Hierarchical twinning governed by defective twin boundary in metallic materials

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Dense networks of deformation twins endow metals and alloys with unprecedented mechanical properties. However, the formation mechanism of these hierarchical twin structures remains under debate, especially their relations with the imperfect nature of twin boundaries (TBs). Here, we investigate the intrinsic deformability of defective TBs in face-centered cubic metallic materials, where the inherent kinks on a set of primary TBs are demonstrated to facilitate the formation of secondary and hierarchical nanotwins. This defect-driven hierarchical twinning propensity is critically dependent on the kink height, which proves to be generally applicable in a variety of metals and alloys with low stacking fault energies. As a geometric extreme, a fivefold twin can be constructed via this self-activated hierarchical twinning mechanism. These findings differ from the conventional twinning mechanisms, enriching our understanding of twinning-mediated plasticity in metallic materials.

INTRODUCTION

Defects naturally endow materials with a conspicuous diversity of microstructural characters, which enable the manipulation of material properties (1–4). Among different defects, coherent twin boundary (TB), a symmetrical planar defect widely existed in both engineering and biological materials (5–7), brings about unprecedented properties and functionalities (8). In principle, the twin-related superior mechanical properties of materials mainly arise from the intrinsic stability of coherent TBs (with perfect coherency and extremely low excessive energy) (9), efficient barriers to dislocation motion (10, 11), and orientation-dependent interactions with dislocations (12–14). Notably, coherent TBs in all types of twins (including deformation twins, growth twins, and annealing twins) typically contain plenty of steps or kinks (15), suggesting that they are essentially imperfect. These defective TBs can facilitate the plastic deformation under different loading conditions by activating TB sliding/detwinning via the motion of twinning dislocations/kinks (16, 17) or provoking heterogeneous dislocation nucleation from the kinks (18). Normally, an inherent maximum strength prevails among nanotwinned (NT) materials, which is dependent on TB spacing (19).

Instead of pushing the limit of TB spacing–governed strengthening in well-aligned primary twins, hierarchically twinned materials with dense networks of coherent TBs have been emerging as a new class of NT materials, in which the spaces in the primary twin lamella have been subdivided by spontaneously implanted barriers to multiple slip systems, bringing about potentials to further enhance the deformability of materials (20, 21). These hierarchical architectures have been frequently observed in a variety of metallic materials with low stacking fault energies (SFEs), including face-centered cubic (fcc) metals (22), twinning-induced plasticity steels (23), shape memory alloys (24), and high-entropy alloys (HEAs) (25). Unfortunately, the construction processes of these hierarchical twin structures remain largely unknown. Several theories have been proposed, such as the coincidental collision of multiple twinning systems (26) or the multifold twinning mediated by grain boundary (GB) migration (27), all of which require highly specific conditions and may not sufficiently account for the frequent occurrence of hierarchical twins. On the other hand, recent experimental and simulation results implied a hierarchical twinning mechanism from the primary TBs (28), given the ample space for the hierarchical plastic deformation inside the primary twin lamella (15, 29). However, the atomistic deformation kinetics that correlate the TB defects and frequently observed hierarchical twins remain an open question, which hinders the manipulation of hierarchically twinned structures toward breakthrough properties in metallic materials.

Here, we unravel the structural and mechanistic origins of hierarchical twinning processes governed by defective TBs in metallic materials with low SFEs, using multiscale in situ transmission electron microscope (TEM) nanomechanical testing and molecular dynamics (MD) simulations. The preexisting or deformation-induced kinks on defective TBs can serve as efficient nucleation sites of secondary twins and dominate the hierarchical twinning processes in several representative fcc systems with low SFEs, including Au, Cu-Al alloys, 304L stainless steel, and CoCrFeMnNi HEA. As a geometric extreme, a fivefold twin configuration can be generated via the sequential activations of this hierarchical twinning dynamics that encompass the TB kink. These findings clarify a general built-in propensity for hierarchical twins in metals and alloys with low SFEs, shedding light on potential development of high-performance metallic materials via defect engineering.
RESULTS

TBs in metals and alloys are inherently imperfect at the atomic scale due to the prevalent existence of defects (steps or kinks) on these coherent boundaries. As exemplified in Fig. 1A, multiple kinks exist on the coherent TBs in an as-deformed polycrystalline Au (see Materials and Methods), similar to the defective TBs in as-grown NT copper (15). Compared with the atomic structure of perfect TBs (Fig. 1B), the lattice coherency along the defective TBs is interrupted by the kinks with different heights and elastic strains (Fig. 1C and fig. S1). Because of the accumulated elastic strain at the core regions, these TB kinks are expected to be highly active upon further straining, contributing to preferential hierarchical twinning during the plastic deformation of NT materials.

