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ORIGINAL REPORT



Thermo-mechanical response of single-phase face-centered-cubic $\text{Al}_x\text{CoCrFeNi}$ high-entropy alloy microcrystals

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ABSTRACT

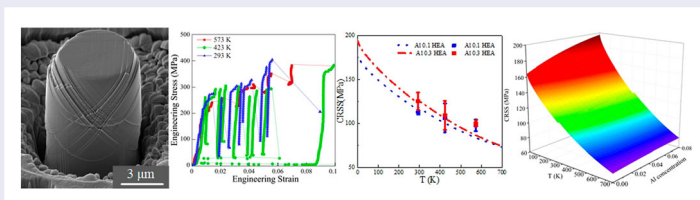
The response of [100]-oriented single-crystal face-centered-cubic $\text{Al}_{0.1}\text{CoCrFeNi}$ and $\text{Al}_{0.3}\text{CoCrFeNi}$ high-entropy alloy (HEA) microcrystals tested from 293 to 573 K by means of *in situ* micro-compression is reported. At all temperatures, plasticity is governed by dislocation slip with significant strain hardening and intermittent strain bursts observed. A model, which is in good agreement with experimental measurements, is also developed to predict the effects of Al concentration, temperature, and crystal size on the strength of HEAs. The model interestingly predicts a softening response with an increase in Al concentration when the crystal size is $\leq 0.1 \mu\text{m}$. Finally, this model can guide the development of advanced HEAs for small-scale applications.

ARTICLE HISTORY

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KEYWORDS

High-entropy alloys; solid-solution strengthening; *in situ* experiments; high-temperature deformation



IMPACT STATEMENT

In situ scanning electron microscopy (SEM) experiments are performed to quantify the thermo-mechanical response of $\text{Al}_x\text{CoCrFeNi}$ microcrystals. A physics-based model is also proposed to predict the strength of high-entropy alloys as a function of crystal size, temperature, and Al concentration.

High-entropy alloys (HEAs) are nominally single-phase random solid solutions formed from equal or nearly equal atomic concentrations of multiple constituents [1–7]. These innovative alloys have drawn substantial attention in recent years due to the vast available composition space for tuning their properties. Several HEA systems have been shown to demonstrate immense potential for a wide range of functional and structural applications. Examples include the metastable dual-phase FeMnCoCr HEAs that demonstrate extensive hardening and massive strengthening [8], NbMoTaWV HEAs that exhibit better high-temperature strength than currently available commercial superalloys [9], and AlCoCrCuFeNi

HEAs that possess significantly improved fatigue resistance [10].

Despite extensive efforts that have been devoted to study the mechanical behavior of various HEA systems [11–17], the underlying deformation mechanism, in particular at high temperatures [18,19], is not yet fully addressed. The Fleischer model for dilute concentration alloys cannot be applied to HEA systems, and statistical-based Labusch-type models that may work for all concentrations are still in their infancy [20–22].

In $\text{Al}_x\text{CoCrFeNi}$ HEA systems, the Al atoms have a much larger radius, compared to the other atoms comprising this alloy. Therefore, the increase in the Al content

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would lead to a significant increase in the lattice constant and distortion of this HEA [23]. On the other hand, increasing the Al concentration will also lead to solid-solution hardening via the formation of strong covalent bonds with neighboring atoms, and the generation of hard atomic clusters [24].

In this paper, the effect of Al concentration on the temperature-dependent mechanical response of [100]-oriented single-crystal face-centered-cubic (FCC) $\text{Al}_x\text{CoCrFeNi}$ HEA (in molar ratio) microcrystals is investigated using *in situ* SEM micro-compression experiments. If the Al composition is such that $x > 0.4$, a secondary body-centered-cubic (BCC) phase will solidify [25,26], the chemical compositions are thus limited in this study to $x = 0.1$ and $x = 0.3$ to ensure that the crystal is single-phase FCC.

The $\text{Al}_{0.1}\text{CoCrFeNi}$ HEA, referred to hereafter as the $\text{Al}_{0.1}$ HEA, was procured by Sophisticated Alloys Inc. in the form of cast blocks. Electrical discharge machining was used to cut a block into ~ 4 -mm thick sheet. From that sheet, ~ 12.7 mm diameter cylinders were machined using the Tormach PCNC 1100 mill. The $\text{Al}_{0.3}\text{CoCrFeNi}$ HEA, referred to hereafter as the $\text{Al}_{0.3}$ HEA, was prepared by arc-melting pure elements under a high-purity argon atmosphere on a water-cooled Cu hearth [27].

