Deformation-Induced Recrystallization of Magnesium Single Crystals at Ambient Temperature

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Abstract. Specially oriented magnesium single crystals were subjected to plane strain compression along the $<11\bar{2}0>$ direction in $c$-axis extension at ambient temperature. The samples exhibited outstanding formability deforming up to a logarithmic final strain of $\approx 1$. Investigations by optical and orientation imaging microscopy revealed that massive $\{10\bar{1}2\}$ extension twinning at low strains consumed the whole sample and resulted in new soft orientations for slip. Observations also indicated that additional twinning took place in the completely twinned matrix by secondary and tertiary twinning events. At advanced stages of deformation newly formed, equiaxed small grains were observed within numerous bands related to former deformation twins. These “recrystallized” grains characterized by a low grain orientation spread of less than $1^\circ$ generated new orientations, which led to a substantial weakening and randomization of the texture during deformation up to very large strains. The reported results in this paper are discussed with regard to the microstructure evolution arising from multiple twinning and continuous dynamic recrystallization at room temperature.

1. Introduction

Due to their hexagonal closed packed (HCP) crystal structure with a limited number of independent slip systems, the ductility of wrought polycrystalline magnesium is usually poor at ambient temperature and the material shows strong anisotropy in mechanical behavior. For ambient temperature deformation the primary deformation modes available are usually basal slip and extension twinning. Both modes have critical resolved shear stress values of less than 5 MPa, and are hence very easy to operate at low temperatures [1-3]. In Mg alloys, the most commonly observed twin types are the $\{10\bar{1}2\}$ extension and $\{10\bar{1}1\}$ contraction twins, which can accommodate extension or contraction along the hexagonal ($c$) - axis. In practice, unlike extension twins, contraction twins in magnesium are rather scant. Hence due to their low volume fraction, they do not generally cause significant changes in the global texture during deformation but in certain cases they can give rise to continuous dynamic recrystallization (CDRX) [4] owing to crystallographic softening within the twinned material. Continuous DRX (also denoted rotation DRX [5, 6]) is based on progressive misorientation of subgrains during recovery until high angle grain boundaries are formed.

In terms of formability, one very important characteristic of deformation twinning is that grains unfavorably oriented for basal slip can be reoriented into a more favorable position upon twinning. In addition, by the onset of numerous double twinning events [7], the total plastic strain can be increased considerably provided that the latter does not lead to void nucleation, as reported by Hartt and Reed-Hill [8], who investigated the significance of deformation and fracture along $\{10\bar{1}1\}$-$\{10\bar{1}2\}$ double twins in polycrystalline Mg. Recent research has, however, indicated that not all types and variants of double twins provide equivalent void nucleation sites. It has been actually observed that voids are formed preferentially at boundaries between primary and secondary twins rotated around the same $<11\bar{2}0>$ axis [9]. It is thus pointed out that the formation of such voids, in principle, can be avoided if the factors responsible for twin variant selection were understood. For HCP crystal plasticity this criterion can be extremely important, as was observed in [10], where it was demonstrated that the variant selection of extension twins can greatly harden the material under certain loading conditions, leading to high fracture strength and limited ductility or soften it under others, giving rise to CDRX and very large ductility. The present work focuses on the deformation and recrystallization behavior of a magnesium single crystal compressed...
along the <11\(_2\)0> axis in a channel-die at ambient temperature, so as to form \{10\(_1\)2\} extension twins. The deformation experiments were terminated at predefined strains so that the microstructure and texture evolution could be tracked throughout the deformation as a function of imposed strain. The obtained results are discussed with respect to the multiplicity of deformation twinning and its consequent crystallographic softening effect that gave rise to deformation-induced recrystallization at ambient temperature.

### 2. Experiments

High-purity magnesium single crystals (99.95%), oriented with the basal plane parallel to the loading direction, were deformed in plane strain compression (PSC) at ambient temperature, such that the stress axis was parallel to the <11\(_2\)0> crystal direction and extension was confined to the c-axis. The misalignments between the crystallographic axes and the sample reference frame were less than 1°. All PSC tests were performed at a constant strain rate of 10\(^{-3}\) s\(^{-1}\) using a conventional mechanical testing machine equipped with an automated data acquisition system. For minimization of friction between the sample and channel-die, lubrication oil was used. The microstructure in the mid-layer of the compression plane was characterized by means of optical microscopy and electron backscatter diffraction (EBSD). EBSD measurements were performed with a scanning electron microscope equipped with a field emission gun and an HKL-Nordlys II EBSD detector. X-ray pole figure (PF) measurements were conducted using a Bruker D8 Advance diffractometer, equipped with a HI-STAR area detector. The MTEX toolbox [11] was employed to analyze and visualize the texture data collected by X-ray diffraction and EBSD methods.

