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In-situ atomic-scale observation of dislocation and deformation twin interaction in FeMnCoCr high-entropy alloy

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ABSTRACT

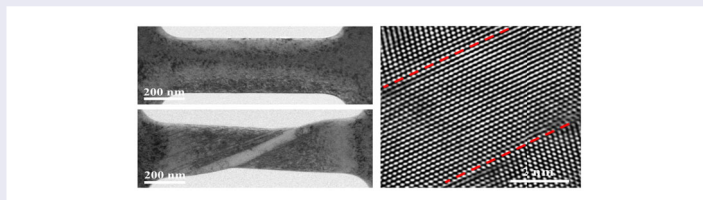
The deformation of a single-crystal Fe₄₈Mn₃₂Co₁₀Cr₁₀ alloy is captured in situ at the atomic scale. The results show that full and partial dislocations, along with deformation twins, are involved in the deformation process. Partial dislocation activities result in the formation of nanotwins, high-density coherent twin boundaries, and incoherent twin boundaries. Full dislocations interact with the coherent/incoherent twin boundaries, and partial dislocations, thereby producing high strength and remarkable strain hardening. The observed high activity of full dislocations can be attributed to the inhomogeneous distribution of solid solution atoms in the alloy.

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KEYWORDS

High-entropy alloys; in-situ; dislocation; deformation twin



Introduction

Recently, there has been a global surge in interest in high-entropy alloys (HEA) owing to their outstanding mechanical properties [1–6]. Among these alloys, FeMnCoCr is of particular interest owing to its high ductility and remarkable strain-hardening [7–10]. The mechanical properties of the materials are directly related to their deformation mechanisms. Understanding deformation mechanisms is essential to facilitate the design of tailored HEAs with exceptional mechanical properties. Therefore, numerous studies have investigated the deformation mechanisms of FeMnCoCr alloys. Many interesting ex situ studies and nanoscale resolution investigations have suggested that the plastic deformation of FeMnCoCr alloys is governed by partial dislocations [11–13]. For example, Li et al. [14] showed that partial dislocation results in a phase transition from a

face-centered cubic (FCC) to a hexagonal close-packed (HCP) structure and that deformation twinning occurs in the Fe₅₀Mn₃₀Co₁₀Cr₁₀ alloy after plastic deformation. Many researchers have concluded that FeMnCoCr alloys have low stacking fault energies and that partial dislocation and deformation twinning are known to frequently nucleate, whereas full dislocation activity during deformation is rare. However, most previous studies were performed at a nanoscale resolution and were therefore unable to clearly distinguish between full and partial dislocations. It is unclear whether full dislocation occurs during deformation, owing to the lack of direct atomic-scale evidence. Moreover, deformation twins not only provide plasticity but also interact with mobile dislocations, resulting in strong strain hardening [15–17]. Picak et al. [18] suggested that the deformation of Fe₄₀Mn₄₀Co₁₀Cr₁₀ alloy was governed by partial

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dislocations, which led to the formation of high-density nanotwins. Further deformation increases the prevalence of twin-twin and twin-dislocation interactions, leading to strong strain hardening in these systems. Without in-situ atomic-scale observations, the mechanism by which these deformation twins cause strain-hardening remains unclear owing to a lack of in-situ atomic-scale observations; therefore, such in-situ atomic-scale results are required to elucidate the deformation mechanism of FeMnCoCr alloys.

In this study, the deformation behavior of an Fe₄₈Mn₃₂Co₁₀Cr₁₀ single-crystal nanoplate was observed in situ using transmission electron microscopy (TEM) with a tensile device. We provide a direct in-situ observation of the atomic-scale deformation behavior of an Fe₄₈Mn₃₂Co₁₀Cr₁₀ high-entropy alloy. Full and partial dislocations along with deformation twins are involved in the deformation process. In addition, partial dislocation activities led to the formation of high-density nanotwins, after which full dislocations interacted with the coherent twin boundary (CTB), incoherent twin boundary (ITB), and partial dislocations, thereby increasing the strain hardening.

