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Co-zone $\{10\bar{1}2\}$ Twin Interaction in Magnesium Single Crystal

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Using *in situ* optical microscopy, electron backscatter diffraction analysis, and atomistic simulations, we studied co-zone $\{10\bar{1}2\}$ twin interaction in magnesium single crystal under compression–tension along the $[10\bar{1}0]$ direction. Two co-zone twins are activated and interact with each other, resulting in two types of tilt boundaries that have habit planes (0001) and $(10\bar{1}0)$ and prevent twin-across-twin transmission but facilitate the propagation of a basal slip band. Upon strain reversal, the unfavorable dissociation of dislocations in the formed tilt boundaries hinder de-twinning.

Keywords: Twinning, De-twinning, Twin–Twin Junctions, Compression–Tension, Magnesium

Magnesium (Mg) and its alloys are the lightest structural metals [1] and understanding their plastic deformation behavior provides insight into fabricating structural components and predicting their performance under mechanical loading. The stress–strain response, texture evolution, and microstructure evolution under cyclic loading and non-proportional loading paths have been extensively studied using experiment and modeling.[2–6] Twinning and de-twinning are recognized to play crucial roles in developing the asymmetric stress–strain hysteresis loop, texture evolution, and initiation of fracture.[3, 5,7] In properly oriented single crystals, multiple twins can be activated and interact with each other. As a consequence, their interaction and reactions develop a characteristic locked ‘quilted looking’ twinning structure (Figure 1(a)) which affects subsequent twinning and de-twinning, causes twinning-induced hardening, and potentially results in crack initiation.[3–7] Jiang et al. [8] and Yang and Zhang [9] reported that different twin variants and twin–twin intersections are associated with the deformation stage with a higher strain hardening rate in AZ31 Mg alloy which is subjected to compression along the extrusion direction. Ma et al. [10] and Oppedal et al. [11] found that multivariants are activated to accommodate large straining and the subsequent twin–twin interaction contributes to a significant strain hardening. El Kadiri et al. [12] compared twin growth behavior in two grains that contain different numbers of

twin variants. They found that a higher twinning nucleation rate is exhibited in a grain containing a twin–twin interaction with two variants while the twinning propagation rate is faster in the grain having only a single variant. Roberts and Partridge [13] experimentally characterized the accommodation during a twin–twin interaction in Mg. The twin–twin boundary between two $\{10\bar{1}2\}$ twin variants was identified as a common interface bisecting two twinning planes. They pointed out that a local rotation of the twin lattice in the vicinity of the contact site could result from the stress relaxation process across the contact interface accompanying the formation of the common interfaces between two twins, not from twin crossing or transmission that has been proposed by Cahn.[14] Nevertheless, the other critical questions regarding a twin–twin interaction still remain unanswered, including (1) does one twin transmit across the other? (2) if yes, what is the mechanism involved? (3) if not, why and what kind of reactions take place? (4) how does the twin–twin reaction influence the further twinning and later de-twinning?

There are two typical twin–twin interactions, two twin variants sharing the same zone axis (Type I, co-zone twins) and two twin variants with different zone axes (Type II). Under compression along the $[10\bar{1}0]$ direction, two twin variants that share the same zone axis have the maximum Schmid factor, offering the best situation to study the Type I co-zone twin interaction. In this Letter,

