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To cite this article: Kenta Yamanaka, Manami Mori, Daishin Yokosuka, Kazuo Yoshida, Yusuke Onuki, Shigeo Sato & Akihiko Chiba (2024) Effect of matrix dislocation strengthening on deformation-induced martensitic transformation behavior of metastable high-entropy alloys, Materials Research Letters, 12:1, 1-9, DOI: [10.1080/21663831.2023.2281593](https://doi.org/10.1080/21663831.2023.2281593)

To link to this article: <https://doi.org/10.1080/21663831.2023.2281593>



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Effect of matrix dislocation strengthening on deformation-induced martensitic transformation behavior of metastable high-entropy alloys

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ABSTRACT

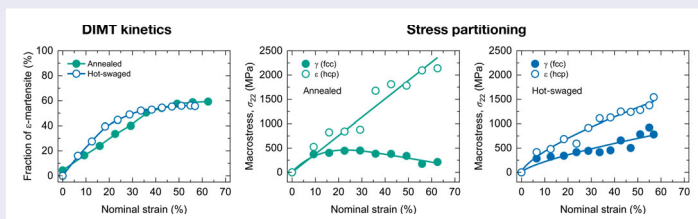
In this study, we investigate the influence of dislocation strengthening in the metastable parent phase on the deformation-induced martensitic transformation behavior of a face-centered cubic (fcc) $\text{Co}_{20}\text{Cr}_{20}\text{Fe}_{34}\text{Mn}_{20}\text{Ni}_6$ high-entropy alloy. Annealed and hot-swaged specimens were prepared. In-situ neutron diffraction experiments captured an accelerated transformation kinetics in the hot-swaged specimen due to accumulated dislocations. The phase-specific macrostress development showed that the hexagonal close-packed ϵ -martensite predominantly accommodated the macroscopic plastic deformation of the annealed counterpart. Conversely, the matrix dislocation strengthening promoted cooperative plasticity of the γ -matrix and the ϵ -martensitic phase, thus enhancing yield stress while preserving ductility.

ARTICLE HISTORY

Received 8 September 2023

KEYWORDS

Metastable high-entropy alloy; thermomechanical processing; deformation-induced martensitic transformation; plastic deformation; in-situ neutron diffraction



IMPACT STATEMENT

This paper firstly uncovers the role and significance of matrix dislocation strengthening on the mechanical behavior of metastable high-entropy alloys via in-situ neutron diffraction experiments.

1. Introduction

High-entropy alloys (HEAs) are multicomponent alloys composed of five or more elements in approximately equimolar proportions [1–3]. The enhanced configurational entropy associated with this unique alloying concept stabilizes solid solutions [4], such as in face-centered cubic (fcc) [2,5,6], body-centered cubic [7,8], and hexagonal close-packed (hcp) [9,10] structures. Moreover, HEAs exhibit exceptional properties, such as outstanding solid-solution strengthening [3,11–14], excellent damage tolerance [5,15], and good corrosion resistance [16–19], thereby attracting considerable attention in the materials science community. Recent studies have introduced precipitation strengthening in HEAs, achieving exceptional high strength [20–24].

Among HEAs, Cantor alloy, an equimolar fcc CoCrFeMnNi HEA [2,5], has been widely studied. This alloy consists of a single-phase fcc γ -microstructure and exhibits dislocation-mediated plasticity [25]. Li et al. [26–28] proposed an alternative design strategy of combining transformation-induced plasticity (TRIP) effect [29–31] with HEAs by introducing non-equimolar compositions to reduce the stacking fault energy (SFE). TRIP-assisted dual-phase (TRIP-DP) HEAs, such as $\text{Fe}_{50}\text{Mn}_{30}\text{Co}_{10}\text{Cr}_{10}$ [26,28] and $\text{Co}_{20}\text{Cr}_{20}\text{Fe}_{34}\text{Mn}_{20}\text{Ni}_6$ [27], undergo γ (fcc)-to- ϵ (hcp) deformation-induced martensitic transformation (DIMIT) to demonstrate an exceptional combination of strength and ductility. This outcome is attributed to their inherent solid-solution strengthening characteristics and the TRIP effect, which

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Supplemental data for this article can be accessed online at <https://doi.org/10.1080/21663831.2023.2281593>.

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delays the onset of plastic instability under tensile loading. Despite their outstanding ductility, the yield strength of these HEAs (approximately 200–300 MPa at room temperature [26–28,32–34]) is even lower than that of the Cantor alloy, which restricts their application as structural materials.

