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On the intrusion-like co-zone twin-twin structure: An *in situ* observation

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ABSTRACT

An *in situ* optical microscopy combined with *ex situ* electron backscatter diffraction testing was applied to a pristine single-crystal magnesium specimen under monotonic tension along the *c*-axis. An intrusion-like co-zone twin-twin structure is observed for the first time at the micron scale. *In situ* observation reveals that the intrusion-like twin-twin structure consists of multiple twin-twin boundaries (TTBs) and incoherent twin boundaries (I-CTBs) following energetically favorable formation sequences. The initial interaction results in the impinging TTB_I and the acute-angle TTB_A. In the local junction region on the obtuse angle side, the impinging twinning dislocations (TDs) further deposit near TTB_I due to the preferred local twinning shear stress, leading to the incoherent curve of the impinging twin boundary adjacent to TTB_I. Shortly after, the barrier twin boundary on the obtuse angle side migrates and encompasses the incoherent impinging twin boundary. The combination of sequential TTB_I, TTB_A, and I-CTBs formed locally on the obtuse angle side shapes the final configuration of the intrusion-like twin-twin structure at the micron scale.

1. Introduction

Magnesium (Mg) and its alloys are highly desired materials for engineering applications due to their low density. Extensive research on the deformation mechanism of Mg has been focused on {10–12} tension twinning [1–4] due to its low critical resolved shear stress (CRSS) and accommodation of *c*-axis extension. Of particular interest is the interaction of different tension twin variants [5–11]. The interaction of two variants results in three possible types: co-zone type I, non-cozone type II(a), and non-cozone type II(b) [6,7]. The co-zone type I interaction is of particular interest given how commonly it is observed under simple loading paths at low stress levels.

There are three possible twin-twin boundary (TTB) formations in type I interactions. The acute-angle twin-twin boundary (TTB_A) is energetically favorable by the zipping of the two twins' twinning dislocations (TDs) at the junction [6,7]. The impinging twin-twin boundary (TTB_I) can form by deposition of the impinging TDs on the barrier twin boundary. The obtuse-angle twin-twin boundary (TTB_O) was suggested to develop by dissociation of TDs from one twin onto the other [7]. However, formation of TTB_O is considered energetically unfavorable [6]. When both TTB_A and TTB_O form, a shallow penetrating structure develops, as revealed by experiment [7] and atomic simulations [11]. In the case where only one TTB is formed, a transmission electron microscope (TEM) observation reveals a nano-scale twin-twin structure showing a knife-like deep penetration [10].

Almost all the experimentally observed intrusion-like twin-twin structures were characterized by post-mortem methods. There is a lack of direct observation of the corresponding formation process. In particular, the formation of a deep intrusion-like structure is the least understood [11]. There is no experimental evidence at the micron scale. The current study provides the first-time experimental evidence of intrusion-like twin-twin structure at the micron scale using *in situ* optical microscopy (OM) and *ex situ* electron backscatter diffraction (EBSD) characterization on a pristine single-crystal magnesium under tension. Our experiments allow for the mechanistic understanding of the formation process for the intrusion-like twin-twin structure in Mg.

2. Experimental

A small dog-bone shaped plate specimen with gage section dimensions of $7 \times 2.85 \times 1.76$ mm^3 was fabricated from a single-crystal magnesium rod by acid saw using 35% diluted nitric acid in water. The specimen was machined such that the loading direction is approximately parallel to the *c*-axis and the observation plane is approximately parallel to the $\left(\bar{1}2\bar{1}0\right)$ *a*-axis (Fig. 1).

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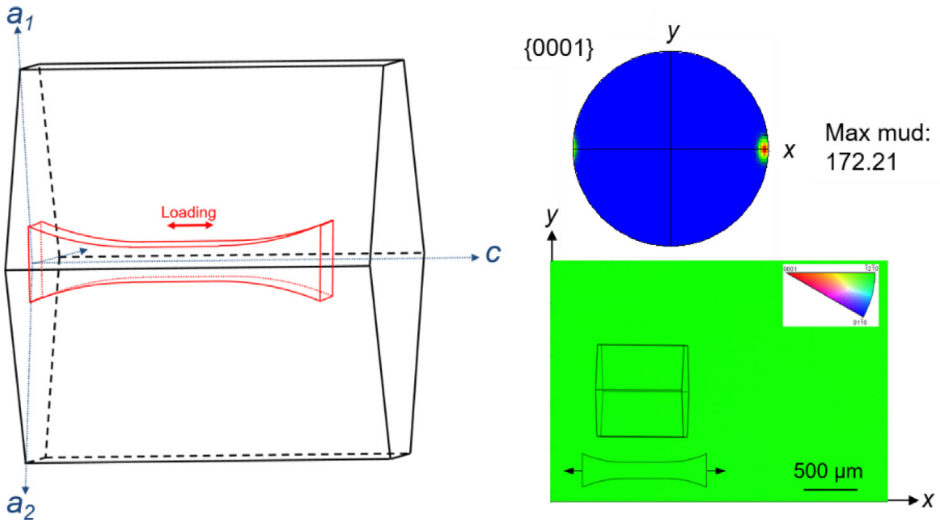

Fig. 1. Specimen orientation for a single-crystal magnesium dog-bone shaped specimen.

