Scripta Materialia 117 (2016) 32–36

Contents lists available at ScienceDirect

Scripta Materialia

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

Electron backscatter diffraction pattern analysis of the deformation band formed in the Mg-based long-period stacking ordered phase

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ARTICLE INFO

Article history:
Received 14 January 2016
Received in revised form 10 February 2016
Accepted 11 February 2016
Available online 2 March 2016

Keywords:
Magnesium alloys
Electron backscattering diffraction (EBSD)
Deformation structure
Dislocation
Deformation kink band

ABSTRACT

A newly proposed analysis protocol using electron backscatter diffraction pattern analysis in scanning electron microscopy (SEM-EBSD) clarified that the deformation bands formed in the Mg-based long-period stacking ordered (LPSO) phase are predominantly deformation kink bands. The kink band contains many additional boundaries within it, and the coalescence of these boundaries varies the crystal rotation angle and rotation axis at the kink band boundaries during their development. The reason for the appearance of a "beak-like" shape in deformation kink band in the LPSO phase was clarified by proposing a unique three-dimensional morphology.

Recently, the long-period stacking ordered (LPSO) phase has received considerable attention as a possible strengthening phase in Mg alloys [1–14]. Mg/LPSO two-phase alloys show superior mechanical properties compared with conventional Mg alloys; thus, practical applications of these alloys are greatly expected. The formation of a deformation band, in addition to basal slip, has been reported as the deformation mode of the LPSO phase. Further, the possibility of a deformation kink band was proposed as the origin of the deformation band [15–17]. The kink band in the hexagonal crystals is considered to be formed by the cooperative initiation and/or operation of basal dislocations and following their arrangement, to be aligned perpendicular to the basal plane [18–25]. However, conflicting opinions attempting to explain the origin of the deformation bands in the LPSO phase by the formation of a deformation twin have also been recently reported [26,27]. To clarify the origin of the deformation band in the LPSO phase, elucidation of its crystallographic features must be useful. In this regard, we recently proposed a protocol to distinguish deformation kink bands and deformation twins in hexagonally structured materials via electron backscatter diffraction pattern analysis in scanning electron microscopy (SEM-EBSD) on the (0001) specimen surface, by using a Zn single crystal as a model material [28]. In this study, the proposed observation protocol was applied to the LPSO phase, and the nature of the deformation band in the LPSO phase was discussed.

To prepare the specimens, master ingots with a composition of $Mg_{85}Zn_6Y_9$ (at.%) were fabricated by induction melting in carbon crucibles. Using the master ingots, directional solidification (DS) was conducted using a vertical Bridgman furnace in an Ar-gas atmosphere at a growth rate of 10 mm/h. Via transmission electron microscopy (TEM), the DS crystal was confirmed to almost only consist of the 18R-type LPSO phase [29]. From the obtained DS crystal, rectangular specimens with dimensions of $2 \times 2 \times 5$ mm^3 were cut by electrical discharge machining for compression tests. The surface of the specimen was polished by emery paper, and then chemically polished with 20 vol.% nitric acid/80 vol.% ethanol solution. The loading axis was set to be parallel to the growth direction. As reported previously, the LPSO-phase grains show plate-like shapes with the interface parallel to (0001), and the grains were well aligned so as to their interfaces become parallel to the growth direction in the DS crystal [15,29]. Thus, the loading orientation was almost parallel to (0001) in most of the grains in the prepared specimen. More precisely, the growth direction was found to be close to $<11\bar{2}0>$ in many of grains [29]. Compression tests were performed in order to introduce deformation bands in the specimen at a nominal strain rate of 1.67×10^{-4} s^{-1} at room temperature (RT) in vacuum. Variations in the crystal orientation due to the formation of deformation bands were examined by SEM-EBSD analysis

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http://dx.doi.org/10.1016/j.scriptamat.2016.02.016
1359-6462/