Both as-forged polycrystalline HEA alloys were mechanically polished using the silicon carbide grinding paper (400, 600, 800, and 1200 grit) and $0.02\text{ }\mu\text{m}$ colloidal silica formula. The chemical composition was confirmed using energy-dispersive spectroscopy and the crystal orientation of the HEA was characterized using electron backscatter diffraction inside a Tescan Mira Field Emission-Scanning Electron Microscope (FE-SEM).

Pillar-like microcrystals having a diameter $D = 5.0\text{ }\mu\text{m}$ were fabricated within a [100]-oriented single grain by the focused ion beam (FIB, FEI Strata DB235) annular milling. This size was chosen as an intermediate pillar size to avoid complications associated with smaller pillar sizes in terms of large taper and FIB-induced damage, while not being too large to avoid excessive fabrication times [28]. An aspect ratio of 2:1 (height:mid-plane diameter) was chosen to avoid buckling for higher aspect ratios and non-uniform stress along the length for lower aspect ratios [29].

Micro-compression tests of the single-crystal HEA microcrystals were performed using the InSEM HT (Nanomechanics Inc.) *in situ* indenter, equipped with a $15\text{-}\mu\text{m}$ diameter diamond flat punch tip in a Tescan Mira FE-SEM. All experiments were carried out at a nominal strain rate of 10^{-3} s^{-1} and at three temperatures: $T = 293$, 423 , and 573 K . While the instrument is inherently load controlled, the displacement rate was controlled via feedback from the load signal in order to

achieve a constant strain rate. If a displacement jump larger than 10 nm was recorded during the deformation (e.g. due to a large strain burst), the microcrystal is unloaded by 70% for the 293 and 423 K case, and by 10% for the 573 K case, then reloaded with the same procedure discussed above. Engineering stress and engineering strain were calculated by dividing the load and displacement by the initial mid-plane cross-sectional area and pillar height, respectively. The raw displacement data, measured at a data acquisition rate of 100 Hz , were corrected to account for the additional compliance due to deformation of the base along with the pillar during loading [30].

Representative stress-strain curves at different temperatures and the typical deformed morphologies at 293 and 573 K for the $\text{Al}_{0.1}$ and $\text{Al}_{0.3}$ HEAs are shown in Figures 1 and 2, respectively. The average and standard deviation of the CRSS at 2% and 5% strains from all tested samples are summarized in Table 1 and Figure 3, and accelerated *in situ* videos can be found in Supplementary Files. The apparent vibrations in the videos at higher temperatures are only shimmering effects. Irrespective of the test temperature, the *in situ* experiment revealed that as the load is applied, the microcrystals elastically deform until the onset of plasticity, at which point the first slip band and strain burst are observed. After plastic flow initiates, the microcrystals exhibit intermittent plasticity events that are evident by the successive strain bursts (i.e. load drops). Since the microcrystals are oriented in a multi-slip orientation, both samples exhibit strong work hardening. Furthermore, for both compositions, the flow stresses are observed to decrease modestly with increasing temperature. It is also observed that the $\text{Al}_{0.3}$ HEA microcrystals are on average stronger than the $\text{Al}_{0.1}$ HEA ones at all tested temperature due to the solid-solution strengthening effect of Al. However, since the difference in Al concentration is only less than 5% between the two HEAs (i.e. 2.4% versus 7%), this enhanced strengthening effect is small.

As expected for the deformation of [001]-oriented crystals, symmetric-slip deformation from all four {111} slip planes is observed. Twinning has also been commonly observed in coarse-grained (CG) polycrystalline $\text{Al}_x\text{CoCrFeNi}$ HEAs after large deformation and extensive work hardening at a wide range of strain rates (10^{-3} to 10^3 s^{-1}) [31]. However, in the current study, TEM lift-outs from the center of the deformed microcrystals did not show any indication of deformation twinning. This lack of deformation twinning can be rationalized by the low strain levels reached in the current experiments ($\sim 10\%$ strain), as compared to bulk crystals. In addition, it is commonly observed in other crystal systems that the twinning

