Materialia 15 (2021) 101044

Contents lists available at ScienceDirect

Materialia

journal homepage: www.elsevier.com/locate/mtla

Understanding the interaction of extension twinning and basal-plate precipitates in Mg-9Al using precession electron diffraction

Kelvin Y. Xie[a,1,*], Dexin Zhao[a,1], Brandon Leu[b], Xiaolong Ma[a,c], Quan Jiao[d], Jaafar A. El-Awady[d], Timothy P. Weihs[e], Irene J. Beyerlein[b], M. Arul Kumar[f]

[a] Department of Materials Science and Engineering, Texas A&M University, College Station, TX 77843, United States
[b] Department of Mechanical Engineering, UC Santa Barbara, Santa Barbara, CA 93106, United States
[c] Pacific Northwest National Laboratory, Richland, WA 99354, United States
[d] Department of Mechanical Engineering, Johns Hopkins University, Baltimore, MD 21218, United States
[e] Department of Materials Science and Engineering, Johns Hopkins University, Baltimore, MD 21218, United States
[f] Materials Science and Technology Division, Los Alamos National Laboratory, NM 87545, United States

ARTICLE INFO

Keywords:
Twinning
Precipitate
Precession electron diffraction
Micropillar
Mg-9Al

ABSTRACT

Precession electron diffraction is used to characterize the interaction between $\{10\bar{1}2\}$ tensile twins and basal plate-like precipitates in a post-deformed, precipitate-dispersed Mg-9Al micropillar. We observed a heterogeneous distribution of precipitates in the micropillar sample, which enabled the study of the different stages involved in twin-precipitate interactions. We show that twin nucleation was promoted, taking place on the surface, as well as from the interior of the micropillar. Twin tip propagation and twin growth were hindered by the precipitates. Twin tips either were arrested by precipitates and new twins formed on the other side of the precipitate, or continued to grow around the precipitate without the re-nucleation events. During twin thickening, the precipitates did not significantly alter the shear-dominant twin boundary migration mechanism, as evidenced by the relatively flat twin boundaries around the partially embedded precipitates. However, the twin boundary migration was retarded by the precipitates, especially in regions confined by closely-spaced precipitates.

1. Introduction

Introducing precipitates is an effective method for engineering the mechanical properties of alloys. For metal alloys with high symmetry crystal structures (e.g., face-centered cubic and body-centered cubic), plasticity is primarily mediated by dislocation slip, and dislocation-precipitate interactions give rise to strengthening, with the mechanisms relatively well understood [1]. For metal alloys with the low symmetry hexagonal close-packed (HCP) crystal structure (Mg, Ti, etc.), deformation twinning can also contribute substantially to plasticity [2]. The interaction of deformation twins and precipitates is, therefore, important in understanding the deformation behavior of HCP alloys. However, the interaction phenomena and underlying mechanisms are poorly understood compared to those of dislocation-precipitate interactions.

Recently, twin-precipitate interactions have received considerable attention, both experimentally [3–9] and computationally [10–13], with a specific focus on understanding the interaction between precipitates and $\{10\bar{1}2\}\langle\bar{1}01\bar{1}\rangle$ extension twins in magnesium alloys. Mg-Al alloys, in particular, have been used as one model material systems. Within super-saturated Mg-Al alloys, β-$Mg_{17}Al_{12}$ basal plates precipitates form upon aging [14,15], and the presence of these precipitates can strongly affect twin growth processes and thus, the stress-strain response of the alloy [14,16]. At a microstructural level, these precipitates reduce the twin volume fraction slightly, but substantially increase the twin number density (and consequently reduce twin size) [3,6,8,17–19]. Moreover, transmission electron microscopy (TEM) observations reveal that basal plate precipitates are not twinned or sheared by the extension twins. Instead, they slightly reorient and are elastically bent when the precipitates are partially embedded in the twin. The incompatibility strain between the elastically deformed precipitates and the twin generates back stresses, which limit the twin boundary migration and offers strengthening [3,5–7, 9].