To gain an insight into the deformation modes of these defective TBs, we performed in situ nanomechanical testing on an NT Au (see Materials and Methods), as demonstrated in Fig. 1 (D to G). Before the nanomechanical testing, a [111]-oriented Au nanocrystal (with an average diameter of 16.6 nm) containing an inclined TB was fabricated via nanowelding inside TEM (see Materials and Methods). Multiple defects exist on the TB, including a six-atom-layer kink and several single-atom-layer steps. Under a uniaxial tensile load along the longitudinal direction of this Au nanocrystal (Fig. 1E and movie S1), the plastic deformation was accommodated neither by the surface nucleation of dislocations commonly occurred in nanocrystals (30) nor by the motion of kinks along the TBs in NT metals (15). Instead, a Shockley partial dislocation with a trailing lattice mismatch (see the horizontal strain map in Fig. 2J). Associated with the successive emission of partial dislocations were the increase in kink height from five to seven atom layers and the development of a stepwise kink configuration (marked as I to III). Because of continuous stress release of the TB kink via partial dislocation emission and local atom rearrangement, a clockwise tilt occurred at the kink interface for approximately 18.5°, as schematically illustrated in Fig. 2K.

To understand the atomistic dynamics of this self-activated twinning process, we performed MD simulations on an Au nanocrystal with an identical sample geometry and TB orientation to our experiment (Fig. 2, E to H). Note that before the partial dislocation emission, a single-atom-layer step readily decomposed from the six-atom-layer kink to reduce the total excessive energy at the kink (Fig. 2, E and F, and fig. S2). In line with our experimental observations, a secondary twin T2 was nucleated as Shockley partial dislocations emitted consecutively from the TB kink (Fig. 2, G and H), which was governed by the elastic strain at the TB kink (fig. S2). During this twinning process, TB kink underwent different structural evolutions upon the nucleation and thickening of the secondary twin, respectively.

![Fig. 1. Defective TBs and TB kink–activated secondary nanotwin in Au. (A) TEM image showing parallel nanotwins in an as-deformed Au. The arrows indicate the TB defects. (B and C) Atomic resolution TEM images showing the perfect TB and defective TB containing multiple kinks, respectively. (D to G) Deformation snapshots showing the nucleation and growth of a secondary nanotwin from a TB kink in an Au nanocrystal. (D) As-received [111]-oriented Au nanocrystal containing an inclined coherent TB, as confirmed by the inset fast Fourier transform pattern. M and T1 denote the matrix and primary twin, respectively, on either side of the TB, where a six-atom-layer kink and several single-atom-layer steps preexisted on the TB. (E) Emission of a partial dislocation with a trailing SF into T1 from the TB kink under longitudinal tensile loading (shown by the white arrow). (F to G) Nucleation and growth of a secondary nanotwin (T2) via the consecutive emission of partial dislocations on the neighboring (111) plane in T1. The light blue arrows indicate the gliding of twinning partials away from the kink. The white dashed line delineates a tilt GB segment (with a misorientation of 45°) transformed from the original TB kink. Scale bars, 20 nm (A) and 2 nm (B to D).]
Specifically, the dihedral angle between the kink facet and primary TB increased from 104° to 123° during the consecutive nucleation of a three-layer T2 embryo (Fig. 2, E to G), which is consistent with our experimental observation of kink tilt for 18.5°. Upon the growth of T2, the bottom junction between the kink and primary TB tended to translate along the primary TB to reduce the excessive energy (schematically illustrated in Fig. 2L). Accordingly, the kink facet further tilted for 24° (Fig. 2G), which led to continuous adjustment of the kink structure from a distorted interface to a low-energy tilt GB segment with a misorientation of ~46° (Fig. 2H), matching well with our experimental observations (Fig. 2D). This eventual deformed boundary is a vicinal of Σ11(113) symmetrical tilt GB with θ = 50.5°, which deviates from the theoretically predicted Σ9(114) tilt GB with θ = 38.9° (9), due to local lattice distortion at the TB kink.