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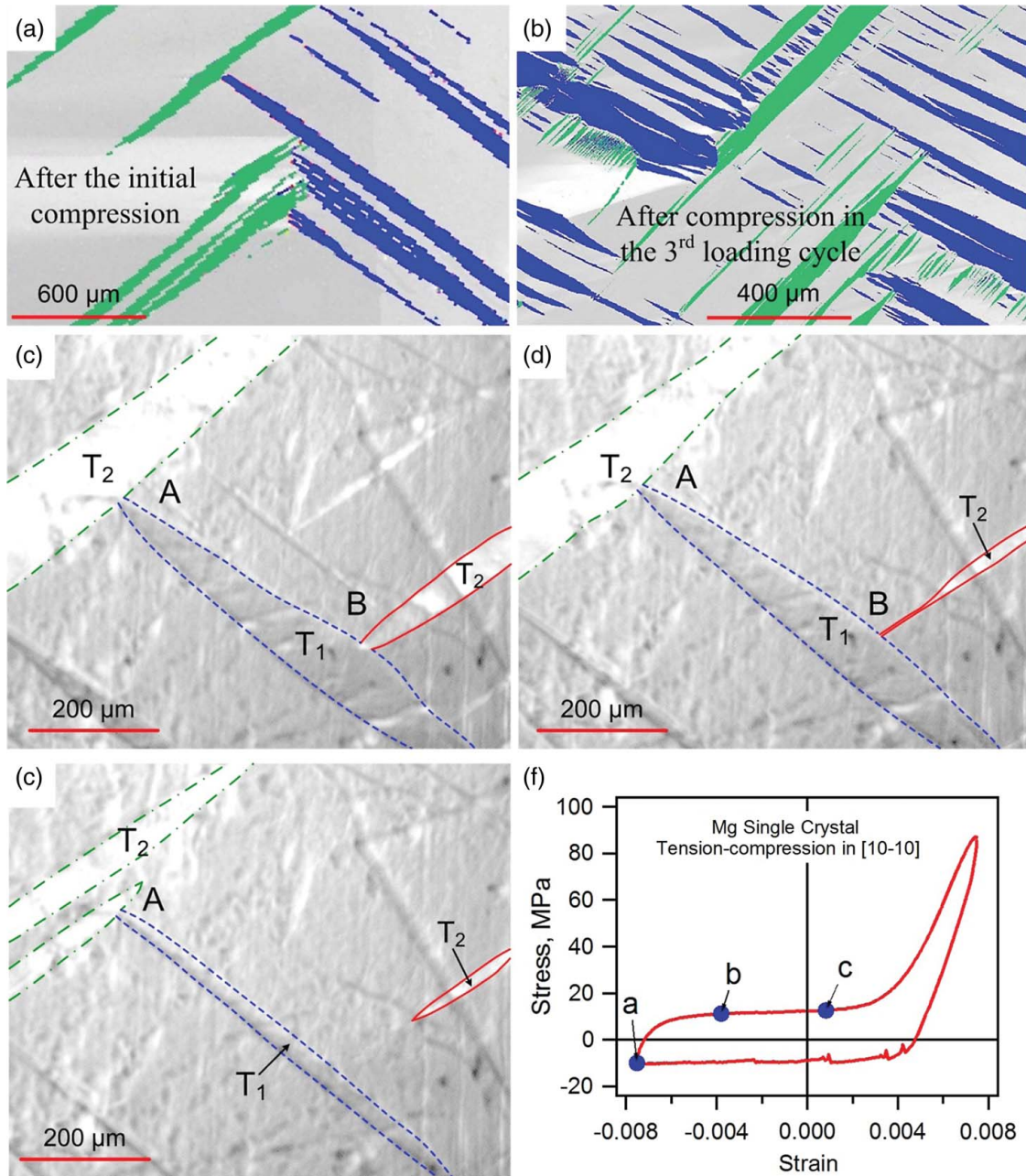


Figure 1. (a) and (b) EBSD analysis showing ‘quilted looking’ twin-twin structures at a compression strain of -0.75% with respect to after the initial compression and after the third loading cycle. (c), (d) and (e) *In situ* optical micrographs showing different stages (a, b, and c) of de-twinning and their corresponding stress-strain states (f).

we performed compression-tension tests in Mg single crystal along the $[10\bar{1}0]$ direction. Using *in situ* optical microscopy and *ex situ* electron backscatter diffraction (EBSD) analysis, we observed the co-zone twin interactions and characterized the twin-twin boundary. The boundary resulting from a twin-twin intersection is analyzed using dislocation theory and atomistic simulations. The strength of the boundaries and their influence on subsequent twinning, slip, and de-twinning are characterized experimentally and discussed in terms of dislocation reactions.

