Effects of recrystallization on texture, microstructure and mechanical properties in HPT-deformed pure Mg

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Abstract. Mg of purity 99.8 wt% was deformed by High Pressure-Torsion at hydrostatic pressures 1 to 4 GPa and RT, up to plastic shear strains of 120. X-ray texture analysis showed up deviations from expected shear texture, which increased with increasing shear strain and hydrostatic pressure. According to TEM and SEM investigations these deviations can be understood in terms of recrystallization. The current paper aimed at the differences of the recrystallization processes which occur during HPT deformation and unloading (dynamic recrystallization, DRX), and those after deformation (static recrystallization, SRX). For this purpose, two sorts of samples were investigated: (i) such being stored at RT immediately after HPT deformation, and (ii) such being stored at 77 K immediately after HPT deformation, and stored at RT for a minimum and constant time needed for preparation. The results show that SRX brings the texture closer to the ideal shear texture and to higher strength values, but to smaller ductilities than DRX does. The mechanical properties can be attributed to changes of texture rather than to those of grain size.

1. Introduction
Magnesium is considered to be a promising material for engineering in times when energy consumption reduction achieved by weight reduction gains much importance. By wide-range application of Mg and its alloys mostly in automotive and aircraft industry \cite{1, 2} a lot of attention has been devoted to investigations of the physical deformation behavior of magnesium. An efficient way to improve the mechanical properties of Mg is a significant reduction of grain size, which enhances its strength and can be achieved by methods of Severe Plastic Deformation (SPD) \cite{3}.

2. Experimental methods
Polycrystalline magnesium of technical purity (99.8 wt.%) samples were hot extruded (350°C) into rods of 8 mm diameter and then deformed by High-Pressure Torsion (HPT, \cite{4}) at hydrostatic pressures 1-4 GPa up to shear strains $\gamma=120$ \cite{5}. During HPT processing a Cu-ring has been attached in order to achieve homogeneity (for further details see \cite{6}).
X-ray texture measurements of HPT-deformed samples were performed on radial cross sections of the samples (RD, Fig. 1a) using a CuKα beam collimated to a spot size of 300 μm, being part of a BRUKER-AXS DISCOVERY D-8 diffractometer equipped with a GADDS area detector. Pole figures (0002), (1010), (1011) and (1012) were measured with Bruker-AXS D-8 Discovery diffraction system supplied with GADDS area detector. Complete pole figures were calculated from ODF by the Arbitrary Defined Cells (ADC) method [7] using monoclinic symmetrization using LaboTex 3.0 software.

Pole figures $\{0002\}$, $\{10\overline{1}0\}$, $\{10\overline{1}1\}$ and $\{10\overline{1}2\}$ were measured with Bruker-AXS D-8 Discovery diffraction system supplied with GADDS area detector. Complete pole figures were calculated from ODF by the Arbitrary Defined Cells (ADC) method [7] using monoclinic symmetrization using LaboTex 3.0 software.

Fig. 1. (a) Setup of X-ray texture measurement with respect to HPT sample geometry and shear directions (b) tension samples as prepared from HPT-deformed specimen. ND =Normal Direction = torsion axis, RD =Radial Direction, TD = Tangential Direction.

Tension tests were performed on samples cut from HPT material by spark erosion (as shown in Fig. 1b) at RT, at a constant strain rate $\varepsilon = 1.4 \cdot 10^{-3} [s^{-1}]$ and RT. TEM imaging was carried out with thin foils being prepared from the radial cross sections used for the texture investigations, within a TEM Philips CM200 operated at a voltage of 200kV (for further details see [5, 8]). EBSD investigations were carried out by a system HKL-OXFORD INSTRUMENTS being part of a SEM Hitachi S-3400N operated at 20keV.

