Interactions between Dislocations and Penta-Twins in Metallic Nanocrystals

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Abstract: Dislocation interactions with twin boundary (TB) have been well-established in nanotwinned metals. Penta-twins, as an extreme of crystal twinning, are tacitly assumed to be more effective at blocking dislocation motions than conventional single or coplanar nanotwins. However, the mechanism underlying the interactions between dislocations and penta-twins remains largely unclear. Here, by combining in situ transmission electron microscope (TEM) nanomechanical testing and atomistic simulations, we rationalize the fundamental interactions between dislocations and penta-twins in Au nanocrystals. Our results reveal that the interactions between dislocations and penta-twins show some similar behaviors to the ones in the cases of coplanar nanotwins, including dislocation impedance at TBs, cross-slip into the twinning plane and transmission across the TB. In addition, penta-twins also exhibit some unique behaviors during dislocation interactions, including multiple cross-slip, dislocation-induced core dissociation and climb-induced annihilation/absorption at the penta-twin core. These findings enhance our mechanistic understanding of dislocation behaviors in penta-twins, shedding light on the accessible design of high-performance nanomaterials with multi-twinned nanostructures.

Keywords: penta-twin; dislocation; twin boundary; interaction; in situ nanomechanical testing

1. Introduction

Nanotwins have been widely reported to significantly improve the strength and ductility of nanostructured face-centered cubic (FCC) metals and alloys due to dislocation interactions with and accumulation at the coherent twin boundaries (TBs) [1,2]. A number of factors can determine the feasibility of a TB-dislocation reaction, such as the stacking fault (SF) energy, dislocation characteristics (screw or non-screw), the energy barriers for dislocation reactions at TBs, dislocation density, twin thickness, loading orientation and magnitude of the applied stress [3–8]. By tuning these controlling factors, dislocation reactions at TBs can proceed in different ways, including cross-slip into the twin plane to cause twinning or de-twinning, formation of a sessile stair-rod dislocation at the TB and transmission across the TB [3,4].

As an extreme of crystal twinning, penta-twin emerges when five TBs simultaneously converge at a line parallel to their common {110} axis. Penta-twins have been widely observed in various of FCC metals and alloys in the form of nanowires, nanoparticles and thin films [9–15], which were found to be capable of effectively enhancing Young’s modulus, yield strength and strain hardening capability of metallic nanomaterials [14,16–19]. Such enhanced mechanical properties of penta-twinned nanomaterials were often ascribed
to dislocation nucleation and impedance, as well as dynamic dislocation reactions in penta-twin networks, distinct from dislocation activities in conventional single or coplanar nanotwins [20,21] that are mostly restricted to the free paths parallel to TBs. Thus, penta-twins have attracted numerous attention in past decades. In metallic nanowires with a longitudinal penta-twin, surface-nucleated partial dislocations commonly slip inclined to the axial direction of nanowires, which then propagate and interact with the constituent TBs and the penta-twin core, inducing the formation of SF decahedrons [22,23]. In other loading conditions, penta-twins in synthesized nanoparticles [24,25], as well as the ones in thin films processed through extreme mechanical [26–28] or annealing conditions [29,30] in other nanomaterials, are expected to interplay with edge-on dislocations slipping parallel to the common \{110\} axis. Within the bulk of these penta-twins, dislocation motions and their interactions with the constituent TBs and the penta-twin core are expected to be significantly affected by the intrinsic disclination stress field of penta-twins, inducing some unique interaction features other than the ones in conventional single or coplanar nanotwins. However, a comprehensive understanding of the interactions between dislocations (especially non-screw ones) and penta-twins in metallic nanocrystals remains largely lacking, especially under some extreme loading conditions.

Here, we unravel the interactions between dislocations and penta-twins in Au nanocrystals by conducting in situ nanomechanical testing inside transmission electron microscope (TEM), supported by molecular dynamics (MD) simulations. Fundamental interactions between dislocations and penta-twins are elucidated, showing some similar behaviors to the well-acknowledged TB-dislocation interactions, including dislocation impedance at TBs, cross-slip into the twinning plane and transmission across the TB. In the meantime, dislocation interactions with penta-twins can also exhibit some unique characteristics, including multiple cross-slip, dislocation-induced core dissociation and climb-induced annihilation/absorption at the penta-twin core. These atomistic insights into the interactions between dislocations and penta-twins advance our understanding of dislocation behaviors within penta-twinned nanomaterials, which will inspire the accessible design of multi-twinned nanomaterials with high strength and good ductility.

