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ORIGINAL REPORT



Novel Co-rich high entropy alloys with superior tensile properties

Daixiu Wei^{a,b}, Xiaoqing Li^c, Weicheng Heng^{a,d}, Yuichiro Koizumi^a, Feng He^{b,e}, Won-Mi Choi^f, Byeong-Joo Lee^f, Hyoung Seop Kim^f, Hidemi Kato^a and Akihiko Chiba^a

^aInstitute for Materials Research, Tohoku University, Sendai, Japan; ^bDepartment of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, USA; ^cDepartment of Materials Science and Engineering, KTH – Royal Institute of Technology Stockholm, Sweden; ^dDepartment of Materials Processing, Tohoku University, Sendai, Japan; ^eState Key Laboratory of Solidification Processing, Northwestern Polytechnical University, Xi'an, People's Republic of China; ^fDepartment of Materials Science and Engineering, Pohang University of Science and Technology (POSTEC), Pohang, Republic of Korea

ABSTRACT

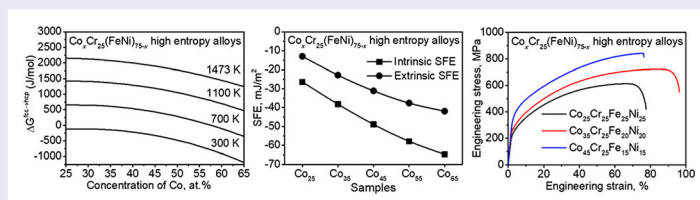
We developed a series of Co-rich $\text{Co}_x\text{Cr}_{25}(\text{FeNi})_{75-x}$ ($x = 35, 45, 55, 65$) high entropy alloys with improved strength and/or ductility, derived from lowering the stacking fault energy (SFE) and reducing the fcc phase stability of the equiatomic CoCrFeNi alloy. Thermodynamics and *ab initio* calculations demonstrated that increasing Co while decreasing Fe and Ni concentrations lower the SFE and reduce the fcc phase stability. The $\text{Co}_{35}\text{Cr}_{25}\text{Fe}_{20}\text{Ni}_{20}$ and $\text{Co}_{45}\text{Cr}_{25}\text{Fe}_{15}\text{Ni}_{15}$ alloys with single fcc phase, exhibit superior tensile properties, contributing to the twinning and fcc $\rightarrow$ hcp martensitic transformation. The present study offers a guideline for designing high-performance high entropy alloys.

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KEYWORDS

High entropy alloy; metastable; stacking fault energy; martensitic transformation; deformation twinning



IMPACT STATEMENT

A series of novel Co-rich non-equiatom high entropy alloys with enhanced tensile properties were developed by lowering the stacking fault energy and reducing the phase stability of equiatomic CoCrFeNi alloy.

1. Introduction

High entropy alloys (HEAs) comprise four or more multiple principle elements have attracted extensive academic interests, which was firstly proposed in the year 2004 [1,2]. Thereinto, the equiatomic FeMnCoCrNi alloy is one of the most thoroughly studied HEAs because of the exceptional fcc phase stability, good combinations of strength and ductility, and superior fracture resistance at cryogenic temperatures [2,3]. The interesting properties are considered as an outcome of heavy lattice distortions and sluggish diffusion by mixing the multiple principle elements [1,4–6]. Dislocation slip is the sole plastic deformation mode of the alloy at room temperature, but deformation twinning occurs at cryogenic temperatures

because of the lowered stacking fault energy (SFE). The SFEs of FeMnCoCrNi and FeNiCoCr alloys are estimated to be 20–30 mJ/m² at room temperature but decreased to approximate zero at 0 K, meanwhile the hcp phase tends to become more stable with decreasing temperatures [7,8].