Thermomechanical processing (TMP) has emerged as a promising treatment to optimize the microstructure and resulting mechanical properties of HEAs [35–39]. The grain size dependence of the mechanical behavior of metastable HEAs has been investigated. Li et al. [26] highlighted that a finer grain size of $\sim 4.5 \mu\text{m}$ results in a superior strength–ductility combination of $\text{Fe}_{50}\text{Mn}_{30}\text{Co}_{10}\text{Cr}_{10}$ alloys. In another study, the grain refinement of a TRIP-DP $\text{Cr}_{20}\text{Mn}_6\text{Fe}_{34}\text{Co}_{34}\text{Ni}_6$ HEA to 3–10 μm was reported to increase its yield stress to higher than that of Cantor alloy [40]. Chen et al. [41] indicated an accelerated $\gamma \rightarrow \epsilon$ DMT in metastable $\text{Fe}_{40}\text{Co}_{20}\text{Cr}_{20}\text{Mn}_{10}\text{Ni}_{10}$ HEAs upon increasing the grain size from 12 to 41 μm . TMP, which is performed at elevated temperatures at which the parent phase is mechanically stable, could strengthen metastable γ -matrices via grain refinement and dislocation hardening [42]. However, the effect of initial dislocation substructures in the γ -matrix on the DMT behavior remains poorly understood.

Therefore, the aim of this study is to examine the interplay between the initial dislocation substructure and DMT and clarify the multiphase mechanical behavior of a model TRIP-assisted metastable $\text{Co}_{20}\text{Cr}_{20}\text{Fe}_{34}\text{Mn}_{20}\text{Ni}_6$ HEA [27], focusing on the stress partitioning between the parent γ -matrix and ϵ -martensitic phases and transformation kinetics. To this end, in-situ neutron diffraction (ND) experiments were conducted under tensile loading, and the macrostress [43] for each phase and phase fraction were analyzed via Rietveld texture analysis (RTA) [44–46]. The results demonstrated that the dislocation strengthening of the metastable γ -matrix phase enhances cooperative plasticity during DMT, improving the yield stress while maintaining excellent strain hardening and ductility.

2. Materials and methods

A 30-kg ingot of $\text{Co}_{20}\text{Cr}_{20}\text{Fe}_{34}\text{Mn}_{20}\text{Ni}_6$ alloy was produced using conventional high-frequency vacuum induction melting. The purities of the raw materials were $\geq 99.9\%$ for Co and Fe, $\geq 99\%$ for Cr and Mn, and $\geq 99.99\%$ for Ni. Table 1 summarizes the chemical composition of the prepared alloy. The ingot was homogenized at 1200 °C for 4 h, hot forged, and subjected to hot caliber rolling to produce bars with a diameter of approximately 15 mm. Additionally, several hot-swaged

Table 1. Chemical composition of the studied alloy (at.%).

Co	Cr	Fe	Mn	Ni	C	N	O
19.93	19.70	Bal.	19.83	6.00	0.23	0.00	0.03

bars were annealed at 1100 °C for 30 min in vacuum, followed by Ar gas quenching.

The microstructure was analyzed using field-emission scanning electron microscopy (FE-SEM; JSM-7100F, JEOL, Japan) at 15 kV. Electron backscatter diffraction (EBSD) measurements were obtained using HKL Channel 5 software (Oxford Instruments, UK). Elemental maps were acquired using a field-emission electron probe microanalyzer (FE-EPMA; JXA-8430F, JEOL, Japan) operated at 15 kV. Standard metallographic procedure was followed to prepare the samples for the microstructural characterizations. The dislocation substructures of the specimens were examined through transmission electron microscopy (TEM; JEM-2000EXII, JEOL, Japan) at 200 kV. Samples were extracted from specimens subjected to nominal strains (ϵ) of 20% and 40%. TEM thin foils were prepared by ion-beam milling (Model 1010, Fischione, USA).

Uniaxial tensile tests were conducted at room temperature. Samples with a gauge diameter of 2.5 mm and length of 12.5 mm were prepared following the ASTM E8M Standard. Each specimen was strained to failure at an initial strain rate of $1.3 \times 10^{-4} \text{ s}^{-1}$ using an Instron 5969 dual-column tensile testing system. The strain during tensile deformation was measured using a non-contacting video extensometer (AVE2, Instron, USA). Each specimen underwent a minimum of three sets of tensile tests to confirm reproducibility.