In most experimental studies, the effect of precipitates on twinning is observed using polycrystalline samples. The presence of grain boundaries, as well as grain orientation and grain size variation, can complicate the interpretation of twin-precipitates interaction observations. In addition, different samples have to be prepared at various deformation stages to capture the effect of precipitates on twin nucleation, twin tip propagation, and twin growth, which demands substantial ef-

* Corresponding author.
E-mail address: kelvin.xie@tamu.edu (K.Y. Xie).
[1] These authors contributed equally to this work.

https://doi.org/10.1016/j.mtla.2021.101044
Received 12 February 2021; Accepted 21 February 2021
Available online 25 February 2021
2589-1529/

fort. In this work we characterized the post-deformed microstructure of single-crystal micropillar to exclude the complication of grain boundaries. In this study, the micropillar was oriented along the $\langle 01\bar{1}0\rangle$ direction, from which the tension twinning variants can be easily predicted. Moreover, the selected micropillar has a varying precipitate distribution [14], which allows for the capture of the different stages of twin-precipitate interactions. It is also worth noting we employed precession electron diffraction (PED) to characterize the twin-precipitate interaction in a Mg-9Al micropillar. In addition to offering the crystal orientation information, PED removes dynamical contrast for imaging, resulting in a "cleaner" microstructure compared to conventional TEM, and elucidates the effects of basal-plate precipitates on twin nucleation, twin tip propagation, and twin boundary migration in Mg alloy more effectively.

2. Materials and methods

Mg-9 wt.%Al is used as a model material in this study. The sample was first hot-rolled to develop a strong basal texture, and solution-treated at 450 °C to dissolve any Al-rich precipitates. To grow precipitates in a controlled way, the solution-treated sample was aged in silicone oil at 200 °C for 12 h. Discs from the aged sample were prepared with their surface normal parallel to the transverse direction of the hot-rolled plate. The disc surfaces were then mechanically and electrochemically polished to remove the strained surface layer. An EDAX electron backscattered diffraction (EBSD) system equipped in a Tescan Mira 3 scanning electron microscope was used to map the grain orientations in the discs. Grains with the near-$\langle 01\bar{1}0\rangle$ zone axis were selected to fabricate micropillars approximately 5 μm in diameter and 12 μm in height using an FEI Quanta focused ion beam (FIB). Single crystal micropillar samples allow one to capture twin-precipitate interactions without having any effect from inter-grain interactions and grain boundaries [16,20]. The micro-compression experiment studied here was conducted using a Nanomechanics InSEM nanoindenter with a flat punch at a strain rate of 10^{-3} s^{-1} as reported in an earlier study [16]. Compression close to the $\langle 01\bar{1}0\rangle$ zone axis induces extension twins [16,21]. The test was stopped at ~4% engineering strain. The deformed micropillar was prepared into a TEM foil using the FIB lift-out method, and the post-deformed pillar microstructure was revealed by regular transmission electron microscopy (TEM, FEI Tecnai G2 F20) and PED (NanoMEGAS). PED maps were acquired with a 0.3° precession angle and a 3 nm step size.

3. Results and discussion

Fig. 1 shows the overall microstructure of the deformed micropillar revealed by conventional bright-field TEM and PED. The micropillar was compressed close to the $[01\bar{1}0]$ zone axis and profuse $\{10\bar{1}2\}\langle\bar{1}01\bar{1}\rangle$ extension twins were activated. The TEM specimen was tilted to the $[2\bar{1}\bar{1}0]$ zone axis, where $(01\bar{1}2)$ and $(0\bar{1}12)$ twin planes are parallel to the electron beam direction. Fig. 1a shows the contrasts from the conventional bright-field TEM is difficult to interpret. In contrast, PED micrographs (Fig. 1b–d) reveal the microstructure with higher clarity. Fig. 1b shows the index map (analogous to band-contrast maps in the EBSD scans) of the deformed pillar. The contrast is determined by how well the acquired diffraction patterns match the calculated ones. The pixels from the interior of the grains usually match well and appear bright, whereas the pixels from the boundaries and precipitates are dark due to overlapping diffraction patterns. Fig. 1c shows the cross-correlation map, in which the diffraction pattern of each pixel is compared to its first-neighbor pixels. No difference will lead to bright pixels, whereas large differences result in dark pixels. Thus, the cross-correlation map highlights interfaces. Fig. 1d shows the in-plane, y-axis inverse pole figure (IPF) superimposed with the index map. In the IPF, the matrix is blue, while twins are red (colors refer to the inverse pole figure key), with their corresponding crystal orientations illustrated.

