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Microstructure and mechanical properties of ultrafine-grained Mg-Zn-Ca alloy

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Abstract. In this paper the influence of high pressure torsion (HPT) on the structure of Mg-Zn-Ca alloy is studied. The microstructure before and after HPT and after additional annealing has been studied by the scanning electron microscopy, transmission electron microscopy and X-ray diffraction. The results of microhardness measurements and tensile tests of HPT samples are discussed.

1. Introduction

It is well known that low density, high strength-to-weight ratio, comparable elastic modulus to bone, and relative biocompatibility of corrosion products make magnesium and its alloys suitable for several biodegradable implant applications. However, conventional magnesium alloys which contain Al, Zn, Mn are toxic for a human body. Therefore, in this work biocompatible alloy of the Mg-Zn-Ca system is chosen for studies [1], as toxicological effects on a human body are minimized. At the same time despite all the advantages biocompatible magnesium alloys are characterized by rather weak mechanical properties. Recent studies have shown that application of severe plastic deformation results in formation of an ultrafine-grained structure in conventional magnesium-based alloys containing Al, Zn, Mn etc, which is accompanied with significant enhancement of strength characteristics [2-5]. This work deals with formation of a UFG structure in magnesium-based alloy Mg-Zn-Ca by high pressure torsion and study of influence of its peculiarities on mechanical properties.

2. Materials and experiment

Samples of Mg–1%Zn-0.13%Ca alloy subjected to homogenizing annealing at 430°C for 1 h and subsequent cooling in water were taken as initial material. Disk-shaped samples 20 mm in diameter and 0.9 mm thick were subjected to high pressure torsion (HPT) (Fig.1) at room temperature under a pressure of 6 GPa (5 rev of anvils).

Thermal treatment of samples was conducted in electric furnaces of a SNOL-type. The temperature in a furnace was controlled with a chromel-alumel thermocouple, the accuracy of control of which was ± 5 ^{o}C.

The objects of research of initial structure and microstructure after SPD were microspecimens after mechanical polishing with subsequent chemical etching in 20% HNO_3 water solution.

The alloy microstructure was studied on a scanning electron microscope (SEM) JEM6390 with an accelerating voltage 10 kV. The microstructure of the samples after HPT was studied on a

transmission electron microscope (TEM) JEM2100 with an accelerating voltage of 200 kV. The average grain size was determined by the intercept method and was calculated from more than 250 grain measurements.

The discs with a diameter of 3 mm for TEM foils were cut out after mechanical thinning to 0.1 mm. Then they were subjected to twin-jet electropolishing on a Tenupole-5 set. Polishing was conducted with the help of the solution of HCl acid - 10% and acetic acid - 90% at ambient temperature.

The X-ray diffraction (XRD) analysis was carried out using a Rigaku diffractometer. The XRD method was applied to determine the lattice parameters *a* and *c*, the root-mean square strains, and the size of coherent domains *D*. The survey X-ray diffraction patterns were obtained by scanning with a step 0.05° and an exposure time of 5 s at each point. The X-ray diffraction-line profiles were analyzed using a software package based on the method of harmonic analysis of the physical broadening of diffraction lines (00.2) and (00.4).

The microhardness *HV* of samples was measured by the Vickers method using a Micromet-5101 device with a load of 1 N and a loading time of 10 s. Each sample was measured along a diameter more than 20 times to provide reliable results.

Mechanical tests were conducted on a specially designed set for tension of small-sized samples. The force on the set was registered by a Dacell UMM-K200 sensor with 0.8% accuracy. Special computer program was developed to control the deformation process of small samples. The specimens (Fig.1) with a gage length of 2 mm and a cross section of 0.6×1 mm were subjected to tensile tests at room temperature and a constant strain rate of 10^{-3} s^{-1}.

3. Results and discussion

The alloy after homogenization had an equiaxed coarse-grained structure with an average grain size 30 μm. Two types of particles were observed in the macrostructure. The first-type particles had an elongated shape (Fig.1a) with a length 20 μm, the particles were located mostly in grain boundaries, but rarely were observed in grain bodies. The second-type particles (Fig.1b) also had an elongated shape with a length up to 1 μm and width up to 0.5 μm, and were observed only in grain bodies. Their volume fraction was 7%. One may assume that these particles have complex composition and contain Zn and Ca. In [6] it was mentioned that intermetallic phases of complex composition containing Ca could precipitate during crystallization of alloys for example of the Mg-Al-Ca system.

