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B. J. BONARSKI*,**, B. MIKUŁOWSKI*, E. SCHAFLER**, CH. HOLZLEITHNER**, M. J. ZEHETBAUER **

CRYSTALLOGRAPHIC TEXTURES OF SINGLE AND POLYCRYSTALLINE PURE Mg AND Cu SUBJECTED TO HPT DEFORMATION

TEKSTURA KRYSTALOGRAFICZNA MONO- I POLIKRYSZTAŁÓW Mg ORAZ POLIKRYSZTAŁÓW Cu ODKSZTAŁCONYCH METODĄ HIGH PRESSURE TORSION (HPT)

The main feature of processing metals by *Severe Plastic Deformation* (SPD) is the achievement of an ultra fine or even nanometre sized grain structure. Among the common SPD methods, *High Pressure Torsion* (HPT) is suited best because of the high amount of hydrostatic pressure (HP), which also allows reaching of very high deformations. The intention of this work was to systematically investigate the effect of HPT deformation on the evolution of texture at different HPs. Such an effect may arise from the marked presence of edge dislocations affecting the slip geometry but at the same time being sensitive to the hydrostatic pressure. Magnesium has been chosen since only a few investigations of texture evolution in SPD metals are available for hexagonal lattice.

As Mg based metals are quite soft, RT deformation by HPT is difficult. Thus, a special HPT tool has been designed which prevents from uncontrolled material flow and provides conditions of real hydrostatic pressure. Single- and polycrystalline samples of Mg and Cu have been deformed to shear strains $\gamma = 0.4\ldots 150$ at HPs between 1 to 8 GPa. Crystallographic textures which developed during HPT of Mg single- and polycrystals, and Cu polycrystals are presented. Concomitantly the flow stress during HPT could be measured. The results are discussed in terms of their dependence on strain and hydrostatic pressure. Increasing the latter both the variations of textures and flow stress are shifted to lower shear strains. In HPT deformation of Mg there are also some indications for the occurrence of dynamic and/or static recrystallisation which does not come true for the case of Cu.

Keywords: magnesium, copper, single crystals, HPT, high pressure torsion, texture

Główną zaletą odkształcania metali metodami ze zmienną drogą deformacji, tzw. SPD (ang. Severe Plastic Deformation) jest możliwość osiągnięcia struktury o bardzo małym rozmiarze ziarna, nawet rzędu nanometrów. Pomiędzy kilkoma metodami SPD, za najbardziej efektywną uważana jest metoda skręcania pod wysokim ciśnieniem hydrostatycznym – HPT (ang. High-Pressure Torsion), która pozwala na odkształcanie metalu do najwyższych możliwych obecnie wielkości. Celem niniejszej pracy jest zbadanie zmian struktury oraz tekstury krystalograficznej w mono i polikrystalicznym magnezie oraz polikrystalicznej miedzi odkształcanych metodą skręcania pod wysokim ciśnieniem hydrostatycznym (HPT) w temperaturze pokojowej, przy różnych wartościach ciśnienia hydrostatycznego zastosowanego podczas procesu odkształcenia. Wpływ zmiany ciśnienia odkształcania na teksturę spodziewany jest ze względu na obecność dyslokacji krawędziowych w dużej gęstości, które oddziaływują na geometrię poślizgu, będąc jednocześnie czułe na wielkość ciśnienia hydrostatycznego. Badania prowadzone były na magnezie technicznej czystości, ponieważ niewiele jest wyników tego typu doświadczeń dla metali o strukturze heksagonalnej.

