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In-situ TEM Dynamic Observation and Multi-scale Dissipation Mechanisms of Damage Evolution at Crack Tips in Hard Domains of Heterostructured Al-Cu Alloys

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Abstract

The damage initiation and propagation mechanisms within the "hard domains" of heterostructured Al-Cu alloys under load are crucial for their service performance. This study employed in-situ TEM tensile testing to dynamically observe crack propagation behavior within these hard domains at the atomic scale. It was found that inside the hard domains, the crack tip continuously emits dislocations. These dislocations interact with grain boundaries and second-phase particles, establishing a dynamic "emission-pile-up-annihilation" equilibrium. This process is identified as the core micro-mechanism for stress relaxation and crack-tip blunting. Notably, within the hard domains, which typically possess high SFE, the extreme local stress/strain gradients at the crack tip drive the anomalous dynamic nucleation and annihilation of stacking faults. This unconventional deformation mode directly releases the stress singularity and can induce crack deflection by generating localized shear strain. Furthermore, this in-situ study captured the critical moment of nano-void coalescence for the first time. These results indicate that the hard domains are not merely zones of brittle fracture. Instead, they achieve effective toughening dissipation through coordinated micro-nano mechanisms. Meanwhile, this research has deepened the understanding of the damage tolerance and the intrinsic strengthening-toughening synergy in heterostructured materials.

Keywords: In-situ TEM; Heterostructured Al-Cu Alloys; Hard Domains; Crack Propagation; Amorphization Transition; Stacking Fault

1 Introduction

Heterostructured Al-Cu alloys exhibit a combination of high strength and multifunctionality due to component synergy, making them widely applicable in fields such as new energy and aerospace [1-9]. Their internal microstructure heterogeneity, arising from factors like grain size, the size and distribution of second-phase particles, and compositional segregation, leads to the formation of regions with significantly different mechanical properties [10-15]. Among these, "soft domains" represent areas of high plasticity and low strength, while "hard domains" form a sharp mechanical contrast, possessing markedly higher hardness and elastic modulus but relatively lower plasticity compared to the surrounding matrix [16-21].

Crack propagation behavior is central to determining a material's fracture toughness, and this is especially true for heterostructured materials [21-25]. The internal "hard domains" typically serve as core

regions for stress concentration and damage evolution. The crack propagation behavior within these regions is influenced by the synergistic effects of soft domains and interfacial bonding strength, thereby directly affecting the overall strength of the heterostructured alloy [26-28]. Li et al. [29] further verified through molecular dynamics simulations that intermetallic compounds (IMCs) like AlCu, Al₂Cu, and Al₄Cu₉, formed during the rolling and annealing of Cu/Al composites, can enhance interfacial heat transfer efficiency. However, due to their inherent brittleness, they also reduce interfacial shear strength. Interfaces enriched with such IMCs constitute typical "hard domains". Zhang et al. [30] observed that a 380 nm thick "hard domain" of Al₂Cu at the interface was the preferred site for crack initiation. During tensile loading, cracks first initiated at multiple points within the "hard domain". They propagated into the softer Cu and Al layers only after the constraining effect of the "hard domain" reached its limit. Therefore, an in-depth investigation into the atomic-scale dynamic evolution mechanisms of crack initiation and propagation within "hard domains" is crucial for advancing the performance control and breakthroughs of heterostructured alloys.

The development of in situ characterization techniques provides key means for dynamically revealing the crack propagation process within "hard domains" [31-36]. Kou et al. [37] studied the deformation twinning behavior at pure Al crack tips using in situ TEM and molecular dynamics simulations. They found that crack-tip twinning has a dual effect on fracture: the intrinsic brittleness of coherent twin boundaries can promote cracking, while the stress concentration relief induced by twinning can hinder crack propagation. Yan et al. [38] used in situ TEM to study crack propagation in aluminum. They observed the formation of multiple Frank-Read dislocation sources in a crystal-thinned region. Nano-cracks nucleated on dislocation loops, and perfect dislocations emitted from the crack tip decomposed to form twin boundaries and stacking faults. For high-strength aluminum-based alloys, Jiang et al. [39] clarified the regulatory role of second-phase particles on crack propagation via in situ TEM. Large-sized dispersoids caused crack deflection by pinning dislocations, while fine precipitates were sheared by dislocations. Vacancy nucleation at phase interfaces led to discontinuous crack propagation. Regarding aluminum matrix composites, Liu et al. [40] demonstrated through in situ TEM tensile tests that the thickness of the MgAl₂O₄ reaction layer at the SiC/Al interface significantly influenced crack propagation. A reaction layer of appropriate thickness could delay fracture by enhancing interfacial bonding. Thus, in situ tensile testing within TEM provides an indispensable dynamic experimental platform for validating theoretical models and revealing new mechanisms of crack propagation within "hard domains".

Based on this, this paper employs in situ TEM tensile testing to systematically investigate the micro-mechanisms of crack propagation within the "hard domains" of a heterostructured Al-Cu alloy. The experiment will directly track the dynamic evolution of the crack tip. It will focus on analyzing how dislocation activities and their interactions with grain boundaries and second-phase particles regulate crack propagation. The study will also explore potential anomalous deformation structures at the crack tip under the unique constraints and high-stress environment of the "hard domain" and their effects. Furthermore, it aims to reveal the dynamic sequence of nano-void nucleation, growth, and coalescence ahead of the crack, with particular attention to the microstructural evolution during coalescence. This research seeks to deepen the understanding of fracture mechanisms in heterostructured aluminum alloys through direct atomic-scale

evidence.

2 Experiment

This study utilized a heterostructured Al-Cu alloy as the research material. Its elemental composition is listed in Table 1. First, rectangular billets measuring $10 \times 8 \times 5 \text{ mm}^3$ were obtained from the alloy by wire-electrical discharge machining. The billets were then ground, polished, and cold-rolled to a final thickness of 1 mm. This corresponds to a thickness reduction of 80%. Circular specimens with a diameter of 15 mm were subsequently cut from the rolled sheet. Part of these specimens was kept in the 80% cold-rolled condition (labeled CR). The remaining specimens were vacuum-annealed at 575°C for 1 hour (labeled AR). After grinding and ultrasonic cleaning, the CR and AR specimens were stacked in a symmetrical, alternating sequence (CR-AR). The stack was wrapped in tantalum foil, vacuum-sealed, and subjected to a hot-pressing bonding treatment using a Gleeble-3800 thermo-mechanical simulator. Subsequently, the bonded stack was compressed along the stacking direction to a thickness of 1 mm using a hydraulic press. Thereafter, thin slices with dimensions of $8 \times 3 \times 1 \text{ mm}^3$ were cut from the plate along the thickness direction by wire-electrical discharge machining. These slices were mechanically ground and polished step-by-step to a thickness below $100 \mu\text{m}$. They were then trimmed into $4 \times 2 \text{ mm}^2$ ultra-thin foils. The foils were ultrasonically cleaned in absolute ethanol, flattened to remove wrinkles, and further thinned at their central regions using a Leica EM RES102 ion-milling system. Finally, the thinned foils were mounted onto a dedicated in-situ tensile holder, as shown in Fig. 1(a). Fig. 1(b) presents an EBSD image illustrating the heterogeneous microstructure of the sample. Fig. 1(c) shows HAADF-STEM image of the interface between the “soft domain” and the “hard domain”. The corresponding elemental distribution maps of Al and Cu across this interface are displayed in Fig. 1(d) and (e), respectively. The in-situ tensile experiments were carried out inside a JEM-F200 transmission electron microscope. A Gatan Model 671 single-tilt, side-entry straining holder cooled by liquid nitrogen was employed. During the test, the specimen was subjected to uniaxial tensile loading.

