



# The role of deformation twinning in the fracture behavior and mechanism of basal textured magnesium alloys



D. Ando\*, J. Koike, Y. Sutou

Department of Materials Science, Tohoku University, 6-6-11-1016 Aoba, Aramaki, Aoba-ku, Sendai 980-8579, Japan

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## ABSTRACT

AZ31 magnesium alloys were deformed to 10% and to failure strain by tensile loading at room temperature. Scribed grids were drawn by a focused ion beam system (FIB) to visualize the local deformation in each grain. This showed that the magnitude of the strain was distributed non-uniformly in each grain. It was found that the low-strain grains accompanied {10–12} twins, while the severely strained grains accompanied {10–11}–{10–12} double twins. Cracks nucleated at the double twins and tended to propagate along {10–12} twin interfaces as well as within grains. Furthermore, fractography revealed three types of microstructural features: dimples, elliptic facets and sheared dimples. Most abundant were the dimples formed by ductile failure. The elliptic facets appeared to be due to crack propagation along the {10–12} twin interfaces. The sheared dimples were frequently observed in connection with localized shear deformation within the double twins. These results led us to conclude that premature and catastrophic failure of Mg alloys is mainly associated with double twins. Prevention of double twinning is essential to improve the ductility of Mg alloys.

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## 1. Introduction

Magnesium alloys have only four slip systems and form many types of deformation twins during plastic deformation. These twins play an important role in deformation and failure at ambient temperature. It has been widely reported that {10–12} twins and {10–11}–{10–12} double twins are formed in Mg alloys. {10–12} Twins are formed easily because of their small critical resolved shear stress (CRSS) of merely 2–4 MPa [1]. {10–12} Twinning induces a tensile strain along the *c* axis of the hexagonal lattice, and a compressive strain along the *a* axis. Normally, {10–12} twins are formed so as to conform to external stress. For example, when extruded Mg alloys are compressed along the extrusion direction, {10–12} twinning occurs and becomes a major deformation mechanism because the samples are compressed in the *a* direction. In contrast, when the samples are deformed in tension along the extrusion direction, {10–12} twinning is not expected to occur. However, {10–12} twins are also formed and induce a strain whose sign is opposite to that of the external stress. The formation of this odd twin, or negatively strained twin, has been explained in our previous paper [2]. We reported a high correlation between the

tendency for basal dislocation slip and the tendency for odd {10–12} twinning. This correlation is thought to be due to the anisotropy and inhomogeneity of dislocation plasticity among neighboring grains. For instance, if basal slip occurs easily in one grain but not in the surrounding grains, negatively strained {10–12} twinning would prevail so as to reduce the incompatibility of plastic strain caused by the basal slip. So far, the formation of odd {10–12} twins has been explained only by the indirect evidence of microstructural observation. Thus, the first purpose of this study is to show that odd {10–12} twins are formed to satisfy the strain compatibility condition.

In the case of the double twin, many twin types are formed and have been proposed to be related to catastrophic failure. Reed–Hill and his colleagues have reported that various twins, viz. {30–34}, {11–24}, {10–14}, and {10–15}, were associated with fracture planes [3,4]. Later, the double twins of {10–11}–{10–12} were reported to be fracture initiation sites [5]. According to this fracture model, small voids form along {10–11}–{10–12} double twin interfaces and grow to become microcracks as the stress increases. Eventually, fracture occurs along the weak twin interface. Other twin types have also been reported to act as crack initiation sites, namely {11–24} [6] and {10–13} twins [7]. However, there have been ambiguities in previous reports because the twin types were often determined by trace analysis of habit planes, which are not necessarily the same as those of the twinning shear planes.

\* Corresponding author.

E-mail address: [dando@material.tohoku.ac.jp](mailto:dando@material.tohoku.ac.jp) (D. Ando).

Recently, we found that sharp surface steps led to crack formation. Transmission electron microscopy (TEM) of a substructure of more than 30 surface steps indicated that all these steps accompanied {10–11}–{10–12} double twins [8]. We also investigated {10–11}, {10–12} and {10–13} twins, and found that only {10–11}–{10–12} double twins could cause a large enough localized deformation to lead to crack initiation [9]. Other researchers have also recognized the relevance of double twins with regard to premature catastrophic failure [10–12]. However, current knowledge is limited to the individual roles of the various twin types at different stages of the deformation process, and does not provide information regarding their relationships and possible interactions throughout the deformation and failure process as a whole. Therefore, the second purpose of this study is to observe the microstructure before deformation and after fracture, and to understand the role of the various twin types in the deformation and failure mechanisms.

The present work was designed to have three steps. In the first, a grid pattern of scribed lines was formed on the sample surface. After deformation to a nominal strain of 10%, the displacement of the grid pattern was measured to determine the magnitude and inhomogeneity of the plastic strain. Also, the surface microstructure was observed to correlate the strain inhomogeneity and twinning. In the second step, the microstructure of a fractured sample was examined. In order to understand the mechanisms leading up to failure, the microstructure of the sample's top surface was observed in a region slightly away from the fracture surface. In the third step, the fracture surface of the same sample was observed to determine the failure mechanisms.