In both experiments and MD simulations, TB kinks with multi-atom layers are favored for the nucleation of secondary twin, while those with single or double atom layers (i.e., steps) tend to move along the TB, indicating a height-dependent competition of twinning dynamics between different defects. To quantitatively address this competition, we calculated the height-dependent activation energy of distinct deformation mechanisms at the TB defects based on the TB kink model (Fig. 2M). The nucleation energy for partial dislocation decreases slightly with the TB kink height, while the kink migration barrier rises sharply within the same range of kink height. A transition from kink motion along the primary TB to dislocation emission into the primary twin lamella appears at a kink height of five atom layers, which is supported by our dynamic observations in Fig. 2. Furthermore, this critical kink height can be reduced with increasing tensile strain, suggesting a stress-assisted propensity for secondary twinning. As the emission of the first partial dislocation significantly reduced the critical stress for subsequent partial nucleation on the neighboring planes (31), the formation of a secondary twin in the primary twin lamella is energetically favorable under further loading (discussion S1). In this process, kink height is not only the governing factor of the nucleation of secondary nanotwin but also that of critical importance for its growth. Geometrically, the maximum thickness of the secondary nanotwin is governed by the kink height, while the preexisting small TB steps merging at the kink can activate further partial nucleation from the kink (Fig. S2).

From the thermodynamic perspective, the average excessive energy of the TB kink drops continuously with the growth of secondary nanotwin, which is mainly correlated with the tilt of kink interface (Fig. 2N). When the thickness of secondary twin increased to more than five atomic layers, structural transition at the kink in terms of the formation of a tilt GB segment occurs, resulting in more prominent reduction of the excessive energy. Thus, self-activated twinning behaviors were frequently observed in Au nanocrystals containing...
defective TBs (figs. S3 and S4 and movie S2). In elemental metals with distinct SFEs, an identical TB kink may exhibit different behaviors, as shown by the dislocation emission in Ag and Cu with lower SFEs (fig. S5) and the kink migration in Ni with higher SFE, underscoring an SFE-dependent critical stress for dislocation emission from the defective TB.

In addition to elemental metals, alloys with low SFEs typically show hierarchical twins after plastic deformation as well (25). To further demonstrate the broad impact of TB kinks (either preexisted or deformation induced) on the formation of hierarchical twins, we studied the twinning behaviors in several low SFE systems, including Cu-Al alloy, 304L austenite stainless steel, and CoCrFeMnNi HEA. Figure 3A shows the deformation microstructures of a Cu–7 atomic % Al micropillar (diameter ~ 740 nm and \( \gamma^d \approx 17 \text{ mJ m}^{-2} \)) (32) after in situ compression inside TEM. Two different twinning systems were activated, both near and away from the stress concentration region at the bottom surface. The magnified TEM image (Fig. 3B) and inset electron diffraction pattern clearly demonstrate the coexistence of both primary twins and several secondary nanotwins. The short secondary nanotwins (T2) embedded inside the primary twin (T1) lamella were likely to be nucleated from the kinks of primary TBs (Fig. 3B), given the preferential twinning path under this compressive loading direction (indicated in Fig. 3A) and the absence of other defect activity nearby. This result is consistent with the self-activated twinning mechanism observed in Au nanocrystals under tensile loading (Fig. 1), which dominates over the conventional surface nucleation mechanism in nanoscale metals (30).