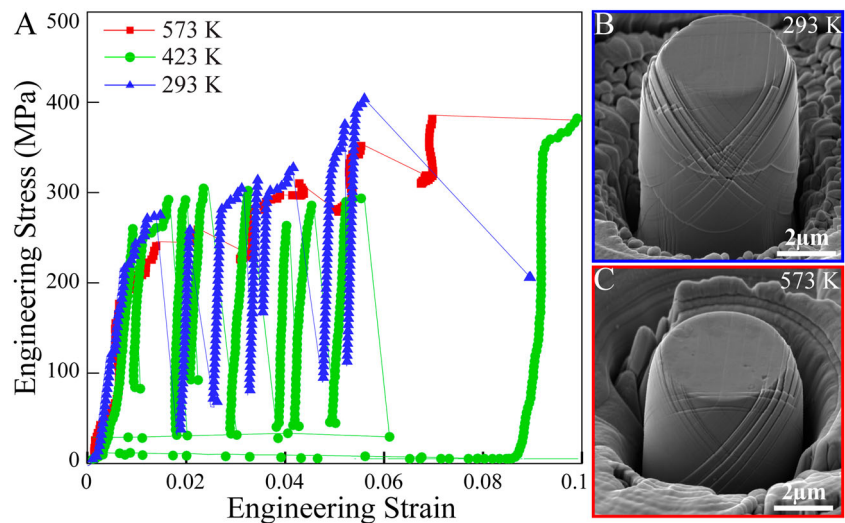


Figure 1. (a) Representative stress–strain curves for the Al_{0.1}CoCrFeNi HEA tested at various temperatures. Representative SEM micrographs of the deformed microcrystals at (b) 293 K and (c) 573 K.

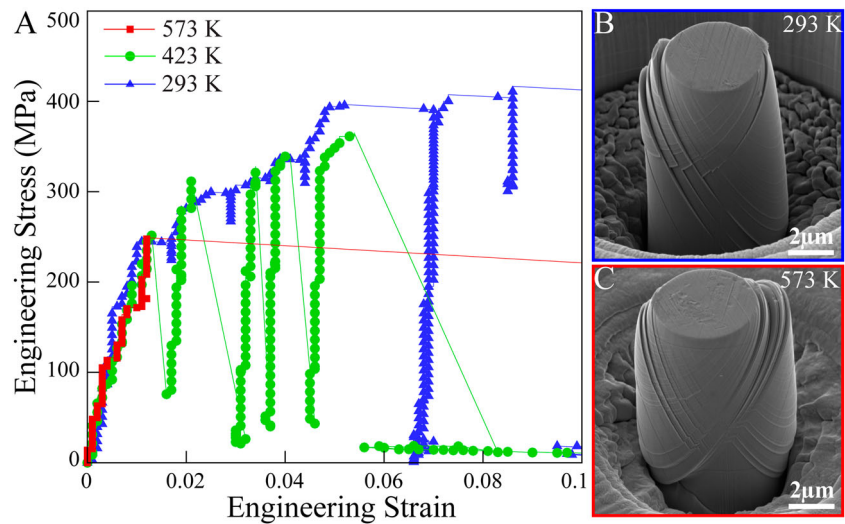


Figure 2. (a) Representative stress–strain curves for the Al_{0.3}CoCrFeNi HEA tested at various temperatures. Representative SEM micrographs of the deformed microcrystals at (b) 293 K and (c) 573 K.

Table 1. Summary of the experimentally measured critical resolve shear stress (CRSS) as well as predicted from Equation (1) for both tested compositions at different temperatures.

HEA system	Temperature (K)	2% Flow stress (MPa)	5% Flow stress (MPa)	CRSS ^a (MPa)	τ_{ss} (MPa)	$\tau_{athermal}$ (MPa)	CRSS from Equation (1) (MPa)
Al _{0.1} CoCrFeNi	293	276 ± 7	352 ± 40	112.6 ± 2.9	48.9	69.6	118.5
	423	260 ± 40	313 ± 37	106.1 ± 16.3	33.7	66.3	100.0
	573	239 ± 16	356	97.5 ± 6.5	21	62.4	83.4
Al _{0.3} CoCrFeNi	293	307 ± 24	395 ± 19	125.3 ± 9.8	58	64.8	122.8
	423	267 ± 40	307 ± 40	108.9 ± 16.3	43.9	61.7	105.5
	573	244 ± 9	244 ± 9	99.6 ± 3.7	28.1	58.1	86.2

