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Mechanisms of exceptional ductility of magnesium single crystal during deformation at room temperature: Multiple twinning and dynamic recrystallization

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Abstract

Specially oriented magnesium single crystals were subjected to plane strain compression along the $\langle 11\bar{2}0\rangle$ direction in *c*-axis extension at ambient temperature. The samples deformed up to a logarithmic final strain of -1, illustrating exceptionally high formability, even though basal and prismatic slip were initially inhibited due to the loading orientation with respect to the deformation geometry. Macroscopic $\{10\bar{1}2\}$ extension twins forming massively during early stages of deformation completely consumed the whole sample, resulting in new soft orientations for slip. Additional twinning events took place in the form of secondary and tertiary twinning. At somewhat advanced stages of deformation, newly formed (recrystallized) grains were observed within numerous bands associated with former $\{10\bar{1}1\}$ contraction twins within the primary extension twinned matrix. Recrystallized grains were rotated around the *c*-axis of their parent twin by an average angle of 30°, resulting in a shift of orientations from the second type ($\langle 11\bar{2}0\rangle$) prismatic fiber of ideal twin orientations towards the first type ($\langle 10\bar{1}0\rangle$) prismatic fiber of recrystallized grains. This led to a substantial weakening of the texture intensity at the final strain, as well as a high frequency peak of 30° grain boundaries in the misorientation distribution. The obtained results are discussed with respect to the texture evolution during multiple twinning in conjunction with continuous dynamic recrystallization at room temperature.

Keywords: Magnesium; Single crystal; Texture evolution; Deformation twinning; Dynamic recrystallization

1. Introduction

The deformation of hexagonal close-packed (hcp) materials is typically very "orientation-dependent" owing to the strong directional anisotropy of the hcp single crystal. Despite the technological significance of hcp materials (i.e. Mg, Ti, Zr and Be) in numerous industrial applications, the crystallographic mechanisms of hcp plasticity are still poorly understood in various aspects. Compared to cubic metals such as Al and steels, the mechanical behavior of hcp metals is considerably more complicated due to the effects of deformation twinning. Deformation twins are utterly significant in hcp metals and alloys: They (i) provide an important strain accommodation mechanism out of the basal plane for needed general deformation, (ii) strongly modify the texture and the anisotropy and (iii) affect the hardening response by interacting with dislocations in complex ways [1]. In Mg alloys, the most commonly observed twin types are the $\{10\bar{1}2\}$ extension and $\{10\bar{1}1\}$ contraction twins, with the latter being much harder to nucleate due to energetic prospects of atomic shuffling [2]. Due to their low volume fraction, contraction twins do not cause texture changes during deformation but

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they can decrease the work hardening rate [3] and give rise to dynamic recrystallization (DRX) [4] owing to texture softening within the twinned material. In terms of ductility, the single most relevant feature of deformation twins is that unfavorably oriented grains for slip can be reoriented into a more favorable position upon twinning. Additionally, by the onset of secondary twinning within the primary twins (i.e. double twinning events), the total plastic strain can be increased considerably provided that the latter does not lead to void nucleation, as reported in Refs. [5–7].

In a previous study on magnesium "single crystals" subjected to plane strain compression (PSC) at ambient temperature, we examined the contribution of deformation twinning to fracture strength and ductility as a function of the loading orientation [8]. Controlled mechanical testing of single crystals vis-à-vis polycrystals has the advantage that more direct and quantitative information concerning the role of twinning in the development of microstructure and texture can be obtained. The reported results demonstrated a clear variety in the twinning response, reliant on the activated "twin type" and "twin variant". It was established that deformation twinning can effectively "strengthen" the material under some conditions, leading to high fracture strength and poor ductility (15% fracture strain or less) and "soften" it under others, giving rise to dynamic recrystallization and stable plastic flow, which led to outstanding ductility (logarithmic strain of -1 without failure). Significant strain accommodation took place by profuse "macroscopic" twinning events, whereby the twinned areas were reoriented into soft orientations for basal slip. Basal slip was naturally important for deformation, but it was also important for the development of new, dynamically recrystallized grains. The development of DRX grains at room temperature was based on the formation of subgrain boundaries and their progressive rotation to high-angle boundaries (commonly denoted continuous or rotation DRX), which is different from the classical view of discontinuous DRX involving nucleation and growth of new grains at the expense of the deformed microstructure [9]. Regardless of its nature, DRX is normally expected to operate at elevated temperatures, where non-conservative movement of dislocations is promoted. However, in addition to our own study [8], dynamic recrystallization has also been observed at room temperature during severe plastic deformation of a magnesium single crystal [10].