Moreover, twinning-induced plasticity (TWIP) and transformation-induced plasticity assisted dual phase (TRIP-DP) non-equiatom Fe-rich HEAs were recently developed [9,10], inspired by the plasticity and strengthening mechanisms in many low SFE metals and alloys such as TRIP steel, metastable austenitic steel, and TWIP steel. The two types of HEAs exhibit high tensile strengths and large elongations at room temperature that

CONTACT Daixiu Wei ✉ wei1987xiu@imr.tohoku.ac.jp Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Sendai, Miyagi 980-8577, Japan; Department of Materials Science and Engineering, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, MA 02139, USA; Xiaoqing Li ✉ xiaoqli@kth.se Department of Materials Science and Engineering, KTH – Royal Institute of Technology, 10044 Stockholm, Sweden

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overcome the strength-ductility trade-off, owing to the assistance of deformation twinning and strain-induced martensitic transformation (SIMT), respectively. The low SFE of metastable fcc phase of the alloys gained from tuning the chemical composition and concentration, enables the formation of Shockley partial dislocations, which is a crucial requisite for the activation of twinning and SIMT when the applied stress exceeds a threshold value.

On the other hand, Co-based superalloys have been widely used in manufacturing vanes, gas turbines, and metallic orthopedic implants, owing to the superior mechanical properties and corrosion resistance [11–13]. The alloys generally exhibit two kinds of crystal structures including γ -fcc and ϵ -hcp, where the γ phase is thermodynamically stable at elevated temperatures whereas the ϵ phase is stable at ambient temperature [14]. Generally, the SFE of these alloys is extremely low, reaching negative values at room temperature for some compositions [15]. As a result, the $\gamma \rightarrow \epsilon$ SIMT proceeds during plastic straining of the metastable γ phase, which partially contributes to the exceptional mechanical properties [16,17]. Furthermore, elements of Fe and Ni are γ phase stabilizers for the alloy while Cr is the ϵ phase stabilizer (i.e. alloying with Fe and Ni can stabilize the γ phase and increase the SFE, whereas alloying with Cr will stabilize the ϵ phase and lower the SFE of the alloys) [18]. In the present study, we aimed to design novel Co-rich HEAs with manipulated mechanical properties and deformation behaviors by regulating the SFE and phase stability, achieved by adjusting the elemental concentrations of the equiatomic CoCr-FeNi alloy based on the thermodynamics and *ab initio* calculations.

2. Methodology

We designed quaternary HEAs described as $\text{Co}_x\text{Cr}_{25}(\text{FeNi})_{75-x}$ ($x = 25, 35, 45, 55, 65$, hereinafter denoted by Co_{25} , Co_{35} , Co_{45} , Co_{55} , and Co_{65} alloys, respectively). The Gibbs free energy difference between hcp phase and fcc phase ($\Delta G^{\text{fcc} \rightarrow \text{hcp}}$, which is defined as $\Delta G^{\text{fcc} \rightarrow \text{hcp}} = G^{\text{hcp}} - G^{\text{fcc}}$) of the various alloys, were calculated using Thermo-Calc software with the TCFe2000 thermodynamic database and its upgraded version [19,20]. The generalized stacking fault energy (GSFE) of the alloys were calculated by *ab initio* calculations based on density functional theory (DFT) method [21], and the Kohn–Sham equations were solved using the exact muffin-tin orbitals method (EMTO) [22,23].

Ingots of the alloys were produced by arc melting in a water-cooled copper hearth, followed by homogenization at 1200°C for 5 h in an Ar atmosphere to ensure the chemical homogeneity. Then, the alloys hot forged at 1200°C to

a 50% reduction in thickness, and finally room temperature rolled to 40% reduction in thickness again with subsequent annealing at 1000°C for 6 min. After that, dog-bone-shaped tensile specimens with a gauge geometry of 10 mm $\times$ 2 mm $\times$ 1 mm were sliced by electrical discharge machining. The tensile specimens were grinded and mirror-finished. Then, uniaxial tensile tests were conducted at 25°C with a strain rate of $1.5 \times 10^{-4} \text{ s}^{-1}$. The microstructures of the specimens were observed by scanning electron microscope (SEM) equipped with an electron backscatter diffraction detector (EBSD), using an acceleration voltage of 15 kV. The phase identification was carried out by X-ray diffraction (XRD) with Co $K\alpha$ radiation. The fractured samples were characterized by scanning transmission electron microscope (STEM) with operation voltage of 200 kV, and the thin-foil specimens for STEM observation were prepared by ion milling.