In-situ time-of-flight (TOF) ND experiments during tensile deformation were performed at iMATERIA (BL20) [47] in the Japan Proton Accelerator Research Complex (J-PARC). Tensile specimens with a gauge diameter of 6 mm and length of 12 mm were installed in the sample chamber, enabling uniaxial deformation perpendicular to the incident beam [48,49]. ND data were continuously acquired while the samples were strained at an initial strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$. The phase fraction and macrostress for each phase at different strains were quantified by RTA [44–46] based on the Materials Analysis Using Diffraction (MAUD) software [50]. The peak shifts due to external stress for both the parent γ - and martensite ϵ -phases were fitted using the moment pole stress model [48], which calculates the bulk elastic modulus from the stiffness of a single crystal, considering the texture of the sample. Specifically, we used the weighted Hill model in MAUD to correlate the bulk and single crystalline elastic moduli. The elastic constants for

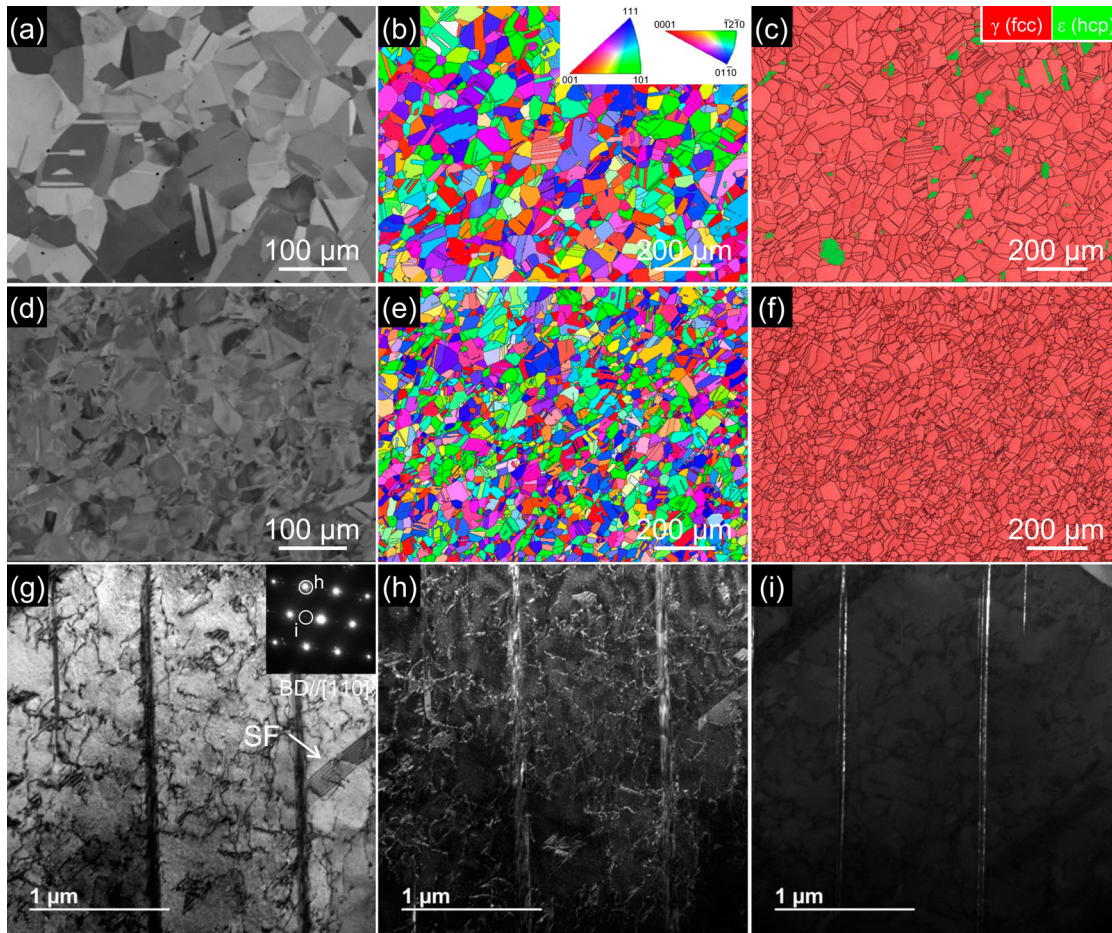


Figure 1. (a, d) SEM-BSE images; (b, e) IPF maps; (c, f) phase maps of the (a–c) annealed and (d–f) hot-swaged specimens prior to tensile loading. (g) TEM-BF and TEM-DF images for the (h) γ -matrix and (i) ϵ -martensite phases in the hot-swaged alloy. The zone axis for the SAED pattern (inset) is parallel to the $[110]_{\gamma}$ direction.

the γ - and ϵ -phases of the $\text{Co}_{20}\text{Cr}_{20}\text{Fe}_{34}\text{Mn}_{20}\text{Ni}_6$ alloy were extracted from the literature [51].