Method

The Fe₄₈Mn₃₂Co₁₀Cr₁₀ single-crystal nanoplate was derived from an annealed polycrystalline bulk Fe₄₈Mn₃₂Co₁₀Cr₁₀ alloy with an FCC structure. Our results reveal that the annealed bulk alloy has an average grain size of approximately 3 μ m, and importantly, there is no HCP

phase present in this alloy (further details can be found in the Supplementary Materials under Method S1). Fig. S1 presents the stress-strain curve of the annealed polycrystalline bulk Fe₄₈Mn₃₂Co₁₀Cr₁₀ alloy. Prior to further examination, the alloy was initially characterized using electron backscatter diffraction (EBSD). Subsequently, using the orientation distribution derived from the inverse pole figure via EBSD, a tensile sample was extracted from grains aligned with the [110] zone axes. This extraction was performed using a Helios G4 UC focused ion beam (FIB). The nanoplate tensile sample was cut into the shape of a dog bone with a length, width, and thickness of 1000, 400, and 100 nm, respectively (more details in Method S1). The nanoplate was then attached to an INSTRON-MT in situ tensile device with double-tilt capability [19,20] and observed using an FEI Titan environmental transmission electron microscope (TEM). The EBSD characterization was performed using an FEI-Quanta 650 field-emission scanning electron microscope equipped with an EBSD system from TexSEM Laboratories.

Results and discussion

Figure 1a shows the EBSD pattern of the polycrystalline Fe₄₈Mn₃₂Co₁₀Cr₁₀ alloy used for fabricating the single-crystal nanoplate for the in situ tensile test. A single-crystal sample for in-situ tensile testing was fabricated from grains with a [110] zone axis identified in the EBSD image. The TEM image captured before deformation (Figure 1b), in which the viewing direction was along

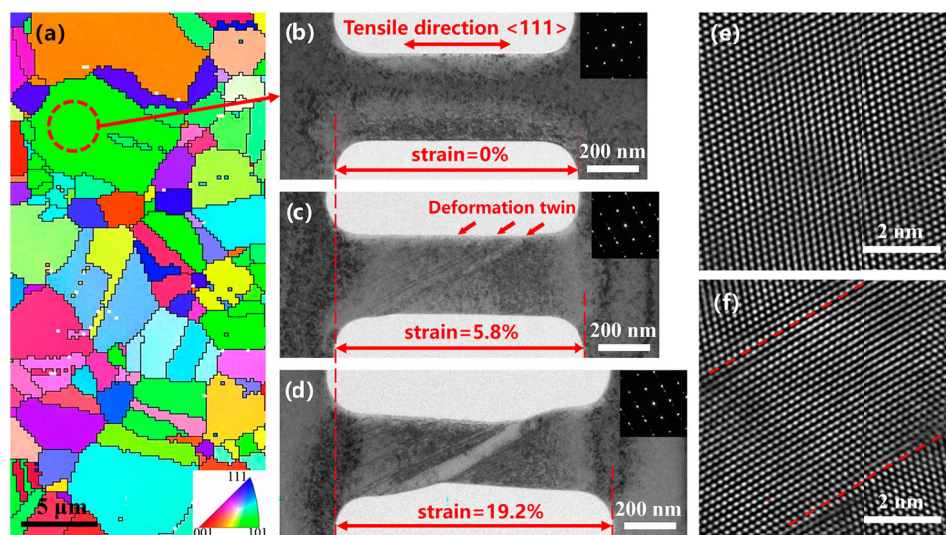


Figure 1. (a) EBSD characterization of Fe₄₈Mn₃₂Co₁₀Cr₁₀ alloy. (b) Nanoplate fabricated from a grain of the alloy using FIB. (c) Nanoscale deformation twins generated during straining. (d) The number and thickness of the deformation twins increased with strain. The insets in parts b-d show the corresponding SAED pattern of the deformed region. (e, f) HRTEM images captured before and after plastic deformation.