2. Experimental and Simulation Methods

2.1. In Situ TEM Nanofabrication and Nanomechanical Testing

In situ nanofabrication/nanowelding of penta-twinned Au nanocrystals was conducted inside a Cs-corrected FEI Titan G² 60-300 TEM operated at 300 kV, using PicoFemto® TEM electrical holder (Zeptools Co., Beijing, China). Before experiments, bulk Au wires (99.99 wt.%) with a diameter of 0.25 mm were cut by a ProsKit wire cutter (ProsKit Co., Shenzhen, China) in order to obtain a pair of fresh fracture surfaces with multiple nanoscale tips. The fractured Au wires were then loaded onto the static and movable (probe) sides of the TEM holder, respectively. Subsequently, in situ nanowelding can be achieved by controlling a piezo-manipulator behind the Au probe with a pre-applied voltage potential of ~1.5 V in order to contact and melt the nanoscale tips on both sides together. By selecting nanoscale tips with a specific \{110\} zone axis and nanostructures with TBs, nanocrystals embedded with penta-twinned structures can be fabricated by fast nanowelding. Further in situ nanomechanical testing can be accomplished by controlling the backward/lateral motion of the Au wire on the probe side with a built-in piezo-manipulator (behind the probe). As such, tensile/shear loading was performed on as-fabricated samples at a constant velocity of ~0.005 nm s⁻¹, which gave rise to a strain rate at the level of ~10⁻³ s⁻¹. Throughout the mechanical loading, the zone axes of these nanocrystals were kept nearly unchanged, albeit with some inevitable vibrations induced by deformation. A charge-coupled device (CCD) camera (Gatan Inc., Pleasanton, CA, U.S.A.) was applied to record videos at a rate of ~0.3 s per frame. Moreover, mechanical testing was conducted under a low beam current condition in order to minimize the potential beam effect on deformation.
2.2. Simulation Methods

MD simulations were performed on an Au penta-twinned model using the Large-Scale Atomic/Molecular Massively Parallel Simulator (LAMMPS) [31] and the embedded atom method (EAM) potential for Au [32]. In detail, an Au prismatic model with a diameter of 20 nm and a total height of 20 nm was created by constructing five separate crystals with corresponding crystallographic misorientations of 70.53° and joining them along the radial direction. Three boundary layers of atoms at the top and bottom of the system were fixed as rigid slabs. The remaining dynamic atoms were allowed to adjust their positions in a Nose–Hoover thermostat at 300 K. Free boundary conditions were applied in all three directions. The sample was first equilibrated by energy minimization, and then it was freely relaxed for 10 ps. The time step of the MD simulations was 1 fs. Subsequently, tensile/shear loading was performed on the bottom rigid slab at a constant velocity of $v = 1 \text{ m s}^{-1}$ in the $x/y$-direction, with the top slab fixed, to apply deformation. A velocity profile with a linear gradient from 0 to 1 m s$^{-1}$ along the axial direction was assigned to the dynamic atoms. Ovito [33] was employed to visualize the nanocrystalline model, and common neighbor analysis (CNA) was utilized to identify the TB behaviors during the simulations. Atoms with FCC, hexagonal close-packed (HCP) and disordered structures were marked in blue, red and cyan, respectively.

3. Results and Discussion

Before deformation, several Au nanocrystals with penta-twins were successfully fabricated via in situ nanowelding inside TEM (see Methods). Figure 1 presents several cases of general reactions between dislocations and penta-twins. For example, a partial dislocation is emitted from free surface and is blocked by one of the TBs that constitute a penta-twin, as shown in Figure 1a. With further straining, a thin twin lamellae forms and grows via sequential twinning on neighboring {111} planes, along with the enlargement of a kink-like step resulting from the interaction (Figure 1a). This observation agrees with the conclusion that penta-twins can strongly confine the motions of lattice dislocations in single subunit [34], which extensively exists in the penta-twinned nanocrystals. Previous studies also showed that an incident dislocation can become fully pinned when penetrating toward a TB of penta-twins unless the strain increases, producing a much higher strengthening effect than that of parallel twins through the cage-like network of penta-twins [35]. During the interactions, some partials may also transmit across TBs of penta-twins by dissociating the incident partial into two new partials slipping across or along the TB, if sufficiently high stress is presented (Figure 1b). Similar transmission behavior is also captured for the interaction between a dissociated full dislocation (with a pair of leading and trailing partials) and a penta-twin, which leaves behind some residual partials that glide along the transmitted TB (Figure 1c). The above observations suggest that, in most cases, the interactions between dislocations and penta-twins are nearly identical to TB-dislocation interactions in the sample with parallel nanotwins, including dislocation impedance, cross-slip into the twinning plane and transmission across the TB, depending on the characteristics of dislocations and deformation geometry [3].
Figure 1. General reactions between dislocations and penta-twins. (a) Dislocation impedance in single subunit of a penta-twin. With further straining, a thin twin lamellae forms and grows along with the enlargement of a TB step. (b) Transmission of a partial dislocation across a TB of penta-twins. (c) Transmission of a dissociated full dislocation across a TB of penta-twins. The directions of dislocation propagation and mechanical loading in (a–c) are indicated by the white and red arrows, respectively. The insets present circuits of Burgers vectors of the propagating dislocations and the corresponding fast Fourier transform (FFT) patterns of penta-twinned nanocrystals.