Based on the PED results, three salient observations can be made. First, multiple twins of two different variants were activated in the micropillar, which are labeled as T_{var1} and T_{var2} and are illustrated in Fig. 1d. The activation of multiple twins and variants lies in stark contrast to the largely single-twin variant observed in the precipitate-free micropillars with the same crystal orientation and loading condition [16,22]. Such differences will be elucidated shortly in the discussion on the effect of basal-plate precipitates on twin nucleation. Second, a number of twin tips can be seen in the upper part of the pillar, but not in the lower part of the pillar. Third, the twins are thinner in the upper part of the pillar compared to the lower part. Together, the second and third observations suggest that the twin tip propagation and growth is less favorable in the upper part of the pillar compared to the lower part, which is counter-intuitive. Since all the FIB-fabricated micropillars through annual milling exhibit small tapering angles (~1–3°), such geometries generally cause non-uniform deformation, in which the upper part of the micropillar deforms more than the lower part [23]. Based on this, more twinning (easy propagation and growth) in the upper part of the pillar than in the lower part would be expected. Interestingly, we observed the opposite trend. We suspect that the disparity can be correlated with the non-uniform distribution of precipitates. The area fraction of precipitates in the top boxed region in Fig. 1b is 8.4%, while the area fraction in the bottom boxed region is only 4.8%. We argue that the much higher density of precipitates in the upper part of the pillar hinders the twin propagation and growth compared to the lower part, resulting in the heterogeneous twin network structure.

The presence of more basal-plate precipitates allows for more twin nucleation, which is inferred by the observation of a large number of twin lamellae seen in Fig. 1. In precipitate-free micropillars, a single twin would form and grow because the twin nucleation stress is higher than the stress required for twin tip propagation and twin boundary migration [16,22]. In precipitate-dispersed micropillars, precipitates can elevate the stress levels for twin tip propagation and twin boundary migration, limiting twin growth and thus rendering twin nucleation more favorable [3–9]. Here, the use of PED provides a holistic view of the possible locations for twin nucleation. Case in point, while the free surface of the micropillar seems to be the most favorable nucleation site (see Fig. 1), we find that with closer inspection PED reveals that twins can also nucleate within the micropillar interior (see the dashed-boxed region in Fig. 1d). This observation was only made in the precipitate-bearing micropillars, not in the precipitate-free ones [16]. This suggests that the interaction of twin boundaries and precipitates can lead to the formation of twins of a different variant. It is suspected that twin-precipitate interactions create local stress concentrations, which serve as nucleation sites for alternative twin variants.

Once nucleated, the extension twin tip in Mg generally propagates across the volume to form a thin lenticular shape twin [24]. The presence of basal-plate precipitates was observed to hinder the twin tip propagation. A number of twin tips can be seen in the upper part of the micropillar (Fig. 1). Twin tips are usually difficult to capture in deformed precipitate-free micropillars because they can easily shear across the entire volume and terminate/disappear at the free surface. The fact that multiple twin tips were observed in the precipitate-bearing micropillar offers strong evidence that the basal-plate precipitates hindered the twin tip propagation.