An Mg single crystal was grown using the Bridgman method. A dog-bone specimen of gage length 5.0 mm and a square cross-section of 3.0 mm $\times$ 3.0 mm was fabricated using electric discharge machining. EBSD analysis on the specimen surface showed that the loading axis (z -axis) is close to the $[10\bar{1}0]$ direction. The observation surface is the y - z plane ($\bar{1}2\bar{1}3$), being tilted approximately 43° from the plane ($\bar{1}2\bar{1}0$). Under compression along the $[10\bar{1}0]$ direction, twin variants $(10\bar{1}2)$ and $(\bar{1}012)$ that share the same zone axis have the maximum Schmid factor of 0.499 and are expected to be

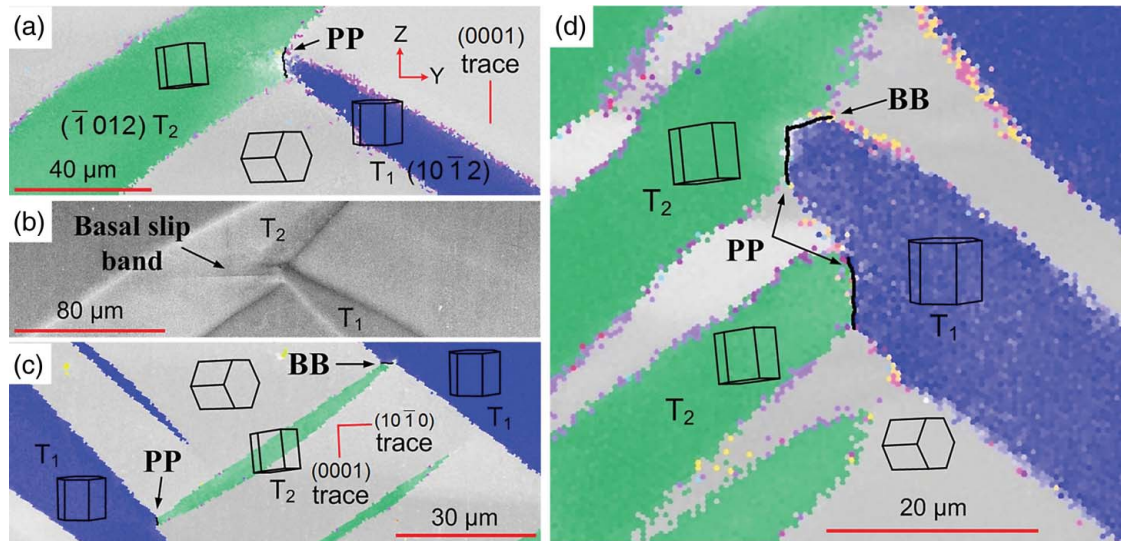


Figure 2. (a), (c) and (d) High-magnification crystal orientation maps illustrate two types of low-angle twin–twin boundaries, PP (Prism–Prism) and BB (Basal–Basal), between $T_1 = (10\bar{1}2)$ and $T_2 = (\bar{1}012)$ twins. (c) BSE micrograph of the same region in (b) shows a basal slip band. Gray region indicates the parent domain, and blue and green regions distinguish variants T_1 and T_2 , respectively. The wire-frame unit cell depicted in the figures indicates the crystal orientation.

more active than the other four twin variants, which have a Schmid factor of 0.125. In the following description twin variants $(10\bar{1}2)$ and $(\bar{1}012)$ are referred to as T_1 and T_2 , respectively. Before mechanical testing, the observation surface of the specimen was chemically polished using a solution of one part of nitric acid, two parts of hydrochloric acid, and seven parts of ethanol.[15] The details of the mechanical testing setup can be found in [5]. The specimen was initially compressed to a strain of -0.75% and slightly unloaded to a strain of -0.725% , where we characterized the specimen surface by scanning electron microscopy and EBSD with a scan resolution of $0.5\ \mu\text{m}$. The specimen was subsequently loaded under fully reversed strain-controlled tension–compression at a strain amplitude of 0.75% . During the cyclic loading, the morphological change of deformation twins was traced using *in situ* long-distance optical microscopy and the orientation of the twins was characterized based on EBSD analysis.