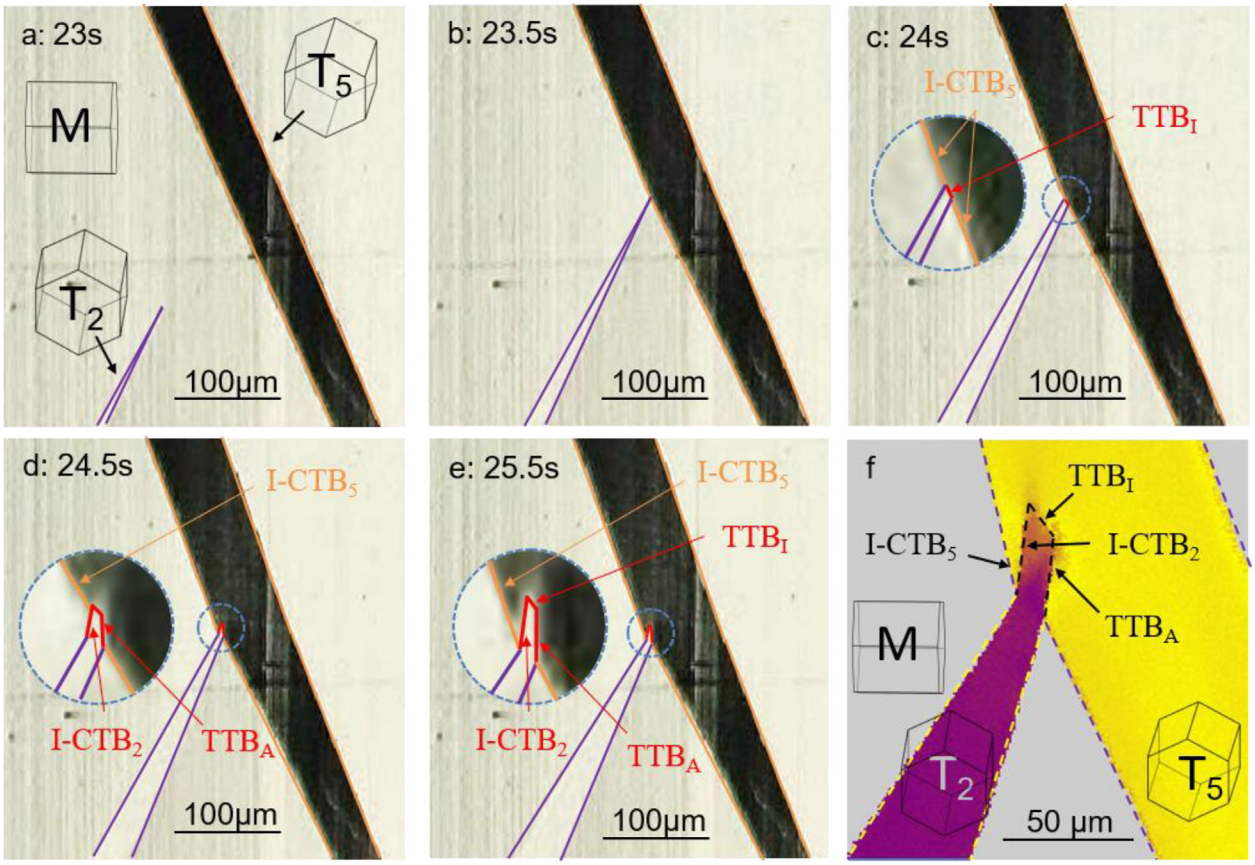

Fig. 2. Optical micrographs highlighting the $T_2 \leftrightarrow T_5$ twin-twin interaction that results in an intrusion-like structure.

The actual orientation has a 4° misorientation between the *c*-axis and the loading axis. The observation surface was prepared by silicon carbide grinding down to 1200 grit, followed by 1-μm diamond suspension polishing, and 50-nm alumina final polishing. Surface quality was confirmed with 10 s etching by 3% nital. Deformation twinning was absent in the undeformed state.