When twins interact with the basal precipitates, two types of twinned-microstructures can result. In the first case, when the twin tip is completely blocked by the precipitate, a stress field is generated on the other side of the precipitate, triggering the nucleation of a new twin of the same variant. This observed re-nucleation of a new twin supports the prediction from Leu et al. using the Fast Fourier Transform based crystal plasticity calculations [25]. The authors pointed out that, when the twin tip is interacting with a basal plate precipitate, a stress concentration on the other side of the precipitate develops, potentially helping to nucleate a new twin. In addition, the new twin can nucleate on the other side

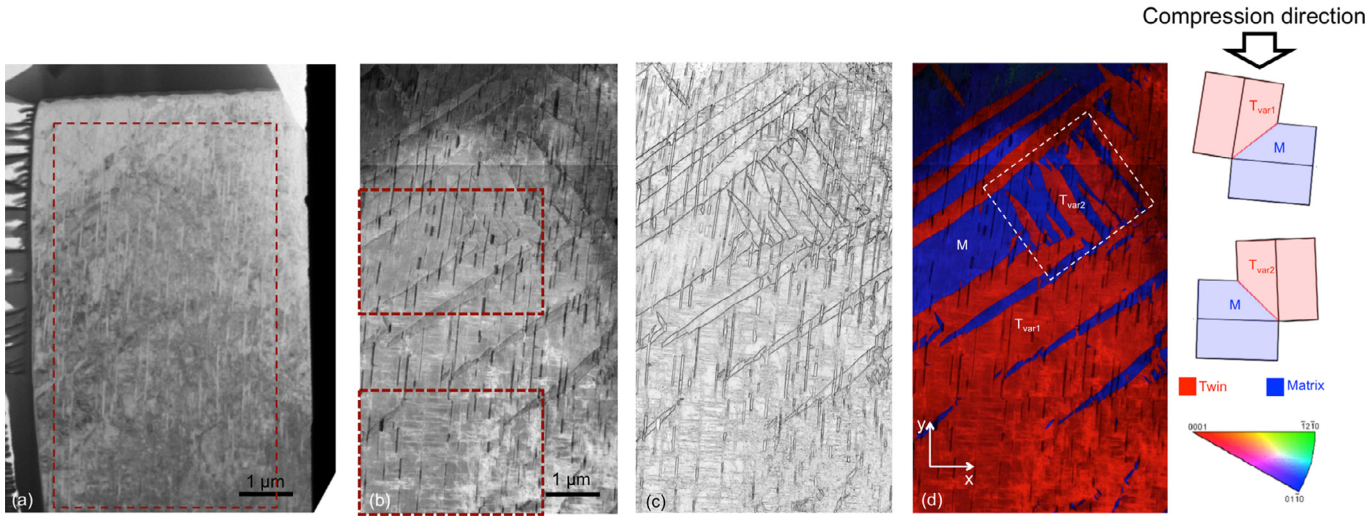

Fig. 1. (a) Bright-field TEM micrograph of the compressed micropillar. The box encloses the region mapped using PED. (b) Index map, (c) cross-correlation coefficient map, and (d) in-plane (along the *y*-axis) inverse pole figure of the boxed region in (a). The schematics illustrate the crystal orientations of the matrix and twins in the micropillar (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.).