3. Results and discussion

The Gibbs free energy difference curves of $\Delta G^{\text{fcc} \rightarrow \text{hcp}}$ in Figure 1(a) illustrates that an increase in Co content leads to a decrease in fcc phase (γ phase) stability in the $\text{Co}_x\text{Cr}_{25}(\text{FeNi})_{75-x}$ alloys. Moreover, γ phase is more stable at elevated temperatures whereas the hcp phase (ϵ phase) tends to be more stable at lower temperatures, which is in accordance with traditional Co-based alloys. The curves of GSFE of the alloys at 0 K in Figure 1(b) show the initiation of one whole stacking fault by shifting along $1/6 \langle 112 \rangle_\gamma$ at close-packed $\{111\}_\gamma$ planes. The unstable SFE of γ_{usf} , which represents the lowest energy barrier for dislocation nucleation [24], increases gradually from 343.2 to 412.5 mJ/m² accompanied by the increase of Co content. It indicates that the Co-rich alloys will exhibit higher resistance for dislocation nucleation (i.e. higher yield strength than the Co-lean alloys). On the other hand, the values of intrinsic and extrinsic SFEs marked by γ_{isf} and γ_{esf} are compared in Figure 1(c). It indicates that both of the γ_{isf} and γ_{esf} are lowered by increasing Co content, and the values of γ_{isf} are smaller than γ_{esf} . It has been found that the Fe and Ni stabilize the γ phase in Co-based superalloys. In addition, independent reduction of Ni concentration or an increase in the Co concentration does significantly lower the SFE [18]. Therefore, the coordination lowers the SFE and reduces the γ phase stability, which will affect the deformation behaviors.

Figure 2(a–e) depict the EBSD IPF maps containing average grain sizes of the Co_{25} (Figure 2(a)), Co_{35} (Figure 2(b)), Co_{45} (Figure 2(c)), Co_{55} (Figure 2(d)) and Co_{65} (Figure 2(e)) alloys. The grains are fully recrystallized without obvious distortions, and the grain size decreases gradually from 14.9 μm to 6.2 μm with the

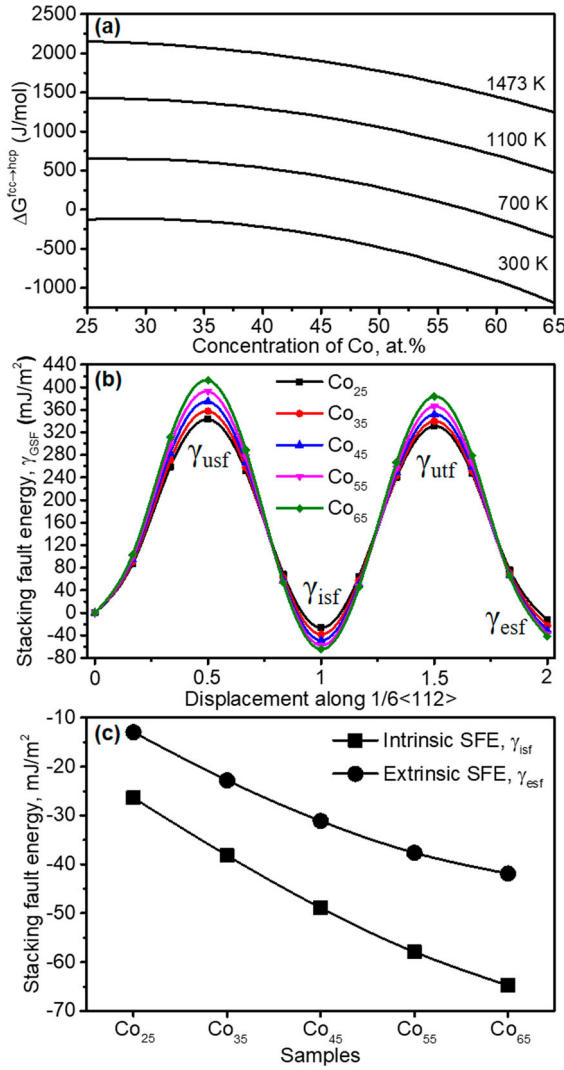


Figure 1. (a) The $\Delta G^{\text{fcc} \rightarrow \text{hcp}}$ of $\text{Co}_x\text{Cr}_{25}(\text{FeNi})_{75-x}$ (x : 25 ~ 65) HEAs calculated by Thermo-Calc; (b) generalized-stacking fault energy of $\{111\}(112)$ slip and (c) the intrinsic and extrinsic stacking fault energies of the HEAs at 0 K calculated by DFT method.