After dislocation-TB interactions, residual dislocations can be generated on the TBs, which, when moving on the TBs, can induce TB migration and twinning/detwinning of conventional coplanar nanotwins [3,4,36,37]. In the penta-twins, these residual dislocations can further glide toward and react with the penta-twin core. This process can induce the splitting of the penta-twin core as well as the displacement of another constituent TB by one atomic layer (Figure 1b,c). Figure 2 further elucidates the underlying mechanism during this core dissociation process, where a twinning partial interacts with the penta-twin core and subsequently transfers to other constituent TBs. A double Thompson tetrahedron and its representation in five adjacent Thompson tetrahedrons are schematically illustrated in Figure 2a,b, respectively, to help understand the dislocation dynamics across different constituent TBs of a penta-twin hereinafter. Upon straining, a twinning partial (Cβ', b = 1/6[112]) is stimulated to glide along a constituent TB (TB4) and further interacts with the penta-twin core (Figure 2c,d) by dissociating into a partial dislocation Cβ' and a sessile stair-rod dislocation β'β (Cβ → β'β + Cβ') (i.e., the transmission of the 30° partial dislocation across the TB3). Associated with this process is the migration of TB2 and TB4 with a distance of one atomic layer and the dissociation of the penta-twin core, as experimentally verified in Figure 2c,d and schematically illustrated in Figure 2e,f. In theory, this partial can transmit across TB5 and glide into TB1 as well depending on the local stress state. As such, twinning partials are able to cross-slip onto different constituent TBs of a penta-twin. Previous studies have shown that penta-twin core can often impede dislocations, inducing restricted migration of a TB of the penta-twin with only slight oscillation around its original location [16]. On the other hand, some simulation studies reported layer-by-layer TB migration induced by twinning partial propagation during annealing [9] or stress-relaxed [38] evolutions of penta-twins. However, these studies were less focused on the coordinated motions of twinning partials along different constituent TBs. Here, our experiments directly visualize the coordinated slip of twinning dislocations within...
penta-twins by interacting with the penta-twin core, which is often associated with the dissociation of the penta-twin core.

Figure 2. Dislocation-induced dissociation of the penta-twin core. (a,b) Double Thompson tetrahedron and its representation in five adjacent Thompson tetrahedrons used for dislocation analyses within penta-twins. (c,d) Twinning partials transmitting across the penta-twin core induces the dissociation of the penta-twin core, as well as migrations of TB2 and TB4 by one atomic layer. Frank circuit in (e) ascertains the dislocation content of a twinning partial/disconnection. The inset in (d) shows the corresponding fast Fourier transform (FFT) pattern of the penta-twin. (e,f) Schematic diagrams of the dislocation-induced dissociation of the penta-twin core.

