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On the diversity of the plastic response of magnesium in plane strain compression



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ABSTRACT

Mg single crystals of selected 'hard' and 'soft' orientations were deformed at ambient temperature in channel-die compression. Specimens with a 'hard' basal starting orientation displayed a limited ductility and fractured along {11 $\overline{2}4$ } planes. Compression perpendicular to the *c*-axis of orientations well aligned for {10 $\overline{1}2$ } extension twinning revealed a very contrasting deformation behavior. Specimens compressed along the crystallographic (11 $\overline{2}0$) direction exhibited an exceptionally high room temperature ductility, whereas compression along (10 $\overline{1}0$) resulted in failure after an initial sigmoidal stress–strain response. In both cases {10 $\overline{1}2$ } extension twins were found to fully consume the matrix at low strains and convert the starting orientations into orientations corresponding to the respective high Schmid factor twin variants. High strains have also been attained for a prismatic starting orientation where the *c*-axis was constrained. In specimens, favorably oriented for basal slip, anomalous {10 $\overline{1}2$ } extension twins formed, clustered in bands parallel to the constraint direction. These twins produced a strain opposite to the imposed plane strain deformation and provided a pronounced dynamic grain boundary strengthening effect.

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1. Introduction

Owing to its hexagonal structure and the tendency towards prevalent sharp textures, magnesium displays a radically anisotropic deformation behavior. A fundamental understanding of the orientation dependent deformation mechanisms and microstructure evolution on the basis of individual grains is therefore key to deduce and predict the deformation texture, and ultimately the mechanical behavior of a polycrystalline aggregate. Pioneer studies concerning this topic were performed nearly 60 years ago on single crystals to quantify the operating slip and twinning mechanisms [1–4]. Nevertheless, studies employing single crystals still provide a valuable asset to uncover the underlying mechanisms of deformation and recrystallization due to the model case nature of single crystal experiments [5–12].

In general, grain orientations in Mg can be classified into 'soft' or 'hard' orientations, depending on whether they are initially favorably oriented for basal slip or not. However, as will be discussed in what follows, not all 'hard' orientations inherit the same deformation behavior. While compression along or perpendicular to the *c*-axis is expected to yield a contrasting response due to

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http://dx.doi.org/10.1016/j.msea.2015.10.108 0921-5093/© 2015 Elsevier B.V. All rights reserved. activation of different twin types, i.e. contraction or extension twinning, a mere rotation around the *c*-axis can also greatly impact the selected twin variants. Moreover, the mechanical response of individual orientations (single crystal specimens) is liable to change drastically during straining, concurrently with a conspicuous change of microstructure. With the advent of modern characterization techniques, new possibilities were evolved to effectively study these microstructural changes in detail. Thus, to cover a wide range of deformation scenarios, specimens of all orientations were deformed up to large strains in the present study, where it was assured that no failure occurred.

Another point of interest in plasticity is the slip induced orientation change. The emergence of a 'hard' basal texture component due to basal slip in 'soft' orientations is known to account for reduced room temperature ductility of polycrystalline Mg in plane strain deformation. Mechanisms for avoiding the formation of a 'hard' basal component are therefore of crucial value.

The present work focused on providing an overview and comparison of the mechanical behavior as well as microstructure evolution of Mg single crystals with 'hard' as well as 'soft' orientations, deformed up to large strains in plane strain compression at ambient temperature. The role of $\{10\overline{1}2\}$ extension twinning with respect to the texture evolution and its impact on ductility is discussed in detail.

2. Experimental procedure

Plane strain compression (PSC) tests were conducted on magnesium single crystals using a channel-die as illustrated in Fig. 1a. Conically shaped single crystals with a base diameter of 34 mm, a length of 56 mm and an opening angle of 4° (Fig. 1b) were grown in a steel mold under argon atmosphere using specifically oriented single crystal seeds. While the top part of the mold was made from stainless steel, the steel used for the bottom part which was in contact with the molten Mg did not contain Ni to prevent contamination. Mg of commercial purity (min. 99.95%) was used as the starting material. The chemical composition of a grown crystal is given in Table 1. Each grown single crystal was attached to a goniometer and aligned according to the desired final orientation of the specimens using the Laue X-ray back-diffraction method [13].