Three microstructural features (Figure 2) are frequently observed in association with twin–twin intersections: (1) two low-angle tilt boundaries with a misorientation of approximately 6.6° ; (2) no twin transmission beyond the tilt boundaries; (3) a basal slip band originating at a tilt boundary. Figure 2(a) shows a low-angle twin–twin boundary that is referred to as PP and indicated by the black line when T_1 encounters T_2 . This twin–twin boundary plane is nearly parallel to the traces of the prismatic plane $(10\bar{1}0)$ in the two twins, and is referred to here as PP. The misorientation angle across the boundary is approximately 6.6° according to EBSD analysis. The trace of the twin–twin boundary nearly coincides with the trace of the basal plane (0001) in the matrix. Crystallographic

analysis according to the twinning-induced reorientation of the two twin variants shows that the PP boundary forms when the angle between the two primary twinning planes is acute (Figure 3(b)). Figure 2(b) shows a backscattered secondary electron (BSE) micrograph that is taken at the same region in Figure 2(a). A slip band connected with the twin–twin boundary was observed inside the T_2 twin. The trace of the slip band aligns with the trace of the basal plane (0001) in the T_2 twin, indicating that this slip band is of a basal type. Figure 2(c) reveals two twin–twin boundaries. One is the same as PP. The misorientation angle across the other boundary is approximately 6.6° according to EBSD analysis. The boundary plane is closely parallel to the basal plane (0001) of both twins and is referred to here as BB. Crystallographic analysis shows that the BB boundary forms when the angle between the two primary twinning planes is obtuse (Figure 3(b)).

These experimental observations can be explained based on twinning dislocation (TD) reactions.[16–20] When two twins meet, two low-angle symmetrical tilt boundaries may form depending on the reaction location, as schematically visualized in Figure 3(a) and 3(b). On the left of Figure 3(b), the prism plane in T_1 is nearly parallel to the prism plane in T_2 , with a deviation angle of 7.4° . On the right of Figure 3(b), the basal plane in T_1 is nearly parallel to the basal plane in T_2 with the same deviation angle of 7.4° . Because of the lower energy and higher compactness associated with symmetric tilt boundaries, the twin–twin boundary in the right adopts the habit plane (0001) in the two twins (referred to as BB) and the one in the left adopts the habit plane $(10\bar{1}0)$ in the two twins (referred to as PP). From the viewpoint of TD reaction, the two twin–twin boundaries can be