increase of Co content. Because of the tiny difference in grain sizes of the $\text{Co}_{25\sim 45}$ samples, the effect of grain size on the tensile properties of those samples can be neglected. On the other hand, the corresponding phase maps in Figure 2(f–j) show the phase composition of the samples. The $\text{Co}_{25\sim 45}$ samples are single γ phase containing some annealing twin bands, while the $\text{Co}_{55\sim 65}$ samples are $\gamma + \epsilon$ dual phase with lath morphologies. The amount of ϵ phase in the Co_{55} and Co_{65} samples are 62.8 and 91.3%, respectively. The phase composition would affect the tensile properties dramatically, because the γ phase is generally soft and ductile but the ϵ phase is hard and brittle.

The engineering stress–strain curves in Figure 3(a) and the strengths and elongations in Figure 3(b) demonstrate the tensile properties of the alloys at room

temperature. The $\text{Co}_{25\sim 45}$ samples comprising single γ phase show superior fracture elongation larger than 76%, but the $\text{Co}_{55\sim 65}$ samples consisting of a large amount of ϵ phase have very limited elongations less than 21%. On the other hand, the yield strength and ultimate tensile strength (UTS) increase co-instantaneously accompanied by the increase of Co content, with the exception of Co_{55} sample. The enhanced strength is probably due to the increase of γ_{usf} and the different amount of ϵ phase. It is noteworthy that the fracture elongation firstly increases and then decreases with a maximum value of 96.1% (Co_{35}), and the strength enhances without the expense of ductility by changing the Co content from 25 to 45%. The Co_{35} and Co_{45} samples exhibit UTS of 721 and 841 MPa with fracture elongations of 96.1% and 76.2% respectively, which are superior to the Co_{25} sample. The strain hardening curves in Figure 3(c) indicate that the Co-rich samples show relatively higher strain hardening rates, despite all the $\text{Co}_{25\sim 45}$ samples are single γ phase. Moreover, the $\gamma + \epsilon$ dual phase samples show much higher hardening rates than single γ phase samples, due to the lack of operative slip systems of the ϵ -hcp phase. Figure 3(d) is the tensile properties of Co_{35} and Co_{45} samples compared with other single fcc phase HEAs reported previously [3,8–10,25–30], which indicates an exceptional combination of strength and ductility is obtained in the two samples. It sheds light on the development of Co-rich HEAs with superior mechanical properties.

Figure 4 shows the XRD patterns of the samples both before (Figure 4(a)) and after (Figure 4(b)) tensile fracture. The $\text{Co}_{25\sim 45}$ samples comprise single γ phase meanwhile the $\text{Co}_{55\sim 65}$ samples consist of $\gamma + \epsilon$ dual phases before tensile in accordance with the EBSD results in Figure 2. However, the peaks of the ϵ phase detected in the fractured Co_{45} sample and the enhancement of the ϵ phase peaks in the fractured $\text{Co}_{55\sim 65}$ samples indicate that the $\gamma \rightarrow \epsilon$ phase transformation proceeded in those samples during plastic deformation. In contrast, the transformation did not occur in the $\text{Co}_{25\sim 35}$ samples, because of no ϵ phase peak is found in those fractured samples. It can be concluded that the room temperature plastic deformation behaviors of the samples are significantly influenced by the Co content, i.e. the SFEs and phase stabilities of the new designed HEAs alloys.

Figure 5(a–c) shows the bright filed image (Figure 5(a)), high-magnification HADDF image (Figure 5(b)), and diffraction pattern (Figure 5(c)) of the fractured Co_{25} sample. A high density of dislocations and deformation-induced nanotwins less than 10 nm thick indicated by arrowheads are observed. The twinning elements are $K_1 = (111)_\gamma$, $\eta_1 = [11\bar{2}]_\gamma$, $K_2 = (11\bar{1})_\gamma$, $\eta_2 = [112]_\gamma$ as indicated in Figure 5(b,c), which is the common