3. Results

Figure 1 shows the specimen microstructures prior to tensile loading. Both the annealed and hot-swaged specimens exhibited equiaxed grains with annealing twins, as observed in the SEM-backscattered electron (BSE) images (Figure 1(a, d)). No precipitates were detected except for minor oxide inclusions. The inverse pole figure (IPF) maps (Figure 1(b, e)) indicated that the texture did not change significantly during subsequent annealing, consistent with the RTA results based on the ND data (Figure S1(a, b)). The phase maps (Figure 1(c, f)) indicate that the γ -phase was stable in the investigated specimens, although small amounts of the ϵ -martensite were detected. The γ -grain sizes for the annealed and hot-swaged specimens were approximately 52.6 and 40.5 μm , respectively. Figure 1(g–i) show the TEM bright-field (BF) image, selected area electron diffraction (SAED)

pattern, and dark-field (DF) images of the hot-swaged specimen. The TEM-DF images were obtained leveraging the $\langle 002 \rangle_{\gamma}$ reflection. A dislocation substructure showing a fringe contrast of stacking faults (SFs) was observed. The TEM-DF image (Figure 1(i)) obtained using the $\langle 0001 \rangle_{\epsilon}$ reflection indicated the presence of a small amount of ϵ -martensite, formed upon cooling of the hot-swaged specimens. Figure S2 demonstrates a homogeneous distribution of alloying elements across the microstructure.

Figure 2(a) shows the nominal stress–nominal strain curves of the alloy specimens. Table 2 summarizes the tensile properties of the specimens. The 0.2% proof stress and ultimate tensile strength (UTS) of the annealed alloy were 202 ± 13 and 708 ± 8 MPa, respectively. These values were higher than the ones reported previously (155 ± 10 MPa and 545 ± 11 MPa, respectively) [27], which can be attributed to differences in the alloy manufacturing process. The hot-swaged specimen exhibited an improved yield stress (245 ± 14 MPa) and comparable UTS. Both specimens exhibited considerable

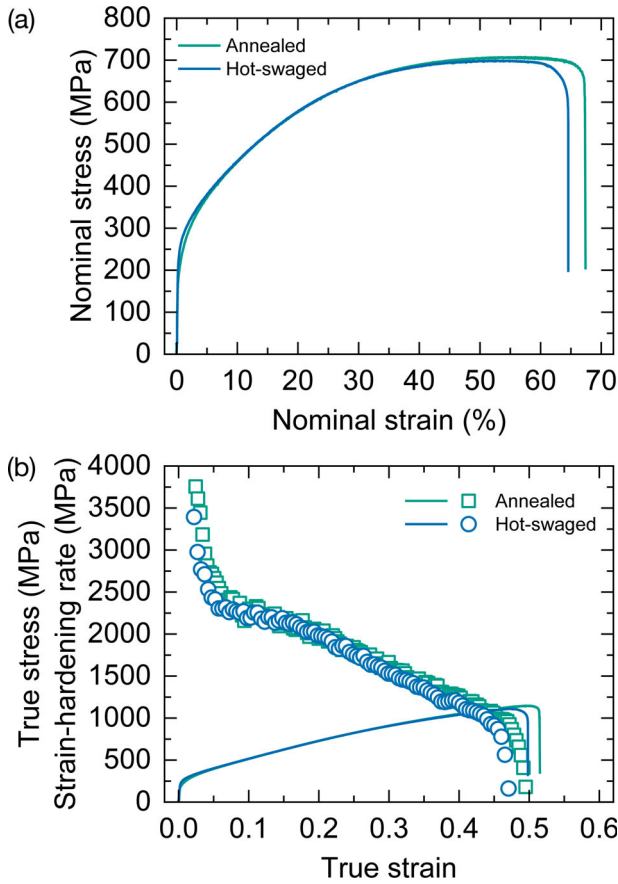


Figure 2. (a) Nominal stress–nominal strain curves and (b) corresponding true stress and strain-hardening rate as a function of the true strain of the annealed and hot-swaged specimens.

Table 2. Tensile properties of the studied alloy.

Specimen	0.2% proof stress (MPa)	Ultimate tensile strength (MPa)	Uniform elongation (%)	Total elongation (%)
Annealed	202 ± 13	708 ± 8	54.3 ± 1.4	66.2 ± 1.1
Hot-swaged	245 ± 14	704 ± 8	52.9 ± 1.0	63.4 ± 0.9

ductility (i.e. > 50% in uniform elongation and > 60% in total elongation) with that of the annealed specimen being slightly higher. Figure 2(b) shows the true stress (σ_t)–true strain (ε_t) curves and corresponding strain-hardening rate ($d\sigma_t/d\varepsilon_t$) curves as a function of ε_t of the alloy specimens. Both specimens exhibited similar behaviors, characterized by significant strain hardening during tensile deformation. Failure occurred when Considère criterion ($d\sigma_t/d\varepsilon_t \leq \sigma_t$) was satisfied. However, the annealed specimen displayed a slightly higher strain-hardening rate in the initial stage of tensile deformation ($\varepsilon_t \sim 0.1$), eventually reaching similar stress levels as the deformation progressed.

