In situ atomistic mechanisms of detwinning in nanocrystalline AuAg alloy

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ABSTRACT Detwinning is an important plastic deformation mechanism that can significantly affect the mechanical properties of twin-structured metals. Although many detwinning mechanisms have been proposed for pure metals, it is unclear whether such a deformation model is valid for nanocrystalline alloys because of the lack of direct evidence. Here, the atomic-scale detwinning deformation process of a nanocrystalline AuAg alloy with an average grain size of ~15 nm was investigated *in situ*. The results show that there are three types of detwinning mechanisms in nanocrystalline AuAg alloys. The first type of detwinning results from grain boundary migration. The second type of detwinning occurs through combined layer-by-layer thinning and incoherent twin boundary migration. The last one occurs through incoherent twin boundary migration, which results from the collective motion of partial dislocations in an array.

Keywords: twin-structured, detwinning, *in situ*, transmission electron microscopy, atomic scale

INTRODUCTION

Face-centered cubic (FCC)-structured metals with grown nanotwins have attracted great interest because of their ultrahigh strength and excellent ductility compared to their twin-free counterparts [1–4]. Because the mechanical properties of twin-structured metals are directly related to their deformation mechanisms, a large number of studies have been conducted to study the deformation mechanism of twin-structured metals in the last decades [1–8]. It is well established that the strengthening of twin-structured metals results from dislocations intersecting with coherent twin boundaries (CTBs), which was confirmed by both molecular dynamics (MD) simulations and experimental investigations [1,5,6,9–12]. In contrast, for the softening mechanism, *in situ* atomic-scale observations of the detwinning process have rarely been studied, especially for twin-structured nanocrystalline (NC) metals with grain sizes of ~15 nm [3,9,10,13–16]. In addition to dislocation-CTB intersection, several MD simulations and experiments have indicated that detwinning can also significantly affect the mechanical properties of twin-structured metals [7,10,11,17]. The atomic-scale mechanism of detwinning is crucial, which may provide clues for designing twin-structured metals with high strength and excellent ductility [1,10,16–19].

Previous MD studies have proposed that the detwinning process results from partial dislocation emission from CTB-grain boundary (GB) intersection and glide along the plane adjacent to the CTB [12–15]. This process leads to CTB migration, which in turn leads to twin thinning in a layer-by-layer manner [16,18,20–23]. Other studies have suggested that detwinning can also result from incoherent TB (ITB) migration [8,19,24,25]. For twins in FCC-structured metals, an ITB consists of an array of Shockley partial dislocations on each {111} plane [19,24–27]. The detwinning process results from the partial dislocation array moving back to the boundary or free surface [13,16,19,25]. In contrast, previous experiments have indicated that this ITB migration can originate from partial dislocations moving successively at the ITB, which results in steps on the TBs [14,15,19,28,29]. However, previous modeling has also predicted that ITB migration can occur *via* the collective glide of partial dislocations in an array from the ITB [19,24,29–31]. However, most previous studies are based on the results of coarse-grained or ultrafine-grained metals, and whether other detwinning mechanisms exist for NC metals with grain sizes of ~15 nm remains unclear. More importantly, the proposed detwinning mechanism is based on experiments on pure metals, and its validity for alloy metals has not been confirmed. Thus, it will be interesting to provide *in situ* atomic-scale evidence of the deformation mechanism of detwinning in NC metallic alloys.

In this study, the deformation behavior of an NC AuAg alloy with an average grain size of ~15 nm was investigated *in situ* at the atomic scale using a custom-built device [32,33]. The results show that there are three types of detwinning mechanisms: GB migration-induced detwinning, combined layer-by-layer thinning and ITB migration, and ITB migration. We provide direct *in situ* atomic-scale evidence that full dislocations can hinder ITB migration, which has only been predicted in modeling thus far.