Ponieważ magnez jest relatywnie miękkim metalem, odkształcanie go w temperaturze pokojowej do wysokich wartości odkształcenia rzeczywistego nie zawsze jest możliwe w temperaturze pokojowej, specjalne narzędzie ograniczające niekontrolowany wypływ materiału zostało zaprojektowane oraz z powodzeniem zastosowane. Monokryształy Mg oraz polikrystaliczny Mg i Cu odkształcono do wartości odkształcenia rzeczywistego od 0.4 do 150 przy zastosowaniu ciśnień hydrostatycznych o wielkości od 1 do 8 GPa w temperaturze pokojowej. Opisano tekstury krystalograficzne, które wykształciły się w tak odkształconych metalach oraz omówiono ich zależności w funkcji zastosowanego ciśnienia oraz wielkości nadanego odkształcenia. Komponenty tekstury charakterystyczne dla odkształcenia metodą HPT obserwowane są wcześniej w funkcji odkształcenia przy zastosowaniu wyższego ciśnienia hydrostatycznego. W przypadku magnezu zaobserwowano występowanie składowych pochodzących od rekrystalizacji statycznej/dynamicznej, czego nie stwierdzono w przypadku miedzi.

* FACULTY OF NON-FERROUS METALS, AGH – UNIVERSITY OF SCIENCE AND TECHNOLOGY, 30-059 KRAKÓW, 30 MICKIEWICZA AV., POLAND
** UNIVERSITY OF VIENNA, PHYSICS OF NANOSTRUCTURED MATERIALS, FACULTY OF PHYSICS, BOLTZMANNGASSE 5, A-1090 WIEN, AUSTRIA

1. Introduction

During the last years, Severe Plastic Deformation (SPD) has gained increasing importance in achieving bulk nanostructured metals and alloys [1, 2]. In comparison to conventional nanocrystalline materials, those processed by SPD exhibit not only enhanced strength values but also considerable extents of ductility [3]. In contrast to ECAP and most of the other SPD methods known, High Pressure Torsion (HPT) offers a number of advantages which are (a) the precise control of important deformation parameters such as the hydrostatic pressure and the deformation induced strain, (b) the possibility to measure in-situ SPD stress and strain characteristics, and (c) the realization of highest hydrostatic pressures and strains of all known SPD methods [4]. As concerns the SPD processed materials studied so far, hexagonal metals have been investigated much less than cubic ones [5], not at least because of the strong anisotropy of slip systems acting in them [6].

This work aimed at the investigation of microstructure, namely of textures of HPT deformed Magnesium and Copper. HPT textures may develop differently to those of conventional torsion deformation, due to the presence of marked hydrostatic pressure affecting the slip geometry because of interaction with edge dislocations being present in high densities. Moreover, for the hexagonal nanostructured materials it is expected that deformation induced changes of texture will have much more influence to the macroscopic mechanical properties, due to the high anisotropic behavior of slip systems [7, 8]. These effects have to be carefully separated from other influences like changes of grain size, dislocation and vacancy density when aiming at the clarification of strength and ductility beneficial effects.

For better understanding of the texture evolution in HPT deformed polycrystalline materials, Mg single crystals with certain orientations have been investigated, too [9, 10].

2. Materials and methods

Materials investigated. Single- and polycrystal samples of Magnesium with 99.8wt% purity have been investigated after deformation by High Pressure Torsion (HPT). For the experiments with polycrystals, materials of two initial states have been used. (i) Mg as-cast, with grains of average size of ~1 mm, and (ii) hot extruded Mg (at 350°C) with average grain size of ~20 μm. Hot extruded Mg revealed a strong texture typical of extrusion with basal slip {0001} perpendicular to the extrusion axis (and sample surface) and prismatic slip $\{10\bar{1}0\}$ parallel to the surface.

For the single crystal experiments, crystals were grown by using a modified Bridgman method. A special furnace allowing slow directional crystallization of Mg from liquid phase has been projected and constructed. Using a technique for directed crystallization, Mg single crystals could be grown, two different orientations of Mg single crystals have been achieved which were subjected to HPT deformation and texture investigation.

Samples of polycrystalline Cu have been gained from extruded rods of 99.99wt% purity, and 20 mm in diameter. Then the samples were mechanically machined and shaped for HPT deformation (see below). Before HPT experiments, samples have been annealed at 600°C for 1 h.