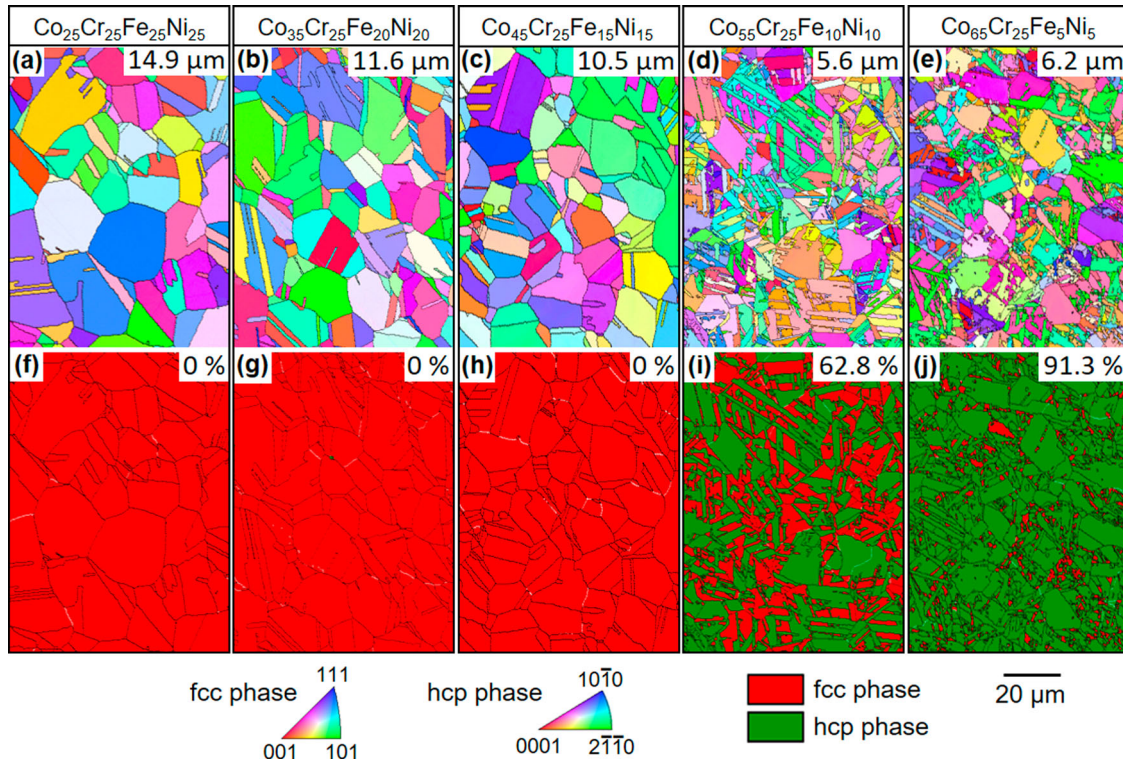


Figure 2. EBSD (a–e) IPF maps and (f–j) phase maps of the $\text{Co}_x\text{Cr}_{25}(\text{FeNi})_{75-x}$ HEAs before tensile, with the average grains sizes and fractions of hcp phase inserted in the IPF and phase maps, respectively.

operative twinning system in fcc metals formed by the motion of Shockley partial dislocations on every close-packed $\{111\}$ planes. Figure 5(d–f) is the bright field image (Figure 5(d)), high-magnification HADDF image (Figure 5(e)), and diffraction pattern (Figure 5(f)) of the fractured Co_{45} sample. A lamellar structure consisting of strip-shaped γ and ϵ phases is observed. The atomic stacking sequences are ABCABC_γ and ABABAB_ϵ along the $\langle 111 \rangle_\gamma$ and $[0001]_\epsilon$ orientations, respectively. These two phases follow an orientation relationship described as: $\{111\}_\gamma // (0001)_\epsilon$, $\langle 110 \rangle_\gamma // [11\bar{2}0]_\epsilon$, which is analogous the lamellar structure consisting of γ and α_2 phase in lamellar Ti–Al alloys [31,32]. The ϵ phase was formed in the γ matrix during plastic deformation by SIMT, which is achieved by the nucleation and propagation of Shockley partial dislocations on every second $\{111\}_\gamma$ planes. On the other hand, it can be deduced that twinning is relatively easier to be activated in the Co_{35} sample, because the SFE is lower than Co_{25} sample meanwhile no ϵ phase was detected in the fractured Co_{35} sample by XRD. The higher probability of activation of deformation twinning partially contributes to the improved strength and elongation.