EXPERIMENTAL SECTION

An NC AuAg alloy thin film with a uniform composition (Figs S1 and S2) was deposited on a single-crystal NaCl substrate using a co-sputtering strategy with an ATC-1800F direct-current magnetron sputtering system. Au and Ag targets (>99.99% purity) were used. Before sputtering, the air pressure of the chamber was $\sim 2 \times 10^{-7}$ Torr (1 Torr = 133.3 Pa). During sputtering, high-purity Ar gas (99.995% purity) was fed into the chamber, and the chamber pressure was maintained at $\sim 2 \times$

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10^{-3} Torr. The sputtering powers of Au and Ag were ~ 19 and ~ 23 W, respectively, corresponding to a nominal deposition rate of ~ 8.5 nm min $^{-1}$. The AuAg alloy film with a thickness of ~ 25 nm was deposited on the NaCl substrate and then annealed at 200°C for 1 h. Transmission electron microscopy (TEM) was used to reveal the boundaries and interiors of the grains (Fig. S2). Then, the grain size was determined from the TEM images by measuring the length of lines drawn between GBs as shown in Fig. S2. Subsequently, the statistical grain size distribution was obtained by measuring more than 300 grains from different regions of the NC thin film.

Under an optical microscope, the thin film on the NaCl substrate was attached to a bimetallic extensor using epoxy resin. Subsequently, the NaCl substrate was etched with water, and the AuAg thin film became free-standing and remained on the bimetallic extensor. The thin film was thinned further to a thickness of ~ 20 nm using a Fischione 1040 NanoMill. Next, the bimetallic extensor was loaded in a heated holder with a double-tilt capability and placed in an FEI Titan G2 TEM (300 kV). The bimetallic extensor was composed of two thin layers of materials with a large mismatch between their coefficients of thermal expansion, which could produce large displacements at relatively low operating temperatures ($<50^\circ\text{C}$). During testing, the bimetal extender was controlled to exert a tensile force on the sample by accurately raising the temperature using a temperature controller, which could achieve slow and gentle deformation with a strain rate below $\sim 4 \times 10^{-3}$ s $^{-1}$.

RESULTS AND DISCUSSION

Fig. 1 shows a series of high-resolution TEM (HRTEM) images, which show that GB migration resulted in detwinning during the deformation of the NC AuAg alloy thin film. To track the detwinning mechanism more clearly, G_{i-j} is defined as the GB between grains G_i and G_j , and the GB is marked by a white dotted line. As can be seen, the plastic deformation of a nanotwin grain (G_1) is governed by GB migration, leading to a decrease in the twinned area. Fig. 1a shows a typical HRTEM image captured at the early stage of tensile loading, and it can be seen that both G_1 and G_3 exhibit the $[1\bar{1}0]$ axis lattice of FCC AuAg. As indicated in Fig. 1a, a nanotwin (T_{1-2}) with a thickness of nine atomic layers crosses G_1 and intersects the GBs of G_{1-2} and G_{1-3} ; the TBs are indicated by yellow solid lines. With increasing the strain, as shown in Fig. 1b, G_{1-3} moves toward the left side, decreasing the lengths of T_1 and T_2 from 4.5 to 2.6 nm and from 10.2 to 8.4 nm, respectively. With continuous strain-

ing, as shown in Fig. 1c, G_{1-3} moves toward the left side, and G_{1-2} moves toward the right side, decreasing the lengths of T_1 and T_2 to ~ 2.4 and 5.6 nm, respectively. With further straining, the migration of G_{1-2} and G_{1-3} leads to the annihilation of T_1 and T_2 . In addition, there was no noticeable change in the misorientation angle between G_1 and G_3 during the GB migration process, indicating detwinning was mainly governed by GB migration. This GB migration allowed the detwinning process to occur without the need for partial dislocation nucleation from the CTB-GB intersection or the movement of partial dislocations at the ITB. Thus, this type of detwinning process is important in NC metals because GB migration is more easily triggered in small-sized grains [7,12]. During the deformation process, the lattices in G_1 and G_3 exhibited no obvious changes, indicating that there was no obvious out-of-plane rotation or global tilt of the specimen during deformation.