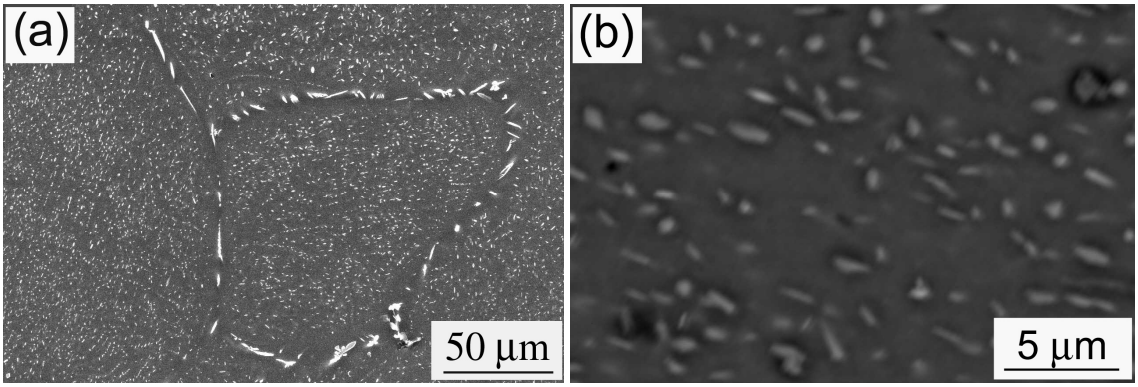

Figure 1. Initial structure of Mg-Zn-Ca: (a) first-type particles – in grain boundaries; (b) second-type particles – in grain bodies.

Electron microscopy studies demonstrated that as a result of HPT a nanocrystalline grain structure with an average grain size about 150 nm formed in the samples of the studied alloy (Fig.2). High density of defects and distortions of a crystalline lattice connected with it caused inhomogeneous diffraction contrast in grain interiors. The X-ray studies demonstrated that the value of mean-square

microstresses increased from 0.0057 in the initial state to 0.234 after HPT (Table 1). The first-type particles were not observed in the structure after HPT. The second-type particles retained in the structure (Fig. 2b), their average size did not change.

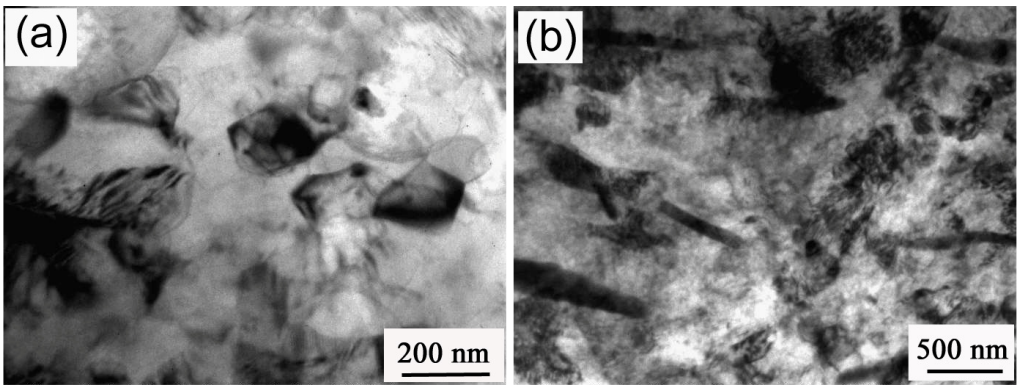

Figure 2. Bright-field TEM images of the microstructure of Mg-Zn-Ca after HPT: (a) average grain size; (b) size of second-type particles.

