles.

Figure 9: (a) Hardening angles vs pillar diameter (μm) (b) Strain hardening exponent plot of the FCC phase of the $\text{Al}_{0.7}\text{CoCrFeNi}$ HEA

We also calculated the strain hardening exponent, n, for the nanopillars in the BCC phase, and found n to be in the range between values of 0.22 to 0.35 with the smallest pillar size of 400 nm having the highest n. This observed size effect on the strain hardening exponent may be explained in terms of the initial dislocation densities present in the nanopillars, with more cross slip events of the screw dislocations in the smaller pillars.

Figure 10: Strain hardening exponent plot of the BCC phase of the $\text{Al}_{0.7}\text{CoCrFeNi}$ HEA

5. Conclusion

We studied the microstructure and mechanical properties of the $Al_{0.7}CoCrFeNi$ high entropy alloy, which revealed the presence of two distinct crystallographic phases: (1) FCC phase which is the Co-Cr-Fe rich region or “the interdendrite” and (2) BCC (A2 + B2) phase which is the Al-Ni rich region a.k.a. “the dendrite”. We fabricated nanopillars ranging from 400 nm to 2 μ m and uniaxially compressed them to study their mechanical response. We observed very high strengths in both phases; $\sim$ 2.2 GPa for the 400 nm nanopillars in the BCC phase and $\sim$ 1.2 GPa for equivalent size nanopillars in the FCC phase. The yield strength of the BCC phase is 1.5 times higher than that of the bulk. The yield strength of the FCC phase is a factor of 1.2 greater than the bulk. We found that the size dependence of strength on pillar diameter in the FCC phase of the HEA is similar to pure FCC metals, with a strengthening exponent of - 0.66. We discovered that the BCC phase also exhibits a “smaller is stronger” size effect with an exponent of - 0.28 between strength and pillar diameter, which is lower than all size dependence reported for pure BCC metals with only Tungsten W approaching it at - 0.32. The absolute strength of the nano-pillars fabricated from the FCC phase is a factor of 1.5 higher than those of nickel (which is the strongest of the commonly studied FCC metals). We also observed pronounced strain hardening during the plastic deformation of the BCC phase, which in itself is size dependent, with smaller nano-pillars having greater strengthening with strain.

The strain hardening observed in the FCC phase is marginal and not size dependent, likely due to a lack of dislocation interactions and multiplication. We postulate that the difference in the size dependence between the two phases is as a result of the difference in the lattice resistance coming from higher presumed distortion energy in the BCC phase compared to the FCC phase.

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