High Pressure Torsion deformation. For the HPT experiments, all samples have been cut by means of spark erosion to discs with 6 mm in diameter for Mg-single crystals, 10 and 14 mm for Mg-polycrystals, and 8 mm for Cu, with thicknesses between 0.7 and 2 mm. HPT experiments were carried out at the Erich Schmid Institute in Leoben (Austria). In-situ flow curves of HPT deformation have been recorded during the deformation process by means of continuous torsion torque measurement. Single crystal samples of Mg have been deformed under hydrostatic pressure of 1.5 GPa up to 0.75 rotations, whereas polycrystalline ones were deformed at hydrostatic pressures from 1 up to 4 GPa, with 0.5 to 4 rotations. The Cu samples have been deformed at 8 GPa, too. The rotation of plungers has been achieved continuously in one direction, by a constant rotation speed of 0.2 rot/min. All samples have been deformed at ambient temperature.

While the HPT deformation of single crystals could be achieved well, those with polycrystals revealed some experimental problems. After applying 2 or more rotations, the deformation became inhomogeneous, and much of deformed material flowed out of the plungers, compare Fig. 1a. In this figure one can recognize the inhomogeneities of deformation being represented by some shift of the upper part of sample with reference to the lower one, suggesting the occurrence of inner cracks. In order to avoid these problems, a Cu ring has been attached to the sample (see Fig. 1b) which prevented the flow of material out of the plungers, and allowed to deform pure Mg up to 4 rotations much more homogeneously The success of this technique is also documented by the characteristics of in-situ flow stress curves (Fig. 2), which show a markedly higher stability of flow stress when the Cu ring has been attached.

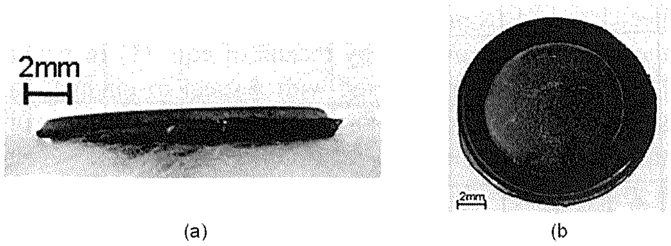

Fig. 1. HPT Magnesium sample without (a) and with (b) Cu ring attached.

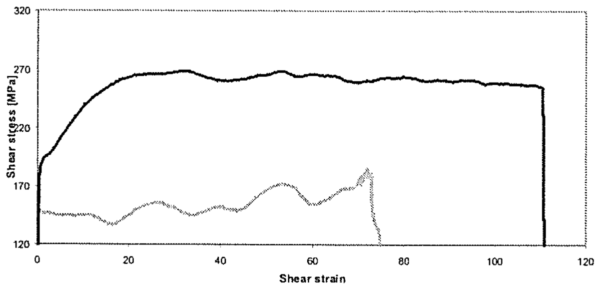

Fig. 2. In-situ recorded HPT flow curves, during deformation of polycrystalline Mg at 2 GPa. The grey (lower) curve indicates deformation instabilities, which is not the case with the black (upper) one, because of Cu ring attached

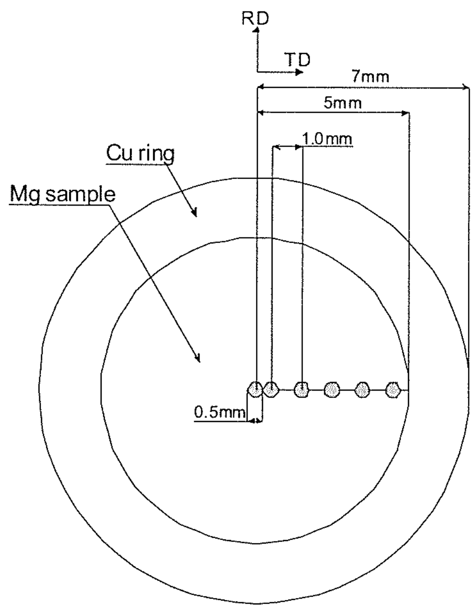

Fig. 3. Spots of texture investigations at the surface of HPT deformed Mg sample with Cu ring attached