Table 1 Content of main elements in Al-Cu alloy (%)

Content of main elements (%)						
Sn	P	Sc	Ca	Cu	Fe	Al
<0.001	<0.001	0.009	0.0011	2.69	0.012	balance

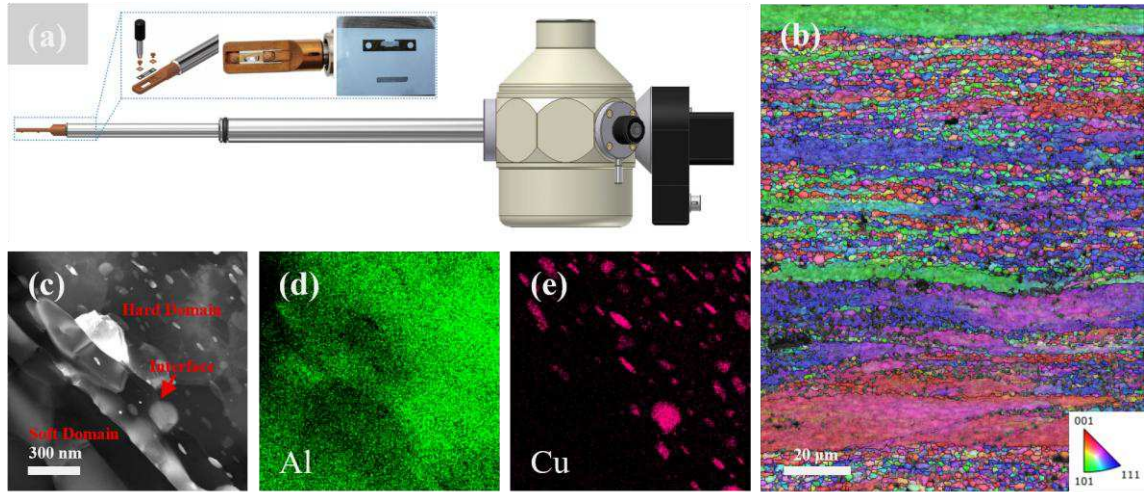


Fig. 1 Specialized Stage for In-situ Tensile Testing: (a) In-situ stretching of the storage platform and sample; (b) EBSD images showing the heterogeneous structures of the in-situ TEM samples; (c) HAADF-STEM images at the interface; (d,e) Show element distribution maps of Al and Cu, respectively.

3 Results

3.1 Dislocation Multiplication Phenomenon at the Crack Tip

Fig. 2(a) displays a crack propagation image observed within a “hard domain” during the in situ tensile experiment. It can be seen that, as the in situ tension progressed, a relatively large crack formed inside the “hard domain”. Fig. 2(b) presents a magnified morphology of the crack tip. A significant stress concentration is evident at the crack tip. A high density of dislocations is present ahead of it.

Fig. 3 presents TEM bright-field images of crack propagation in the heterostructured Al-Cu alloy during the in-situ tensile experiment, in which the tensile loading direction is perpendicular to the crack propagation direction.

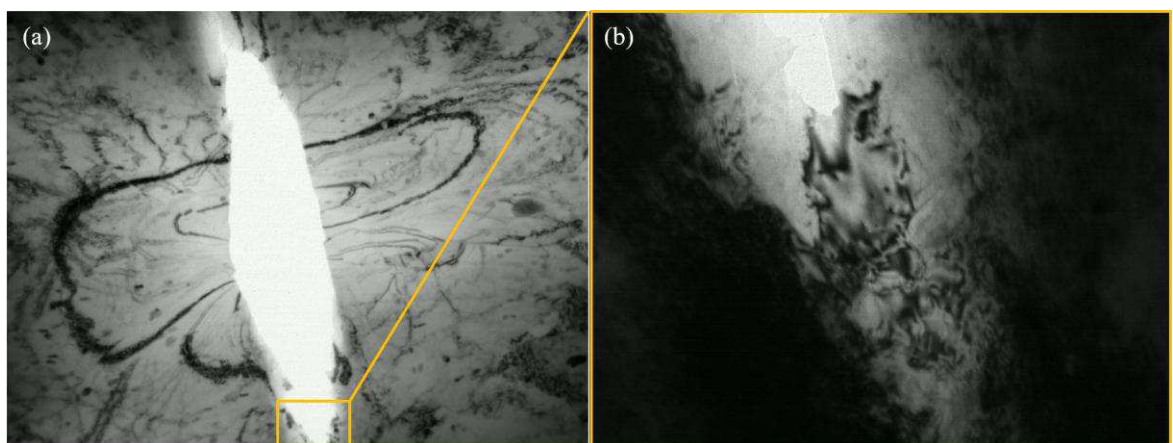


Fig. 2 Crack region in the "hard domain" observed during the in-situ tensile experiment: (a) Crack morphology images in the "hard domain"; (b) Magnified view of the observed region

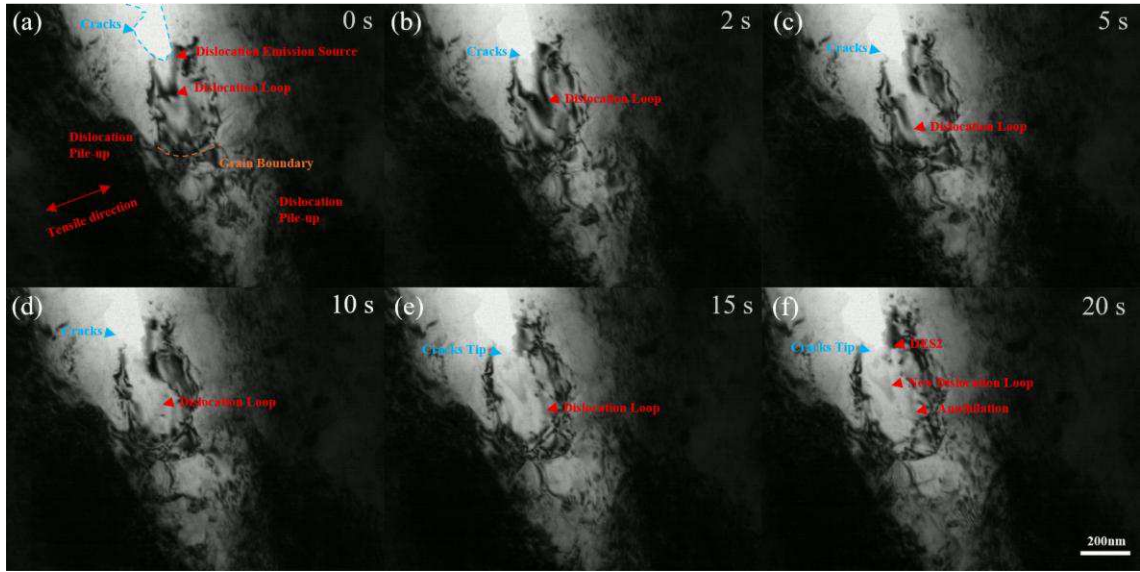


Fig. 3 TEM images showing crack propagation at different time points during in-situ tensile testing: (a) $t=0$ s; (b) $t=2$ s; (c) $t=5$ s; (d) $t=10$ s; (e) $t=15$ s; (f) $t=20$ s