Fig. 2 displays several typical magnified HRTEM images along the $[1\bar{1}0]$ axis that show the combined layer-by-layer thinning and ITB migration. This detwinning process occurred in two steps: partial dislocation nucleation from the CTB-GB intersection, followed by ITB migration. Fig. 2a shows the image obtained at the initial time (0 s), where the loading direction is labeled by the double white arrow. Fig. 2b–g provide six magnified HRTEM images that correspond to the yellow framed region in Fig. 2a, and the times are 0, 6.5, 15.5, 37, 43.5, and 51 s. In Fig. 2b, there is a full dislocation D_1 interacting with the CTB. By employing Burgers circuits around the dislocation cores in the magnified HRTEM image in Fig. S3, it is found that there is an extra half-plane on $\{111\}$ planes. This configuration represents a typical full dislocation with a Burgers vector of $\mathbf{b} = \frac{1}{2} \langle 011 \rangle$, which was frequently observed in previous studies [33–35]. With increasing strain, as shown in Fig. 2c, two steps on the CTB can be found at different positions, and step 1 is an obstacle created by full dislocation D_1 . As can be seen, on the left side of step 1, there are three atomic layers associated with the twin, and on the right side of step 1, the twin has two atomic layers. This feature should be associated with a partial dislocation emitted from the right side of the GB-TB intersection and move in the left direction during straining. The glide of the partial dislocation diminished an additional twinned layer behind its movement, resulting in one-atomic-layer thinning. For step 2, the structural feature was nearly the same as step 1, indicating that its origin was also a partial dislocation emission and glide from the right side of the GB-TB intersection. With continuous straining, step 1 remained because it was obstructed

Figure 1 *In situ* atomic-scale observation of the detwinning deformation dominated by the GB migration in nanograins of AuAg alloys. The GB is denoted by the white dotted line. The CTBs are denoted by the yellow solid line. The white double arrow indicates the loading direction. The misorientation angle between G_1 and G_3 is denoted by the green solid line. (a–c) With the GB migration, the nanotwin suffered the plastic deformation, and both the length and the thickness were reduced.

Figure 2 *In situ* atomic-scale observations of detwinning dominated by the combined layer-by-layer thinning and ITB migration in nanograins of AuAg alloys. (a) HRTEM image showing the morphology of nanotwins. The misorientation angle between G_1 and G_3 is 9° . The scale bar is 5 nm. (b–g) Series of magnified HRTEM images showing the detwinning process through layer-by-layer thinning in AuAg alloy specimens. The scale bars are 2 nm.

by full dislocation D_1 , whereas step 2 moved in the left direction (Fig. 2d). As the strain was increased, a third partial dislocation emission from the right GB-TB intersection and glide toward the left side occurred. The movement of these three dislocations was prevented by D_1 . Thus, we can observe an ITB consisting of three partial dislocations confined in G_1 . This is direct evidence that a full dislocation can hinder ITB migration and delay the detwinning process, which was only predicted in previous classical modeling [10,25,36]. With further straining, two partial dislocations moved toward G_{1-2} and were annihilated. As a result, a stacking fault (SF) was observed in G_1 . With extensive straining, the third partial dislocation moved toward G_{1-2} and was annihilated again, leading to the defect-free grain. Thus, our *in situ* atomic-scale results show that when a full dislocation creates an obstacle, the detwinning process is more likely to undergo two stages: combined layer-by-layer thinning and ITB migration. In the first stage, partial dislocation nucleation occurs because of the GB-TB intersection, and then an obstacle is created by the full dislocation, resulting in an ITB confined in the grain. In the second stage, the detwinning process results from successive movement of partial dislocations at the ITB toward the GB, which ultimately leads to the grown twin annihilation.