In bulk 304L stainless steel (\( \gamma^d \approx 20 \text{ mJ m}^{-2} \); Fig. 3C) (33) and Cu–11.6 at % Al alloys (\( \gamma^d = 10 \text{ mJ m}^{-2} \); fig. S6A) (32), numerous secondary nanotwins were also generated inside the primary twin lamellae after deformation, challenging the hierarchical twinning model based on the collision of GB-emitted twins on multiple systems (26). Moreover, the secondary nanotwins were preferentially formed at the larger kinks of primary TBs (Fig. 3D), consistent with our theoretical predictions of kink height–dependent tendency for hierarchical twinning (Fig. 2M). Furthermore, hierarchical twinning can serve as an important plastic mode in low SFE alloys in cryogenic deformation. As exemplified in Fig. 3E, pronounced secondary twinning was exhibited in the ultrafine-grained (UFG) CoCrFeMnNi HEA after tensile testing at 77 K, while the plastic deformation at room temperature was dominated by dislocation dynamics (34). This frequent activation of secondary nanotwins, in conjunction with the widely acknowledged transition from dislocation dynamics to (primary) deformation twinning at low temperature, is expected to provide both extra deformability and slip barriers, which is confirmed by the simultaneous improvement of ultimate tensile strength and ductility at 77 K compared with that at room temperature (Fig. 3F). Moreover, the strain hardening rates of UFG CoCrFeMnNi HEA are readily increased at 77 K, despite certain amount of inherent instability at the onset of yielding in UFG metals (35).

Hierarchical twinning in low SFE metallic materials may also induce high-order twin structures (fig. S6B), especially in the regions under complex stress state (e.g., near the fracture surface). To fully understand the self-activated twinning behaviors beyond secondary
twins, we performed in situ nanomechanical testing to compare the distinct atomistic mechanisms associated with each step toward high-ordered twins in Au (Fig. 4). Starting from the twofold twin (Fig. 4A), additional partial dislocation emissions from the faceted kink interface (evolved from the flat kink interface after the formation of T2; see fig. S7) initiated the formation of a threefold twin configuration (Fig. 4B). Meanwhile, the upper boundary of T2 continued to migrate upward via the consecutive partial nucleation from both the kink and the surface. With further deformation, a new twin nucleated from the joint interface between T2 and T3 (Fig. 4, C and D), reversing several layers of lattice in T2 back to T1 and generating a fourfold twin structure. With the simultaneous growth of T3 and T1, the atomic structure of the joint interface among T2, T3, and T1 evolved into an extensively faceted configuration (Fig. 4E) (19). This faceted interface provided an extra nucleation site for additional partial dislocations, favoring the generation of T4 in T2 (note that T4 did not extend much because of the blockage of the upper boundary of T2 ahead; Fig. 4F). Eventually, a crystallographic extreme of fivefold twin was generated, consistent with pentatwin structures reported in previous literatures (36, 37). However, our in situ experiments provide a direct atomistic understanding of TB defect–induced formation of fivefold twin, which differs fundamentally from previously proposed mechanisms involving either complex GB activities or coincidental collision of TBs under consecutively hanging orientations (26, 27, 38). MD simulations further confirm this hierarchical twinning mechanism facilitated by TB kinks under tensile loading (fig. S8). This crystallographic extreme of fivefold twin structure indicates a potential of constructing complex twin structures through a common multiplication route driven by defective TBs.

To understand the driving force for the generation of a fivefold twin, we calculated the excessive energies associated with each increase in twinning fold in MD simulations, as shown in Fig. 4G. A cylindrical model (with a diameter of 7.5 nm) was adopted, and the TB junction was aligned along the axial direction of the model (see the insets in Fig. 4G). Because of the intrinsic low-energy feature of the coherent TB, primary twinning corresponds to an extremely low excessive energy of 40 eV. Besides the primary twin, a geometrically necessary Σ9 GB (θ = 38.9°) formed in the two-order twin, which results in a marked increase of excessive energy. In real circumstances, the insufficient stress relaxation at the intersection site may lead to a deviated misorientation [as shown in Fig. 2D and an additional example in nanocrystalline Pt (39)], adding to the excessive energy of the twin system. However, further increasing the twinning order to three and above leads to continuously decreased system energy, which arises from the reduced GB misorientation and the structural transition from a high-energy GB into two coherent TBs with substantially lower energy (38) upon the eventual formation of a fivefold twin, respectively. This quantitative calculation indicates that the fivefold twin can be spontaneously generated beyond the threefold twin structure, providing thermodynamic explanations for our experimental observations in Fig. 4 (A to F). However, a disclination