the [110] zone axis, showed that the tensile sample contained no grain boundaries or growth twins. The selected area electron diffraction (SAED) pattern of the nanoplate (Figure 1b, inset) shows typical single-crystalline features, which can be well indexed to the FCC-structured $\text{Fe}_{48}\text{Mn}_{32}\text{Co}_{10}\text{Cr}_{10}$ alloy. Several nanoscale deformation twins with thicknesses ranging from 1 to 10 nm were formed as the strain increased to 5.8% during tensile processing (red arrows in Figure 1c). These deformation twins resulted from partial dislocations, and passed through the entire tensile sample. With further loading, both the number and thickness of the deformation twins increased (Figure 1d). The increase of thickness of these deformation twins was also attributed to the typical layer-by-layer gliding of partial dislocations, which is consistent with the results of previous studies on FeMnCoCr alloys [11–17]. As the strain increased, many twinned spots appeared in the SAED patterns (Figure 1c and 1d, insets), further confirming the generation of deformation twins. The single-crystalline features of the nanoplate before deformation are clearly observed in the high-resolution TEM (HRTEM) image (Figure 1e). The HRTEM image captured after plastic deformation shows that deformation twins were generated (Figure 1f). Furthermore, no HCP phase is generated during the loading of nanoplate.

In addition to the formation of nanoscale deformation twins, full dislocation and deformation-twin interactions were directly observed. Figure 2 shows a typical example of in situ atomic-scale HRTEM images of the twin-dislocation interaction during straining. The CTB and reference area for the in situ observations are indicated by red dashed lines and orange dashed rectangles, respectively. A deformation twin approximately 5 nm in thickness was observed (Figure 2a), along with many full dislocations pinned on the top side of the CTB (indicated by the symbol ‘T’). As observed, there is an additional (111) plane inserted on the CTB (detailed in Figure 3e), indicative of a typical full dislocation structure with a Burgers vector of $1/2 \langle 110 \rangle$ and a glide plane of (111) [21]. Moreover, numerous atomic-scale steps are evident on the CTB. These steps emerge due to the emission and glide of partial dislocations on the CTB. When these partial dislocations stop at the CTB, they lead to the formation of steps. Therefore, in this study, these types of steps on the CTB directly correspond to partial dislocations. These partial dislocations possess a Burgers vector of $1/6 \langle 112 \rangle$ and a glide plane of (111) [22,23], and notably, they do not introduce additional (111) planes (as seen in Figure 3f). Full dislocations often hinder the glide of partial dislocations on the CTB. Therefore, many atomic-scale steps are pinned on the CTB. The bottom of this deformation twin varies in thickness, with that

of the left-side and right-side twins being ~ 4.4 nm and ~ 5.5 nm in thickness, respectively, owing to a step with 7 atomic layers. This large step represents an ITB [24–26], and several full dislocations are pinned in this region. With increasing strain, as shown in Figure 2b–d, many full dislocations escaped from the CTB, whereas many new full dislocations were simultaneously pinned onto the CTB. These pinning and unpinning processes frequently occur in the CTB during deformation.

In the initial stages of deformation, the successive emission of partial dislocations on 111 plane resulted in twin nucleation. Subsequently, numerous partial dislocations glided along the CTB, generating atomic-scale steps. As the loading progressed, many gliding partial dislocations were obstructed by full dislocations, resulting in the formation of complex steps due to the interaction of full dislocations with the steps. Figure 3 presents a series of magnified HRTEM images that demonstrate the interactions between the full dislocations and the CTB during deformation. Two full dislocations (denoted ‘1’ and ‘2’ in Figure 3a) are pinned by the CTB (as shown in Figure 3e, there is an extra (111) plane insert on the CTB), leading to localized distortions in the lattice. This type of dislocation-CTB interaction increases the stress of dislocation sliding, leading to strain-hardening [27,28]. In addition, several steps (denoted ‘A’ and ‘B’ in Figure 3) are formed on the CTB at the location of full dislocation. These complex steps result from the interaction of partial dislocations on the CTB and full dislocations with the (111) glide plane. Our in-situ atomic-scale observations show that the movement of these types of complex steps is severely inhibited until the full dislocation escapes from the CTB. Thus, the motion of these step types typically requires a relatively long time. As the strain increases (Figure 3b), the full dislocation ‘1’ escapes from the observed area, and the step resulting from this partial dislocation quickly glides right to left, causing step ‘A’ to disappear. In contrast, step ‘B’ shows no obvious change because the full dislocation ‘2’ remains pinned. This observation further confirmed that the full dislocation-CTB interaction obstructed partial dislocations in the CTB and inhibited CTB migration. As the strain is further increased (Figure 3c), the full dislocation ‘2’ escapes from the CTB, resulting in the rapid disappearance of step ‘B’ owing to the removal of the obstruction caused by the full dislocation. Meanwhile, two new steps of one atomic layer (denoted ‘C’ and ‘D’) and full dislocation ‘3’ were generated. The rapid gliding of partial dislocations on the CTB lead to the formation of steps ‘C’ and ‘D’ (Figure 3d), which combine to form the two-atomic-layers thick step ‘E.’ The results presented above indicate that deformation-induced nanotwins not only anchor the full dislocation but also hinder the movement