As revealed above, a twinning partial can experience one-time cross-slip at the penta-twin core by transferring to another constituent TB of the penta-twin (Figure 2). For an incident lattice dislocation into the bulk of penta-twins, however, it is expected to undergo several iterations of cross-slips due to the presence of multiple slip systems within a penta-twin. Figure 3 shows two typical examples. As shown in Figure 3a–c, a surface-emitted dislocation experiences cross-slips numbering three times within a penta-twin. Firstly, it changes its slip route in the bulk of the penta-twin (Figure 3a,b). Once it approaches TB5, the propagation of this dislocation is blocked by TB5, which then changes its motion direction toward the penta-twin core by cross-slipping onto the [111] plane parallel to TBs. With further straining, this dislocation continues to slip across the penta-twin core, followed by its propagation onto TB3 and the final annihilation to free surface. These sequential processes result in the migration of TB5 and TB3 by one atomic layer and the dissociation of the penta-twin core. The remarkable change of surface steps (see insets in Figure 3b,c) unambiguously validates the complete annihilation of this dislocation at the TB3-surface junction. In addition to the cross-slip along constituent TBs, our MD simulation results show that dislocation cross-slip across different constituent TBs via transmission mechanism may also occur within a penta-twin, as indicated in Figure 3d–f. During this process, an incident lattice dislocation successively transmits across two neighboring constituent TBs (i.e., TB1 and TB5) of the penta-twin (Figure 3d–f). However, such transmission-mediated dislocation cross-slip in the penta-twin is mainly achieved by [001] slip rather than the conventional dislocation slip on [111] planes. Previous studies have reported that the energy barrier for [100] slip gradually decreases with decreasing twin thickness, where, below a critical twin thickness, [100] slip can be more favored than normal [111] slip transmission (which remains identical for all twin sizes) [5]. In our simulation, dislocation slip occurs close to the core, thus favoring the activation of [100] slip (Figure 3d–f). Given the high energy barrier for [100] slip, however, the commonality of such [100]-transmission-related cross-slip in experiments still needs to be confirmed. Furthermore, it should be noted that aside from the multiple slip systems, the variations
of stress distributions within penta-twins may also affect the operable route for dislocation cross-slip. The stress-free penta-twin typically possesses a symmetrical disclination stress field, with the local stress maxima uniformly distributed along each TB and the penta-twin core [19,34]. Such stress distribution renders dislocation cross-slip preferred at constituent TBs and the penta-twin core. Nevertheless, the local stress state within a deformed penta-twin is far more complicated than expected [28,39]; therefore, the detailed routes for dislocation cross-slip within a penta-twin are difficult to precisely predict based merely on the rule of Schmid factor.

**Figure 3.** Multiple dislocation cross-slip in penta-twins. (a–c) A dislocation experiences cross-slips three times sequentially at the bulk of penta-twins, TBs of penta-twins and the penta-twin core. Insets in (b,c) show the variation of atoms at free surface due to the final annihilation of this dislocation at the TB-surface junction. Note that the white and black dashed arrows track the cross-slip routes of two different dislocations that nucleate before and after, respectively. (d–f) Transmission-mediated dislocation cross-slip in a penta-twin facilitated by [001] slip mode. Atoms with FCC, HCP and disordered structures were marked in blue, red and cyan, respectively.

The penta-twin core may also act as a preferential site for dislocation annihilation (Figure 4), as experimentally verified in Figure 4a–d and schematically illustrated in Figure 4e–h. When an incident dissociated full dislocation (d1, a leading partial Cβ and a trailing partial βD bounded with a SF) is blocked by a TB, it tends to contract its dissociated length by forming a full dislocation CD (Figure 4a,b). Interestingly, the compact dislocation (CD) subsequently climbs toward the penta-twin core along TB1 (Figure 4b,c). Finally, this dislocation annihilates at the penta-twin core, which further triggers partial emission along another constituent TB (TB3). Accompanying this process is the migration of TB3 by one atomic layer (CD $\rightarrow$ β′A + 1/3{001}), as verified by the conspicuous change of surface steps (see the insets in Figure 4c,d). MD simulations also show dislocation annihilation/absorption at the penta-twin core via a mechanism of dislocation climb (Figure 4i–k). As shown, an extended dislocation contracts its dissociated length upon being blocked by a constituent TB (Figure 4i,j), which subsequently climbs toward the penta-twin core due to the absorption effect of the penta-twin core (Figure 4k). The annihilation of this dislocation at the penta-twin core should increase the distortion of the core lattice, which further stimulates partial emission from the penta-twin core (Figure 4k). These simulation results are in good agreement with our experimental observations in Figure 4a–d, which further validate the feasibility and rationality of the occurrence of such climb-induced annihilation/absorption at the penta-twin core.
Figure 4. Dislocation annihilation/absorption at the penta-twin core via dislocation climb. (a–d) Sequential TEM snapshots showing dislocation annihilation at the core of a penta-twin. (a,b) A full dislocation (d1) contracts its dissociated length when approaching TB1 of the penta-twin. Insets in (a,b) show the dissociated and compacted structures of this propagating dislocation, respectively. Note that some dislocations are irrelevant to our analyses (such as later-nucleated d2 and pre-existed immobile d3). (b,c) Dislocation climbs along TB1 toward the penta-twin core. (c,d) Dislocation annihilates at the penta-twin core, which further stimulates partial emission from the core along TB3. Insets in (c,d) show the variation of atoms at free surface due to partial emission from the penta-twin core. (e–h) Schematic illustrations presenting the corresponding mechanisms of dislocation behaviors in (a–d), respectively. (i–k) MD simulations showing similar dislocation annihilation at the penta-twin core. Atoms with FCC, HCP and disordered structures were marked in blue, red and cyan, respectively.