Mechanical tests were conducted using an *in situ* loading stage the same as described in [12], where the loading was performed horizontally relative to the optical axis. The equipment used in this procedure is detailed in the Supplementary Information. Tension was performed to a pre-defined displacement at room temperature under displacement control at 0.1 mm/min and then unloaded to 0 N at the same speed. The specimen surface in the middle of the gage section was tracked *in situ* by optical microscope, covering an area of ~3. 1 × 2.3 mm^2 with a pixel resolution of 2 μm. A framerate

of 15 frames/second was used to capture deformation. After the test, EBSD scans were performed on the observation area with 4 μm step size.

3. Results and discussion

The development of a twin-twin structure is presented in Fig. 2 where one twin appears to penetrate its co-zone pair. Additionally, a schematic delineating the formation process is provided in Fig. 3. Twin variants will be identified with "T_i" where the subscript "i" indicates the variant following the same convention in [6]. $T_i \leftrightarrow T_{i\pm3}$ represents a co-zone interaction. The key interaction is observed 23 s after the test starts, where T_2 is observed propagating toward T_5 (Fig. 2a and Fig. 3a).

Initial contact is observed at 23.5 s (Fig. 2b), where the needle-like tip of T_2 meets the T_5 boundary. T_5 boundary is noted to deviate from the coherent twin boundary (CTB) as it reaches out to contact T_2 (Fig. 3b). This "reach out" phenomenon was explained by local variation of the twin resolved shear stress (TRSS) during twin-twin interaction [9]. At a critical distance between the two twins, the TRSS for T_2 becomes negative, and T_2 will stop propagating. Alternatively, the TRSS for T_5 will increase sufficiently large near the tip of T_2, driving the local thickening of T_5 by boundary migration. Therefore, instead of T_2 growing toward T_5, it is T_5 that locally thickens towards T_2 (Fig. 3b). Because of the local deviation from the CTB, TTB_I is also affected as seen in Fig. 2c where TTB_I does not align with CTB_5, but with the T_5 local incoherent twin boundary ($I\text{-}CTB_5$) (Fig. 3c).

With further growth of T_2 and T_5, TTB_A is formed as an energetically favorable zipping reaction of the TDs from T_2 and T_5 (Fig. 2d and Fig. 3d). The trace of TTB_A approximately bisects the coherent twin traces of T_2 and T_5, which is consistent with previous observations [6,7], where the acute TTB_A plane bonds the two twins' prismatic planes (termed as PP boundary). On the obtuse angle side, instead of forming the energetically unfavorable TTB_O that bonds the basal planes of the two twins, T_2 thickens locally and transitions TB_2 into $I\text{-}CTB_2$ adjacent to the TTB_I. As inferred by large-scale molecular dynamics simulations (Fig. 7c in [9]), the local distribution of TRSS for T_2 near the junction on the obtuse angle side is positive. This means that the local nucleation and glide of T_2 TD are favorable, leading to the deviation of the TB_2 on the obtuse side as evidenced in Fig. 2d and illustrated in Fig. 3d.

Shortly after, as indicated in Fig. 2e and 3e, T5 boundary migrates on the obtuse angle side and encompasses the prior deviated $I\text{-}CTB_2$, shaping the intrusion-like twin-twin structure. With further loading, TTB_A continues to grow while $I\text{-}CTB_5$ continues to migrate along the $I\text{-}CTB_2$ on the obtuse angle side, making a deeper intrusion-like twin-twin structure.

After unloading, the EBSD inverse pole figure map is characterized for the intrusion-like twin-twin structure (Fig. 2f). Given the small misorientation of the two variants, the color scale is modified where the matrix is gray whereas T_2 and T_5 are violet and yellow, respectively. The color gradient indicates local variation of crystal orientation due to the high local stress concentration. As shown in Fig. 2f, TTB_A has grown significantly, while $I\text{-}CTB_2$ and $I\text{-}CTB_5$ are more clearly observed on the obtuse angle side, forming a deep intrusion-like twin-twin structure.