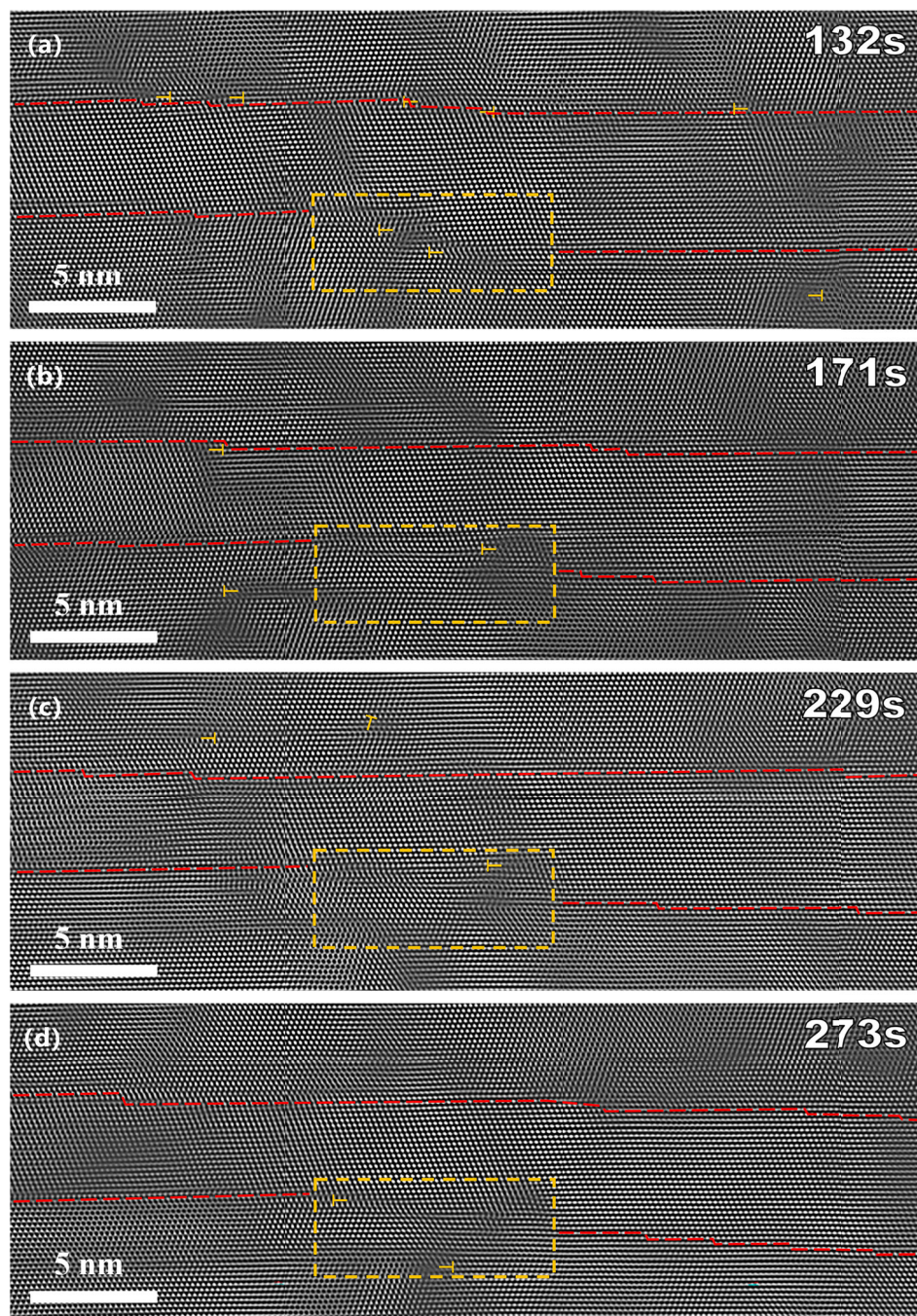


Figure 2. (a-d) In-situ HRTEM images of full dislocation interactions with the CTB and ITB in the $\text{Fe}_{48}\text{Mn}_{32}\text{Co}_{10}\text{Cr}_{10}$ alloy. The CTB is indicated by the red dashed line, and ITB is indicated by the dashed rectangle. Full dislocations are marked with symbol 'T'.

of partial dislocations on the CTB, resulting in enhanced strain hardening. (Two additional examples show full dislocations resting on the CTB and blocking partial dislocations, leading to the formation of complex steps). Figure 3e provides a magnified HRTEM image, clearly displaying both the full dislocation and a step on the CTB (related to Figure 3b). Figure 3f offers a zoomed-in HRTEM image that corresponds to the highlighted