Texture measurements. The crystallographic textures have been investigated at the sample surface normal to torsion axis, at points along the sample radius, with different distances from the sample centre by steps of 1.0 mm, (see Fig. 3). For X-ray diffraction a beam collimator of 0.5mm in diameter was used, with monochromatic X-ray radiation CuKα (λ = 0.1542 nm). Pole figures $(0002), (10\bar{1}0), (10\bar{1}1)$ and $(10\bar{1}2)$ have been registered using a GADDS area detector being part of a Bruker AXS D-8 wide angle diffraction system. Inverted pole figures were recalculated from Orientation Distribution Functions (ODFs). The ODF has been calculated without symmetrization using the ADC method with LABOTEX texture analysis software. For the investigation of HPT Cu, pole figures of types (111), (200), (220), (311) were recorded [11, 12, 13].

3. Results and discussion

3.1. Texture evolution in Mg single crystals in HPT deformation

Two different orientations of Mg single crystals were prepared for HPT deformation: $(10\bar{1}2)$ and $(11\bar{2}2)$ (denoted "I" and "II" respectively), as shown in Fig. 4. Deformation has been taken at a hydrostatic pressure p = 1.5 GPa. 4 samples with the same initial orientation have been subjected to HPT, by 0.1, 0.2, 0.3 and 0.5 rotations, respectively. Texture measurements were carried out at points along the sample radius as described in chapter 2 (please refer to Fig. 3). The respective values of shear strain γ of those points linearly depend on the radius distance from the sample centre r, according to

$$\gamma = \frac{2\pi \cdot n \cdot r}{h}; \qquad (1)$$

where n is the number of rotations and h the sample height after deformation.

Single crystals samples were deformed with an only limited number of rotations (up to 0.5), as the main structure and texture changes take place at the very first stages of HPT deformation. Those can be seen in Fig. 5, where the texture already exhibits a significant basal component. From point of plastic deformation, this basal component ((0001) fibre) indicates easy glide on basal planes (0001).

The analysis of texture after deformation (see Fig. 5 and 6) showed that orientation "II" tends to remain stable with increasing shear strain. There are no significant texture changes, although additional deformation components arised. In contrast, orientation "I" reveals a strong texture change already when applying an external pressure to the sample, indicating that this pressure is not yet hydrostatic. With the onset of torsional deformation s those texture components, which are also characteristic of orientation "II" are becoming stronger. With highest strains another change of texture is observed, probably because of the onset of dynamic or static recrystallization. Similar evidence is shown by the textures of highest strains of the single crystals with initial orientation "II"

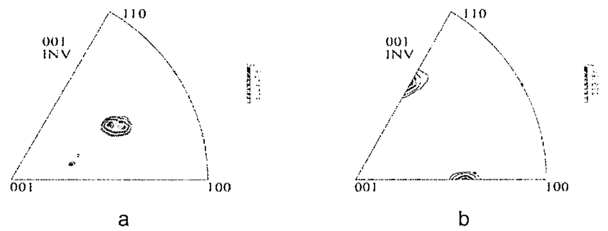

Fig. 4. . Initial Mg single crystal orientations "I" (a) and "II" (b)

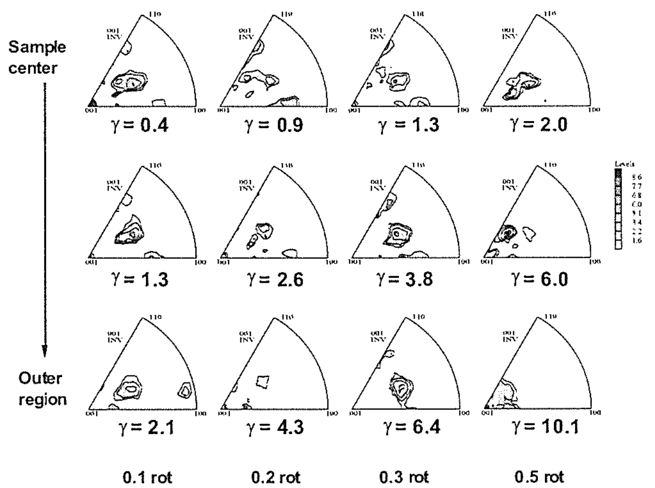

Fig. 5. Evolution of texture during HPT of Mg single crystals of orientation "I". For each texture, values of shear strains calculated by equ. (1) are given