The SFE determines the plastic deformation mechanisms of fcc metals, which switches the deformation mode from dislocation slip to twinning and finally

to $\text{fcc} \rightarrow \text{hcp}$ SIMT via lowering the SFE. Dissociation of a perfect dislocation with Burgers vector $\vec{b}$ of $\frac{1}{2} [\langle 110 \rangle]$ into a pair of Shockley partial dislocations with $\vec{b}$ of $\frac{1}{6} [\langle 211 \rangle]$ is often stimulated in low SFE fcc metals, which can be described as: $\frac{1}{2} [110] \rightarrow \frac{1}{6} [211] + \frac{1}{6} [12\bar{1}]$. If a partial dislocation of $\frac{1}{6} [211]$ glides on a $(111)_\gamma$ plane represented by symbol ‘|’, the stacking sequence along $[111]_\gamma$ changes from ... ABC|ABC ... to ... ABC|BCA ..., which forms an intrinsic stacking fault with local hcp stacking sequence. The gliding of partial dislocations on every second $(111)_\gamma$ planes produce ϵ phase. On the other hand, if the partial dislocations glide on every two consecutive $(111)_\gamma$ planes, the stacking sequence along $[111]_\gamma$ direction changes from ... ABCABC|A|B|C|A|BC ... to ... ABCABC|B|A|C|B|AC ..., generating a local twin with ‘C’ as the plane of symmetry. The SFE governs the dissociation and motion behaviors of the dislocations, where the intrinsic stacking fault prefers to form in metals with lower SFEs than that of extrinsic stacking fault.

The SFE and γ phase stability are lowered by increasing the Co content in the present study, which then manipulate the plastic deformation mechanisms of the alloys. In $\text{Co}_{25\sim 65}$ alloys, dislocations and deformation twinning are formed during plastic deformation. However, the $\text{fcc} \rightarrow \text{hcp}$ martensitic transformations