Since DRX generates new orientations, it can play a key role in avoiding the formation of a basal texture through randomization [11] as long as the orientation change is not simply limited to a rotation around the [0001] direction, i.e. there are a high number of grain boundaries with a random misorientation axis.

This work is focused on the deformation and recrystallization behavior of the highly ductile Mg single crystal [8]. The specimen was compressed along the $\langle 11\bar{2}0\rangle$ axis, such that extension occurred in the c-direction. The deformation experiments were terminated at select strains and the deformed specimens were systematically characterized by X-ray diffraction and electron backscatter diffraction (EBSD) measurements. This allowed identification of twin types and twin variants throughout the deformation, and obtaining explicit information on the evolution of texture and microstructure with respect to various twin generations and DRX. The data so collected shed conclusive light on the mechanisms responsible for the outstanding room temperature formability of the Mg single crystal.

2. Experimental procedures

Conically shaped single crystals of commercially pure (99.95%) Mg were grown in a stainless steel mold in a vertical Bridgeman furnace using specially oriented monocrystalline seeds (c-axis parallel to the growth direction). In order to measure the orientation of the monocrystalline seeds as well as the grown single crystals, the Laue X-ray back diffraction method [12] was utilized. The as-grown single crystals were placed on a goniometer in order to align the crystallographic axes with respect to the specimen coordinate system. For the PSC tests smaller specimens with dimensions of 14 mm × 10 mm × 6 mm were fabricated by means of electrical discharge machining from the specially oriented single crystals. Compression was applied parallel to the $\langle 11\bar{2}0\rangle$ crystal direction, whereas extension was confined to the c-axis. The misalignments between the [0001], $[11\bar{2}0]$ and $[10\bar{1}0]$ crystallographic directions in the specimens and the corresponding compression (CD), longitudinal (LD) and transverse directions (TD) of the channel-die were less than 1°.

Deformation of the single crystal specimens was carried out up to a logarithmic strain (hereafter referred to as true strain) of $\varepsilon_t = -1$; with $\varepsilon_t = \ln(1+\varepsilon)$, where ε is the engineering strain. All PSC tests were performed at ambient temperature and a constant strain rate of $10^{-3}\,s^{-1}$ using a conventional screw-driven ZWICK testing machine. During deformation, the variation of the applied force and displacement in CD was monitored by a computer equipped with an automated data acquisition system. In order to minimize friction in the contact area between the sample and channel-die, lubrication oil was used.

The microstructure on the mid-surface of the LD–TD plane was characterized by means of optical microscopy and EBSD within a LEO 1530 scanning electron microscope with a field emission gun operated at 20 kV. Sample preparation included soft grinding and diamond polishing, followed by electropolishing in a 5:3 solution of ethanol and H_3PO_4 for 30 min at 2 V. Chemical color-etching with a freshly prepared 1:1:7 solution of water, acetic acid and picric acid was performed for optical microscopy examinations using polarized light. Multiple micrographs of the whole specimen surface were acquired manually at 100 × magnification and stitched together to obtain a complete macroscopic image of the sample. X-ray pole figure (PF) measurements were conducted using a Bruker D8 Advance diffractometer, equipped with a high resolution