The microstructure of Mg-Zn-Ca after HPT and additional annealing is displayed in Fig. 4. The average size of grains increased to 1.5 μm (Fig. 3a). The second-type particles remained stable (Fig. 3b) with a length up to 1 μm and width up to 0.5 μm. The volume fraction was 6%. Fig. 4 displays the TEM microstructure, on which the elongated particles are also observed. The density of dislocations reduced significantly (Fig. 4).

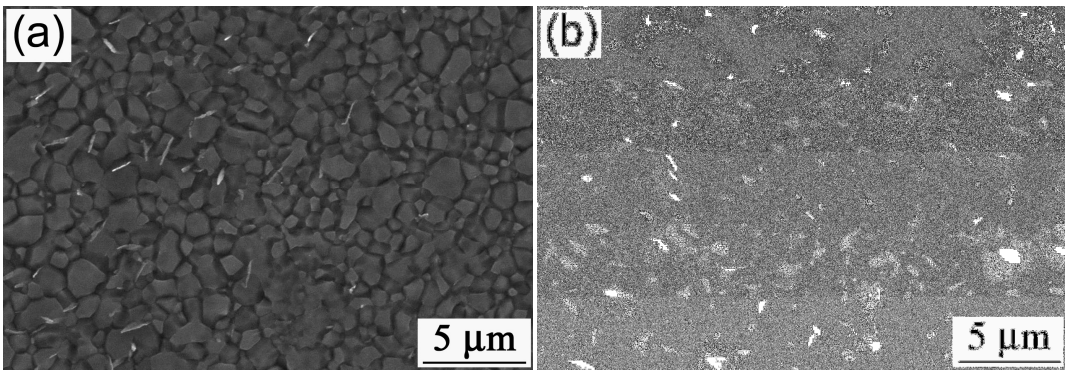

Figure 3. Bright-field SEM images of the microstructure of Mg-Zn-Ca after HPT+annealing at 200 ^{o}C: (a) average grain size; (b) size of second-type particles.

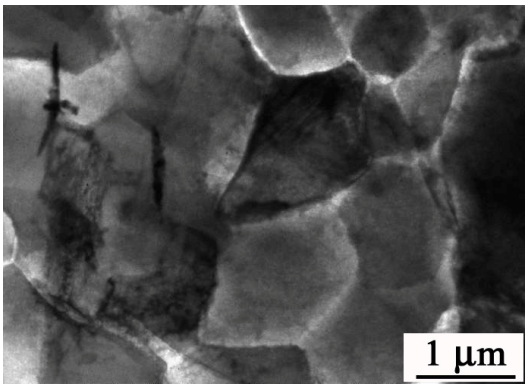

Figure 4. TEM microstructure of Mg-Zn-Ca after HPT+ annealing at 200 ^{o}C.

X-ray studies (Table 1) showed that the average size D (nm) of coherent scattering domains (CSD) of an initial sample was 139.4 nm. Application of HPT resulted in reduction of CSD to 73.4 nm. After additional annealing at 200°C the CSD value was 117.4 nm, and the value of microdeformations of a crystalline lattice of samples with a UFG structure decreased to 0.026%. This testified to redistribution and annihilation of dislocations in the structured and ,therefore, to relaxation of internal stresses and start of grain growth at this temperature, which is confirmed by TEM studies of the microstructure (Fig.4).

Table 1. Values of CSD, mean-square microdeformations, lattice parameters *a* and *c* for crystallographic direction <00.1> for different states of Mg-Zn-Ca.

State	D, nm	Microdistortions, %	*a*, Å	*c*, Å
Initial	-	0.0057	3.2065±0.0001	5.203±0.0004
HPT	73.5	0.234	3.2085±0.0001	5.210±0.0004
HPT+ annealing 200°C	117.4	0.026	3.2066±0.0001	5.206±0.0004

The values of the crystalline lattice parameters in the initial homogenized coarse-crystalline state were $a \approx 3.2065$ Å and $c \approx 5.203$ Å (table 1). The obtained values do not differ from the parameters of the crystalline lattice of pure magnesium ($a \approx 3.2029$ Å and $c \approx 5.200$ Å). This testifies to the fact that the atoms of Zn and Ca are partially dissolved in Mg. The parameters *a* and *c* of the samples with a UFG structure processed by HPT change significantly (table 1), which testifies to further dissolution of particles in the matrix during HPT. The subsequent annealing at 200°C of the HPT-processed samples with a UFG structure resulted in recovery of the crystalline lattice parameters. During annealing of HPT samples some fraction of particles could precipitate.