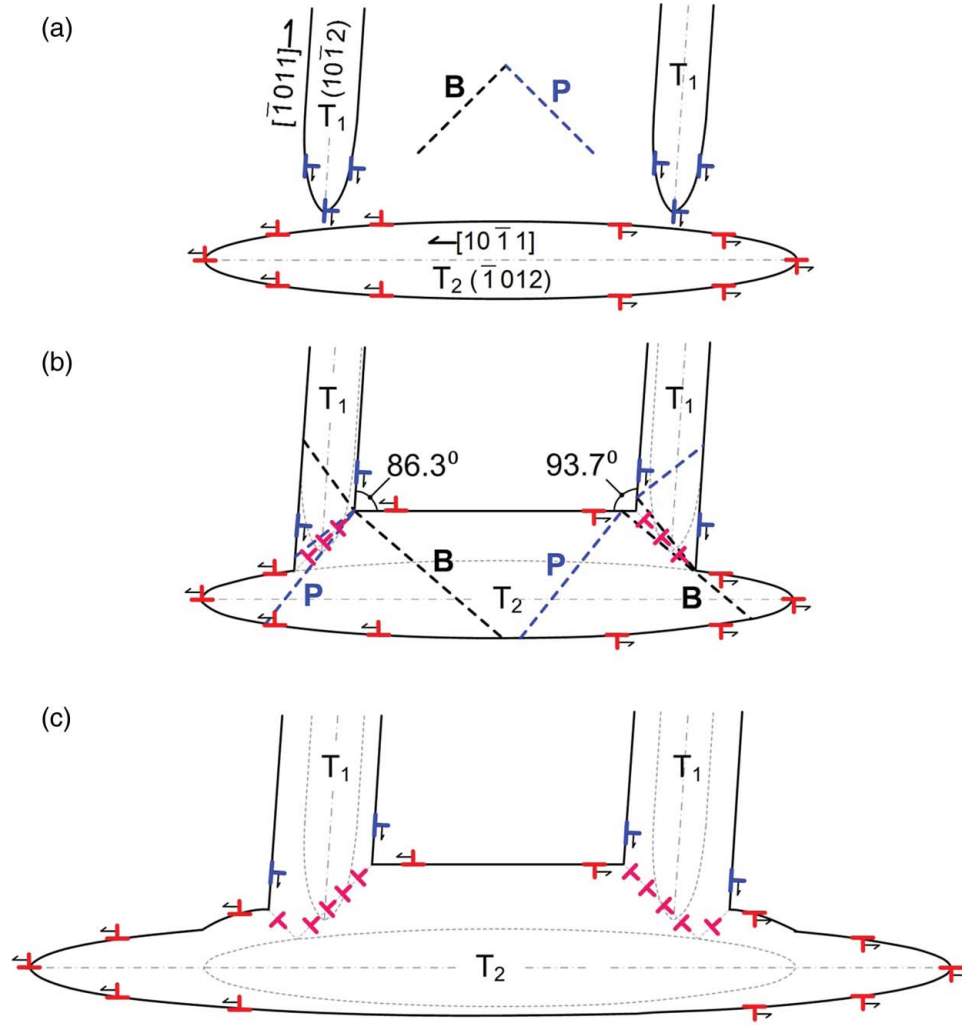


Figure 3. Formation of low-angle tilt boundaries when a $(10\bar{1}2)$ twin interacts with a $(\bar{1}012)$ twin: (a) two twin variants; (b) twin–twin boundaries; (c) twin growth after the formation of twin–twin boundaries. TDs are red for T₂ twin, blue for T₁ twin, and pink for boundary dislocations. Blue and black dashed lines indicate prismatic plane (P) and basal plane (B), respectively.

described as low-angle tilt boundaries, where the line of TD points out-of-paper. The Burgers vector of TD associated with the T₁ twin, $b_{tw}^{T_1}$, is equal to $\lambda[10\bar{1}\bar{1}]$. The Burgers vector of TD associated with the T₂ twin, $b_{tw}^{T_2}$, is $\lambda[\bar{1}01\bar{1}]$ in the left and $\lambda[10\bar{1}1]$ in the right. λ is calculated as $\lambda = (3 - \kappa^2)/(3 + \kappa^2) = 0.0649$ for Mg where $\kappa = c/a = 1.623$. [18] The possible reactions are:

$$\begin{aligned} (b_{tw}^{T_1} + b_{tw}^{T_2})^{\text{Left}} &= \lambda[10\bar{1}\bar{1}] + \lambda[\bar{1}01\bar{1}] \\ &= 2\lambda[000\bar{1}] = b^{PP} \end{aligned} \quad (1)$$

$$\begin{aligned} (b_{tw}^{T_1} + b_{tw}^{T_2})^{\text{Right}} &= \lambda[10\bar{1}\bar{1}] + \lambda[10\bar{1}1] \\ &= 2\lambda[10\bar{1}0] = b^{BB} \end{aligned} \quad (2)$$

b^{PP} and b^{BB} denote the resultant Burgers vector of the twin–twin boundary dislocations in the PP and BB boundaries with respect to the matrix (Figure 3(b)). The elastic energy associated with the left-hand side of Equations (1) and (2) is proportional to $11.23(\lambda a)^2$ ($0.0473a^2$ for Mg)

according to Frank's law. [16] The right-hand sides of Equations (1) and (2) are proportional to $10.56(\lambda a)^2$ ($0.0445a^2$ for Mg) and $11.97(\lambda a)^2$ ($0.0504a^2$ for Mg), indicating that the reaction is favorable in forming the PP boundary and unfavorable in forming the BB boundary when only the change in the line energy of dislocations is considered. However, the formation of tilt walls will further reduce the net elastic energy due to the core–core interaction among these dislocations. [16] Thus the formation of PP and BB boundaries (or two types of tilt walls) will be energetically favorable; in particular, the difference of the line energy is small in Equations (1) and (2).