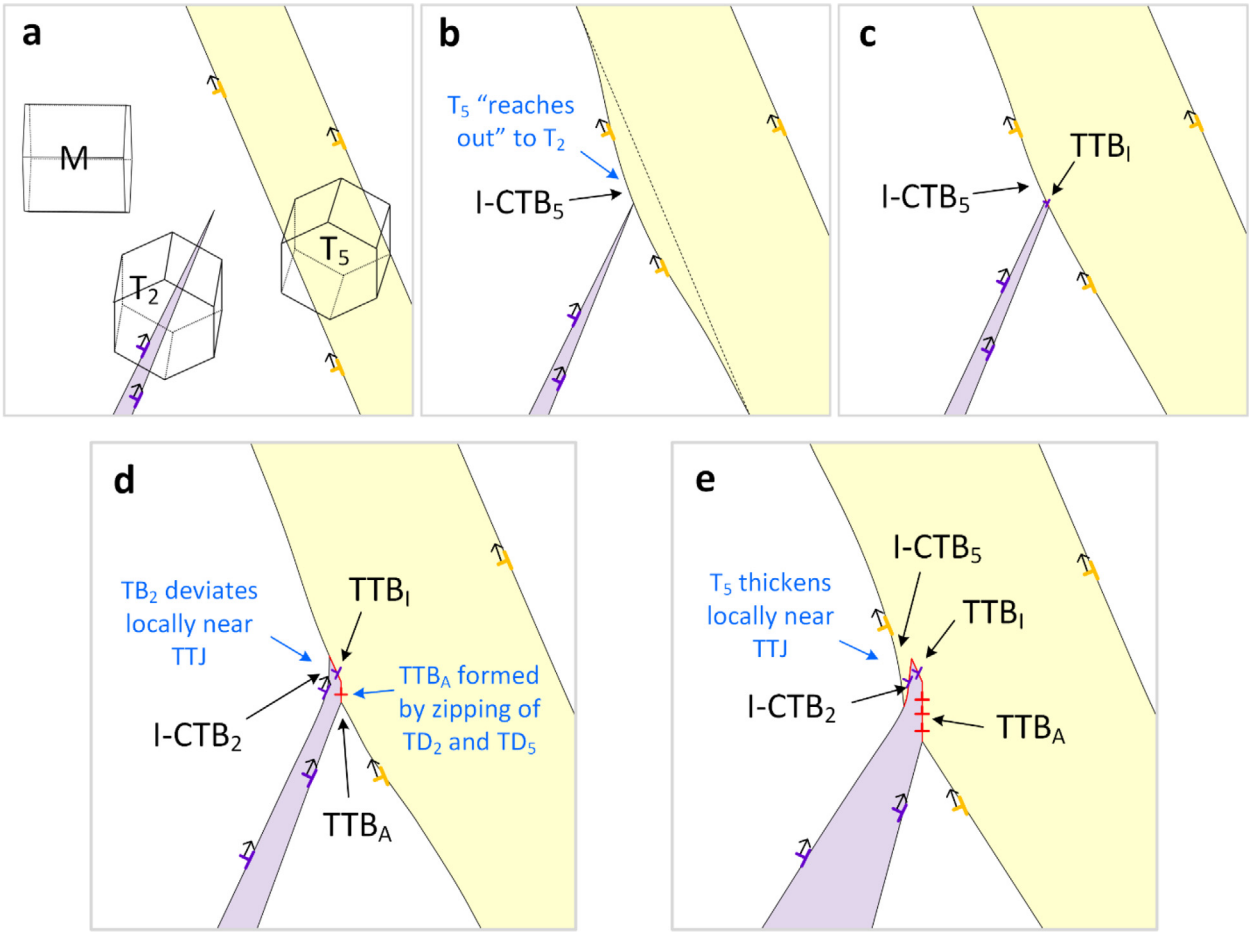

Fig. 3. Schematic showing the formation process for the intrusion-like co-zone twin-twin structure.

This intrusion-like twin-twin structure observed in the current study differs from the cases reported in literature. In Fig. 2d from [7], a shallow penetrating twin-twin structure with both TTB_A and TTB_O are formed. In the current study, only TTB_A is formed. On the obtuse side, I-CTB_2 rather than TTB_O is formed. This difference in geometry results in a narrower and deeper intrusion. At the nano-scale, a similar structure was reported where one twin appears to penetrate into its co-zone pair like a dagger (Fig. 2 in [10]). However, the intruded TTBs are also proposed to be BB and PP boundaries which are reconstructed from a single TTB formed earlier. Lastly, it is worth pointing out that previously reported intrusion structures were all characterized by post-mortem methods, where the mechanism may be controversial. Our current work provides the first-time characterization of the intrusion-like twin-twin structure at the micron scale, where the sequential formation process is illuminated by the *in situ* observation.

4. Conclusions

In summary, an intrusion-like co-zone twin-twin structure at the micron scale was characterized by *in situ* OM and *ex situ* EBSD in single-crystal magnesium subjected to monotonic tension along its *c*-axis for the first time. The intrusion-like co-zone twin-twin structure is composed of the sequentially-formed TTB_I, TTB_A, and I-CTBs located on the obtuse angle side. The formation processes of the multiple TTBs and I-CTBs in the intrusion-like structure are all energetically favorable.

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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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matlet.2020.129140.

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