In Fig. 3(a), the crack front morphology is clearly visible, and an active dislocation emission source (marked by the red arrow) is formed at the crack tip. This source continuously emits multiple sets of dislocation loops toward the front. These dislocation loops exhibit distinct dark contrast. As the tensile process progresses, the outer dislocation loops encounter strong obstruction when moving toward the lower grain boundary, leading to significant accumulation at the grain boundary. This accumulation directly causes an increase in the energy of the grain boundary region and a significant rise in the degree of atomic arrangement disorder. The grain boundary exerts obstructive and restrictive effects on dislocation movement, effectively slowing down the crack propagation rate.

Figs. 3(b)-(d) continuously record the dynamic evolution of dislocation loops: these loops expand outward continuously, and after gradually approaching the grain boundary, their contrast gradually fades—indicating that the dislocation loops annihilate after interacting with the grain boundary. Meanwhile, the crack tip continues to extend forward, and a large number of dislocations accumulate on both sides of the dislocation source emission path. Due to severe distortion of the local crystal structure and a significant increase in energy in these regions, the contrast of the regions on both sides gradually deepens and darkens with the tensile process.

In Fig. 3(e), the crack further extends forward, and a new dislocation emission source is formed at its tip. This new source also continuously emits dislocation loops forward, and these newly generated loops gradually annihilate when moving to the front grain boundary. This continuous process clearly demonstrates that during crack propagation, the emission and annihilation of dislocations are always in a dynamic equilibrium state. This equilibrium mechanism not only precisely regulates the stress distribution at the crack tip but also continuously maintains the plastic deformation capacity of the material in the crack tip region.

Furthermore, in Fig. 3(f), a new dislocation source appears on the right side of the initial crack tip. This source continuously emits and accumulates dislocations to the right, resulting in a significant increase in

dislocation density and an aggravated degree of atomic arrangement disorder. During this process, the region in front of the main crack gradually thins and exhibits a transparent characteristic. A DFZ is formed due to dislocation accumulation on both sides of the crack, and the local stress concentration at the crack tip is effectively released through dislocation emission and accumulation, thereby slowing down the crack propagation rate[34-40].

Stress concentration at the crack tip is the core driving force for dislocation emission. Under high stress, atomic displacement leads to the formation of dislocation sources and the emission of dislocation loops. Dislocation expansion not only retards crack propagation but also releases stress. The accumulation of dislocations on both sides of the crack forms a DFZ in front of the main crack, which further releases local stress to slow down crack propagation. From the perspective of grain boundary function: during the expansion of dislocation loops, they interact with grain boundaries. The grain boundaries not only hinder dislocation movement (causing dislocation accumulation, which increases grain boundary energy and atomic disorder degree and alters diffraction contrast) but also trigger dislocation annihilation after intersection—forming a dynamic equilibrium of "generation-disappearance". The annihilation process not only releases stress but also creates conditions for the emission of new dislocations, thus maintaining the plastic deformation capacity of the crack tip.

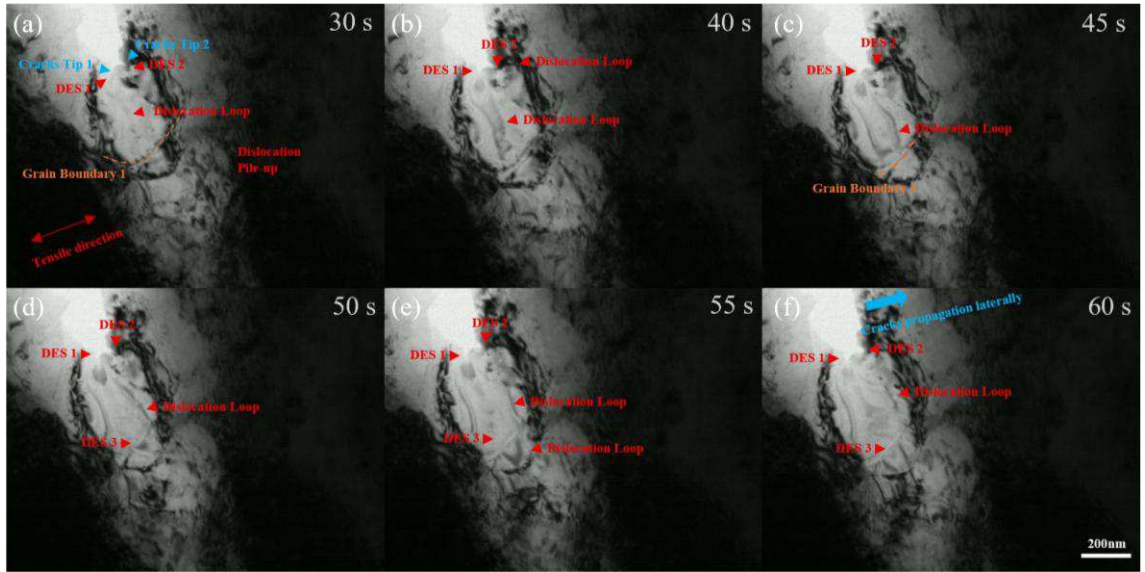


Fig. 4 TEM morphology images showing crack propagation at different time points during in-situ tensile testing: (a) $t=30$ s; (b) $t=40$ s; (c) $t=45$ s; (d) $t=50$ s; (e) $t=55$ s; (f) $t=60$ s

In-depth studies on the sample in Fig. 3 (Fig. 4, directional tracking TEM bright-field images of crack tip propagation) show that: compared with the state at 20 seconds, the two sets of crack tips gradually sharpen, and both generate new dislocation sources that continuously release dislocations forward. Stress concentration is effectively released through dislocation emission (Fig. 4 (a)). Meanwhile, the grain boundary region gradually thins, and its orientation difference with one side of the crack decreases, which weakens the resistance to dislocations—this leads to the gradually brightening contrast at Grain Boundary 1. Such changes not only provide more channels for dislocation motion but also affect the crack propagation path, reflecting

the dual role of grain boundaries: they act not only as obstacles to dislocation motion but also as potential dislocation emission sources.