^aCRSS is calculated from the 2% flow stress based on a Schmid factor of 0.408.

nucleation stress increases with decreasing sizes [32,33]. In addition, recent studies on single-crystal and polycrystalline materials reported that the power-law exponent for deformation-twinning-mediated plasticity is much larger than that for dislocation-mediated plasticity

[32–36]. That is, deformation twinning shows stronger crystal-size effects. Therefore, even if deformation-twinning-mediated plasticity governs the mechanical response at the bulk scale, it is plausible that there is a crystal size below which dislocation-mediated

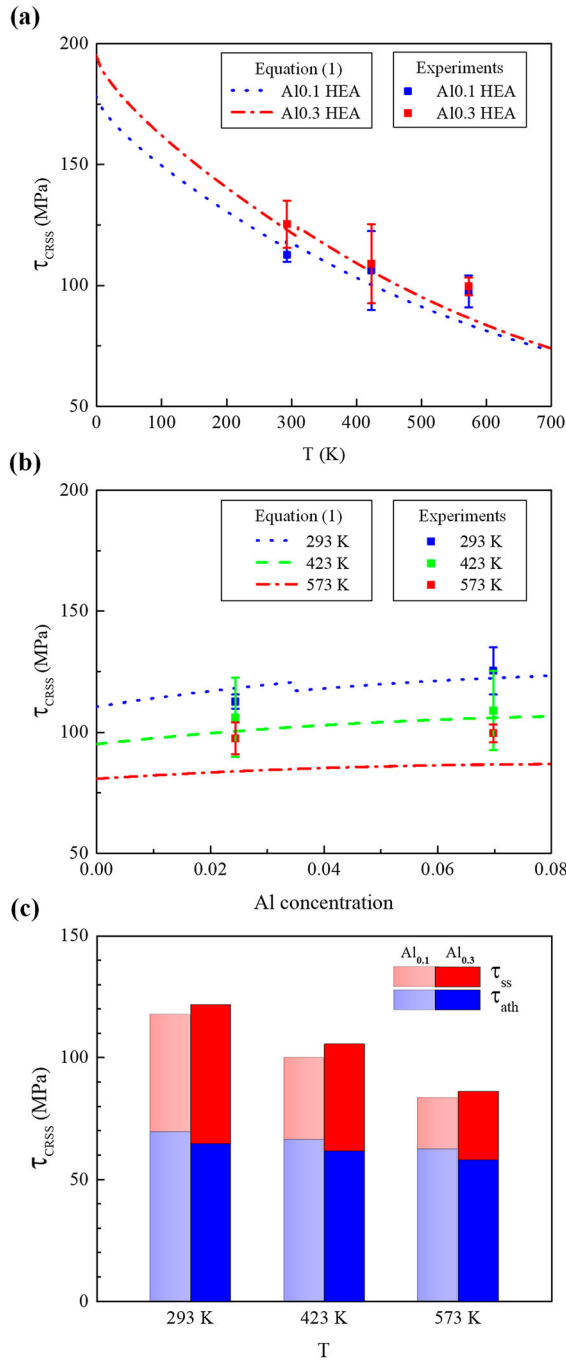


Figure 3. The variation of τ_{CRSS} as a function of (a) temperature and (b) Al concentration for $\text{Al}_x\text{CoCrFeNi}$ microcrystals having $D = 5.0 \mu\text{m}$, $\rho = 3 \times 10^{13}/\text{m}^{-2}$, and $\dot{\epsilon} = 10^{-3}\text{s}^{-1}$ as predicted by Equation (1). The experimental results are also shown for comparison. (c) τ_{ath} and τ_{ss} are represented in stacked bar chart for all tested temperatures and compositions for the demonstration of the contribution of each.

plasticity dominates over deformation twinning (i.e. when $\text{CRSS}_{\text{slip}} < \text{CRSS}_{\text{twinning}}$).