region in Figure 3c, illustrating solely the step on the CTB.

Figure 4 displays a series of enlarged HRTEM images showing the full dislocation-ITB interaction during deformation. In the region enclosed by the orange dashed rectangle, the CTB on the left is seven atomic layers higher than that on the right, forming a typical ITB consisting of seven atomic layers between the matrix and the

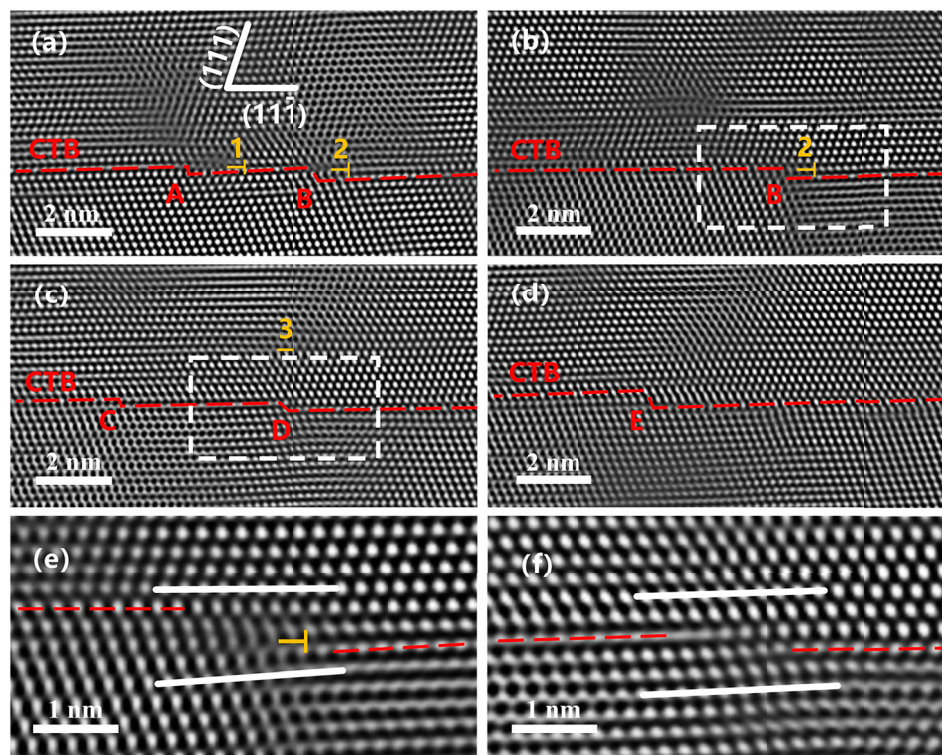


Figure 3. (a–d) Magnified HRTEM images showing the full dislocation-CTB interaction during deformation. (e, f) HRTEM extract from the framed region of (b, c).