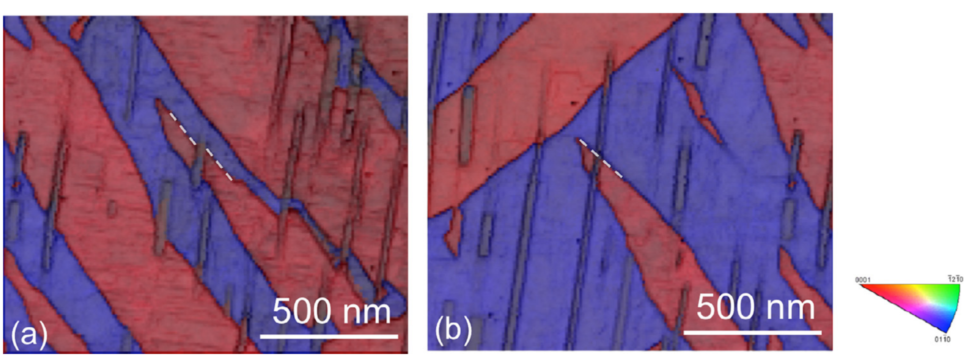

Fig. 2. Superimposed index map, cross-correlation coefficient map, and in-plane inverse pole figure revealing the microstructure as a result of twin tip propagation interacting with basal-plate precipitates. (a) A discontinuity of twin growth was noted as highlighted by the white dashed lines. (b) No apparent discontinuity of twin growth was observed as indicated by the white dashed line.

of the precipitate with some offset (highlighted by the dashed lines in Fig. 2a), causing a staggered twinning structure. In the second case, the twin tip propagation path seems to be undisturbed by the precipitate (highlighted by the dashed line in Fig. 2b). This may be caused by the bowing of the twin boundary around the precipitate [6]. We speculate this process becomes more favorable when the twin tip is only partially blocked by a precipitate.

Following twin tip propagation, next we analyze twin thickening via twin boundary migration. Basal-plate precipitates are known to impede twin boundary migration [3,5–7,9]. Here, we focus on the microstructural details and infer how twin boundaries may have interacted with precipitates dynamically. The early, intermediate, and late stages of twin thickening are illustrated in Fig. 3a, 3b, and 3c, respectively. In all stages, most of the twin boundaries lying between precipitates are not bowed but are relatively flat, with no apparent deviation from the ideal twin plane (examples indicated by the white arrows in Fig. 3). This microstructure of the relatively flat twin boundaries adjacent to the partially engulfed precipitates aligns well with a molecular dynamics prediction [10].

At first sight, such an observation may be surprising, considering dislocation bowing (e.g., Orowan hardening) and regular grain boundary arcing (e.g., Zener pinning) by the precipitates [1,26]. The bowing of dislocations (and grain boundaries) is due to the line tension (and surface tension) balancing the pinning force from precipitates. But one cannot draw the same arguments for twin boundary migration, because the underlying process for twin thickening is different from that for grain boundary migration. Generally, twin boundary migration in Mg can be achieved by shear (e.g., nucleation and glide of twin dislocations) or atomic shuffling [27,28]. The former usually results in a relatively flat twin boundary that is parallel to the $\{10\bar{1}2\}$ twin habit plane, whereas the latter leads to a rumpled twin boundary deviating from the twin habit plane [29]. As twin boundaries remain relatively flat around precipitates in many places, it suggests twin boundary migration is mediated mainly by twinning dislocations when interacting with basal-plate precipitates in this study. Even at a stage where part of the twin boundary has a large deviation by a basal-plate precipitate, mobile defects (e.g., twin dislocations and basal/prismatic or prismatic/basal facets) could readily multiply to restore the twin boundary back to its habit plane [30].

It is worth mentioning much slower twin migration and large deviations from the twin habit planes were noticed in some local areas (highlighted by the dashed ovals in Fig. 3). They are usually associated with closely spaced precipitate plates (spacing generally < 100 nm) and the underlying reasons are not known. We offer two possible explanations. First, both dislocation and twinning exhibit strong size effects [31]. Higher stresses are required to glide twinning dislocations to advance twin boundaries. Second, the small confined volume may trigger other twin boundary migration mechanisms (e.g., atomic shuffling) rather than twinning dislocations, leading to a more sluggish twin boundary movement [29]. We speculate that if the average precipitate spacing were successfully engineered to sub-100 nm in the Mg alloy, the twin boundary migration mechanism could be dramatically altered, leading to very different mechanical responses and post-deformation microstructure.