Although Grain Boundary 1 exerts a blocking effect on the dislocations from Dislocation Source 1, it forms a new Dislocation Source 3 on itself and continuously emits dislocations forward (Figs. 4 (b)-(e)), which further confirms its dual role. It regulates the stress distribution at the crack tip and the plastic deformation capacity by adjusting dislocation behavior. With the progression of the process, the DFZ at the crack front continues to expand. Due to the massive accumulation of dislocations on both sides, increased atomic disorder, and dislocation entanglement, the resistance to crack expansion toward both ends increases. Therefore, in Figs. 4 (a)-(f), although dislocation multiplication occurs at Crack Tip 2, its propagation is not obvious; in contrast, the DFZ at Tip 1 continues to expand and still shows a tendency to extend further. This indicates that the DFZ, as an important symbol of stress release at the crack tip, alleviates stress concentration through dislocation emission and accumulation, thereby effectively retarding crack propagation.

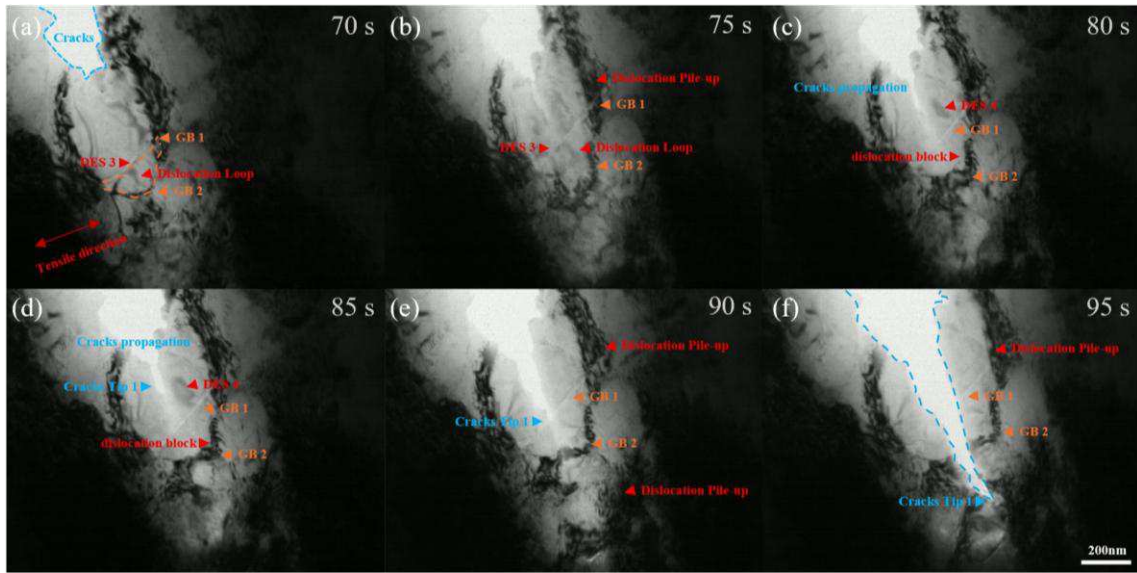


Fig. 5 TEM morphology images showing crack propagation at different time points during in-situ tensile testing:

(a) $t=70$ s; (b) $t=75$ s; (c) $t=80$ s; (d) $t=85$ s; (e) $t=90$ s; (f) $t=95$ s

During the tensile process, the contrast of Grain Boundary 1 (GB1) within the crystal thinning region ahead of the material's crack gradually becomes transparent, while the grain boundaries outside this region persist. This phenomenon indicates that tearing and reorganization of grain boundaries occur within the thinning region — original grains are split into multiple small grains by the crack, and dislocation accumulation accompanies the formation of dislocation walls and subgrain boundaries. Subgrain boundaries not only alter the local crystal structure but also provide new propagation paths for the crack, guiding it to propagate along the subgrain boundaries. This reflects changes in the local stress state and the diversity of crack propagation paths, confirming that subgrain boundaries are a significant microstructural evolution feature during crack propagation.

At 70 seconds, GB1 gradually disappears under the influence of the crack, and Grain Boundary 2 (GB2) below it also thins gradually (Fig. 5(a)). This results from the changed stress state caused by continuous

tension and also demonstrates that the significant changes in grain boundary structure and properties during crack propagation affect dislocation motion and crack paths. The dislocation emission source 3 originally located on GB1 still emits dislocations toward GB2, while the dislocation emission source at GB2 continuously emits dislocations to the dislocation accumulation region below. The dynamic process of dislocation emission and accumulation on adjacent grain boundaries further confirms the stress concentration state at the crack tip. After the disappearance of GB1, the crack rapidly propagates to this region within a short time (Fig. 5(b) – (d)), the disappearance of GB1 provides more channels for the crack, accelerating its propagation rate. This also verifies the changes in stress distribution at the crack tip, dislocation motion, and propagation path. At this point, a new dislocation emission source 4 forms on the right side of the crack tip, continuously emitting dislocation loops outward; in Fig. 5(c) – (f), the crack shows an obvious tendency of transverse propagation with a significant increase in width. The activation of dislocation emission source 4 and the expansion of dislocation loops alleviate the stress concentration at the crack tip to a certain extent, and the direction and rate of crack propagation are regulated through dislocation slip and multiplication. The transverse propagation phenomenon further confirms the complexity and diversity of dislocation behavior during crack propagation (The overall crack propagation process can be observed in Supplemental Video 1).

Frank-Read sources, as a key mechanism for dislocation multiplication, reflect the high-stress state at the crack tip. They alleviate stress concentration and retard crack propagation by emitting and expanding dislocation loops. Previous observations show that dislocations emitted from the crack tip pile up ahead, promoting crack nucleation near the dislocation emission sources at GB1 and GB2, and driving the propagation and cracking of the crack tip. This is related to the continuous emission of dislocation loops by multiple Frank-Read sources formed in the crystal thinning region—dislocation loops alleviate stress concentration and regulate the direction and rate of crack propagation through slip and multiplication; high-energy dislocation loops serve as crack nucleation sites, and perfect dislocations emitted from the crack tip slide and dissociate in the crystal, further regulating the propagation rate and direction. In addition, when there is a grain boundary ahead of the crack, the grain boundary in the thinning region is torn, allowing stress to release here, leading to the gradual disappearance of the grain boundary.

In summary, the significant changes in grain boundary structure and properties during crack propagation affect dislocation motion and crack paths: GB1 gradually disappears with the application of tension and crack propagation, while GB2 below it thins; the disappearance of grain boundaries provides more channels for the crack, accelerating its propagation. The new dislocation emission source 4 on the right side of the crack tip continuously emits dislocation loops outward, causing transverse propagation of the crack and an increase in its width, which confirms that dislocation emission can effectively release stress and retard crack propagation. Dislocations pile up ahead, promoting crack nucleation near the dislocation emission sources at GB1 and GB2 and driving the crack tip to propagate forward and crack. Multiple Frank-Read sources formed in the crystal thinning region, as a key mechanism for dislocation multiplication, alleviate stress concentration at the crack tip by emitting dislocation loops. The results indicate that high-energy dislocation loops act as crack nucleation sites, and the random distribution of dislocation sources enables the regulation of crack propagation rate and direction.

