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Intragranular cross-level twin pairs in AZ31 Mg alloy after sequential biaxial compressions

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ABSTRACT

Sequential double extension twinning in AZ31 Mg alloy happens easily during sequential biaxial compressions. Widespread twin-twin interaction leads to the formation of intragranular cross-level twin pairs, each of which can be defined by a primary twin variant PVi (i = 1–6) and its nearby secondary twin variant PVj-SVk (j, k = 1–6, j ≠ i, k) across the twin-twin boundary between them. Theoretically, 9 possible misorientations exist between the two variants. Misorientation between two crystals can be expressed by a pair of rotation axis (**r**) and the minimum rotation angle (θ). The crystallography of a cross-level twin pair can be described unanimously by the misorientations between PVi and PVj-SVk (i.e., $\mathbf{r}_1$-θ_1), PVi and PVj (i.e., $\mathbf{r}_2$-θ_2), and the host grain and PVj-SVk (i.e., $\mathbf{r}_3$-θ_3), resulting in 13 possible orientation relationships (ORs) that can be expressed explicitly by the three misorientation angles (θ_1, θ_2, θ_3). A cross-level twin pair is a key element of a crossing or crossed twin structure, which is common in the deformed microstructure. Statistical electron backscatter diffraction analysis reveals that OR2 of (49.7°, 60°, 60°) appears the most frequently. Twinning shear transmission is almost irrelevant to the formation of a cross-level twin pair. Although the majority of the identified twins are high Schmid factor (SF) ones with SF > 0.3 and SF ratio > 0.8, SF analysis fails to explain the highly preference of OR2 and the abnormal absence of OR8 of (44.0°, 60°, 60.4°), probably due to the complexity of the formation of a cross-level twin pair.

1. Introduction

Magnesium alloys have attracted much attention for light-weight applications in aerospace and transportation, where weight saving is crucial [1,2]. However, limited ductility and pronounced mechanical anisotropy pose a bottleneck to a widespread usage of Mg alloys. Both the properties bear a close relationship to deformation twinning, which is a necessary deformation mode in Mg with a hexagonal close-packed (HCP) structure. The most common primary twinning modes in Mg and its alloys are {1 0 −1 2}⟨−1 0 1 1⟩ extension twinning and {1 0 −1 1}⟨1 0 −1 −2⟩ contraction twinning [3,4]. The resolved critical shear stress (CRSS) of the former one is much lower than that of the latter one [5–7], which contributes significantly to the anisotropy of yield strength between tension and compression of a wrought Mg alloy with prominent texture. However, such an anisotropy can be largely removed through sequential multi-directional compressions [8,9], the basic process of which is sequential biaxial compressions [10]. During the sequential biaxial compressions, the first loading is usually applied along the rolling direction of a rolled plate, unloaded, and then the second loading is applied along the transverse direction, or vice versa.

Knowledge on microstructure evolution during sequential biaxial compressions lays the foundation for understanding the benefits of sequential multi-directional compressions. In-situ electron backscatter diffraction (EBSD) observations reveal that multiple twinning behaviors happen during sequential biaxial compressions of AZ31 Mg alloy, including primary {1 0 −1 2} extension twinning, {1 0 −1 2}–{1 0 −1 2} double extension twinning, and detwinning [11]. During uni-axial deformation, {1 0 −1 1}–{1 0 −1 2} double twinning is common [12,13], while during biaxial compressions {1 0 −1 2}–{1 0 −1 2} double extension twinning is common [10,14,15]. The crystallography of the double extension twinning has been explored systematically elsewhere [10]. There exist 36 possible double extension twin variants, which can be classified into 4 misorientation groups according to their misorientations with respect to the host grain [10]. Statistical EBSD

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analysis reveals that the majority of the double extension twins belong to group III with a misorientation of ⟨0 14 −14 1⟩ 60° [10].

Twin-twin interaction plays an important role in microstructure evolution during plastic deformation of Mg and its alloys, which results in the formation of twin pairs across a grain boundary or a twin-twin boundary. Long {1 0 −1 2} or {1 0 −1 1} twin bands across several grains can form in AZ31 Mg alloy after uni-axial deformation [16–18]. Previous studies [16,17,19–21] mainly focused on twin pairs of two connected primary twins or two connected secondary twins, which can be called as same-level twin pairs. Logically, a cross-level twin pair should consist of a primary twin and its connected secondary twin.

Although such a cross-level twin pair seldom forms in Mg alloys after uni-axial deformation, it is quite easy to be detected after the sequential biaxial compressions, as will be shown in the present study. Furthermore, an intragranular cross-level twin pair contributes to the formation of a crossing or crossed twin structure, which is a key feature of the deformed microstructure. The present study will shed light on the characteristics of intragranular cross-level twin pairs, which differ from previously reported compound cross-grain boundary twin structures [21] in the following aspects: (1) a compound cross-grain boundary twin structure contains twins from two neighboring host grains, while an intragranular cross-level twin pair contains twins in the same host grain; (2) a compound cross-grain boundary twin structure forms largely due to twinning shear transmission over grain boundary, which is irrelevant for the formation of an intragranular cross-level twin pair as will be shown afterwards.

2. Material and methods

2.1. Material

A commercial hot-rolled AZ31 Mg alloy (Mg-3Al-1Zn in wt.%) plate of 20 mm in thickness was bought from Tianrui United (Beijing) Technology Development Co., Ltd. The rolling, transverse and normal directions of the plate were designated as RD, TD and ND, respectively. It was annealed at 400 °C for 2 h. The annealed plate without twins has a strong basal texture with the basal pole oriented to ND, while the {1 0 −1 0} and {1 1 −2 0} poles randomly oriented in the rolling plane as previously reported [17], which is typical of a hot-rolled AZ31 alloy [10,22,23].

