phys. stat. sol. (c) **4**, No. 10, 3591–3594 (2007) / **DOI** 10.1002/pssc.200675777

Microstructure investigations of ultra-fine grained Mg-Gd alloys prepared by high pressure torsion

J. Čížek[*, 1], **I. Procházka**[1], **B. Smola**[1], **I. Stulíková**[1], **R. Kužel**[1], **Z. Matěj**[1], **V. Cherkaska**[1], **R. K. Islamgaliev**[2], and **O. Kulyasova**[2]

[1] Faculty of Mathematics and Physics, Charles University in Prague, V. Holešovičkách 2, 180 00Prague, Czech Republic

[2] Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa 450000, Russia

Received 23 July 2006, accepted 2 April 2007
Published online 29 June 2007

PACS 61.72.Lk, 78.70.Bj, 81.07.Bc

Mg-Gd represents a promising light hardenable alloy with a high creep resistance even at elevated temperatures. A disadvantage of this alloy consists in a low ductility. Recently it has been demonstrated that ultra fine grained (UFG) metals with grain size around 100 nm can be produced by high pressure torsion (HPT). A number of UFG metals exhibit favourable mechanical properties consisting in a combination of a very high strength and a significant ductility. The HPT processing creates a high density of defects, especially dislocations, in UFG material. These defects play key role in the specific physical properties of UFG materials. Defects studies of UFG materials are, therefore, very important for understanding of their unusual properties. Positron lifetime (PL) spectroscopy is one of the most powerful techniques for investigations of open-volume defects. In the present work we employed PL spectroscopy for defect studies of UFG Mg-Gd alloys prepared by HPT. The PL investigations were combined with transmission electron microscopy, X-ray diffraction, and microhardness measurements. At first, the microstructure of the as-deformed specimens was characterized. Subsequently, the specimens were isochronally annealed and the development of microstructure with increasing temperature was investigated.

1 Introduction Lightweight Mg-based alloys allow for a significant weight reduction especially important in automotive and air industry. However, the applicability of Mg alloys is limited due to a degradation of their mechanical properties at elevated temperatures. This problem was successfully solved using rare Earth alloying elements [1]. Mg-Gd system represents a promising novel hardenable material with high creep resistance even at 300 ^{o}C [2]. A remaining problem of this alloy is relative poor ductility insufficient for industrial applications. Grain refinement could be a way how to improve ductility of this material. Recently it has been demonstrated that UFG metals with grain size around 100 nm can be produced by HPT [3]. A number of UFG metals exhibit favourable mechanical properties consisting in a combination of very high strength and a sufficient ductility. For this reason, it is highly interesting to examine microstructure and physical properties of UFG Mg-based light alloys. Following this purpose, microstructure investigations of HPT deformed Mg-9.33wt.%Gd (Mg10Gd) were performed in the present work using positron lifetime (PL) spectroscopy combined with transmission electron microscopy (TEM), X-ray diffraction (XRD) and Vicker microhardness (HV) measurements.

* Corresponding author: e-mail: jakub.cizek@mff.cuni.cz, Phone: +420 221 912 788, Fax: +420 221 912 567

2 Experimental The Mg10Gd alloy was prepared by squeeze casting followed by solution treatment at 500 °C for 6 hours finished by quenching into water of room temperature. The UFG samples were prepared by HPT at room temperature up to the true logarithmic strain e = 7 under a high pressure of 6 GPa [3]. The HPT deformed samples were disk shaped with diameter of 12 mm and thickness of 0.3 mm. The isochronal annealing was performed step-by-step (20 °C/20 min) in a silicon oil bath and a vertical furnace with protective Ar atmosphere. Each annealing step was finished by quenching and microstructure investigations at room temperature. A fast-fast spectrometer similar to that described in [4] with timing resolution 160 ps was employed in the present work. Diameter of the positron source spot was ≈4 mm and PL measurements were carried out at the centre of the studied samples. The TEM observations were performed on the JEOL 2000 FX electron microscope operating at 200 kV. The XRD studies were carried out with the aid of HZG4 (Seifert-FPM) powder diffractometers using Cu K_α radiation. The HV measurements were performed by the Vickers method at the load of 100 g applied for 10 s using the LECO M-400-A hardness tester.