The studies of microhardness showed that its value in the initial coarse-crystalline state of Mg-Zn-Ca was 450 MPa, which was a bit higher than that of pure magnesium (400 MPa). After HPT the microhardness of the studied alloy increased to 990 MPa (Fig. 5a) due to considerable grain structure refinement to a grain size 200 nm (Fig. 2). The samples were subjected to annealing for 30 min in the temperature range from 100-350°C at 50°C intervals to study thermal stability. Alongside with that the microhardness value decreased after annealing at 350 °C to an initial value 450 MPa, as it can be seen on the microhardness-annealing temperature curve (Fig. 5a).

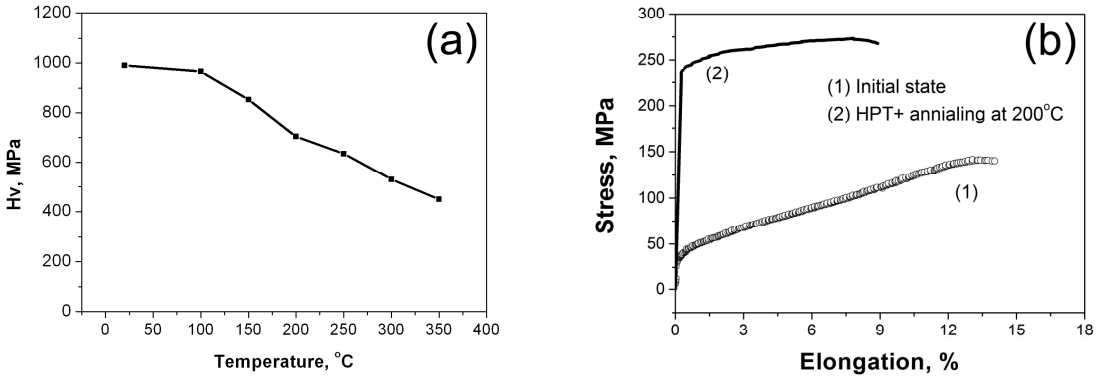

Figure 5. (a) – change of microhardness of Mg-Zn-Ca after HPT depending on the annealing temperature; (b) - tensile tests of Mg-Zn-Ca at room temperature and a strain rate 10^{-3} s^{-1}: (1) initial coarse-crystalline state, (2) after HPT and additional annealing.

The mechanical tensile properties of the initial alloy and the alloy after HPT and additional annealing are displayed in Fig. 5b. The alloy in the coarse-crystalline state has the ultimate tensile

strength 140 MPa and ductility 13%. The microstructure studies of the samples after mechanical tests were not conducted, but according to the published data [2] one may assume that the extended stage of strain hardening in the samples of magnesium alloy can be connected with intensive twinning due to formation of deformation twins during tensile testing. After HPT the samples failed in a brittle manner without achieving the yield stress, as the samples after HPT have a high value of internal elastic stresses. Alongside with that the samples after HPT and additional annealing at 200 ^{o}C exhibited an enhanced ultimate tensile strength 270 MPa and ductility 8.5%. Significant enhancement of the ultimate tensile strength is conditioned by strong grain structure refinement. Retention of observable extension of the strain hardening stage could be caused by activation of dislocation gliding in non-basal planes, as observed on the example of UFG Mg alloy in [7].

4. Conclusions

The use of high-pressure torsion allowed producing an ultrafine-grained structure in the Mg-Zn-Ca alloy with an average grain size 150 nm. During heating of SPD samples to a temperature 200°C the average grain size is observed to increase to 1.5 μm. The samples after HPT and additional annealing at 200 ^{o}C have higher ultimate tensile strength (270 MPa) characteristics as compared to the initial samples (140 MPa), which is connected with refinement of an average grain size. Retention of ductility (8.5%) is conditioned by activation of dislocation gliding in non-basal planes.

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