Under the premise that deformation-twinning-mediated plasticity is excluded from such micron-sized samples, in the following a dislocation-based model is developed to describe the effect of Al concentration (C_{Al}),

temperature (T), dislocation density (ρ), strain rate ($\dot{\epsilon}$), and crystal size (D) on the CRSS of $\text{Al}_x\text{CoCrFeNi}$ microcrystals. In this model, the CRSS, $\tau_{\text{CRSS}}(T, \dot{\epsilon}, D, \rho)$, is decomposed into two components, such that:

$$\tau_{\text{CRSS}}(T, \dot{\epsilon}, D, \rho) = \tau_{\text{athermal}}(D, \rho) + \tau_{\text{ss}}(T, \dot{\epsilon}), \quad (1)$$

where $\tau_{\text{athermal}}(D, \rho)$ is the contribution from athermal mechanisms and $\tau_{\text{ss}}(T, \dot{\epsilon})$ is the contribution from thermally activated mechanisms during plastic deformation. The athermal contribution is directly associated with the long-range elastic interactions of dislocations, which is well described by the generalized size-dependent Taylor-strengthening law [28]:

$$\tau_{\text{athermal}} = \mu \left(\frac{\beta}{D\sqrt{\rho}} \right) + \alpha b\sqrt{\rho}, \quad (2)$$

where $\beta = 1.76 \times 10^{-3}$, $\alpha = 0.57$ are dimensionless constants, and μ is the shear modulus as a function of temperature and Al concentration. The first term on the right-hand side of Equation (2) is the stress required to activate the weakest dislocation source, while the second term accounts for hardening arising from dislocation forest interactions. Experimental and ab initio simulation results suggest that by treating CoCrFeNi as an effective medium, the material parameters can be approximated as a bilinear function of temperature, T , and Al concentration, C_{Al} . By fitting data in the literature, the shear modulus can be shown to be: $\mu(T, C_{\text{Al}}) = (1 - 1.51C_{\text{Al}}) \times (-0.031T + 93.5)$, while Poisson's ratio ν and atomic volume V are relatively temperature insensitive and only depend on the Al concentration according to: $\nu(C_{\text{Al}}) = 0.28 + 0.2C_{\text{Al}}$ and $V(C_{\text{Al}}) = (1 - C_{\text{Al}})V_{\text{CoCrFeNi}} + C_{\text{Al}}V_{\text{Al}}$ [22,37].

The thermal contribution, $\tau_{\text{ss}}(T, \dot{\epsilon})$, originates from the local energy barrier for dislocation slip that can be overcome by local thermal fluctuations. In the current HEA system, τ_{ss} is predominantly dominated by the lattice friction originating from solid-solution strengthening. Here, a recently developed Labusch-type theory is adopted to estimate the solid-solution strengthening [38]. In this theory, τ_{ss} can be computed at a finite temperature, T , and a given strain rate of $\dot{\epsilon}$, by considering each elemental component, n , as a solute atom embedded in an effective matrix of surrounding alloy, such that [22]:

$$\tau_{\text{ss}}(T, \dot{\epsilon}) = \begin{cases} \tau_{y0} \left[1 - \left(\frac{kT}{\Delta E_b} \ln \frac{\dot{\epsilon}_0}{\dot{\epsilon}} \right)^{2/3} \right] & \text{for } \tau_{\text{ss}}/\tau_{\text{ss}}^0 > 0.5, \\ \tau_{y0} \left[-\frac{1}{0.57} \frac{kT}{\Delta E_b} \ln \frac{\dot{\epsilon}_0}{\dot{\epsilon}} \right] & \text{for } \tau_{\text{ss}}/\tau_{\text{ss}}^0 < 0.5, \end{cases} \quad (3)$$

where k is the Boltzmann constant, $\dot{\epsilon}_0 = 10^4\text{s}^{-1}$ is a reference strain rate and the apparent zero-temperature flow

stress, τ_{y0} , is

$$\tau_{y0}^0 = 0.051\alpha^{-1/3}K_{\tau}f_{\tau}\left[\sum_{n=1}^N c_n\Delta V_n^2\right]^{2/3}, \quad (4)$$

while the energy barrier, ΔE_b , is

$$\Delta E_b = 0.274\alpha^{1/3}K_{\Delta E}f_{\Delta E}\left[\sum_{n=1}^N c_n\Delta V_n^2\right]^{1/3} \quad (5)$$

and

$$K_{\tau} = \mu\left(\frac{1+\nu}{1-\nu}\right)^{4/3}b^{-4}, \quad K_{\Delta E} = \mu\left(\frac{1+\nu}{1-\nu}\right)^{2/3}b, \\ f_{\tau} = 0.35, \quad f_{\Delta E} = 5.7. \quad (6)$$

Here, N is the number of elements in the HEA, and the atomic volume can be calculated as $V = b^3/\sqrt{2}$. In this study, f_{τ} and $f_{\Delta E}$ are treated as two constants related to the dislocation core structures [22,38].