3.2 Interaction of Low-Angle Grain Boundaries (LAGBs) and Second Phases with Cracks

Low-Angle Grain Boundaries (LAGBs) are formed by dislocation rearrangement, with a misorientation angle typically less than 15° , and exert a significant influence on crack behavior during crack propagation[41-45]. Their formation not only alters the local stress distribution at the crack tip but also significantly regulates crack propagation behavior by influencing dislocation motion and propagation paths. Fig. 6(a) records the dynamic changes of the DFZ at the crack front, where the in-situ loading direction is perpendicular to the crack propagation direction. A large number of dislocations accumulate on both sides of the DFZ, and two sets of LAGBs are present inside the DFZ. As shown in Fig. 6(b) – (e), when the dislocation loops marked by red arrows move to the dislocation accumulation zone on the right, the contrast of LAGB 1 first darkens and then brightens. Before the crack tip reaches the region, the dislocations emitted by the crack tip continuously entangle at LAGB 1, leading to an increase in the degree of atomic arrangement disorder and elastic strain energy at the grain boundary, which darkens the contrast. When the crack tip approaches, a new DES2 is formed on LAGB 1, emitting dislocations toward DES1. This process enables the continuous release of strain energy of LAGB 1 at crack tip 1, resulting in thinning of the local region and brightening of the contrast. This contrast change directly reflects the variation of the stress state at the crack tip, confirming the important role of LAGBs.

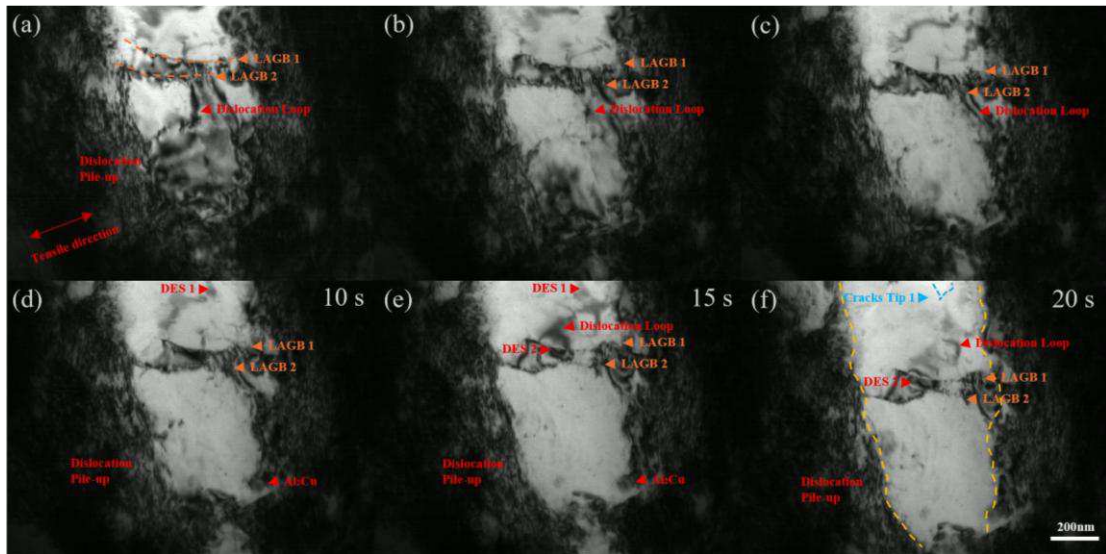


Fig. 6 Interaction Behavior of Cracks with Low-Angle Grain Boundaries and the Second Phase - Stage 1: (a) $t=0$ s; (b) $t=2$ s; (c) $t=5$ s; (d) $t=10$ s; (e) $t=15$ s; (f) $t=20$ s

Notably, with the progression of loading, LAGB 2 continuously moves toward LAGB 1. The contrast of the region between the two sets of LAGBs gradually brightens and tends to be consistent with the outer DFZ. During in-situ loading, the misorientation angle between the region enclosed by the two sets of LAGBs and the outer DFZ continuously decreases, with improved consistency. This dynamic change not only regulates the stress distribution at the crack tip but also influences the crack propagation path and rate through dislocation rearrangement and changes in local stress states, further confirming the critical role of LAGBs in

crack propagation.

Second phases play a key role in crack propagation: they not only act as an obstacle to dislocation motion but also generate a "shielding effect" by accumulating surrounding dislocations[46-52]. This effect modulates local stress-strain conditions to reduce the crack propagation rate, which is conducive to enhancing the material's strength and toughness. As shown in Fig. 6(a) – (f), a large number of dislocations entangle near the Al_2Cu phase at the bottom right corner, and the DFZ around this phase (as indicated by the yellow dashed line) takes the Al_2Cu phase as its boundary—this confirms the regulatory effect of second phases on the direction and rate of crack propagation.

In addition, the fracture of second phases in bulk alloys under tensile loading increases the susceptibility to microcrack or micropore initiation and releases the elastic strain energy at the crack tip. Their sudden and intermittent failure further intensifies stress-strain oscillations at the crack tip, inducing features such as stacking faults. These phenomena affect the crack propagation path by altering the local stress state.

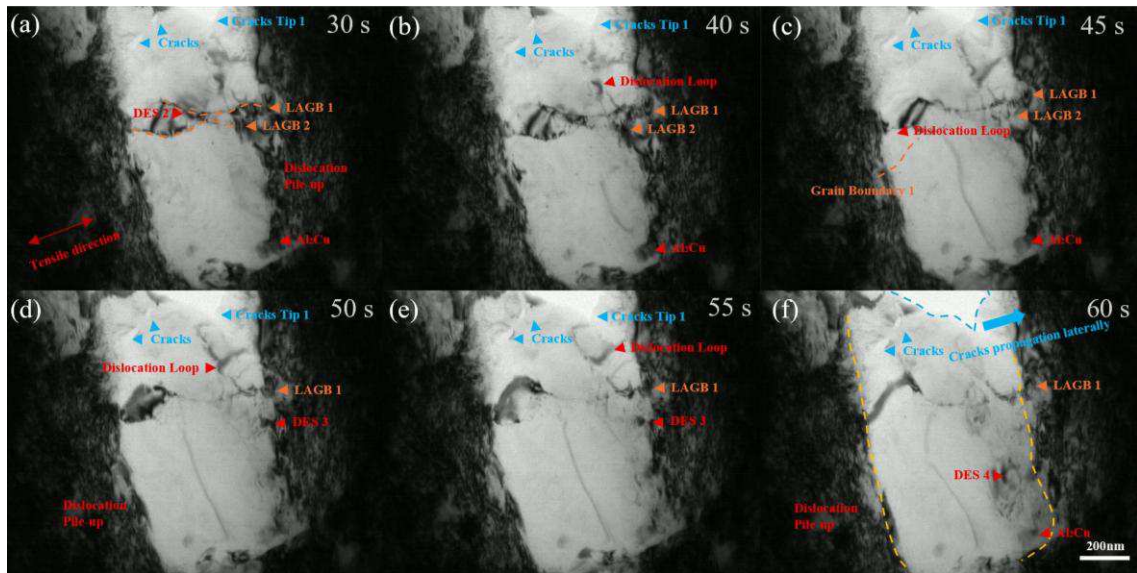


Fig. 7 Interaction Behavior of Cracks with Low-Angle Grain Boundaries and the Second Phase - Stage 2: (a) $t=30$ s; (b) $t=40$ s; (c) $t=45$ s; (d) $t=50$ s; (e) $t=55$ s; (f) $t=60$ s