It is also possible that both types of tilt boundaries (PP and BB) will form when one twin meets the other, as experimentally observed in Figure 2(d). The extension of the tilt boundaries take place if T₁ and T₂ growth can be accounted for by the reaction and dissociation of TDs. When a TD in T₁ glides toward the tilt boundary, it

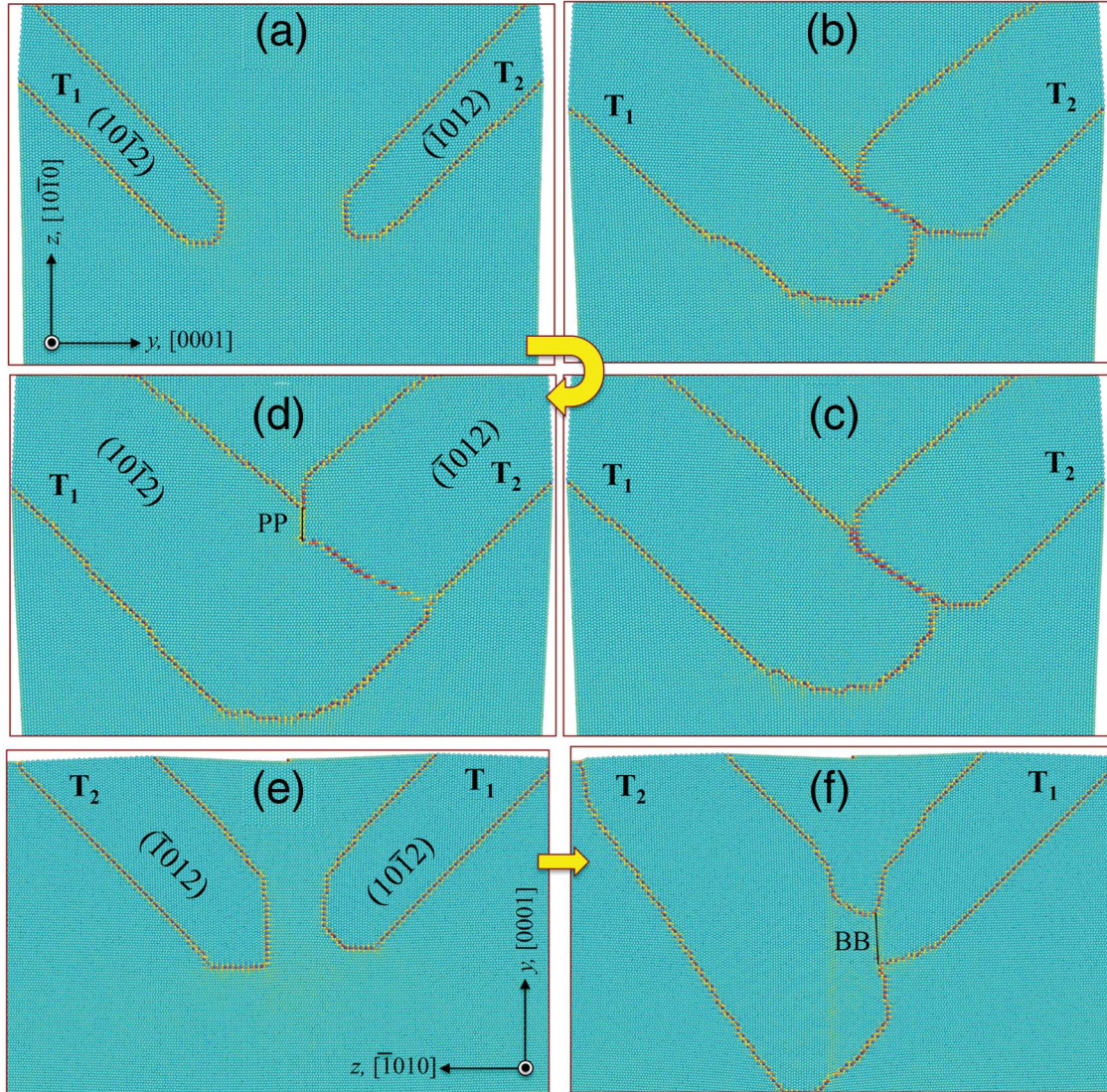


Figure 4. Formation of a PP tilt boundary with the habit plane $(10\bar{1}0)$ in the two twins. The two twins come from two surfaces. Under constant compression of 1.0 GPa along the z -axis, twins grow and propagate via the nucleation and glide of TDs, and meet to form twin–twin boundaries. The twin–twin boundary in (b) and (c) is composed of TDs in association with T_2 . PP in (d) indicates the tilt boundary. (e) and (f) Formation of a BB tilt boundary with the habit plane (0001) in the two twins. Atoms are colored according to their excess energy.

can dissociate into a TD in T_2 and leave a tilt boundary dislocation at the tilt boundary. As a result, the T_2 twin grows and the tilt boundary extends (Figure 3(c)), adding extra atomic layers on one side of the twin–twin junction and creating a ‘plateau’. An example of such a plateau is schematically shown in Figure 3(c), and is experimentally observed in Figure 2(d) (PP tilt boundary at the bottom left interaction). The required reaction involves a permutation of the dislocations in Equations (1) and (2) and is energetically unfavorable.

We examined the structure of the two tilt boundaries by performing molecular dynamics (MD) simulations with an empirical interatomic potential for Mg [21] which has proven to be reliable.[22–30] The MD simulation cell has the dimensions of 1.6, 50, and 100 nm with respect to

the x -, y -, and z -directions. The periodic boundary condition is applied along the x -direction, the free boundary condition for the y -direction, and the semi-fixed boundary condition for the z -direction, parallel to the compression axis. During MD simulations, atoms in the semi-fixed region can move in the x - y plane in order to accommodate the elastic strains due to Poisson effect. To mimic the two interaction locations described in Figure 3, one simulation was conducted with two twin variants that are created by gliding multiple TDs from two free surfaces (Figure 4(a)), and the other with two twin variants that are created from one free surface (Figure 4(e)). The MD simulations were performed at a temperature of 10 K. Under constant uniaxial compression of -1.0 GPa along the z -direction, the MD simulations reveal that