In penta-twinned nanomaterials, the interactions between dislocations and penta-twins should frequently occur and affect the intrinsic deformation behaviors of penta-twins. Hence, it is of great significance to reveal the underlying mechanisms so as to bridge the mechanistic origin of penta-twin deformation and resultant modified performance of nanomaterials. The current work shows that, in most cases, the interactions between dislocations and penta-twins are similar to the well-known TB-dislocation interactions, including dislocation impedance at TBs, cross-slip into the twinning plane and transmission across the TB (Figure 1). With the high-symmetry structure and associated particular intrinsic stress field, however, the interactions between dislocations and penta-twins can be far more complex than those in a conventional single or coplanar nanotwins (dislocation activities are mostly restricted to the free paths parallel to TBs). Our in situ nanomechanical testing intuitively presents real-time evolutions regarding some unique dislocation behaviors within penta-twins that distinguish them from TB-dislocation interactions, including cross-slip onto different constituent TBs (Figures 2 and 3), climb-induced annihilation/absorption at the penta-twin core (Figure 4) and dislocation-induced core dissociation (Figures 2–4). Cross-slip across constituent TBs and the penta-twin core should be partly enabled by the intrinsic disclination stress field generated by the 7.33° gap of five twin units (Figure 3). Complex and changeable routes of dislocation cross-slip suggest that dislocation motions in penta-twins should be more difficult than that in coplanar nanotwins, thereby resulting in a stronger strain hardening effect [16]. On the other hand, vacancy generation may play a role in adjusting the dislocation motions given that vacancies are commonly observed in the vicinity of the penta-twin core in penta-twinned nanomaterials [22,39], which could
facilitate stress relaxation and enhance diffusion-related dislocation motions, e.g., dislocation climb (Figure 4). As such, the penta-twin core can act as a potential site for dislocation annihilation and accumulation. Note that dislocation accumulation at the penta-twin core, as well as TB migration induced by twinning partial propagation, should inevitably induce the delocalization and distortion of the penta-twin core; therefore, this should make the penta-twin core a potential dislocation source throughout the deformation and result in core dissociation (Figures 2–4).

It is also worth noting that all interactions between dislocations and penta-twins in our experiments are enabled under tensile or shear loading condition, distinct from the compression performed in previous literature [16,35]. Tensile or shear loading, to some extent, can readily provide sufficient spaces for dislocation motions within penta-twins, which increases probabilities for the occurrence of multiple dislocation cross-slip within penta-twins. In the penta-twinned nanowires, penta-twins are often axially loaded and dislocations commonly slip inclined to the axial direction of nanowires, where dislocation interactions with the constituent TBs and the penta-twin core often induces the formation of SF decahedrons [22,23]. In contrast, penta-twinned nanocrystals in our study are radially loaded, and dislocations commonly slip parallel to the common [110]; axis of penta-twins, which show distinct dislocation behaviors from that in penta-twinned nanowires. In general, these observed interactions should be applicable for the deformation of penta-twinned nanoparticles. The fundamental mechanisms regarding the interactions between dislocations and penta-twins revealed in this study should play a non-negligible role in modifying the mechanical properties of penta-twinned nanomaterials, shedding light on the tailoring and testing of multi-twinned nanostructures with exceptional performances.

4. Conclusions

In summary, we investigate the interactions between dislocations and penta-twins in Au nanocrystals by using in situ nanomechanical testing integrated with atomistic simulations. We find that the interactions between dislocations and penta-twins show some similar behaviors to the ones in the cases of coplanar nanotwins, including dislocation impedance at TBs, cross-slip into the twinning plane and transmission across the TB. However, penta-twins also exhibit some unique behaviors during dislocation interactions, including multiple cross-slip, dislocation-induced core dissociation and climb-induced annihilation/absorption at the penta-twin core, which are distinct from the ones in the cases of coplanar nanotwins or penta-twinned nanowires. These findings provide a good insight into the interactions between dislocations and penta-twins, which would help to explain potential performance variations of penta-twinned nanomaterials.

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