In Fig. 7(a), two nanocracks appear on the left side of crack tip 1. The DES2 on LAGB 1 continuously emits dislocations, and the stress release through this process delays crack propagation. Meanwhile, LAGB 2 continuously moves toward LAGB 1; a large number of dislocations accumulate on both sides of the DFZ, and dense dislocation entanglement is also observed near the Al_2Cu particles at the bottom right corner. As shown in Fig. 7(b) – (e), with the progression of tensile loading, the middle part of LAGB 1 intersects with LAGB 2 and is split into left and right segments, while crack tip 1 gradually expands. Subsequently, LAGB 2 merges with LAGB 1, DES2 disappears, the contrast on both sides tends to brighten and the orientations become consistent. A new DES3 is formed on the lower right side of LAGB 1, which continuously emits dislocations toward LAGB 1. In Fig. 7(f), as the crack propagates transversely, the width of the DFZ increases and gradually encloses the Al_2Cu particles at the bottom right corner. Under continuous stress, LAGB 1 is pulled to form left and right protrusions facing the crack tip and is on the verge of disappearance—this again

confirms its important role in stress release at the crack tip, dislocation motion, and crack propagation. In addition, there is no significant volume growth in the two newly formed nanovoids at the top right corner. It is inferred that during the propagation of crack tip 1, the dislocations emitted by DES3 and DES4 are preferentially released at this crack tip rather than in the nanovoid region.

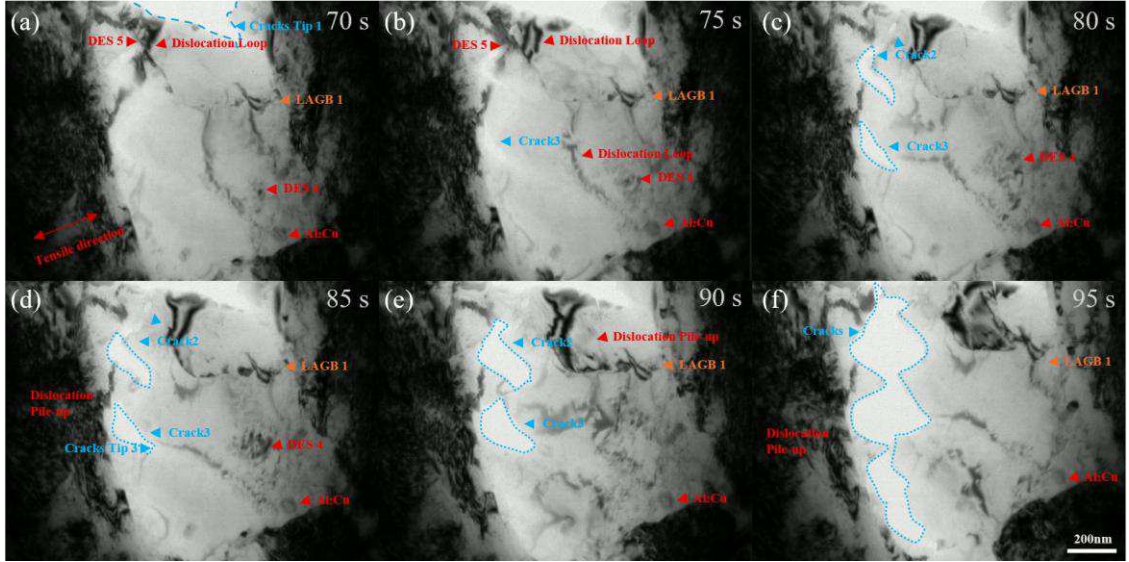


Fig. 8 Interaction Behavior of Cracks with Low-Angle Grain Boundaries and the Second Phase - Stage 3: (a) $t=70$ s; (b) $t=75$ s; (c) $t=80$ s; (d) $t=85$ s; (e) $t=90$ s; (f) $t=95$ s

As shown in Fig. 8(a) – (e), a new DES5 is generated near the nanovoid at the top left corner, which continuously emits dislocations toward crack tip 1, effectively relieving the stress concentration at the tip. At this point, LAGB 1 disappears completely, and a new nanovoid 3 is formed on its lower left side; the dislocation emission source DES4 emits dislocations toward the left, causing the continuous widening of the DFZ. With the progression of tensile loading, the existing nanovoid 2 and the new nanovoid 3 continue to grow, while crack tip 1 at the top right corner shows no obvious change. It is inferred that after long-term stress release in the dislocation accumulation zone on the right, the critical propagation stress is insufficient, so nanovoids are preferentially formed and expanded. During this process, dislocation loops can be observed being released from the bottom of nanovoid 2 to the top of nanovoid 3; the two nanovoids merge in Fig. 8(f), and a new large crack is formed at 95 seconds, which further propagates downward. At this stage, the width of the DFZ on both sides of the crack no longer increases, and the contrast tends to brighten (The overall crack propagation process can be observed in Supplemental Video 2).

Crack propagation modes are mainly classified into mode I (opening mode), mode II (sliding mode), and mode III (tearing mode). In this process, as the crack tip approaches, dislocation lines disappear and irregular slip traces appear. Combined with the thickness differences in local regions and the characteristics of slip trajectories, it is determined that the crack may propagate in a mixed mode I/III. Under tensile loading, the thin region is exposed as the slip trajectories of dislocations emitted by multiple dislocation sources migrate, resulting in the widening of the DFZ; when the crack tip reaches the thin region, the slip system changes, and the propagation mode transitions from mixed mode I/III to mode I. Due to differences in local

crystal orientation, stress concentration, and strain rate, alternating sharpening and blunting of the crack tip occur, leading to dynamic changes in the propagation rate rather than a stable trend. At the moment when the propagation rate decreases, nanovoids are instantly formed in front of the crack tip; these nanovoids gradually grow and coalesce into the main crack with continuous tension, while the blunting of the original crack tip slows down propagation, exhibiting the characteristic of discontinuous propagation.

Further analysis shows that the formation of DES5 is related to the redistribution of the local stress field, and its activation relieves the stress at the crack tip through dislocation emission. The disappearance of LAGB 1 may result from dislocation motion and grain boundary migration, while the formation of the new nanovoid 3 may be associated with vacancy aggregation caused by local stress concentration. The growth and merging of nanovoid 2 and nanovoid 3 reveal an important mechanism of crack propagation: nanovoids merge under the action of loading and connect with the main crack, promoting the propagation of the main crack. The change in DFZ width is a key indicator: an increase in DFZ width reflects that dislocation emission and motion effectively relieve stress concentration; when the width no longer increases, it may be due to decreased activity of dislocation sources or a transition in propagation mechanism (e.g., from mixed mode to mode I). In addition, the alternating effect of crack tip sharpening and blunting collectively affects the propagation rate and path.