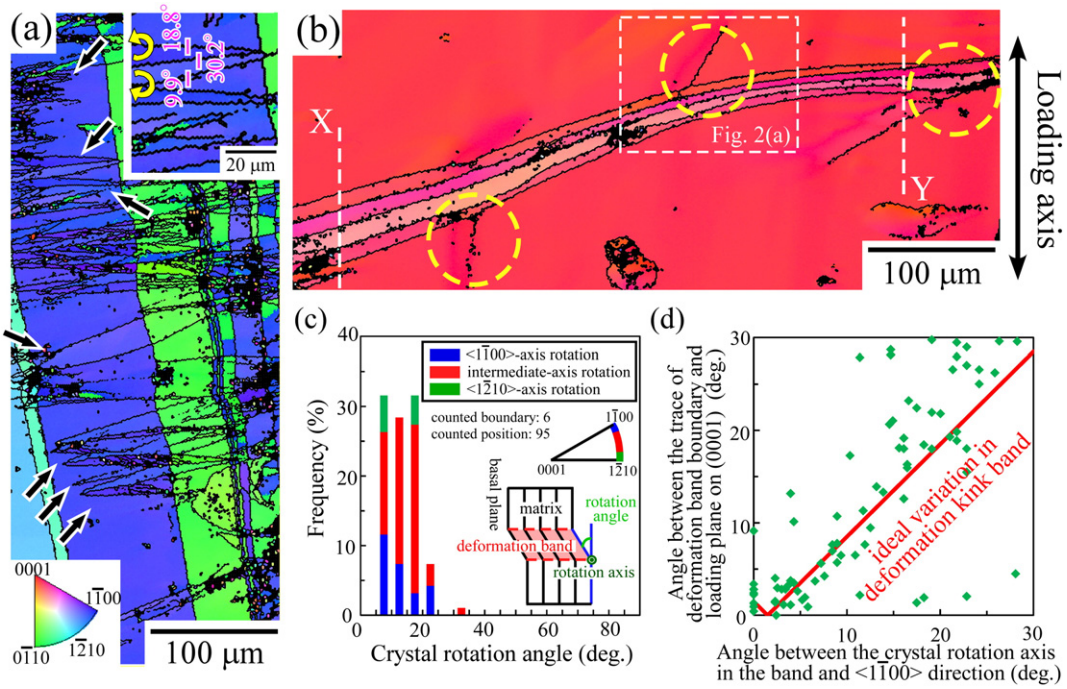

Fig. 1. (a, b) Crystal orientation maps taken on the side surfaces of deformed LPSO-phase DS specimens, focusing on grains whose surface normals are nearly parallel to (a) [1$\bar{1}$00] and (b) [0001]. (c) Distribution of the rotation angle on the deformation band boundaries measured on (0001) in the ~1% deformed specimen shown in Fig. 1(b). The variations in the crystal rotation axis in the bands are also indicated as a difference in the color of the bar, in which the tolerance angle of the deviation was set to be within 5°. (d) Relationship between the crystal rotation axis and the inclination angle of the trace of the deformation band boundary with respect to the direction normal to the loading axis, as observed on the (0001) surface.

at a measured step size of 1 μm. For the analysis, specimens were first polished by emery paper followed by the chemical polishing, and finally polished by colloidal silica.

Fig. 1(a, b) show the typical crystal orientation maps taken in the specimens compressed at RT, showing the variation in the crystal orientation due to the formation of a deformation band. The observations were conducted on the specimen side surfaces and focused on grains whose surface normals were nearly [1$\bar{1}$00] and [0001] as shown in Fig. 1(a, b), respectively. The morphology of the deformation bands was significantly different depending on the observation direction, as previously reported using optical microscopy [30]. In most of the observations other than the direction nearly along [0001], deformation bands frequently show characteristic "beak-like" shapes [15–17,30], as indicated by black arrows in Fig. 1(a). The beak-like shape of the band consisted of two bands that show opposite crystal rotation angles to each other, as indicated in the higher magnification image in Fig. 1(a). The definitions of the crystal rotation angle and rotation axis in the bands are schematically drawn in Fig. 1(c). When the deformation bands are observed along [0001], however, such beak-like morphologies were never seen. On the (0001) side surface, deformation bands macroscopically lay nearly perpendicular to the loading axis, but they exhibited a considerably wavy (rounded) morphology, as shown in Fig. 1(b). In this regard, it was recently reported that the waviness of the bands in a Zn single crystal can be explained by assuming that the deformation band is not a deformation twin but a deformation kink band [28]. To validate this assumption, the use of the analysis protocol using EBSD on (0001) was proposed. The details of this method have been reported in a previous paper [28], but the essence of the analysis is briefly described. Consider the deformation of a crystal whose loading axis is parallel to [11$\bar{2}$0]. Assuming that the deformation kink band boundary is constructed by a basal dislocation array comprising only one kind of dislocation with a Burgers vector parallel to [11$\bar{2}$0], the boundary plane of the deformation kink band must lie on (11$\bar{2}$X) [28]. The value of X varies depending on the density of the dislocations that form the deformation kink band boundary. Note here that, taking geometric considerations into account, the trace of the deformation kink band boundary in this case must be observed to be parallel to [1$\bar{1}$00] on (0001). Therefore, the trace must lie perpendicular to the loading axis on the (0001) specimen side surface independent of the value of X, i.e., independent of the density of the dislocations that form the deformation kink band boundary. On the other hand, if the deformation kink band boundary is constructed by two kinds of basal dislocations, e.g., by equivalent numbers of [11$\bar{2}$0] and [2$\bar{1}\bar{1}$0] dislocations, the kink band boundary must lie on the (10$\bar{1}$X) plane [28]. In this case, the trace of the deformation band boundary on the (0001) side surface is inclined by 30° with respect to the direction normal to the loading axis independent of the value of X. The inclination angle of the traces of the deformation band can vary depending on the ratio of [11$\bar{2}$0] and [2$\bar{1}\bar{1}$0] dislocations. Thus, the variation in the ratio of two-kinds of basal dislocations that construct the kink band boundary depending on the position can induce the waviness of the deformation kink band boundary when observed on (0001) [28].

It is noted that the crystal rotation axis in the kink band also varies depending on the ratio of two-kinds of basal dislocations that construct the boundary, since the crystal rotation axis on the deformation kink band boundary is defined as the direction perpendicular to both the slip plane normal (c axis for basal slip) and slip direction (the summation of the Burgers vector of the dislocations that construct the boundary) [16]. Thus if the observed deformation band is the deformation kink band, the inclined angle of the trace of the boundary at a certain point must be correlated with the variation in crystal rotation axis at the point. Whereas such correlation is not observed in the deformation twin. Thus, by examining the relation between them at each point on the bent deformation band boundary, the features of a deformation band and whether it is deformation twin or deformation kink band can be experimentally clarified.

Fig. 1(c, d) show the obtained analysis results according to this observation protocol for the deformation bands introduced in the grain shown in Fig. 1(b), in which the loading axis is parallel to [9 10 $\bar{19}$ 0]. Fig. 1(c) shows a bar graph exhibiting the variation in the crystal