2 Results and discussion The TEM observations showed that the solution treated Mg10Gd alloy exhibits coarse grains and a low dislocation density ≤ 10^{12} m^{-2}. Two components were found in the PL spectrum of the solution treated alloy, see Table 1. The dominant contribution comes from free positrons. In addition, there is a weak component with lifetime ≈300 ps. It can be attributed to quenched-in vacancies bound to Gd atoms [5].

Table 1 The lifetimes τ_i and corresponding relative intensities I_i of the components resolved in PL spectra (except of the source contribution). HV values are shown in the last column. In case of HPT deformed sample, HV in the centre and at the margin of the sample are given. The errors (one standard deviations) are given in parentheses.

Sample	τ_1 (ps)	I_1 (%)	τ_2 (ps)	I_2 (%)	HV 0.1
Mg annealed 280°C/30 min	225.3(4)	100	-	-	33(2)
Mg10Gd solution treated	220(4)	90.9(6)	301(9)	9.1(7)	68(3)
HPT deformed Mg10Gd	210(3)	34(2)	256(3)	66(2)	167(4)-233(4)

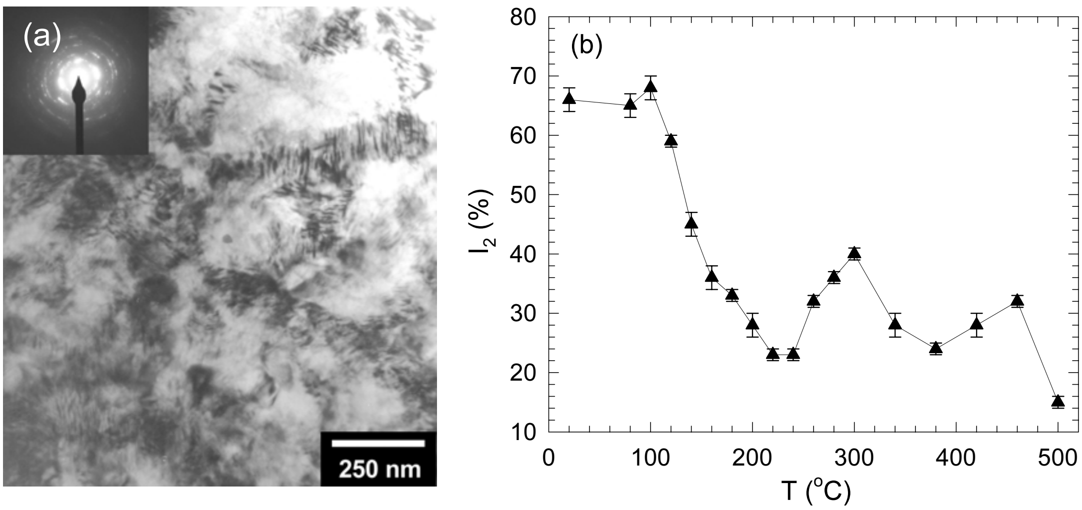

Fig. 1 (a) A bright field TEM image of HPT deformed Mg10Gd, (b) temperature dependence of the intensity I_2 of trapped positrons. The lifetime τ_2 = 256 ps does not change with temperature.

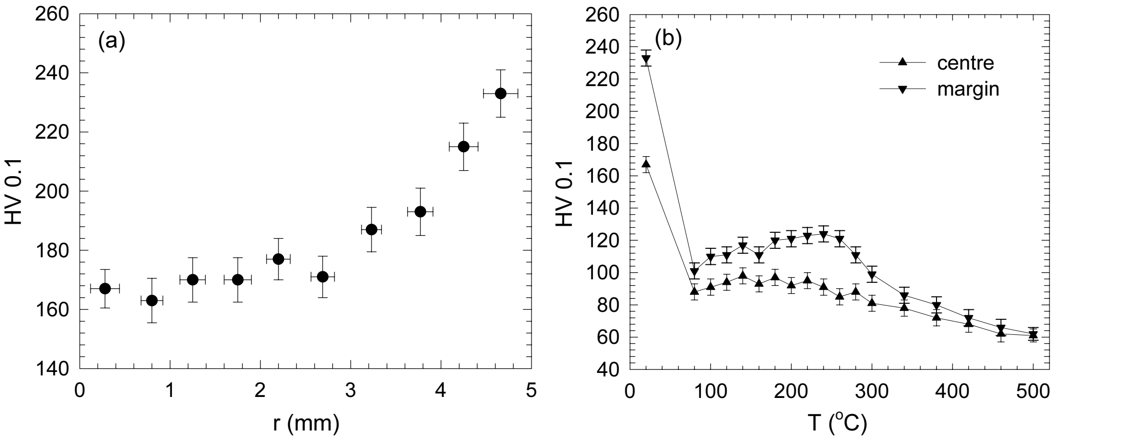

Fig. 2 (a) Dependence of HV on the radial distance *r* from the centre of the specimen for the HPT deformed Mg10Gd, (b) Temperature dependence of HV for the HPT deformed Mg10Gd.