## 2. Experimental procedure

Samples were rolled sheets of AZ31 (Mg–3Al–1Zn in mass%) having a thickness of 0.5 mm, provided by Nippon Kinzoku Co., Ltd. The as-received sheets were annealed at 400 °C for 8 h. The average grain size in the annealed samples, as determined by a linear interception method, was approximately 80  $\mu\text{m}$ . Abnormal grain growth was not observed and the grain size distribution was uniform. Tensile test samples were machined from the annealed sheets to have a gauge length of 15 mm and a gauge width of 4 mm. The sample surface was mechanically polished using abrasive papers #1200–4000, then chemically polished to a mirror finish using a solution of 50 ml of ethanol and 8 ml of nitric acid for 20 s. Subsequently, scribed grids were drawn on the sample's top surface to visualize the local deformation in each grain. The scribed grids were drawn by locally sputtering the sample surface with an energetic Ga ion beam using a focused ion beam microscope (FIB). The grids consisted of 400 squares having an edge length of 10  $\mu\text{m}$ . The grid lines were nominally 100 nm in width and 100 nm in depth.

Before the tensile tests were performed, the crystallographic orientation distribution was investigated with an electron backscatter diffraction (EBSD) apparatus attached to a scanning electron microscope (SEM) using a step size of 0.8  $\mu\text{m}$ . Following the EBSD measurement, tensile tests were performed along the rolling direction at room temperature to an engineering strain of 10% and to a fracture strain of 24% at a constant crosshead speed corresponding to an initial strain rate of  $1.0 \times 10^{-3} \text{ s}^{-1}$ . After tensile testing, each sample was examined with a FIB microscope to determine the displacement of the scribed lines and with an optical microscope to measure the magnitude of the localized deformation.

Furthermore, all fractured samples were observed in secondary electron imaging mode with the FIB apparatus. FIB observation was performed in two different locations: (i) the sample's top surface near the fractured region and (ii) the fracture surface. Both locations were examined with the surface under observation

nearly perpendicular to the incident electron beam. To achieve this, the fractured samples were placed on a tilted sample holder at 45°. This sample alignment was necessary to cut a cross-sectional TEM sample from a specific area of the observed region using FIB. The internal microstructure of characteristic features was thus observed with TEM, and the crystallographic type of twins was determined by analyzing the diffraction patterns. The diffraction patterns were taken with the incident electron beam parallel to the  $\langle 1-210 \rangle$  direction with the aid of Kikuchi diffraction patterns.

## 3. Results and discussion

### 3.1. Relationship between inhomogeneity of dislocation plasticity and twinning

Fig. 1 shows the top surface of a sample observed by optical microscopy (a) before and (b) after deformation to 10% strain. In these figures, TD and RD stand for transverse direction and rolled direction, respectively. Throughout this work, the tensile direction is RD. It can be seen that the square-shaped grid patterns are distorted non-uniformly upon deformation and that deformation is accompanied by the formation of surface roughness, indicating the spatial inhomogeneity of the plastic deformation. Also, many twins are observed, as indicated by the arrows. In order to better understand localized twin formation, the framed regions containing grains A and B will be analyzed in detail later. Fig. 1(c) shows a crystallographic orientation map of the same area as in (a) before deformation. The various colors correspond to the specific crystallographic orientations shown in the inset of the orientation triangle. This orientation map is overlapped with an image quality map so as to show not only the grain orientation but also grain boundaries and scribed grids. In this figure, the original square grid appears to be distorted into a diamond shape because of incomplete tilt angle compensation by the image processing software. Thus, the diamond-shaped grid is taken to be the initial state of the EBSD image. Fig. 1(d) is a Schmid Factor (SF) map for basal slip. The magnitude of the SF is indicated by the colors shown in the inset color bar. Blue corresponds to a small SF, while red corresponds to a large SF. According to the SF map, a large deformation is expected in a red grain, while a smaller deformation is expected as the color deviates from red to blue. However, a comparison between Fig. 1(b) and (d) shows the opposite trend. For example, grain A, which accompanies a lenticular twin, has a high SF for basal slip but shows almost no deformation. In contrast, grain B has a low SF for basal slip but is severely deformed, leading to the formation of surface steps.

Next, we show the relationship between the localized strain and deformation twins. The localized strain value was evaluated by a difference of actual measured length of grids in optical microscope pictures. This method can measure only RD–TD two-dimensional face strain. In this work, the strain of ND was ignored because they were much smaller than the face strain. Fig. 2(a) is a FIB image of the framed region in Fig. 1(b) containing grain A after deformation to 10% strain. The white line indicates the grain boundary, and the yellow lines indicate twin interfaces. The square grid formed by FIB has an edge length of 10  $\mu\text{m}$ . The original size before deformation is shown by the blue square. A comparison between the grid pattern and blue square indicates that almost no deformation is observed in this grain. The total strain estimated from the grid pattern is less than 1%, which is much smaller than the nominal strain of 10%.

Grain A has a large SF for basal slip while the surrounding grains have a smaller SF. Moreover, grain A has lenticular twins while its neighboring grains have no twins. This suggests that

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