Figure 3 shows the SEM-BSE and EBSD results for both alloys strained to 20% and 40%. The SEM-BSE

images indicated the presence of numerous intragranular boundaries within the matrix of both specimens after tensile deformation. The magnified EBSD results captured the formation of a plate-like ε -phase, as confirmed by the TEM observations (Figure 4). The SAED patterns exhibited two sets of diffraction spots corresponding to the hcp ε -phase, which displays the Shoji–Nishiyama orientation relationship with the surrounding fcc γ -matrix: $(111)_\gamma // (0001)_\varepsilon$, $\langle \bar{1}10 \rangle_\gamma // \langle 11\bar{2}0 \rangle_\varepsilon$. This finding demonstrated the $\gamma \rightarrow \varepsilon$ DIMIT in the alloy during tensile loading [27]. Streaks in the SAED patterns indicated the formation of SFs in the γ -matrix. Several ε -martensite plates underwent distortion owing to further straining after the transformation [52], as indicated by the phase maps (Figure 3). Overall, the ex-situ EBSD and TEM analyses could not capture any significant difference in the DIMIT behavior between the annealed and hot-swaged specimens.

During the in-situ ND experiments under tensile loading, the peaks associated with ε -martensite became more pronounced as the applied strain increased (Figure S3). Additionally, an increase in the applied strain led to peak broadening, thereby decreasing the peak intensity of the γ -matrix. Figure 5(a) shows the evolution of the martensite fraction as a function of the applied strain, as obtained through the RTA of the ND profiles. Although the hot-swaged specimen showed higher ε -martensite fractions in the initial stage, both specimens exhibit similar behaviors involving the continuous progress of DIMIT with the ε -martensite fraction saturated at $\sim 60\%$. Figure 5(b) and 5(c) show the phase-specific macrostress (σ_{22} along the tensile direction) as a function of applied strain for the annealed and hot-swaged specimens, respectively. The total macrostress σ_{total} (Equation 1), based on the rule of mixtures [53], was consistent with the experimentally determined σ_t , demonstrating the accuracy of the analysis.

$$\sigma_{\text{total}} = \sigma_\gamma f_\gamma + \sigma_\varepsilon (1 - f_\gamma) \quad (1)$$

where σ_γ and σ_ε denote the macrostress for the γ -matrix and ε -martensite, respectively, and f_γ is the fraction of the γ -phase. As shown in Figure 5(b), σ_γ of the annealed specimen was considerably lower than σ_ε . Specifically, σ_ε of the annealed specimen continuously increased to > 2 GPa, whereas σ_γ increased and then decreased to zero as the applied strain increased. This observation suggested that the plastic deformation of the annealed specimen was governed by high-strength ε -martensite and the stress partition between the parent γ - and martensitic ε -phases became negligible with increasing ε . The hot-swaged specimen exhibited a different behavior. Here, the macrostress of both the γ -matrix and ε -martensite