deformation twin. In situ observations revealed that the ITB also inhibited the movement of full dislocations. In Figure 4a, two full dislocations (labeled ‘1’ and ‘2’) are anchored on the ITB’s left side, revealing an additional (111) plane embedded within the ITB. This structure contrasts with the ITB devoid of a full dislocation, as illustrated in Fig. S2. The ITB is formed by a sequence of partial dislocations with a glide plane parallel to the CTB, and its migration is driven by the movement of these partial dislocations [29,30]. When the full dislocation on the (111) glide plane interacts with the ITB, a high stress is necessary to activate the motion of the partial dislocation array (comprising the ITB), contributing to strain hardening. Our in-situ atomic-scale observations showed that the interaction of the ITB with the full dislocation strongly inhibits its movement until the full dislocation escapes. With increasing strain, the full dislocations ‘1’ and ‘2’ escaped from the observed area (Figure 4b and 4c). Simultaneously, a new full dislocation labeled as ‘3’ emerges and also interacts with the ITB (see Figure 4c). As the strain continues to increase, similar interactions between the full dislocation and ITB, and the escape of the full dislocation from the ITB, are observed once more (Figure 4d). Furthermore, comparing Figure 4a–4d reveals shifts in the position and structure of the ITB, suggesting that the partial dislocation array motion was active during the straining process [29,30]. Typically, ITB

migration takes place when the full dislocation disengages from the ITB, indicating full dislocation-ITB interaction inhibits the ITB movement. Based on images taken during in-situ experiments, we plotted the ITB migration distance against time (refer to Fig. S4 in Supplementary Materials). The results demonstrate that the ITB migration rate drops when full dislocations are anchored to the ITB. Conversely, when these dislocations depart from the ITB, the migration rate of the ITB elevates, further confirming that the full dislocation-ITB interaction inhibits the ITB motion. While ITBs have pinned full dislocations, these dislocations might also impede ITB migration, contributing to pronounced strain hardening. Here, we primarily center on the movement of the ITB, which consists of an array of partial dislocations. It is worth noting, however, that in other regions, numerous partial dislocations glide on the CTB. This is validated by the observed migration of the CTB and the progression of multiple steps on the CTB during loading.

Many previous studies attributed the excellent mechanical properties of FeMnCoCr alloys to partial dislocations, FCC to HCP phase transitions, and deformation twinning [11–17]. Our in-situ observations provide direct evidence that full and partial dislocations and deformation twins are involved in the deformation of Fe₄₈Mn₃₂Co₁₀Cr₁₀ nanoplates. First, partial dislocations result in the formation of many nanotwins, leading to

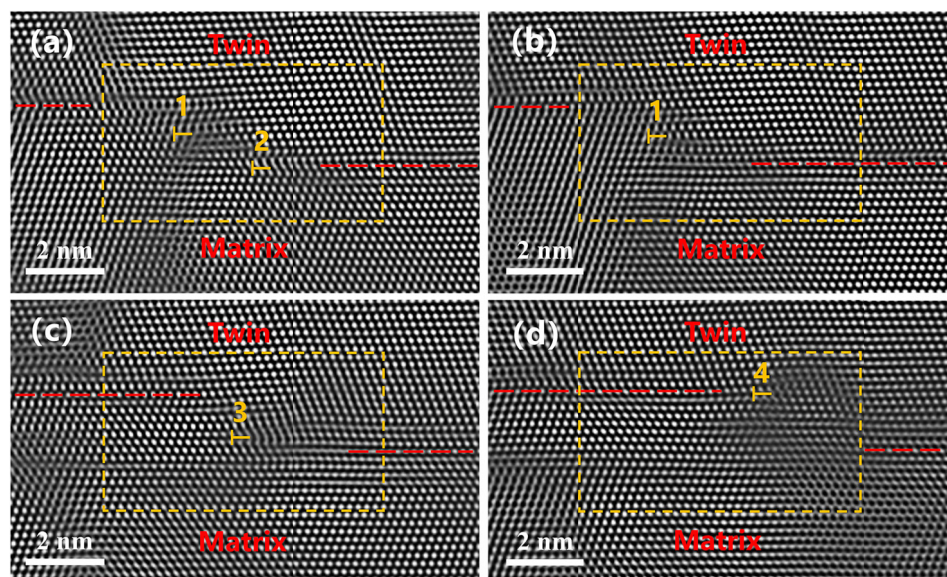


Figure 4. (a-d) Magnified HRTEM images showing the full dislocation-ITB interaction during deformation.