Cubic specimens with 10 mm in length were cut from the annealed plate by electro spark wire-electrode cutting. In order to activate {1 0 −1 2}–{1 0 −1 2} twinning, sequential biaxial compressions, following the principle of a previous study [10], were applied to the specimens. Compressive loading was first applied along RD for 2.0% reduction, unloaded and then applied along TD for 2.0% reduction, at room temperature and a strain rate of 1×10^{-3}/s. Mechanical response of a similar sequential biaxial compressions of 1.8% RD + 1.3% TD was reported previously, which shows a remarkable increase of yield strength during the second TD compression [10]. In order to diminish the barreling effect as much as possible, surfaces of the specimens were carefully ground with sandpaper from 1000 to 4000 grits and then mechanically polished with ethanol to obtain mirror-like surfaces. Graphite sheets were used to lubricate the surfaces and the upper and lower platens of the compression machine.

After the sequential biaxial compressions, the specimens were sectioned in half for microstructure observations. Mirror-like sectioned faces were obtained according to the aforementioned procedure. Then they were electrolytically polished in an electrolyte of 62.5% phosphoric acid and 37.5% ethanol at 1.5 V for 1 min, at −15 °C. EBSD measurements with step sizes of 0.1–0.3 μm were performed using a Zeiss Auriga field emission scanning electron microscope (FESEM) equipped with an Oxford NordlysNano EBSD camera. An area of about 7.4×10^4 μm^2 in total was measured by EBSD for statistical analysis. The measured data were analyzed using the MATLAB™ toolbox MTEX (mtex-toolbox.github.io) [24]. The total deformation of the material was about 4.0%, not highly deformed. Even though, there were unclean locations mainly distributed along grain boundaries. An area containing more than five measured points of similar orientations was considered to be reliable. Then the EBSD maps were cleaned.

2.2. Identification of twin variants

In order to describe multiple parent-twin relationship clearly, G and G-P are used to designate a host grain and its primary twin, respectively. A secondary twin in G-P or equivalently a double twin in G is designated as G-P-S. Such a designation system works very well for describing the microstructure after the sequential biaxial compressions [25]. The six possible {1 0 −1 2} twin variants are defined as follows [10,19,21,26]: 1: (1 0 −1 2)[−1 0 1 1], 2: (0 1 −1 2) [0 −1 1 1], 3: (−1 1 0 2)[1 −1 0 1], 4: (−1 0 1 2)[1 0 −1 1], 5: (0 −1 1 2) [0 1 −1 1], and 6: (1 −1 0 2)[−1 1 0 1]. Consistent with a previous study [10], primary and secondary twin variants are designated as PV and SV, respectively. For example, both PV1 and SV1 refer to variant 1: (1 0 −1 2)[−1 0 1 1], and PV1-SV1 refers to variant (1 0 −1 2)–(1 0 −1 2). The method for determining twin variants using EBSD data has been explained in detail elsewhere [10]. {0 0 0 1} pole figure of all the 6 possible primary {1 0 −1 2} twin variants and 36 possible {1 0 −1 2}–{1 0 −1 2} double twin variants in a grain with Euler angles (0°, 0°, 0°) are drawn in Fig. 1a.

3. Results

3.1. Explore crystallography of an intragranular cross-level twin pair

Due to the basal texture, the first loading along RD can activate primary {1 0 −1 2} twinning in Mg grains. A primary twin generated in a grain is schematically drawn in Fig. 1b, in which the primary twin is G-P1 in its host grain G. The second loading along TD not only can activate primary {1 0 −1 2} twinning in the remained grain G, but also can activate secondary {1 0 −1 2} twinning in the twin G-P1, as verified previously [10,11,15]. The products are primary twin G-P2 and secondary twin G-P1-S1. As shown in Fig. 1b, they have a chance to be connected with each other across the twin-twin boundary (TTB) between G-P1 and G-P2, and thereby form a cross-TTB twin pair of a primary twin and a secondary twin, which is the so-called cross-level twin pair. By contrast, a same-level twin pair consists of two primary or two secondary twins.

According to the crystallography of {1 0 −1 2} twinning, all the possible misorientations between two twins in a cross-level twin pair can be calculated. As shown in Fig. 1b, twin G-P1 corresponds to variant PVj (j = 1–6), twin G-P2 corresponds to variant PVi (i = 1–6, i ≠ j) and twin G-P1-S1 corresponds to variant PVj-SVk (k = 1–6, k ≠ j), in which k ≠ j since PVj-SVj corresponds to detwinning of PVj. Crystallographically, variants PVi (i = 1–6) and PVj-SVk (j, k = 1–6, j ≠ i, k) define a cross-level twin pair. It can be calculated that there are 150 (=6 × 5 × 5) possible combinations of PVi (i = 1–6) and PVj-SVk (j, k = 1–6, j ≠ i, k), which yields 9 misorientation groups, as listed in Table 1. All the rotation axes in Table 1 were shown in Mg crystal (Fig. 1c). Consistently, the 9 misorientation groups can be deduced in another way. As listed in Table 1, there are 3 possible misorientation groups between PVi (i = 1–6) and PVj (j = 1–6, j ≠ i) [27]. There also are 3 possible misorientation groups between the host grain G and PVj-SVk (j, k = 1–6, j ≠ k) [10]. Therefore, according to twinning symmetry, 9