Materials Science & Engineering A 590 (2014) 174–185

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

Experimental study of fracture behavior of magnesium single crystals

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ARTICLE INFO

Article history:
Received 19 July 2013
Received in revised form
9 October 2013
Accepted 12 October 2013
Available online 19 October 2013

Keywords:
Magnesium single crystals
Fracture behavior
Tensile twinning
Fracture toughness
Crack deflection
EBSD

ABSTRACT

In this work, the fracture behavior of magnesium single crystals is studied by conducting experiments with notched three point bend specimens of three crystallographic orientations. In the first and second orientations, the *c*-axis is along the normal to the flat surface of the notch, while in the third it is aligned with the notch front. For all the orientations, in situ electron back scattered diffraction observations made around the notch root show profuse tensile twinning of $\{10\bar{1}2\}$–type. Further, in the first two orientations basal and prismatic slip traces are identified from optical metallography. The width of the most prominent twin saturates at around 120–150 μm, while twins continue to nucleate farther away to accommodate plastic deformation. In all the orientations, crack initiation occurs before the attainment of peak load and the crack grows stably along twin–matrix interface before deflecting at twin–twin intersections. Results show that profuse tensile twinning is an important energy dissipating mechanism that enhances the fracture toughness.

1. Introduction

Magnesium alloys are well known for their mechanical properties such as high specific strength at room temperature and low density as compared to the commonly used aluminum alloys, making them prospective candidates for applications in aerospace and automobile industries. However, poor corrosion resistance and low fracture toughness [1] impedes their usage. Most research pertaining to mechanical behavior of magnesium has focused on understanding tension/compression asymmetry [2,3], stress–strain response [4–6] and texture changes [7,8]. By contrast, few studies have been devoted to investigating the fracture response of Mg.

Deformation twinning is particularly important in crystals of lower symmetry (e.g., HCP metals), where the Von Mises criterion for general deformation may not be satisfied due to the absence of five independent slip systems. Kelley and Hosford [5] and Wonsiewicz [4] investigated the formation of different types of twins along with slip activities by conducting channel die compression experiments on magnesium single crystals corresponding to various orientations. They concluded that extension of *c*-axis is predominantly accommodated by $\{10\bar{1}2\}$ tensile twins (TTs), owing to their low critical resolved shear stress (CRSS), while contraction of *c*-axis is accommodated through pyramidal $\langle a+c\rangle$ slip along with $\{10\bar{1}1\}$ contraction twins (CTs). This claim was further substantiated by Yoo [9] who indicated that only $\{10\bar{1}2\}$ tensile twin-type and $\{10\bar{1}1\}$, $\{10\bar{1}3\}$ contraction twin-types are active in Mg. Recent experimental work has focused on polycrystalline magnesium alloys owing to their application in structural components. Studies by Barnett [2] on alloy AZ31 have indicated that extensive tensile twinning may enhance ductility of the alloy. Further, experimental observations by Knezevic et al. [3] on AZ31 have shown that formation of CTs causes strain hardening while TTs are found to contribute very little to strain hardening. Also, they reiterate that extensive formation of TTs enhances ductility.

In magnesium alloys the fracture toughness can be as low as 7–20 $\text{MPa}\sqrt{\text{m}}$ [1,10] which will impede their application as structural components. Therefore, the operative fracture mechanisms and crack growth resistance of magnesium alloys need to be carefully investigated. To understand whether cleavage of basal {0001} plane [11] and prismatic $\{10\bar{1}0\}$ plane [12] is observed in magnesium, Reed-Hill and Robertson [13] performed tension experiments on Mg single crystals with loading axis on the basal plane. These experiments showed parting along $\{30\bar{3}4\}$ habit plane unlike the commonly observed basal cleavage in HCP metals. Further, they noted that for the crystal orientations considered in their work, the fracture mechanism is unaffected by the presence of $\{10\bar{1}2\}$ twins. From their channel die experiments, Kelley and Hosford [5] reported reduced ductility in Mg single crystals oriented for compression along *c*-axis and attributed it to the formation of $\{10\bar{1}1\}$ twins.

Yan et al. [1] noted that there is a transition in fracture mechanism from brittle to ductile with decrease in constraint level in AM60 Mg alloy. Similar transition was observed by Mukai et al. [14] in AZ31 Mg alloy with refinement in grain structure. Somekawa et al. [15,16] conducted fracture experiments on coarse-grained

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http://dx.doi.org/10.1016/j.msea.2013.10.018

(grain size of 50 μm) and fine-grained (grain size of 5 μm) AZ31 Mg alloy, respectively. They noted that in the fine-grained alloy, sub-grain structures form near the crack tip promoting blunting and ductile fracture occurs by void growth and coalescence. On the other hand, the formation of TTs near the crack tip in the coarse-grained alloy causes premature crack growth along the twin–matrix interface leading to slightly lower fracture toughness. Somekawa et al. [15] attributed the reason for crack growth along the twin–matrix interface to dislocation pile up at the twin boundary and incompatibility in the strains at the interface. By contrast, experiments by Yu et al. [17] on Mg single crystals indicated that nanotwins formed near the crack tip to shield it and promote crack blunting. Similar observations of crack blunting due to formation of tensile twins were made by Govila [18] in Be single crystals.