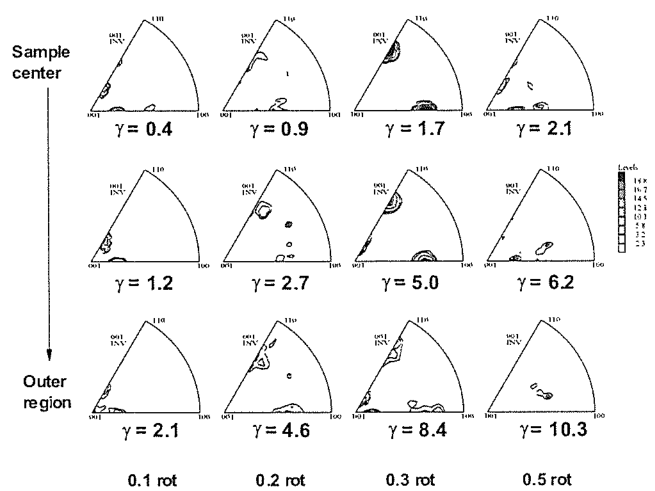

Fig. 6. Evolution of texture during HPT of Mg single crystals with orientation "II". For each texture, values of shear strains calculated by equ. (1) are given

Figs. 7 and 8 show in-situ recorded flow curves of Mg single crystals during HPT deformation. Curves have been derived from torsion torque measurements during the deformation process by means of equ. (1) in paper [14], and were recalculated with respect to changes in sample height during deformation. The shear strains of the curves represent the maximum strains achieved in a given sample – calculated for the outer sample area. To compare the strains for the positions where pole figures were measured. As it has been already expected from the orientations, one finds clear differences in the flow curves of single crystals of orientations 'I' and 'II'. While orientation 'I' plays the role of a "soft" orientation and reveals a low and almost constant level of stress (ca. 160 MPa), orientation 'II' shows marked variations in stress level, but all of them being markedly larger than those of orientation I (up to even 400 MPa). Thus, orientation 'II' activates slip systems which are quite different from those of orientation I, and will therefore be called 'hard' orientation in what follows. In contrast to the flow stress curves of crystals with orientation I, those with orientation II are differing very much. The reasons for those differences may lie in the limited accuracy of orientation directed crystal growth procedure which has much more effect to the stress in "hard" orientations than to that in "soft" ones.

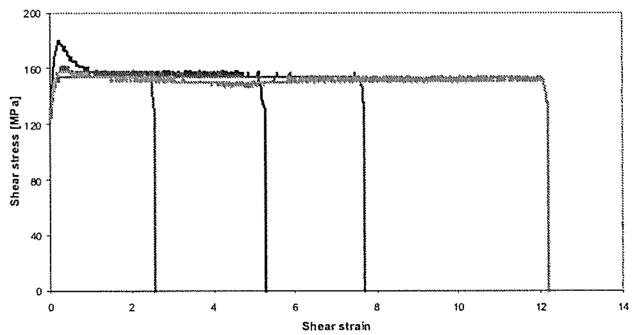

Fig. 7. Shear-stress curves of HPT deformation of Mg single crystal – orientation "I" at 1.5 GPa