Using Equation (1), the variation of τ_{CRSS} as a function of temperature and Al concentration is shown in Figure 3(a,b) for $D = 5.0 \mu\text{m}$, $\rho = 3 \times 10^{13}/\text{m}^{-2}$ (which is a representative value for FIB-milled microcrystals), and $\dot{\epsilon} = 10^{-3}\text{s}^{-1}$. The results are also summarized in

Table 1 for the tested temperatures and Al concentrations for comparison with the experimental predictions. It is clear that the predicted τ_{CRSS} as a function of temperature and Al concentration is in excellent agreement with those measured experimentally. The slightly higher CRSS at 573 K compared to the model prediction can be attributed to possibly a slightly higher dislocation density in those experiments [28]. It is also observed that the effect of Al concentration decreases with increasing temperature, which can be rationalized by the fact that the energy barrier, ΔE_b , is an increasing function of the overall atomic-size misfit from all elements (see Equation (4)). Thus, the increase in the Al concentrations raises the lattice distortion, which consequently leads to a higher energy barrier and stronger temperature dependency for dislocation slip. It should be noted that the small discontinuities in the predicted τ_{CRSS} at different temperatures and Al concentrations are a result of the piecewise Arrhenius-type relation described by Equation (3), depending on the value of $\tau_{\text{ss}}/\tau_{\text{ss}}^0$. Nevertheless, this discontinuity is very small and has no implications for the current discussion.

It is also observed in Figure 3(b) that by increasing the Al concentration up to 8%, the effect of solid-solution

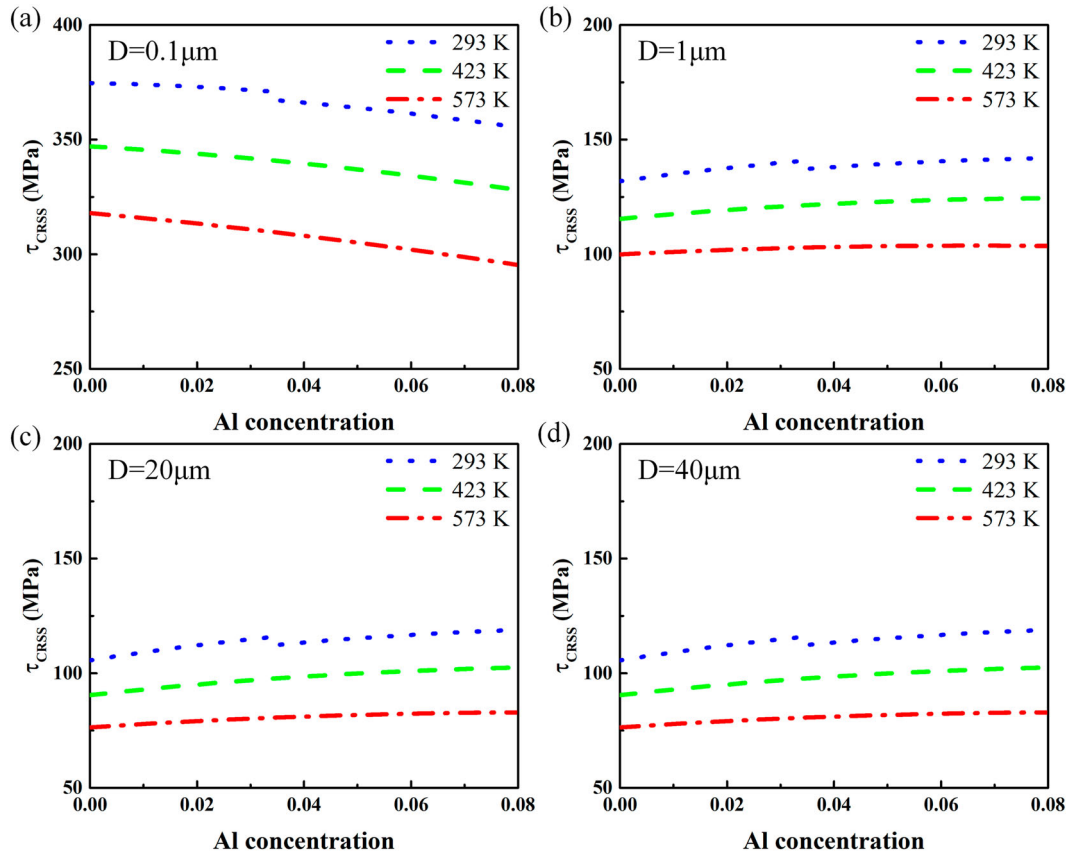


Figure 4. The CRSS for microcrystal having (a) $D = 0.1$, (b) 1, (c) 20, and (d) 40 μm , as predicted from Equation (1) for $\rho = 3 \times 10^{13}/\text{m}^{-2}$, and $\dot{\epsilon} = 10^{-3}\text{s}^{-1}$.