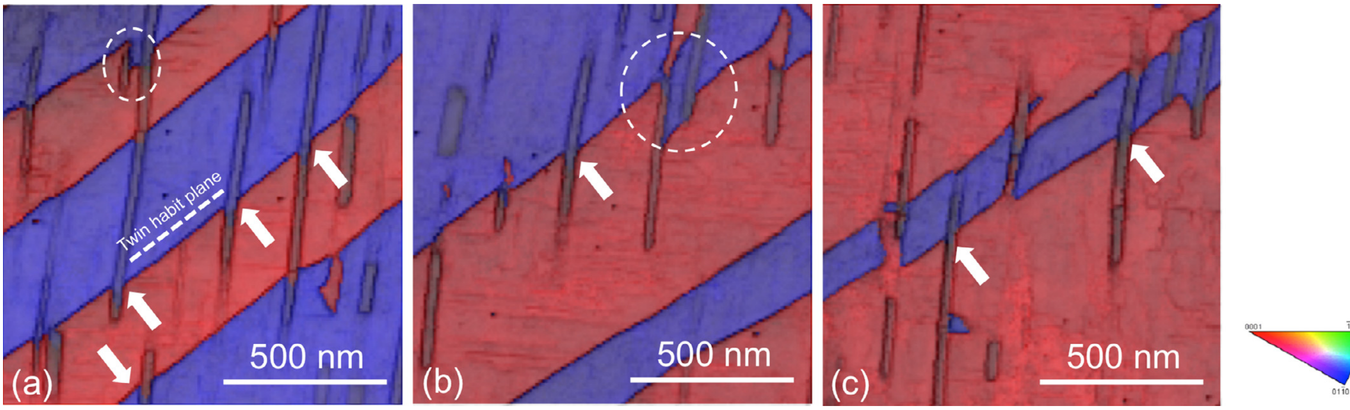

Fig. 3. Superimposed index map, cross-correlation coefficient map, and in-plane inverse pole figure revealing the microstructure as a result of twin boundary migration interacting with basal-plate precipitates at (a) early-stage, (b) intermediate-stage, and (c) late-stage twin growth. White arrows indicate where twin boundaries remain relative flat when interacting with basal-plate precipitates. Dashed ovals indicate where twin boundaries migrate much slower than the adjacent regions.

4. Summary and conclusions

In summary, we have employed PED to characterize the microstructure of a post-deformed, precipitate-dispersed Mg-9Al micropillar to understand the interactions of extension twinning and basal-plate precipitates. Multiple twins of different variants were observed in the volume, four significant conclusions were drawn. First, the observation of twin formation within the micropillar's interior and not just from the surface suggests that basal-plate precipitates promote twin nucleation. Second, changes in twin geometries indicated that twin tips were arrested basal-plate precipitates but then nucleated a new twin on the other side of the precipitate or circumvented the precipitate. Third, observations of relatively flat twin boundaries around the partially embedded precipitates suggest a shear-dominant mechanism for twin boundary migration. Four, dramatically hindered twin boundary migration was observed in regions confined by closely spaced precipitate plates.

Declaration of Competing Interest

We wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

Acknowledgments

K.Y.X. would like to acknowledge the support from the Los Alamos National Laboratory Fellowship Program initiated at Texas A&M University and Texas A&M Experimental Station. M.A.K. acknowledges the financial support from the U.S. Department of Energy, Office of Basic Energy Sciences (OBES) FWP-06SCPE401. B.L. was supported by the Department of Defense (DOD) through the National Defense Science & Engineering Graduate Fellowship (NDSEG) Program. I.J.B. acknowledges financial support from the National Science Foundation Designing Materials to Revolutionize and Engineer our Future (DMREF) program (NSF CMMI-1729887). Q.J and J.A.E. acknowledge support by the National Science Foundation through award #DMR-1609533. T.P.W. acknowledges the support from the Army Research Laboratory under the Cooperative Agreement Number W911NF-12-2-0022. The views and conclusions contained in this document are those of the authors and should not be interpreted as representing the official policies, either expressed or implied, of the Army Research Laboratory or the U.S. Government. The U.S. Government is authorized to reproduce and distribute reprints for Government purposes notwithstanding any copyright notation herein.

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