high CTB and ITB densities in the nanoplate tensile sample. Second, both the CTB and ITB strongly inhibited the motion of dislocations and played important roles in strain hardening. The frequent activity of full dislocations may be a consequence of the inhomogeneous distribution of solid-solution atoms, which leads to variations in the chemical composition at different locations in HEA [31–34]. This variation in chemical composition leads to variations in mechanical properties owing to changes in local stacking fault energy (SFE) [35–37]. Partial dislocations and nanotwins are preferred in regions with low SFE, whereas full dislocation activities frequently occur in regions with relatively high SFE. Therefore, the plastic deformation of the $\text{Fe}_{48}\text{Mn}_{32}\text{Co}_{10}\text{Cr}_{10}$ alloy is governed by both full and partial dislocations.

The deformation mechanism observed in the single-crystal nanoplate is also anticipated to be present in bulk polycrystalline alloys. However, considering the extensive presence of grain boundaries in the latter, it is important to be circumspect when applying our findings to bulk polycrystalline alloys. This distinction becomes evident when comparing the stress–strain curves of a single-crystal nanoplate to those of the bulk polycrystalline $\text{Fe}_{48}\text{Mn}_{32}\text{Co}_{10}\text{Cr}_{10}$ alloy (refer to Fig. S1 and S5). While both curves display clear strain hardening, the single-crystal nanoplate shows higher stress but limited ductility. In contrast, the bulk polycrystalline $\text{Fe}_{48}\text{Mn}_{32}\text{Co}_{10}\text{Cr}_{10}$ alloy exhibits larger plasticity but low strength. In bulk polycrystalline alloys, both pre-existing dislocations and grain boundaries can act as sources of dislocations, thereby enhancing plasticity [38–41]. Furthermore, as noted in our earlier study [10], the HCP

phase also generates during deformation. Consequently, there is an expectation of a high density of deformation twins, both partial and full dislocations, and the HCP phase within the grains. The emergence of these diverse defects and their subsequent interactions contribute to the bulk polycrystalline $\text{Fe}_{48}\text{Mn}_{32}\text{Co}_{10}\text{Cr}_{10}$ alloy's pronounced plasticity and strain hardening. On the other hand, the single-crystal nanoplate, due to its reduced initial dislocation density and smaller size, necessitates higher stress for dislocation nucleation owing to size effects [42,43]. Moreover, dislocations in the nanoplate are easily escaped. As a result, strain hardening primarily stems from the interactions of full dislocations with CTB and ITB after the generation of deformation nanotwins. This leads to the nanoplate displaying high strength, albeit with limited ductility.

Conclusion

In summary, our in-situ atomic-scale observations reveal that, in addition to partial dislocations, full dislocations also significantly influence the plastic deformation of the single-crystal $\text{Fe}_{48}\text{Mn}_{32}\text{Co}_{10}\text{Cr}_{10}$ alloy. Initial partial dislocation activity prompts the formation of deformation nanotwins, resulting in a high density of CTB and ITB, which in turn enhance plasticity. As strain intensifies, full dislocations interact with CTB and ITB introducing two types of strain-hardening. This provides insight into the strain-hardening observed in the $\text{Fe}_{48}\text{Mn}_{32}\text{Co}_{10}\text{Cr}_{10}$ alloy. Moreover, full dislocations on CTB hinder the gliding of partial dislocations on the CTB, which causes

the emergence of complex step formations. These findings are expected to deepen our understanding of the deformation mechanisms in HEAs and guide the development of HEAs with optimized mechanical properties

Disclosure statement

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

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