strengthening does not increase significantly. This can be attributed to two competing effects. On one hand, the relative large atomic misfit of Al atoms leads to solid-solution strengthening, while on the other hand, it leads to elastic softening. Considering that an Al concentration higher than 8% will lead to the formation of a BCC phase [25,26], the focus here is only on the Al concentration below this limit.

The currently proposed model demonstrates a similar strengthening effect of Al at all three testing temperatures and is in good agreement with the current experimental results. There is a slight discrepancy in the overall CRSS level for the highest temperature tested here (i.e. 573 K), although the strengthening effect of Al is well captured qualitatively. Nevertheless, a better approximation could be readily achieved by slightly adjusting f_τ and $f_{\Delta E}$ in Equations (5) and (6), which would give a better dislocation core structure description at elevated temperatures [39].

The relative contribution of τ_{athermal} and τ_{ss} on the overall CRSS of the HEA microcrystal is summarized in Table 1 and as a stacked bar chart in Figure 3(c). It is clear that the significant drop in strength is predominantly associated with the decrease in τ_{ss} , which on the other hand is attributed to thermal softening. It is also clear that for the crystal size studied here, the contribution of τ_{athermal} and τ_{ss} to the total CRSS of the microcrystal is on the same order (tens of MPa) due to the sample size effect. This trend might not be the case for other crystal sizes, for example, for bulk single-crystal or CG polycrystalline sample, the contribution from τ_{ss} will dominate the CRSS.

The model, represented by Equation (1), can give a general understanding of the relative effects of Al composition, temperature, and crystal size on the CRSS of $\text{Al}_x\text{CoCrFeNi}$, as shown in Figure 4. It is clear that for a given concentration and temperature, a decrease in the crystal size leads to an increase in the CRSS in agreement with the ‘smaller is stronger’ phenomenon. It is also observed that for $D \geq 20 \mu\text{m}$, the effect of size becomes irrelevant, and the crystal strength reaches a steady state that is equivalent to the bulk strength. Furthermore, the increase in temperature for a given crystal size and Al concentration will lead to a decrease in the crystal strength. On the other hand, it is interesting to note that due to the elastic softening effect of the added Al, when $D \leq 0.1 \mu\text{m}$, the increase in the Al concentration leads to a decrease in the predicted CRSS (Figure 4(a)). However, for larger crystals, the increase in Al concentration will always lead to strengthening in agreement with the solid-solution hardening effects. It should be noted that although in the current study only experiments on micron-sized samples are used to verify the

model, the model could be extended to bulk sample naturally. By assuming a dislocation density of 10^{12} and using a sample diameter of 2 mm, this model yields a prediction of 68.2 MPa CRSS which is very close to 75.2 MPa measurement [40].

In summary, *in situ* micro-mechanical testing technique was utilized to study the effect of Al composition and temperature on the mechanical properties and deformation mechanism for $\text{Al}_x\text{CoCrFeNi}$ microcrystals. The experimental results show that, unlike bulk samples, plasticity is mediated by dislocation slip without any observation of deformation twinning. In addition, a physics-based model was developed to predict the effect of Al concentration, temperature, and crystal size on the strength of $\text{Al}_x\text{CoCrFeNi}$ microcrystals. In this model, the crystal strength is proposed to be dominated by both the long-range athermal and short-range thermal contributions in a linearly additive manner. The model was shown to match well with the experimentally measured CRSS. The model also interestingly predicts that when the sample size decreases below $0.1 \mu\text{m}$, the increase in Al concentration leads to a softening effect. Finally, the currently proposed model is most likely to have a more general validity for a wider range of HEAs, and provides a guide for the development of advanced alloys for small-scale applications.

Disclosure statement

No potential conflict of interest was reported by the authors.

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