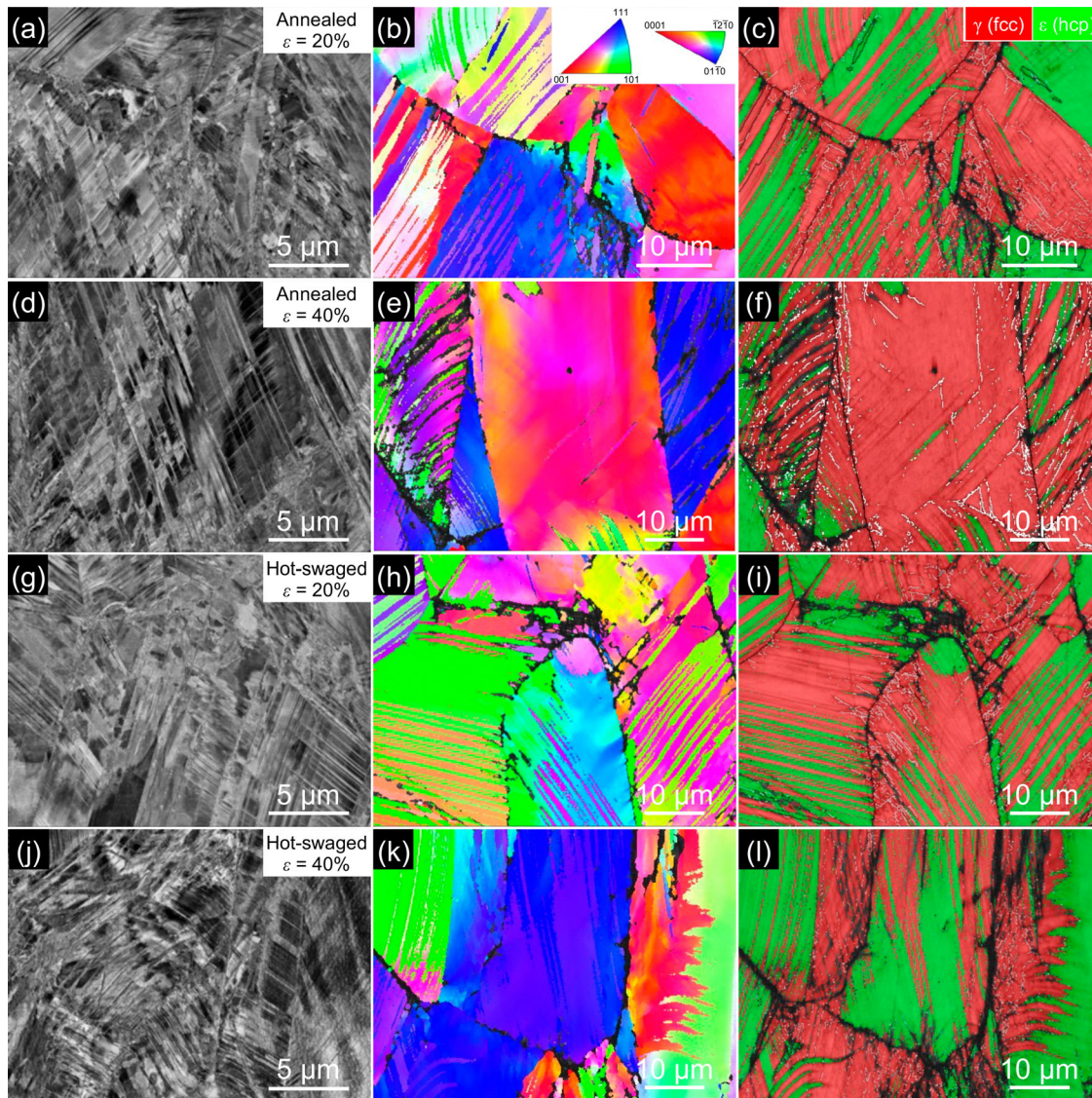


Figure 3. (a, d, g, j) SEM-BSE images, (b, e, h, k) IPF maps, and (c, f, i, l) phase maps of the (a–f) annealed and (g–l) hot-swaged specimens subjected to tensile straining to (a–c, g–i) $\varepsilon = 20\%$ and (d–f, j–l) 40% .

increased with strain, although ε -martensite remained the harder phase. This indicates that the γ -matrix also contributed to macroscopic plastic deformation in the hot-swaged specimen. Moreover, prior hot deformation of the γ -matrix altered the plasticity in the duplex microstructure in a more cooperative manner, although the RTA revealed similar texture evolution during tensile loading (Figure S1).

4. Discussion

In this study, we investigated the tensile deformation behavior and corresponding microstructural evolution of a TRIP-aided metastable HEA in both annealed and hot-swaged conditions. Notably, hot swaging did not compromise the strain-hardening capability or the resultant ductility relative to the annealed alloy (Figure 2).

Given that hot swaging elevated the yield stress of the alloy, this approach could be employed to improve the strength–ductility balance in metastable HEAs.

In a duplex microstructure, stress and strain partitioning, which varies throughout the deformation process, plays a key role in strain hardening. The significant strain hardening in TRIP steels can be ascribed to the evolution of the stress and strain partitioning with the appearance of the hard martensite phase [54]. We characterized the macrostress of each phase by in-situ ND measurements. Consequently, we established that the flow stress of the annealed specimen was dominated by the ε -martensitic phase during tensile loading (Figure 5(b)). Thus, the γ -matrix in the annealed specimen did not act as a stress-bearing phase, and the external load partitioned to it was exclusively used for ε -martensite production, resulting in further strain hardening.