3.3 Amorphization Transition in the Ligament Region of Nanovoids at the Crack Tip during In-situ Tensile Testing

Fig. 9 (a) shows the crack tip morphology in the hard domain during in-situ tensile testing, Fig. 9 (b) displays the morphology of nanovoids ahead of the crack tip, and Fig. 9 (c)-(e) are high-resolution TEM images of the nanovoid coalescence process. Observations reveal that multiple nanovoids appear in the region ahead of the crack tip; these nanovoids gradually coalesce and merge with the main crack, driving the crack tip to propagate forward. During the coalescence of two nanovoids, an "amorphization transition" occurs in the ligament region between them. (The overall crack propagation process can be observed in Supplemental Video 3)

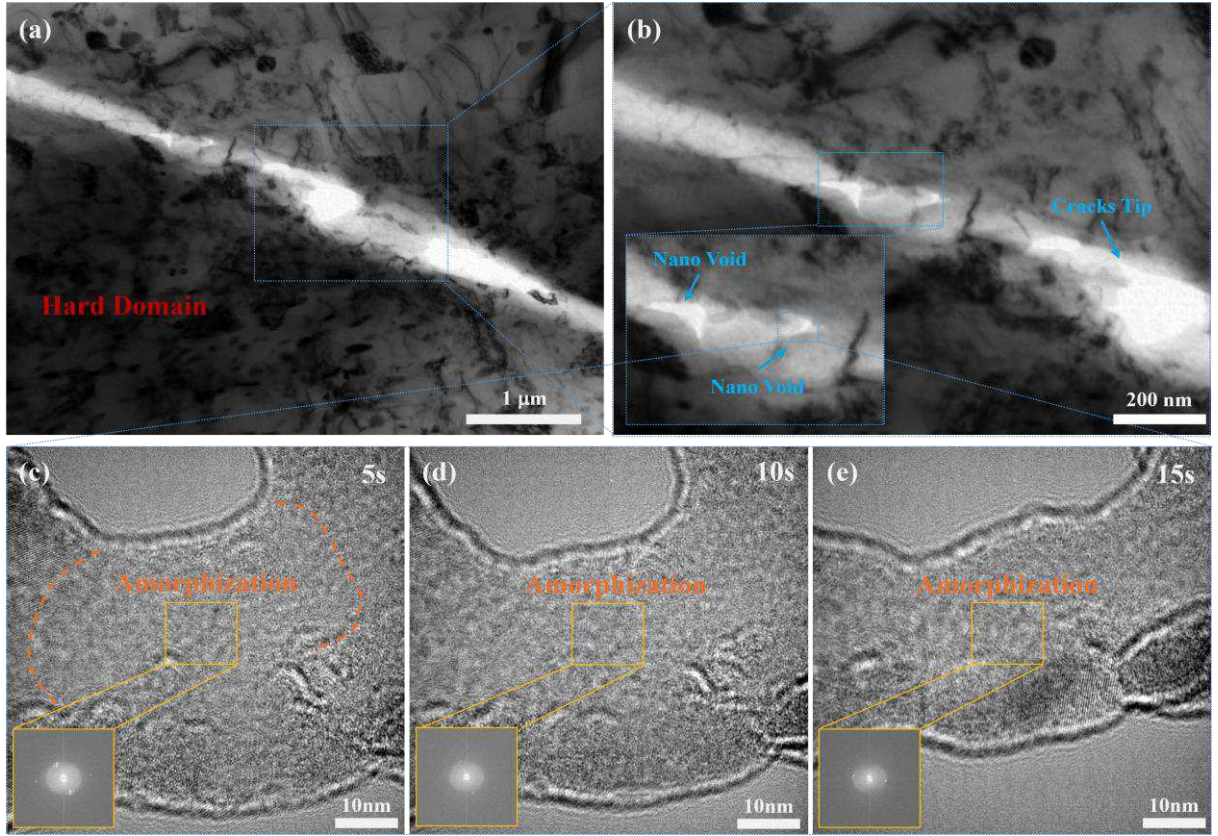


Fig. 9 (a) Crack morphology; (b) Nanovoid morphology; High-resolution TEM images of the nanovoid coalescence process: (c) $t=0$ s; (d) $t=5$ s; (e) $t=10$ s

This phenomenon may be attributed to the significant stress concentration at the crack tip under tensile loading. The nanovoids ahead act as stress amplifiers, further distorting the surrounding stress field. The ligament region between the two voids bears extremely high biaxial tensile stress, which may no longer follow the classical von Mises yield criterion. This stress state may inhibit conventional dislocation slip and multiplication (as dislocation multiplication requires deviatoric stress) while greatly reducing the activation energy barrier for atomic diffusion. In addition, void coalescence requires extreme plastic contraction in the ligament region, similar to "necking". This deformation occurs over an extremely short distance at the nanoscale, generating a huge strain gradient. When the critical value is exceeded, the traditional lattice structure can no longer be maintained, leading to the complete loss of long-range lattice order. Atoms are agitated and displaced from their equilibrium lattice positions, forming an amorphous region. In summary, the amorphization between two nanovoids is essentially a highly localized solid-state phase transformation driven by extreme strain gradients. It represents an "energy dissipation shortcut" adopted by the material when traditional void coalescence mechanisms (such as dislocation emission and internal necking) fail or are insufficient to accommodate such severe and rapid deformation.

3.4 Evolution of stacking faults near nanovoids ahead of the crack tip during in-situ tensile testing

Figure 10(a) shows a typical crack propagation morphology within a “hard domain” of the heterostructured Al-Cu alloy. The crack propagates perpendicular to the tensile direction. A nano-void is present ahead of the crack tip. Figures 10(b–e) clearly reveal, through in-situ high-resolution images, the significant dynamic growth and annihilation of stacking faults around the nano-void at the crack tip in the “hard domain”. This behavior of the stacking faults directly relaxes the stress singularity at the crack tip. It accelerates energy dissipation and promotes crack-tip blunting. Furthermore, as shown in the geometric phase analysis (GPA) strain maps (f3, g3), intense local shear strain exists around the stacking faults. The internal stress field generated by this strain interferes with the stress distribution ahead of the main crack. This can cause the crack to deflect or branch, thereby increasing the tortuosity and resistance of crack propagation. Finally, the stacking faults nucleated within the “hard domain” can effectively accommodate the strain concentration at the interface. This delays interface debonding or micro-void growth caused by deformation mismatch. This effect is particularly important for suppressing interface-dominated crack propagation.