3. Results and discussion

As reported in previous works [5, 8], the texture of pure magnesium after HPT reveals components expected for deformation by shear (‘B-fiber’, see [9]), but the positions of these components seems surprising – they are deviating from the position of ideal shear (by a certain angle $\alpha$ (Fig. 2cd)). This deviation is apparently caused by recrystallization during and after the HPT deformation, as it was confirmed by TEM investigations reported in [5, 8]. The deviation has been found to increase with the hydrostatic pressure applied during HPT which suggested but not proved that the deviation is arising from the dynamic part of recrystallization: While this dynamic recrystallization (DRX) is assumed to operate during HPT and the unloading process, the part of static recrystallization (SRX) is to occur afterwards, i.e. in entirely load-free condition. Special experiments have been performed in order to clarify the specific effects of DRX and SRX to microstructure and mechanical properties. For this purpose two sets of samples have been prepared investigated: (i) those which have been stored at RT after HPT processing for a few months as long as no change of microstructure has been observed (called ‘Recrystallized – RX’ samples in what follows), and (ii) those which have been stored at 77 K (in liquid nitrogen) immediately after HPT processing (called ‘Frozen – F’ samples in what follows). While the first set was expected to show mainly effects of static recrystallization (SRX), the second one should reflect mostly influences from dynamic recrystallization (DRX). Both groups of samples were subjected to the same investigations, such as X-ray diffraction, SEM and TEM imaging as well as tension tests, with a minimum and approximately constant time of RT storage required for sample preparation and investigations. The texture investigations of the F-type samples showed that deviations $\alpha$ of the B-fiber from ideal shear texture get even larger than that of the RX-type samples, confirming the suspicion that it is mainly the dynamic part of recrystallization which is responsible for the deviation $\alpha$ of B-fiber from its ideal shear position (see Fig. 3a, and [9]). From the same results,
however, it can be concluded also that the static part of recrystallization (SRX) exhibits a clear tendency to move the position of B-fiber towards the ideal shear position.

![Fig. 2](image)

**Fig. 2.** ‘Ideal’ shear deformation components (B-fiber) in HPT deformed Mg shown schematically by (a) (0001) and (b) (10\(\bar{1}\)0) pole figures [9], and experimental pole figures of (c) (0001) and (d) (10\(\bar{1}\)0) planes, with angle \(\alpha\) indicated which describes the deviation of the position of B-fiber from the ideal position.

These findings of effects specific to DRX and SRX seem to be correlated with the results of the tension tests. Fig. 3b compares the stress-strain characteristics obtained for both the RX-type and the F-type samples. The RX-type samples show a much higher strength, while at the same time their ductility is lower than that of the F-type samples. It seems that the static part of recrystallization (SRX) increases the strength whereas the dynamic one (DRX) is beneficial to the ductility. This can be explained if one takes a closer view on the microstructure of differently treated samples (please see [8] for complete results).

![Fig. 3](image)

**Fig. 3.** (a) Deviation angle \(\alpha\) indicating the deviation of B-fiber from its ideal (shear) position and (b) stress-strain characteristics of HPT-deformed Mg samples. Recrystallized (RX) samples show higher strength but lower ductility than ‘Frozen’ (F) ones. Data of initial Mg before HPT deformation are shown for comparison.

All HPT deformed samples of Mg reveal a strong bimodal microstructure with large, recrystallized grains beyond micron-size coexisting with submicron-size grains produced during severe plastic deformation (see [5]). However, irrespective of the pressure applied, RX-type samples show a much larger volume fraction of the recrystallized grains than the F-type samples, and their resulting average grain size is markedly larger than that of the F-type samples (see [8]). From both of those tendencies, one would expect a lower strength as well as a lower ductility of the RX-type samples compared to the F-samples, but the tensile tests tell a contrary story. From that it is concluded that the grain size and/or distribution does play only a minor role for the observed stress-strain characteristics and that it is the grain orientation rather than the grain size which govern the mechanical properties observed. Also if one considers the microstructures resulting from HPT deformation performed at different pressures (Fig. 4), these conclusions are confirmed. The average grain size \(d\) and its distribution is markedly...
larger in case of pressure relaxation after HPT at 4 GPa (d=1.8 µm) than after HPT at 2 GPa (d=2.4 µm), although the strength values demonstrated in Fig. 3b is larger and the ductility is lower.

Fig. 4. EBSD images of RX-type samples after HPT deformation at different hydrostatic pressures: Left hand side: 2GPa; right hand side: 4GPa.

4. Summary and Conclusions
Commercial purity magnesium subjected to HPT deformation at room temperature develops textures close to those expected for pure shear deformation, but distinct deviations occur due to recrystallization. Particular experiments have been performed which showed that it is mainly the dynamic part of recrystallization which is responsible for the deviation from ideal shear texture, and that the static part taking place after dynamic recrystallization turns the texture closer to the ideal shear position. The behaviour of mechanical properties are in line with that picture: While the dynamic recrystallization lowers the strength but increases ductility, the static recrystallization after HPT processing enhances the strength and decreases the ductility which can be explained by the large differences in texture during dynamic and static recrystallization rather than the variations in grain size and/or distribution.

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