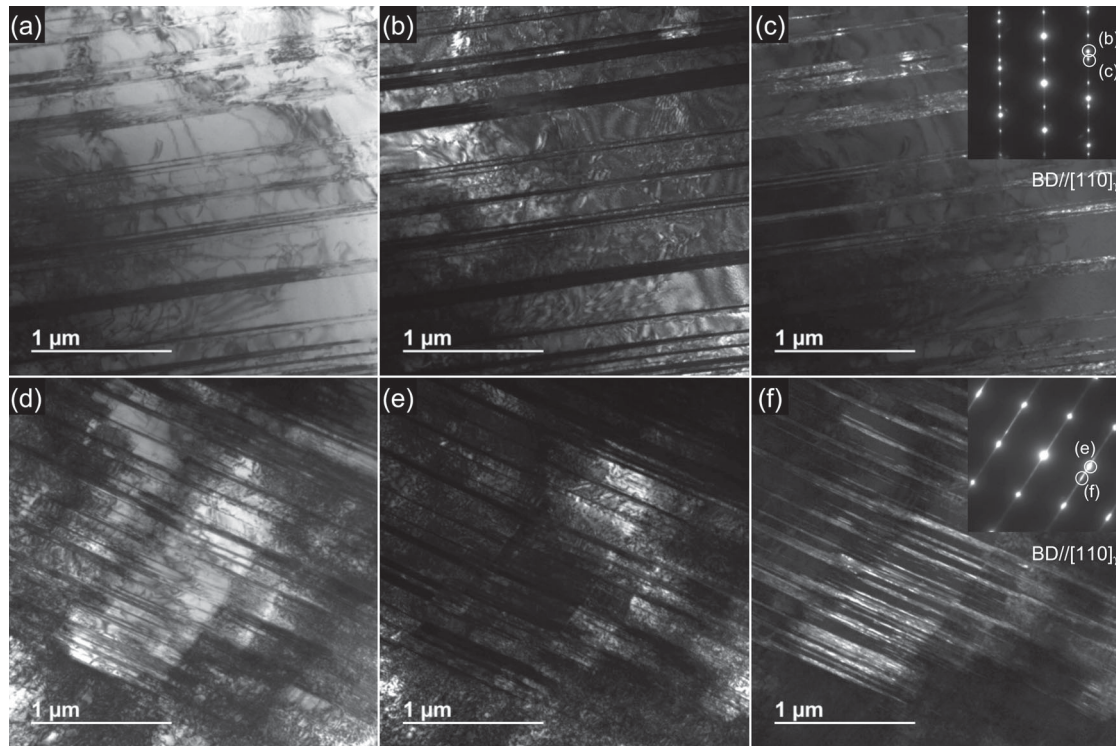


Figure 4. (a, d) TEM-BF and TEM-DF images for the (b, e) γ -matrices and (c, f) ε -martensite plates in the (a–c) annealed and hot-swaged specimens subjected to tensile straining to $\varepsilon = 20\%$. The insets show the corresponding SAED patterns with the diffraction spots used for capturing the TEM-DF images.

Conversely, we observed that the external stress applied to the hot-swaged specimen was partitioned to the ε -martensite and metastable γ -matrix (Figure 5(c)), demonstrating the cooperative deformation between the γ -matrix and ε -martensite. Thus, the dislocation substructure developed during hot swaging, i.e. dislocation strengthening, rendered the γ -matrix a load-bearing phase alongside the ε -martensite. This phenomenon altered the phase-specific plasticity, in which $\gamma \rightarrow \varepsilon$ DIMIT played a key role.

The influence of the microstructure on the DIMIT and TRIP effect has been extensively investigated. In steels, grain refinement increases the mechanical free energy required for the transformation of martensite to austenite [55]. Conversely, the TRIP-DP-HEA, $\text{Fe}_{50}\text{Mn}_{30}\text{Co}_{10}\text{Cr}_{10}$, exhibited enhanced strain hardening responses, which are associated with accelerated DIMIT kinetics in the presence of a finer grain size [26]. In contrast, dislocation strengthening typically results in reduced ductility and heightened brittleness [56]. Notably, the hcp ε -martensite phase is formed by the overlap of intrinsic SFs on alternate 111_{γ} planes [57]. Consequently, the formation of SFs was observed through the TEM images (Figure 4). Mori et al. [58] reported that the $\gamma \rightarrow \varepsilon$ DIMIT behavior

in biomedical Co–Cr–Mo alloys is accelerated through the consumption of lattice defects, including dislocations and SFs [59], introduced upon hot rolling. Similarly, the DIMIT kinetics of the hot-swaged specimen was accelerated by the accumulated dislocations in the γ -matrix, also contributing to the strain hardening. However, in spite of the lower ε -martensite fraction, the enhanced strain hardening behavior at the initial stage of the annealed specimen indicates a higher dislocation accumulation in the γ -matrix of the specimen than that of the hot-swaged specimen during tensile loading.