### 3. Results and discussion

The mechanical response of the investigated monocrystalline specimen during room temperature PSC along the <11\(_2\)0> direction (c-axis extension) is reported elsewhere [12]. In summary, the specimen achieved a remarkable logarithmic strain of -1 without mechanical failure. During initial deformation the flow curve showed a sigmoidal work hardening regime consistent with predominant \{10\(_1\)2\} extension twinning at low strains. After reaching a peak stress of 220 MPa at 11% strain, there was an unexpected drop of stress with strain (flow softening) down to 200 MPa. During further straining the stress-strain curve depicted a nearly steady-state behavior consistent with the formation of new recrystallized grains. Fig. 1 shows the microstructure development of the originally monocrystalline specimen as a function of different strain levels from 3% to 100%. The EBSD maps are represented in terms of the Schmid Factor (SF) for basal slip considering a stress of \(\sigma\) and \(\sigma/2\) in the compression and transverse direction, respectively. The latter passive stress component takes into account the contribution of back stress due to the channel-die constraints. At 3% strain, extensive twinning was evident throughout the whole specimen. The twin area fraction was approx. 53 pct. Two opposite sets of \{10\(_1\)2\} twins were observed to emanate from one TD edge of the specimen (or from a preexisting twin boundary), and extend diagonally to the opposite side, occasionally penetrating other twins (Fig. 1a). The twinned areas exhibited characteristic orientations corresponding to crystallographic rotation of 86° about <11\(_2\)0> from the initial single crystal (parent) orientation. This rendered a large portion of the microstructure favorable for basal slip, as indicated by a significant increase of the SF for basal slip from zero to approx. 0.2. Within the large twins that seemed to form first (primary twins), secondary small twins of the \{10\(_1\)2\} extension type (for details see [12]) possessed even higher SF values of about 0.3. At 8% strain (Fig. 1b), the remaining parent microstructure was completely consumed by the \{10\(_1\)2\} primary twins. The only twins observed were the secondary twins that appeared isolated within a “new” parent matrix. Additionally, there were other small twins with very high SF values of approx. 0.45 that seemed to form inside the secondary twins (i.e. third generation \{10\(_1\)2\} twins). At 40% strain, the second generation of \{10\(_1\)2\} twins appeared to have undergone significant growth compared to what was observed at low strains (Fig. 1c). When reaching the final strain of 100%, the fraction of recrystallized
regions, characterized by fine equiaxed grains with grain orientation spread (GOS) values below 1°, was very prominent throughout the whole sample (Fig. 1d). Although not shown in Fig. 1, it is noted that the formation of recrystallized grains was already seen at 11% strain [12].

Fig. 1: EBSD maps of the deformed magnesium crystal colored according to the Schmid factor for basal slip. (a): 3%, (b): 8%, (c): 40%, (d): 100% strain.

Fig. 2 shows recalculated (0002) and \{10\(\bar{1}\)0\} pole figures that present the macrotexture development in correspondence with the microstructures given in Fig. 1. The initial texture of the single crystal prior to deformation corresponded to the orientation (\(\phi_1\), \(\Phi\), \(\phi_2\)) = (90°, 90°, 0°) in Euler space (Bunge notation). After 3% strain, primary \{10\(\bar{1}\)2\} twinning imparted significant texture changes, seen by two new texture components (0, 30, 0) and (180, 30, 0) with an angle of 30° between the direction of their respective c-axes and the compression direction (Fig. 2a). After 8% deformation the initial orientation of the monocrystalline matrix was completely converted into the new twinning orientations with soft slip conditions (Fig. 2b), which was crucial for the operation of further deformation mechanisms beyond primary twinning. With increasing strain up to 40% the twinning components appeared to accumulate some scatter and were shifted from the characteristic (0/180, 30, 0) orientations (Fig. 2c).

Fig. 2: X-ray texture development in the deformed magnesium single crystal as a function of strain. (a): 3%, (b): 8%, (c): 40%, (d): 100%. ODF calculations have been performed with a kernel half-width of 5°.
The significant growth of secondary $\{1012\}$ twins at this strain level gave rise to an additional weak texture component further away from the center of the basal pole figure. At the final strain of 100% the advanced state of DRX resulted in a massive decrease in texture intensity associated with significant texture spread around the twin orientations (Fig. 2d). Fig. 3 shows a representative example of a recrystallized region at 100% deformation, represented in terms of EBSD microstructures. From the SF map in Fig. 3a it is evident that the majority of recrystallized grains had a favorable basal slip condition. Fig. 3b depicts additionally, that these grains possessed a broad orientation spectrum that contributed to the observed weakening of the overall macrotexture. As revealed by the results reported in the current paper and elsewhere [10, 12], the crucial mechanism of the deformation-induced DRX was based on dynamic recovery characterized by high slip activity of dislocations in the soft twinned regions, and the formation of subgrains by dislocation rearrangement. These subgrains, as indicated in Fig. 3b, continuously transform into recrystallized grains with high-angle boundaries by means of subgrain coarsening and coalescence.

Fig. 3: (a) Schmid factor map for basal slip with slip trace overlay. (b) Grain boundary map with crystal lattice overlay. The color key corresponds to inverse pole figure coloring with respect to the compression axis. Light-colored boundaries 5°-15°, dark-colored boundaries > 15°.

4. Conclusions

Although easy slip modes were suppressed for the initial single crystal orientation investigated in this study, it was still possible to deform the specimens at room temperature up to 100% without failure. Initial deformation proceeded by profuse $\{1012\}$ extension twinning, converting the whole sample into softer orientations for slip and further twinning. This gave rise to deformation-induced dynamic recrystallization that led to a randomization of the overall texture, which was vital for enhancing the room temperature ductility.

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