Fig. 3 shows the *in situ* atomic-scale observation of the third type of detwinning process in the NC AuAg alloy specimen, where detwinning occurs through ITB migration that results from the collective motion of partial dislocations at the ITB. In

Fig. 3a, one can observe a nanotwin with a thickness of seven atomic layers and a length of 6.8 nm. The left side of the nanotwin intersects with the GB, and the right side is embedded in the nanograin featuring an ITB that consists of an array of partial dislocations on each $\{111\}$ plane. In order to track the detwinning process, the position of the CTB is indicated by yellow lines, and the ITB is indicated by a dashed yellow line. During loading, the nanotwin suffered plastic deformation, as shown in Fig. 3b, and the thickness changed from seven layers to six layers, resulting from the movement of partial dislocations on the ITB toward the left side of the GB. With further straining, detwinning continued through ITB migration. As shown in Fig. 3b–d, the ITB moved toward the left side, decreasing the length of the nanotwin from 6.8 to 4.8 nm. With further loading, the ITB continued to move to the left, decreasing the length of the nanotwin to ~ 3.0 nm (Fig. 3e). Further straining led to twin annihilation, as shown in Fig. 3f. During straining, the ITB was nearly straight without an obvious irregular shape or a step on the CTB, indicating the detwinning process was governed by the collective motion of partial dislocations in an array on the ITB [19,24].

To estimate the detwinning rate in the alloy specimens, Fig. 4 shows the length of the nanotwins as a function of time. The black square, red circle, and blue triangle represent detwinning via combined layer-by-layer thinning and ITB migration, ITB migration, and GB migration, respectively. As can be seen, the rate of detwinning resulting from GB migration is the slowest.

Figure 3 *In situ* atomic-scale observation of the ITB migration-induced detwinning process in the twin lamellae. (a, b) Nanotwin with seven atomic layers in thickness underwent thinning through straining, and the thickness decreased to six atomic layers. (c–e) During straining, the ITB migrated in the left direction, decreasing the length of the nanotwin from 5.9 to 3.0 nm. (f) With the extensive loading, the ITB moved to the left continuously until it contacted the GB, and the nanotwin disappeared. The detwinning deformation completed, and the crystal returned to the ideal lattice at this moment. The white double arrow in the picture indicates the loading direction, along the crystal direction of the [001]. The scale bars are 2 nm.

Figure 4 Length of the nanotwins as a function of time after the detwinning process occurred in three different deformation modes. Three different symbols represent the mechanisms of detwinning, and dashed lines of different colors represent the fitting curves of the corresponding experimental data.

This is because the detwinning rate is controlled by the GB migration, which should be relatively slow at room temperature.

For the combined layer-by-layer thinning and ITB migration, the rate falls into two regions corresponding to the two stages of detwinning. In the first stage, the detwinning rate is relatively slow because the full dislocation on the CTB hinders the partial dislocation motion. In the second stage, the ITB is generated, and the detwinning rate becomes very fast. This is also consistent with the rate of detwinning resulting from ITB migration (red circle) being faster than the other two detwinning mechanisms. These results indicate that the dislocation-CTB interaction can delay the detwinning process and contribute to strain hardening, whereas ITB migration can easily occur, which can supply large plasticity [1,7,10,11].

According to previous studies, the plastic deformation will switch from dislocation behavior to GB plasticity as the grain size is below ~15 nm [34]. This strong grain size effect on deformation can significantly affect the observed detwinning mechanisms. In the current study, the grain size of the thin film was in the range of ~3–38 nm (with an average grain size of ~15 nm). For twin-structured grains with sizes above ~15 nm, intragrain dislocation activities are more likely to occur [37,38], in which the driving force for dislocation motion is smaller than that for GB plasticity [39,40]. Thus, the detwinning resulting from the collective motion of partial dislocations (Fig. 3) or combined layer-by-layer thinning and ITB migration (Fig. 2) is more likely to occur in relatively large-sized grains. In addition,