![Fig. 4. Formation of fivefold twin from a defective TB. (A and B) Multiplication of T3 from the kink interface (delineated by the white dashed line). (A) A Shockley partial b1 with a trailing SF was emitted from the kink interface and glided along the [111] plane, as shown by the red arrow. Scale bar, 2 nm. (B) Formation and growth of T3 via consecutive emission of b2 partials. (C and D) Formation of a fourfold twin structure via the multiplication of T4 (equivalent to T1 in terms of crystallography) inside T2. (C) Partial b2 nucleated from the junction between T3 and the kink interface. (D) Structural evolution of the kink interface that leads to the formation of a fourfold twin. (E and F) Formation of a fivefold twin structure via consecutive emission of b3 partials and the associated structural evolution of the TB intersection region. The inset demonstrates the core structure of the fivefold twin and the dihedral angles between neighboring TBs. (G) Excessive energy of different orders of twins calculated from MD simulations. A geometrically necessary GB embedded in the two-order twin (i.e., TB-GB structure) sharply increases the total excessive energy, while further twin multiplications are favored because of reduced system energy. (H) Grain size–dependent excessive energy of different TB-GB structures and an ideal fivefold structure twin predicted by the disclination model.](image-url)
of 7.35° at the junction should be bridged in the fivefold twin, resulting in an accumulated strain energy (40). Quantitative analysis (Fig. 4H and discussion S2) shows that the excessive energy associated with this disclination increases parabolically with the radius of cylindrical model (i.e., grain size) compared with linear increment of the GB energy, rendering a critical grain size of approximately 50 nm. Therefore, the formation of fivefold twin should be energetically favorable in nanosized grains according to theoretical predictions (38). In UFG materials, the formation of high-density primary twins readily refines the grains, thereby reducing the geometric threshold of hierarchical twinning from TB defects. Moreover, the elastic strain inherently stored at the disclination can be released among the five constituent twins via defect-induced structural relaxation (37).

**DISCUSSION**

The combined multiscale mechanical testing and atomistic simulations demonstrate the indispensable role of defective TBs in self-activated hierarchical twinning among a range of fcc metallic materials with low SFEs. In principle, numerous kinks left on the TBs after fabrication (e.g., as-deposited NT Cu and Ag) (10, 15) or plastic deformation (e.g., as-deformed Au in Fig. 1) can serve as preferential nucleation sites for hierarchical nanotwins. A critical dependence of this built-in hierarchical twinning propensity on the height of TB defects is unveiled through our investigation, which governs the competition between kink migration and kink nucleation. Specifically, small kinks below five atom layers tend to move along the TB and induce the softening of NT metals (15, 18), while large kinks beyond five atom layers are favored for the nucleation of secondary nanotwins and can contribute to the strain hardening of materials (Fig. 3F). The core of this hierarchical twinning mechanism rests on the widely reported successive partial dislocation motion (6); moreover, similar hierarchical twinning mechanisms can be implied from a great deal of metallic materials in previous work (23). Our real-time atomistic study of this self-activated twinning mechanism and the kink size dependence elucidates a unique intrinsic propensity of hierarchical twinning among metallic materials.

Besides the preexisting kinks on the TBs, several additional mechanisms also contribute to the kink generation, thereby facilitating the formation of hierarchical twins. First, the kink height can change dynamically during the deformation of materials. Specifically, the frequent dynamic interactions between different small TB kinks and steps can leave some large and sessile kinks with pronounced stress concentration (Fig. S2), catalyzing the consecutive emission of Shockley partials and hence the nucleation and thickening of secondary twins. In contrast, large TB kinks are prone to decomposing into smaller steps with higher mobility along the TB. The dynamic transition between these two competing processes enables the storage, transport, and release of elastic strain in concert with local stress evolutions, yielding the possibility for simultaneous nucleation of secondary nanotwins from multiple sites on a defective TB (Fig. S4). On the other hand, the binding SF can substantially reduce the mobility of TB kinks (Fig. 2, A and B, and fig. S2), which is similar to the SF pinning effects on the motion of GB steps (41), favoring the swift transition from individual partial dislocation emission to the nucleation of secondary nanotwins at the kink (see the example in Fig. 2C). Moreover, the emitted secondary partial dislocation/nanotwin can also extend and intersect with the neighboring primary TBs (42, 43), leaving a residual kink and generating additional sources on the originally perfect TB. These extrinsic TB kinks can, in turn, activate more secondary slip systems and generate secondary nanotwins inside the primary twin (figs. S6A and S9). Therefore, the commonly observed TB kinks (both inherent and residual) lay the foundation of self-activated formation of hierarchical twins, which renovates our understanding of twinning routes in metallic materials.