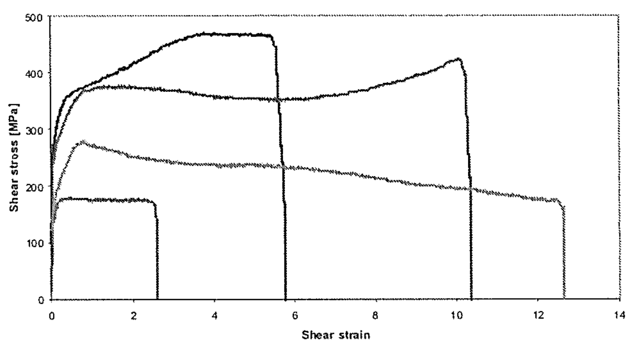

Fig. 8. Shear-stress curves during HPT deformation of Mg single crystal – orientation "II" at 1.5 GPa

3.2. Texture evolution in Mg Polycrystals during HPT deformation

For the experiments with polycrystalline Mg materials two initial states were used, in the as-cast and in the hot extruded (at 350°C) state, respectively. The average initial grain size of as-cast Mg was 1 mm, that of the extruded Mg 20 μm. While the texture of as-cast Mg initial texture was almost random, that of extruded material revealed a strongly axial texture, typical of extrusion processing (see Fig. 9).

Textures of polycrystalline samples after HPT deformation are similar for both as-cast and hot extruded material. The initial grain size and initial texture seem to have no big influence on texture after such high deformation degrees as they have been applied to polycrystalline Mg where the structural fragmentation proceeds much faster than in single crystalline samples. Up to about a shear strain of $\gamma \approx 50$ there are still traces of the initial texture which, however, disappear almost completely, and the basal component (0001) emerges. Further deformation up to very high strains does not show any components left from the initial material state.

Careful analysis of inverted pole figures of deformed Mg reveals an influence of applied pressure on the strain dependent evolution of deformation texture. It seems that increasing hydrostatic pressure advances the development of components which would have arisen under low pressure conditions at higher strains only.

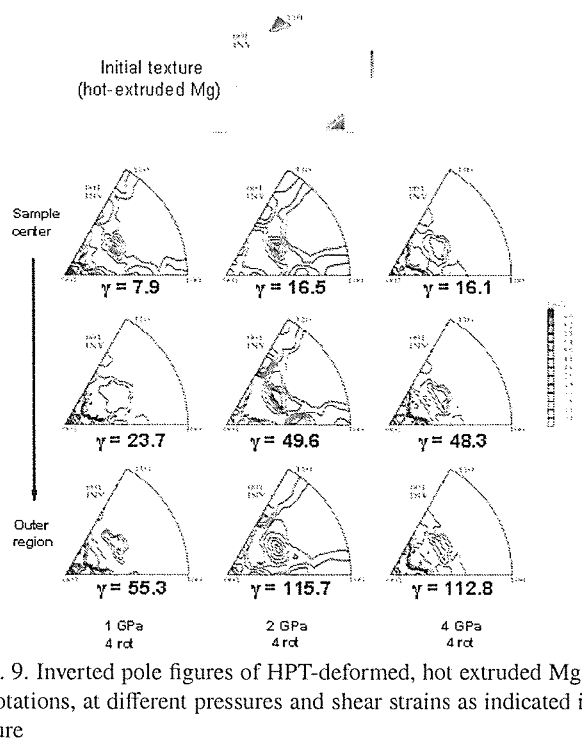

Fig. 9. Inverted pole figures of HPT-deformed, hot extruded Mg after 4 rotations, at different pressures and shear strains as indicated in the figure

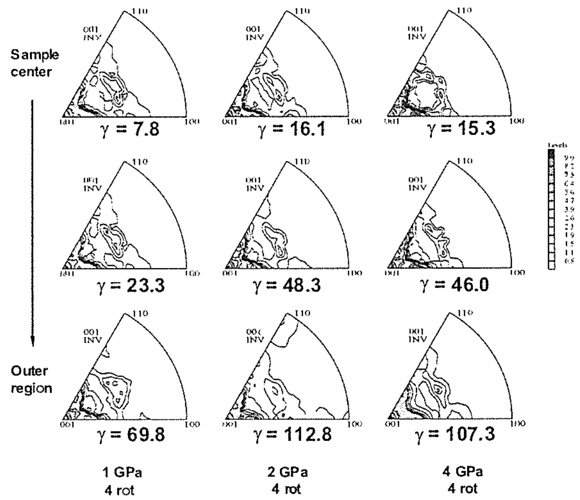

Fig. 10. Inverted pole figures of HPT-deformed as-cast Mg after 4 rotations, at different pressures and shear strains as indicated in the figure