the interactions of partial dislocations or full dislocations with TBs may also affect the detwinning process. In small-sized grains, the driving force for GB plasticity is smaller than that for dislocation activity [35,41,42]; thus, GB migration-induced detwinning is more likely to occur (Fig. 1). Although both GB migration and ITB migration that resulted in detwinning are driven by applied stress, their mechanisms are very different. GB migration-induced detwinning is governed by stress-driven GB atom diffusion and GB atom shuffling coupled to GB migration [43]. The ITB consists of an array of partial dislocations [19,24], and ITB migration-induced detwinning involves the motion of these partial dislocation under an applied stress [13,19,44]. In addition to the grain size, the loading mode can also affect the detwinning mechanism [16,30,45,46]. For example, previous MD simulations indicated that under torsion, the detwinning in twin-structured metallic nanowires results from the rotation of the {111} plane [45]. This rotation enables all the atomic layers above twin plane “C” (CABCBC) to be restored to the perfect FCC structure (CABCABC) after being rigidly twisted at an angle of 60°. Under the conventional deformation model, detwinning resulting from dislocation nucleation and slip or TB migration was also observed in the TiAl alloy [46], which is similar to the results shown in Figs 2 and 3 in the current study.

In the current study, *in situ* atomic-scale observations reveal that there are three types of detwinning mechanisms. The first type is the result of GB migration, which occurs more easily in very small nanograins. The second type is more likely to occur in relatively large nanograins, in which dislocations are highly active and thus have a high possibility of interacting with the CTB [7,10,16,47,48]. Once a dislocation is pinned by the CTB, it can hinder ITB migration and delay the detwinning process, leading to strain hardening, which has only been predicted in classical modeling thus far [12,37,49–51]. The third type of detwinning occurs *via* the collective glide of partial dislocations, which results in ITB migration; this is consistent with the detwinning mechanism previously reported for pure metals [13,19,24,48]. The first two types of detwinning have rarely been reported in binary solid-solution NC alloys. They are also different from previous studies on pure metals, in which detwinning results from partial dislocation glide on the plane adjacent to the CTB in a layer-by-layer manner or from partial dislocation motion on the ITB [11,19,52,53]. It should be noted that the solution atoms, secondary phases, precipitated phases, and local chemical composition segregation in alloys can also affect the dislocation activity [46,51,54,55], which affects the detwinning mechanism. Particularly for those alloys containing more than three types of solution atoms, such as medium-entropy or high-entropy alloys, the detwinning mechanism should be different from those observed in the current study and in pure metals. As shown in previous studies, there are noticeable fluctuations in the elemental distribution in medium- or high-entropy alloys [51,54,56–58]. This local chemical ordering can cause the nucleation energy and dislocation motion pathways to vary between regions [59,60], which would affect the detwinning model. Thus, we believe that the detwinning mechanisms in medium- or high-entropy alloys will exhibit new behaviors, which require further study.

CONCLUSIONS

In summary, using our custom-built device, the atomic-scale and time-resolved detwinning process in NC alloys was observed

in situ. We discovered three types of detwinning mechanisms: GB migration, combined layer-by-layer thinning and ITB migration, and collective motion of partial dislocations in an array, resulting in ITB migration. We showed that dislocation-CTB interaction could delay detwinning and contribute to strain hardening, whereas ITB migration could supply large plasticity. Our experimental results provide deeper insight into the fundamental detwinning mechanism in metallic alloys and also provide important clues for achieving high strength and high plasticity of twin-structured alloy NC metals.

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Conflict of interest The authors declare that they have no conflict of interest.

Supplementary information Supporting data are available in the online version of the paper.

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AuAg合金纳米晶去孪晶机制的原位原子尺度研究

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摘要 去孪晶是影响金属力学性能的重要塑性变形机制. 本文对平均晶粒尺寸为15 nm的AuAg合金纳米晶进行了原位拉伸试验, 首次在原子尺度上研究了合金纳米晶的去孪晶机制. 结果表明合金纳米晶中存在三种去孪晶机制, 第一种是晶界发生相向迁移的结果, 这在小尺寸晶粒中更容易发生; 第二种是通过孪晶逐层减薄和非共格孪晶界迁移相结合的方式进行, 在去孪晶过程中, 去孪晶速率会由于位错与共格孪晶界相互作用的影响而减缓, 这仅在以往的经典建模中预测到; 第三种是由非共格孪晶界迁移控制, 这是多个偏位错集体滑移的结果. 前两种类型的去孪晶过程在过去很少被报道, 这表明固溶体合金纳米晶中的去孪晶机制不同于纯金属.