This self-activated hierarchical twinning mechanism has proven to occur predominantly in a great deal of fcc alloys with low SFEs, as exemplified in Cu-Al monocristalline micropillars after compression, in 304L austenite stainless steel after tensile tests at room temperature, and in CoCrFeMnNi alloys after tensile tests at 77 K in our experiments (Fig. 3 and fig. S6), as well as in some other metals and alloys reported in literature (22–24, 44, 45). With the activation of these built-in twinning sites, defective TBs can frequently stimulate the development of secondary or even hierarchical twins, as demonstrated by the geometric extreme of fivefold twin (Fig. 4, A to F). This fivefold twinning dynamic via defective TB differs fundamentally from either the collision model of multiple GB-activated slip systems or GB migration–mediated mechanism proposed in previous literature (26, 27, 36, 46), which can appropriately explain the frequent formation of secondary or hierarchical nanotwins inside primary twin lamella (with the absence of GB or Frank-Read sources). Besides, we reveal a conspicuous energy drop beyond twofold twins during the hierarchical twinning process (Fig. 4, G and H), underscoring an intrinsic propensity for spontaneous generation of multifold twins in metals and alloys with low SFEs. In principle, both secondary and multifold twins can provide additional plastic deformation dynamics to accommodate the accumulating strain and dense barriers against multiple dislocation slip systems inside the grain (22, 47). Consequently, an improved strain hardening capability is expected for hierarchically twinned materials, which has received increasing attention in the community (20, 23, 43, 44).

Our multiscale in situ experiments and simulations in this work elucidate a built-in avenue for proliferating hierarchical twins via defective TBs in a wide range of metallic materials. The key role of defective TBs and its dependence on SFE can inspire the exploration of new heat treatment or prestraining processes. In addition, the anticipated orientation-dependent competition between kink motion and secondary twin indicates an inherently tunable deformability of metallic materials with low SFEs, which can potentially benefit the design of anisotropic NT materials. Therefore, the findings in this work shed light on defect engineering to potentially improve the mechanical properties of metallic materials.

**MATERIALS AND METHODS**

**Sample preparation**

High-purity Au slugs (99.999 wt %, polished surface) with a diameter and height of 3 mm were ordered from ZhongNuo Advanced Material (Beijing) Co. Ltd., which were compressed to approximately 0.3 mm. Au discs with a diameter of 3 mm were cut from the as-deformed samples and mechanically thinned to around 20 μm. Final thinning to electron transparency was achieved by Ar ion milling using Gatan PIPS II Model 695. Cu–7 at % Al single crystal with <001> orientation was prepared using the conventional Bridgeman method. A 3 mm–by–3 mm–by–0.5 mm sample was cut from the ingot and carefully mechanically ground into a thin piece with a thickness of ~100 μm. Subsequently,
electropolishing was used to make a wedged lamella with one edge thinned to a few micrometers, and then the single-crystalline lamella was carefully glued to a specific specimen mount for in situ deformation. Submicrometer-sized cylindrical pillars with a diameter of around 740 nm and an aspect ratio (length/diameter) of ~3 were fabricated in Zeiss Auriga scanning electron microscope equipped with a gallium ion source.

304L stainless steel dog bone samples (10 mm by 2 mm by 2.5 mm) were cut from commercial slab and mechanically polished for quasi-static tensile testing. Slices with a thickness of ~200 μm were cut near the fracture surface, which were mechanically thinned to ~60 μm. Final thinning to electron transparency was realized by Struers TenuPol-5 twin jet electrochemical polishing machine using a solution of 90% ethanol and 10% perchloric acid at about ~30°C. UFG CoCrFeMnNi HEAs dog bone specimens with gauge dimensions of 10 mm by 2 mm by 2.5 mm were cut from the cold-rolled sheet (34), which were mechanically polished using SiC paper before the tensile tests.

**Supplementary materials**

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**References and Notes**

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