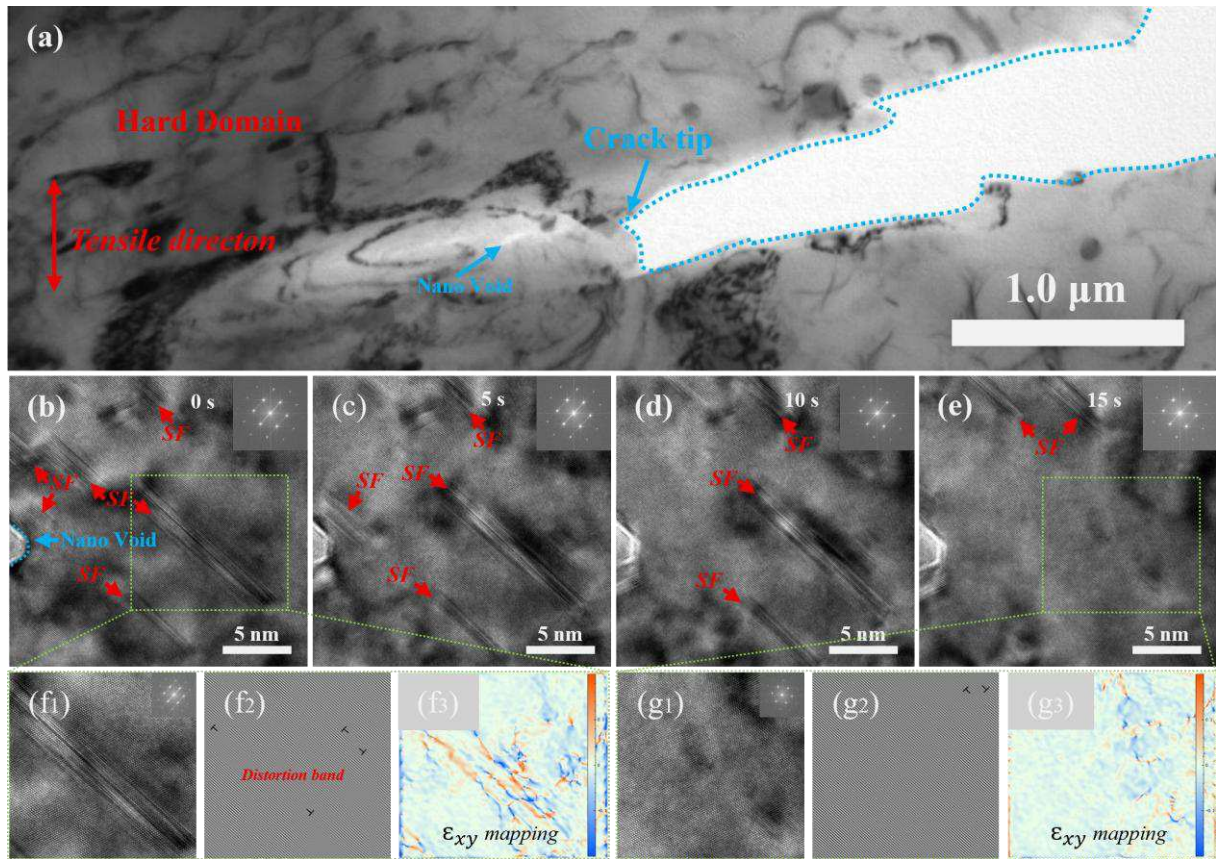


Fig. 10 (a) Crack morphology. High-resolution TEM (HRTEM) images showing the growth of stacking faults near a nano-void: (b) $t = 0$ s, (c) $t = 5$ s, (d) $t = 10$ s, (e) $t = 15$ s. (f1): Locally enlarged HRTEM image (with its FFT) of the stacking-fault region in (b). (f2): Inverse Fast Fourier Transform (IFFT) image of the stacking-fault region in (b). (f3): Geometric Phase Analysis (GPA) map of the stacking-fault region in (b); (g1): Locally enlarged HRTEM image (with its FFT) of the stacking-fault region in (e). (g2): Inverse Fast Fourier Transform (IFFT) image of the stacking-fault region in (e). (g3): Geometric Phase Analysis (GPA) map of the stacking-fault

region in (e).

Pure aluminum and most of its alloys possess a fcc structure with a high intrinsic stacking fault energy [53]. Under conventional plastic deformation, full dislocation slip dominates as the primary mechanism. Consequently, the formation and stabilization of stacking faults are difficult. Therefore, the observation of active stacking fault activity at the crack tip within the "hard domain" indicates the presence of special local micro-conditions. This phenomenon can be attributed to several factors. First, the crack tip is typically a region of highly concentrated stress and strain. Even in high-SFE materials, the extremely high local stress can be sufficient to overcome the SFE barrier. This drives the dissociation of full dislocations and the subsequent nucleation of stacking faults. Simultaneously, a significant property mismatch exists between the "hard domain" and the surrounding softer matrix. This leads to highly incompatible deformation and generates severe strain gradients near their interfaces. To accommodate this incompatibility, a high density of GNDs is produced. The interactions and reactions among these GNDs provide favorable conditions for the nucleation of partial dislocations and stacking faults. Furthermore, within the "hard domain", conventional deformation modes may be constrained. Obstacles such as precipitates can impede the activation of primary slip systems. When dominant slip is hindered, the system may switch to alternative deformation modes to release energy. Under the extremely high strain rate at the crack tip, the activation of stacking faults serves as one such alternative deformation mechanism. This is analogous to the mechanism of deformation twinning observed at crack tips in Al-Cu-Mg alloys, as reported in the literature [32]. (The overall crack propagation process can be observed in Supplemental Video 4)

4 Conclusions

This study employs in-situ TEM tensile testing to reveal the atomistic mechanisms of crack propagation within the "hard domains" of a heterostructured Al-Cu alloy. The key findings are:

1) A dynamic dislocation-mediated coordination mechanism at the "hard domain" crack tip is revealed. The crack tip continuously emits dislocations. Their interaction with grain boundaries and second phases establishes a dynamic "emission-pile-up-annihilation" equilibrium. This process is central to stress relief, DFZ formation, and crack-tip blunting.

2) Anomalous SF activity is discovered in the high-SFE "hard domain". Driven by extreme stress/strain gradients at the crack tip, dynamic nucleation and annihilation of SFs are observed. This unconventional deformation directly relaxes the stress singularity. The induced local shear strain can lead to crack deflection, increasing propagation resistance.

3) In-situ observation of amorphization during nano-void coalescence is achieved. The ligament between two coalescing voids undergoes a solid-state crystalline-to-amorphous transition at the critical coalescence moment, driven by an immense strain gradient. This represents an extreme nanoscale energy dissipation mechanism.

4) The regulatory role of microstructure on crack path is clarified. Cracks preferentially initiate and propagate within the "hard domain". Their path is significantly governed by low-angle grain boundaries and second-phase particles, underscoring the "hard domain" as the core region for damage evolution.

In summary, this work demonstrates that “hard domains” achieve effective toughening dissipation through multiple micro-nano mechanisms: dynamic dislocation coordination, activation of anomalous stacking faults, and void-coalescence-induced amorphization. These findings deepen the understanding of the strengthening and toughening mechanisms in heterostructured materials.

Credit authorship contribution statement

Chuanchuan Ma: Conceptualization, Writing—original draft, writing—original draft preparation. **Tuo Li:** Methodology, Data curation. **Duo Zhang:** Resources, Data curation. **Yuxiang Zhao:** Conceptualization. **Feng Wang:** Validation, Formal analysis. **Yafei Teng:** Validation. **Bo Gao:** Data curation, Resources. **Zhibing Chu:** Resources, Project administration. And all authors reviewed the manuscript.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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