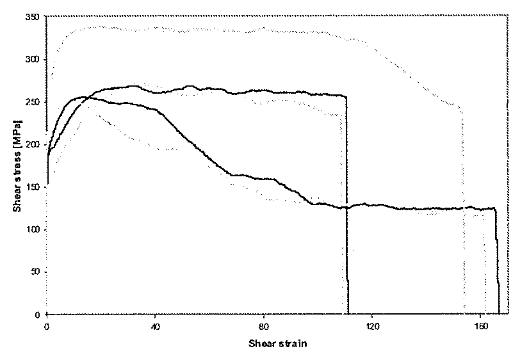

Fig. 11. HPT deformation in-situ curves of polycrystalline as-cast Mg (grey lines) hot extruded Mg (black lines) at different hydrostatic pressures, i.e. 1 GPa, 2 GPa and 4 GPa for the lowest, medium and highest stress level, respectively

Figure 11 shows in-situ recorded stress-strain curves of polycrystalline Mg deformation during HPT experiments. One should remember that some part of the flow stress is related to the Cu ring; however this part can be neglected when restricting the discussion on the difference of flow curves only, as the ring effect is the same in each experiment. From Fig. 11 it can be seen that in-situ deformation curves recorded during deformation of polycrystalline Mg revealed no differences between as-cast and hot-extruded material. This agrees with the findings of texture investigations where no essential differences occurred especially at large HPT strains applied. Small local maxima shown up on the deformation curves (especially with those of most deformed samples) indicate the occurrence of relaxation processes which may help to further plastic deformation.

3.3. Texture evolution in Cu polycrystals during HPT deformation

The HPT deformation of Cu has been performed by applying 4 GPa and 8 GPa hydrostatic pressures, up to shear strains of about $\gamma = 110$. The texture measurements have been recorded along the radius at 0.5, 1, 1.5, 2, 2.5, 3, 3.5 mm distance from the center. Three representative texture measurements at 0.5, 2, and 3.5 mm are being presented by inverse pole figures in Figs. 13 and 14, each for 4 GPa and 8 GPa, respectively. The related torsional shear strains are 2.7, 10 and 18, corresponding to equ. (1).

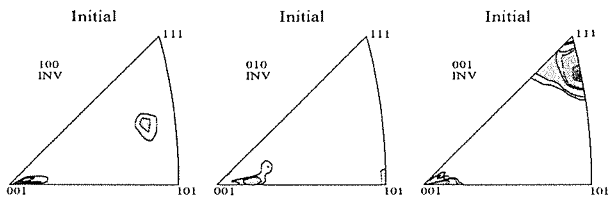

Fig. 12. Inverse pole figures of initial texture of Cu samples. Types of inverted pole figures are (100), (010) and (001) from left to right, respectively)

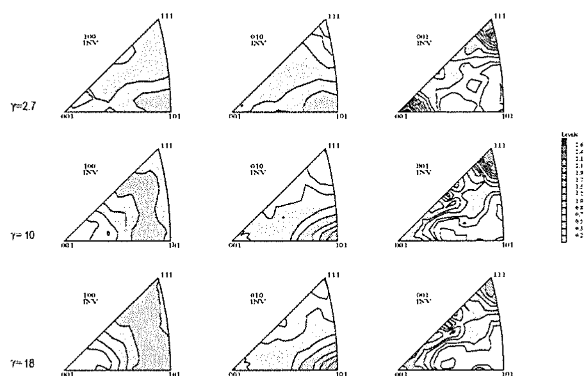

Fig. 13. Inverse pole figures of type (100), (010) and (001) (from left to right, respectively) of Cu samples deformed at 4 GPa with different shear strains (increasing from top to bottom, given on the left)