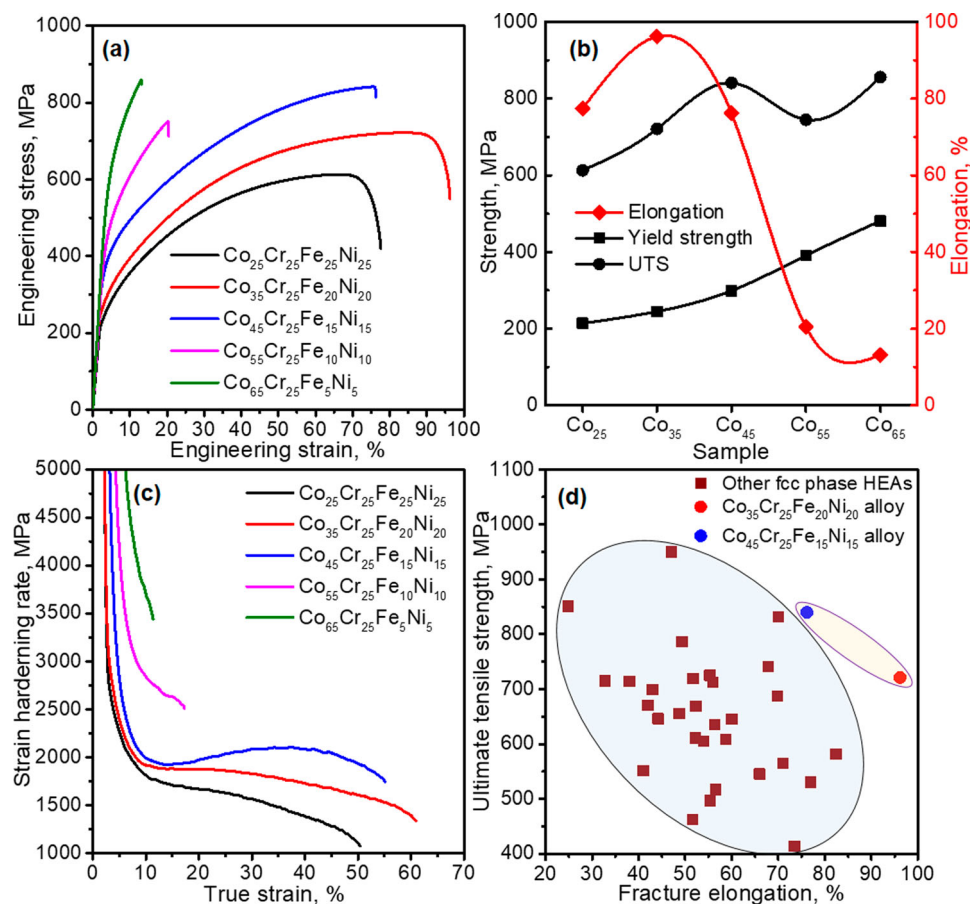


Figure 3. (a) Tensile stress–strain curves, (b) the yield strength, elongation and ultimate tensile strength, and (c) the strain hardening rates of $\text{Co}_x\text{Cr}_{25}(\text{FeNi})_{75-x}$ HEAs; (d) the strength versus elongation of $\text{Co}_{35}\text{Cr}_{25}\text{Fe}_{20}\text{Ni}_{20}$ and $\text{Co}_{45}\text{Cr}_{25}\text{Fe}_{15}\text{Ni}_{15}$ alloys compared with other single fcc phase HEAs.

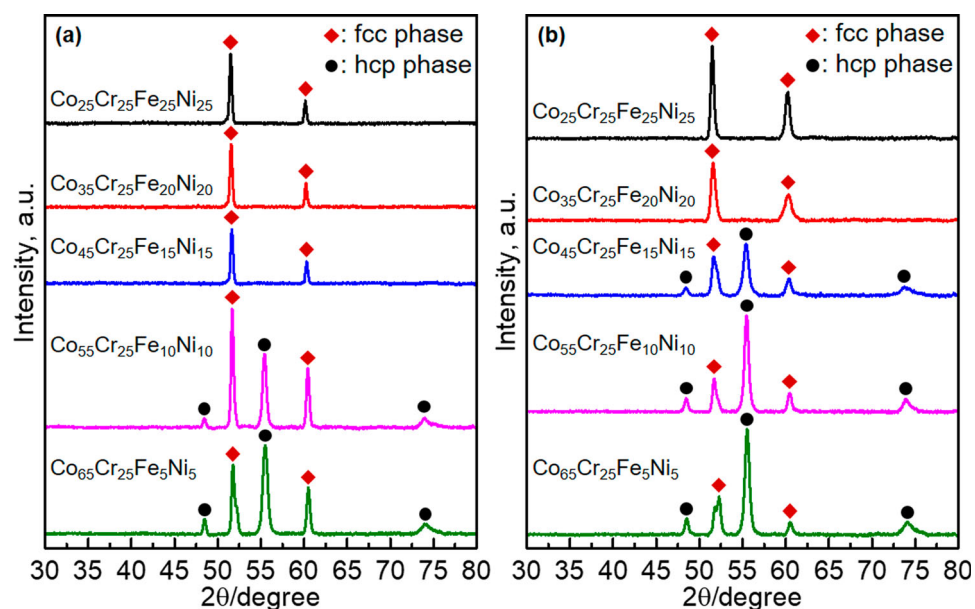


Figure 4. XRD patterns of $\text{Co}_x\text{Cr}_{25}(\text{FeNi})_{75-x}$ high entropy alloys before tensile and after tensile at room temperature.