In order to obtain a clear understanding of the fracture behavior of Mg, a systematic study of interaction of tensile twins with a notch or crack tip under mode I loading needs to be conducted. Also, the dependence of lattice orientation with respect to the notch surface or crack plane on twin evolution and fracture resistance should be examined. These issues may be better studied using Mg single crystal fracture specimens rather than with a polycrystalline Mg alloy. Such a study will allow for tracking the evolution of individual twins, their interaction with neighboring twins of the same and different variants and also with a notch tip. To this end, systematic fracture experiments using pre-notched three point bend specimens of Mg single crystals are conducted within a scanning electron microscope (SEM). Specimens having three different crystallographic orientations are considered. In two of the orientations, the normal to the flat surfaces of the notch coincides with the *c*-axis, whereas in the third the notch front is aligned along the *c*-axis. In situ observations on the evolution of twins near the notch are made using electron backscattered diffraction (EBSD). Also, optical metallography is performed on the unloaded specimens to examine the slip and twin traces and the crack path. Fractographic observations are conducted to understand the operative fracture mechanism. The results show that in all orientations studied, profuse TT formation occurs ahead of the notch tip and contributes to toughening. However, crack growth occurs along boundary of a prominent twin and gets deflected at twin–twin intersections. In a follow-up work [19], finite element simulations are conducted to provide further insights on the mechanics of fracture of Mg single crystals.

2. Experimental procedure

2.1. Specimen details

The experiments are conducted using edge-notched three point bend (TPB) specimens as shown in Fig. 1. Three orientations are chosen in this study. In the first and second orientations, referred to in the sequel as orientations A and B, the normal to the flat surface of the notch (X_2-axis) is aligned along [0001]. In orientation A, the notch front (X_3-axis) is along $[1\bar{2}10]$ direction and the crack growth direction (X_1-axis) is along $[10\bar{1}0]$. In orientation B, the above-mentioned notch front and crack growth directions are interchanged. Orientation C mimics the basal-textured magnesium alloys with the *c*-axis along X_3, while $[1\bar{2}10]$ and $[10\bar{1}0]$ are along X_2 and X_1, respectively. The dimensions of the tested specimens pertaining to the three orientations are summarized in Table 1.

2.2. Specimen preparation and test set-up

The specimens are cut using electric discharge machining (EDM) from short cylinders of Mg single crystals, grown from seed crystals. A notch of radius $r_0 = 200$ μm is machined using EDM up to the center of each specimen along the width. The specimens are chemically polished with 83% ethanol, 10% HCl, and 7% HNO_3, followed by etching and electropolishing. After preparing the specimens, EBSD scans in the form of inverse pole figures (IPF) and image quality (IQ) maps are obtained to check the correctness of the initial lattice orientation.

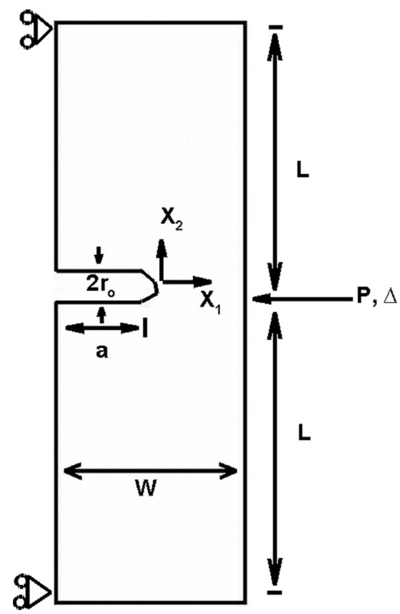

Fig. 1. Schematic of three point bend (TPB) specimen along with the notations used to denote the various dimensions.

Table 1
Various dimensions of the specimens (see Fig. 1) pertaining to the three orientations considered in the study.

Orientation	$2r_0$ (μm)	W (mm)	a (mm)	L (mm)	Thickness t (mm)
A	400	4.0	2.0	12.0	1.63
B	400	4.0	2.2	12.0	1.50
C	400	4.0	2.0	12.0	1.70

The experiments are carried out in LEO-1550 SEM so that it is possible to make detailed in situ observations of crack initiation and growth. It is coupled with EBSD hardware to obtain scans at intermediate loading stages. This hardware, obtained from EDAX, USA, has a resolution of up to 1 μm supplemented with high speed cameras to expedite the data acquisition. The SEM is also fitted with a miniature three point bend (TPB) stage procured from DEBAN, UK, to conduct the fracture tests. The TPB stage is mounted in the SEM such that it is tilted at an angle of 70° with respect to the horizontal plane. This angle is optimum for acquiring good quality EBSD patterns. It is ensured that the load on the specimen remains at zero before conducting the experiment. The load cell of the TPB stage has a maximum capacity of 200 ± 0.2 N. A lead-screw attached to the loading pin, threaded clockwise at one end and anti-clockwise at the other imparts displacement to the loading pin at a rate between 0.1 mm/min and 1.5 mm/min. For the present experiments, a displacement rate of 0.1 mm/min is used. The test is interrupted at different stages to capture EBSD data and SEM images in the loaded condition. It has been noticed that slip traces on single crystal specimens can be better visualized using an optical microscope [20,21]. Hence, in some cases the specimen is unloaded and taken out in order to observe it using an optical microscope. The optical images are taken using Zeiss Axio Vert.A1 microscope. The surface profilometry was conducted using a Vecco profilometer.