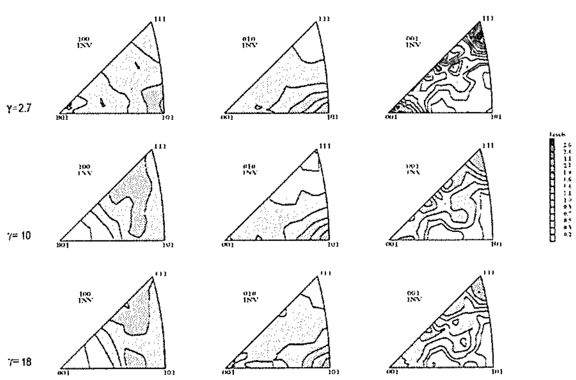

Fig. 14. Inverse pole figures of type (100), (010) and (001) (from left to right, respectively) of Cu samples deformed at 8 GPa with different shear strains (increasing from top to bottom, given on the left edge)

Considering the (001)-inverse pole figure (IPF), the HPT-deformation at 4 GPa reveals quite strong intensities around (001) and (111), the latter partially arising from the initial texture (see Fig.12). The maximum of intensity of this orientation has shifted closer to (111) which corresponds to a rotation by shear on (111) planes. With further deformation this component decreases slowly, while the (001) component drops faster to a low constant value. In the (010)-IPF an increase of the (101)-orientation can be observed which corresponds to the shear direction in the sample, again proving the shear-type character of the deformation.

A similar but still different texture evolution can be observed for 8 GPa. In the (001)-IPF the maximum of intensity near to the (111)-orientation again has shifted closer to (111) – at the smallest deformation degree – but this and the following decrease occurs at smaller deformations than with 4 GPa. At the same time the orientations around (001) which have small intensities, decrease to even lower ones. In contrast to the 4 GPa – deformation, the (101) component in the (010)-IPF has already developed at the smallest deformation and does not change too much with further deformation. So it seems that the higher hydrostatic pressure leads to the same sequence of textures as the lower one, but at clearly smaller shear strains.

This effect of the hydrostatic pressure combines with previous observations of microstructural changes like long range internal stresses determined by X-ray line profile analysis [15]. A drop of these 3^{rd} -order stresses indicated a rearrangement of the dislocation structure by dynamic and static recovery, and this drop occurred at strains which were the lower the higher the hydrostatic pressure was. Dynamic or static recrystallisation, which has been considered as a possible reason of microstructural changes in [11], has been excluded in [15] due to a steady but slow increase of the dislocation density over the wide range of the applied deformation. Also the present analysis of the texture evolution in HPT deformed Cu – in contrast to the case of Mg described before – does not exhibit any hints to recrystallization effects.

4. Summary

- A new HPT technique has been introduced, which allows to homogeneously deform pure magnesium at ambient temperature by HPT up to very high strains.
- As concerns Mg single crystal deformation by HPT, they depend on the initial crystal orientation. While hard orientations close to $(11\bar{2}2)$ tend to remain stable during deformation, others – softer ones – close

to $(10\bar{1}2)$ reveal strongly strain dependent reconstructions. Some components characteristic of hard orientation also arise in soft orientations. Therefore these components are to be considered to be typical of HPT deformation of magnesium.

- With HPT deformation of polycrystalline Mg, initial differences in grain size and texture had no essential effect neither to the texture nor to the flow curves evolving during HPT.
- There exists an effect of the magnitude of hydrostatic pressure applied during HPT deformation, which is to be observed mainly with the texture evolution of polycrystalline samples. The higher the pressure applied, the smaller the shear strain is where specific texture components arise. This effect is observed with both HPT deformations of Mg as well as that of Cu.
- In case of HPT deformed Mg, some components emerge both in single as well as in polycrystal samples which may be interpreted by the onset of dynamic and/or static recrystallization. Nothing similar is observed in the texture evolution of HPT Cu, in accordance with previous observations of the authors.

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