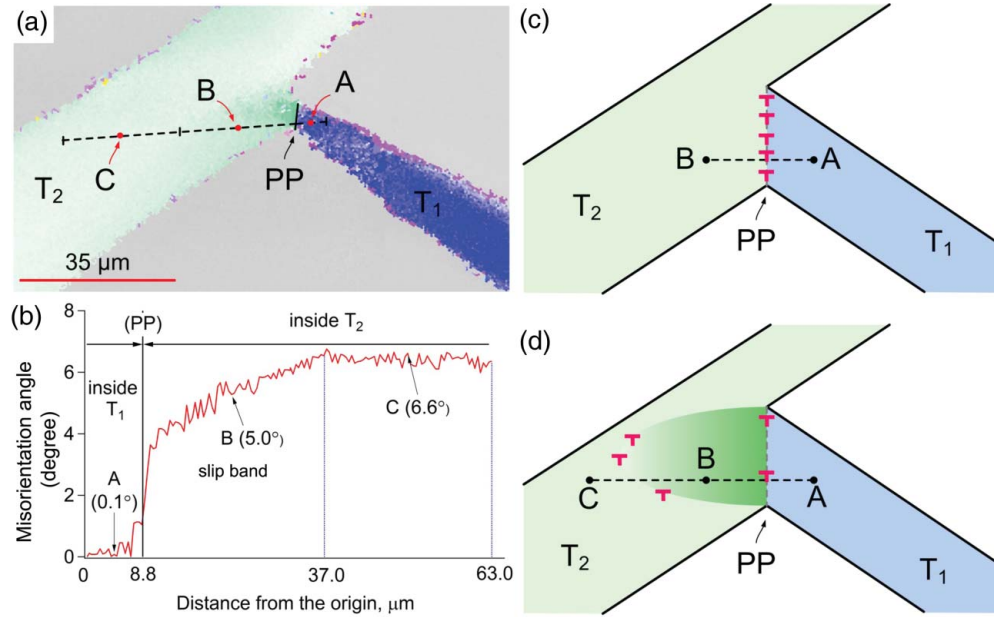


Figure 5. (a) and (b) Variation of the misorientation angle across the PP tilt boundary along a path denoted by a straight line in (a). The misorientation angle measured in T_1 is used as reference. (c) Schematic of an ideal PP tilt boundary without slip bands. All boundary dislocations are located at the boundary. The misorientation angle between A and B is equal to 7.4° . (d) Schematic of a PP boundary with slip bands. The misorientation angle varies with the formation of slip bands.

the growth of twins is accomplished via the nucleation and glide of TDs from surfaces. When the two twins meet (Figure 4(b–d)), twin–twin boundaries form and extend. The symmetrical tilt boundary denoted by PP in Figure 4(d) adopts the habit plane $(10\bar{1}0)$. Figure 4(f) shows the formation of the BB tilt boundary with the habit plane (0001) .

In addition to the formation of tilt boundaries, a basal slip band is usually observed in connection with the tilt boundary with the habit plane $(10\bar{1}0)$ (Figure 2(a)). Figure 5(a) and 5(b) show the misorientation analysis in the slip band region according to EBSD analysis. The misorientation angle between T_1 and T_2 is 6.6° , where the slip band region has misorientation angles of 5.0° with respect to T_1 and 1.6° with respect to T_2 . The associated misorientation axis is between the c -axis and the a -axis. The details can be found in the Supplementary Material. There are two possible mechanisms corresponding to the formation of basal slip bands. First, a simple geometry analysis shows that the angle between the basal planes in T_1 and T_2 is 7.4° . The coherent region associated with the tilt boundary facilitates slip transmission for basal dislocation from the T_1 twin to the T_2 twin, and prevents the formation of a secondary tension twin inside the T_2 twin because TDs in the T_1 twin cannot directly transform into TDs in the T_2 twin. Second, Burgers vectors along the tilt boundary could reassemble, under stress, into a boundary dislocation with Burgers vector $\frac{1}{2}[10\bar{1}0]$ accompanying the extension of the tilt boundary, so reducing the chemical potential energy by forming more coherent segments

along the tilt boundary.[19,20] These reassembled boundary dislocations can act as sources for nucleation and emission of basal dislocations. These basal dislocations are then emitted from the tilt boundary, forming a basal slip band. Accompanying the emission of basal dislocations from the tilt boundary, the tilt angle decreases, as schematically described in Figure 5(c) and 5(d). This is an energetically favorable process.

In summary, co-zone twin–twin interactions in an Mg single crystal under compression–tension loading was studied using *in situ* optical microscopy, EBSD analysis, and atomistic simulations. Two tilt boundaries are observed when two twins meet. One has the habit plane (0001) and the other $(10\bar{1}0)$. Their formation and the influence on twinning, de-twinning, and basal slip bands are discussed based on dislocation theory and MD simulations. The formation of BB and/or PP tilt boundaries is crystallographically preferred and energetically favored. Accompanying the formation, TDs associated with one twin cannot transmit into the other twin to form secondary twins. However, a basal slip band can form due to the easy transmission of basal dislocation across the PP tilt boundary and the nucleation and emission of basal dislocation from the PP tilt boundary. Further, twinning and later de-twinning are affected by the tilt boundary. In particular, de-twinning is obstructed because of the energetically unfavorable dissociation of the tilt boundaries, more so in the case of BB than of PP tilt boundaries because of the higher energy barrier in association with the dissociation of boundary dislocation in the PP tilt boundary.

Supplementary online material. A more detailed information on experiments is available at <http://dx.doi.org/10.1080/21663831.2013.867291>

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