Recently, Liu et al. [60] reported that hot rolling can elevate both the yield stress and ductility of AISI301 austenitic steel. This enhancement is attributed to an increase in the initial dislocation density and dislocation plasticity. Another study on additively manufactured austenitic steel pointed towards an improved strength–ductility synergy stemming from the formation of a dislocation network, which acts as a soft barrier against dislocation slip [61]. The results of our study demonstrate the correlation between the $\gamma \rightarrow \varepsilon$ DIMIT and the initial dislocation structure, a relationship not previously established with certainty. The DIMIT behavior and subsequent strain hardening are possibly

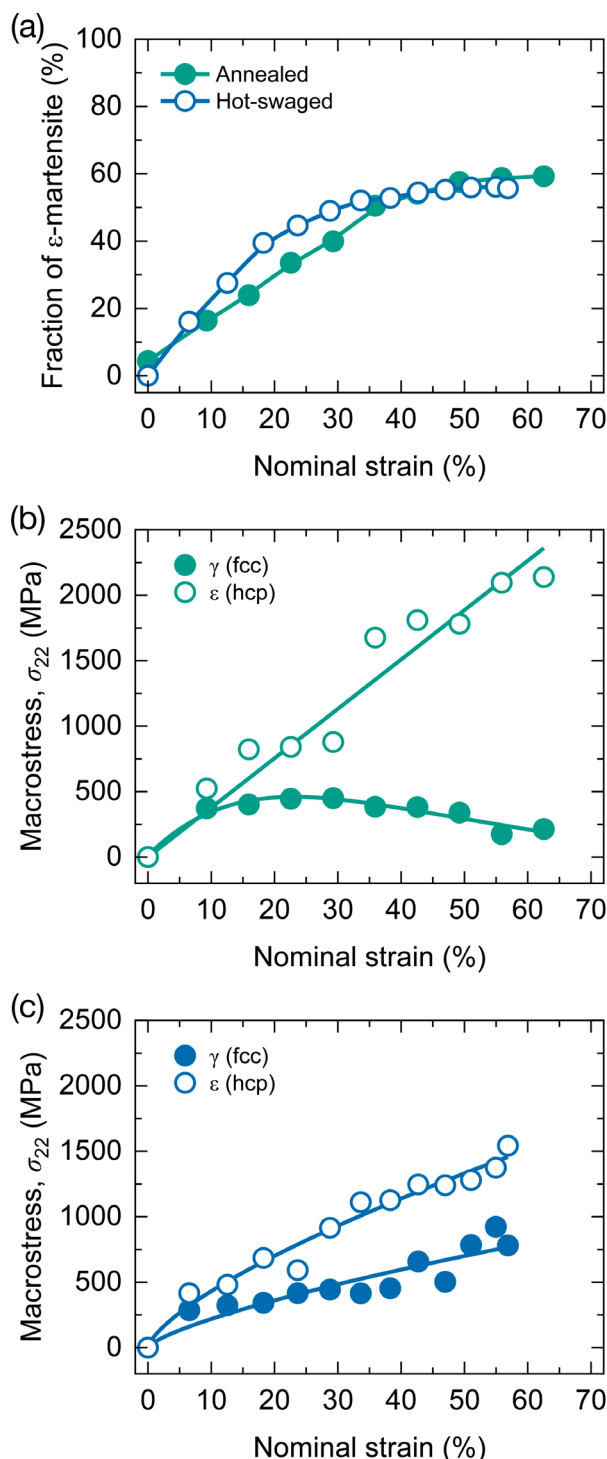


Figure 5. (a) ϵ -martensite fraction of the annealed and hot-swaged specimens, determined via RTA. Macrostress of the γ -matrices and ϵ -martensite of the (b) annealed and (c) hot-swaged specimens.

shaped by multiple aspects of dislocation substructures, including their distribution and the extent of dislocation dissociation. This indicates that by fine-tuning the dislocation substructure via the alloy composition, the DIMT behavior can be effectively modulated.

5. Conclusions

In this study, we clarified the stress partitioning between the fcc γ -matrix and hcp ϵ -martensite during DIMT in metastable $\text{Co}_{20}\text{Cr}_{20}\text{Fe}_{34}\text{Mn}_{20}\text{Ni}_6$ HEAs. In-situ ND measurements and RTA demonstrated that hot swaging of the alloy promoted its deformation with the ϵ -martensite, whereas ϵ -martensite predominantly governs the flow stress during DIMT in the annealed specimen. Furthermore, dislocation accumulation in the metastable γ -matrix enhanced the DIMT kinetics and increased the yield stress, while maintaining excellent strain hardening and tensile ductility of the alloy. These findings highlight a promising approach involving lattice defect engineering for enhancing strength–ductility synergy.

Acknowledgments

The authors thank Masahiro Yamazaki, Eiwa Co., Ltd., for assisting in sample preparation, and Issei Narita, Shun Ito, and Fuyuki Sakamoto, Institute for Materials Research, Tohoku University, for providing technical assistance. EPMA analysis was performed under the GIMRT Program of the Institute for Materials Research, Tohoku University (Proposal No. 19G0414). The neutron experiment at J-PARC was performed as per proposal 2019PM2006.

Disclosure statement

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

Funding

This research was supported by the Japan Society for the Promotion of Science (JSPS) [Grant Nos. 17H04957 and 23H01722].

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