Cuboid specimens with dimension $14 \text{ mm} \times 10 \text{ mm} \times 6 \text{ mm}$ were cut by means of electrical discharge machining. Crystals with different 'hard' orientations, including compression along and perpendicular to the c-axis as well a 'soft' orientation with the caxis inclined at an angle of 45° to the compression direction, were tested. The misalignment between the crystallographic directions and the specimen axes i.e. compression (CD), longitudinal (LD) and transverse direction (TD) of the channel-die were less than 1°. All PSC experiments were performed at ambient temperature and a constant strain rate of 10^{-3} s⁻¹ using a conventional screw-driven ZWICK testing machine. The single crystal specimens were deformed up to various logarithmic (true) strains, defined by $\epsilon_t = \ln(1 + \epsilon)$, where ϵ is the engineering strain. The applied force and displacement in CD were monitored and automatically regulated by a computer equipped with a data acquisition system. In order to reduce friction hydraulic oil was used for lubrication.

Characterization of the microstructure in the mid-surface of the LD-TD plane was performed by means of optical microscopy using polarized light. Several micrographs covering the whole specimen surface were acquired manually at low magnifications and stitched together using a specially developed in-house software to obtain a complete macroscopic image of the investigated plane.

Electron backscatter diffraction (EBSD) measurements to identify the type of twins by their characteristic orientation relationships were conducted on a LEO 1530 scanning electron microscope equipped with a field emission gun and an HKL-Nordlys II EBSD detector. The Matlab toolbox MTEX [14,15] was employed to process the EBSD data and calculate the displacement gradient tensors for the respective twin variants.

3. Deformation anisotropy and microstructural evolution of magnesium single crystals

As depicted in Fig. 2, the investigated single crystal specimens with different initial orientations displayed a very contrasting mechanical behavior during PSC deformation. During *c*-axis compression (orientation A in Fig. 2), the samples failed after reaching a true strain of -7.8% at a flow stress of 310 MPa. The deformation behavior of crystals with this orientation at elevated temperatures is reported in [16]. Owing to the alignment of the basal plane with respect to the compression direction, basal slip was suppressed (Schmid factor m = 0). Fracture occurred along $\{11\overline{2}4\}$ planes as shown in Fig. 3. The $\{11\overline{2}4\}$ fracture plane has also been reported previously by Kelley and Hosford [4] for PSC of Mg single crystals as well as by Reed-Hill and Robertson [1] for tension at -190 °C. More recently, the same fracture plane has been observed during equal-channel angular pressing (ECAP) [9] and *c*-axis compression [10] of Mg single crystals at room temperature.

Unexpectedly, numerous $\{10\overline{1}2\}$ extension twins were found, particularly in the vicinity of cracked regions, despite the imposed *c*-axis compression. The appearance of these extension twins must however be attributed to unloading and residual stresses after failure [3,5].

Both orientations B and C represented the case of *c*-axis extension, i.e. they were favorably oriented for $\{10\overline{1}2\}$ extension twinning. For an in-depth review of the behavior of crystals with these starting orientations the reader is referred to [17–19]. These two orientations differed in essentially a 30° rotation around the *c*-axis. Despite this similarity, the deformation behavior of the respective specimens was decisively distinct. Specimens with orientation B failed at a true strain of -15.7% at 380 MPa, whereas samples with the starting orientation C showed extraordinary high room temperature ductility and deformed up to a true strain of -100%. Analogous to the case of orientation A, basal slip was suppressed for both starting orientations B and C. During early stages of deformation profuse $\{10\overline{1}2\}$ extension twinning was observed (Fig. 4). $\{10\overline{1}2\}$ extension twins consumed about half of the matrix at a true strain of -3% for both orientations B and C. In addition to those primary twins, secondary {1012} extension twins were found inside the primary ones for orientation C (Fig. 4b).

A major difference in the behavior of specimens with the starting orientations B and C at this point was in the selected twin variants. For orientation B the twinning planes were parallel to TD, as evident from the twin trace in Fig. 4a, while for orientation C the TD-LD twin traces were at an angle of $\pm 65^{\circ}$ to LD (Fig. 4b). As will be discussed further, the selected twin variants accounted for the contrasting deformation behavior of specimens with these two



Fig. 1. (a) Schematic illustration of the channel-die used for plane strain compression tests (LD-longitudinal direction, TD – transverse direction, CD – compression direction); (b) conically shaped magnesium single crystal (etched in dilute nitric acid).

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