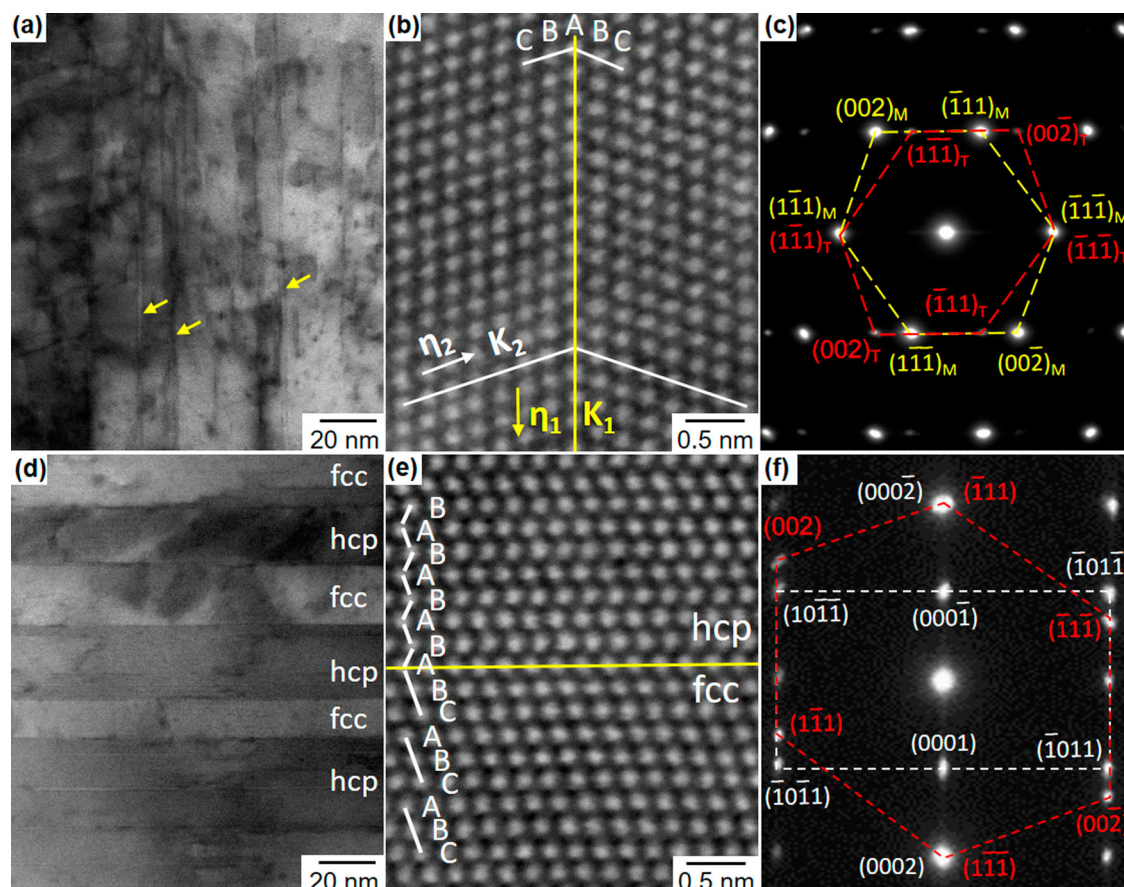


Figure 5. STEM (a, d) bright filed images, (b, e) HADDF images and (c, f) diffraction patterns of (a–c) $\text{Co}_{25}\text{Cr}_{25}\text{Fe}_{25}\text{Ni}_{25}$ alloy and (d–f) $\text{Co}_{45}\text{Cr}_{75}\text{Fe}_{15}\text{Ni}_{15}$ alloy after tensile to fracture at room temperature.

occurred in the Co_{55~65} alloys due to the lowered SFE. As a result, the Co_{55~65} alloys strengthened by TRIP effect exhibit much higher hardening rate and strength than the Co_{25~35} alloys with TWIP effect during plastic deformation at room temperature.

4. Conclusions

A series of novel Co-rich $\text{Co}_x\text{Cr}_{25}(\text{FeNi})_{75-x}$ ($x = 25-65$) HEAs with enhanced tensile properties and manipulated deformation behaviors, were designed by lowering the SFE and fcc phase stability of the equiatomic CoCrFeNi alloy. The increase of Co concentration at the expense of decreasing Fe and Ni concentrations yields a lower SFE and reduced fcc phase stability, accompanied by the deformation mechanism switches from deformation twinning to $\text{fcc} \rightarrow \text{hcp}$ martensitic transformation. Hereinto, the $\text{Co}_{35}\text{Cr}_{25}\text{Fe}_{20}\text{Ni}_{20}$ and $\text{Co}_{45}\text{Cr}_{25}\text{Fe}_{15}\text{Ni}_{15}$ alloys with metastable single fcc phase possess superior combination of strength and ductility compared to other single fcc phase counterparts. The findings here provide a

feasible guideline for developing high-performance high entropy alloys.

Disclosure statement

No potential conflict of interest was reported by the authors.

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ORCID

Hyoung Seop Kim  <http://orcid.org/0000-0002-3155-583X>

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