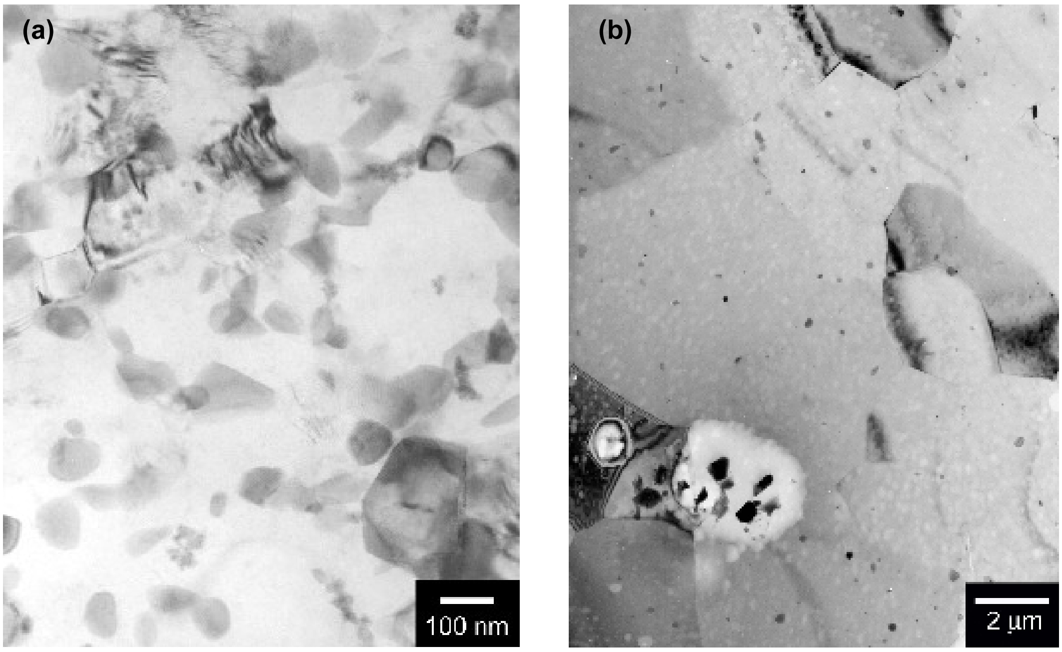

Fig. 3 A bright TEM image of the HPT deformed Mg10Gd alloy annealed up to (a) 300 °C, (b) 380 °C.

A bright-field TEM image and an electron diffraction pattern for the HPT deformed Mg10Gd alloy are shown in Fig. 1a. The sample exhibits uniform UFG microstructure with the mean grain size about 100 nm. The electron diffraction pattern shows high-angle missorientation of the neighboring grains. A high density of homogeneously distributed dislocations was observed. It is not possible to distinguish the individual dislocation line on the TEM images. A high number of dislocations cause a significant broadening of the XRD profiles, see [6] for details. A lower broadening of (000l) profiles with respect to other peaks indicates dominating presence of <*a*> dislocations with Burgers vector $1/3.a.\left[2\bar{1}\bar{1}0\right]$. A weak (0001) texture was found by XRD [6]. The PL spectrum of the HPT deformed Mg10Gd alloy consists of two components, see Table 1. The first component with the lifetime τ_1 comes from free positrons, while the second component with higher intensity and lifetime τ_2 = 256 ps represents a contribution of positrons trapped at dislocations. Figure 2a shows HV as a function of the radial distance r from the centre of the sample. An increase of HV with r indicates that dislocation density increases from the centre of the sample towards the margin. The reason is that the strain in torsion deformation increases with the radial distance from the centre of rotation, i.e. the largest deformation occurs at the margin of the sample.

After characterization of the as-deformed microstructure, the samples were subjected to isochronal annealing. Decomposition of the supersaturated solid solution (sss) in coarse-grained Mg10Gd alloy takes place in the sequence sss $\rightarrow$ β'' (D019) $\rightarrow$ β' (cbco) $\rightarrow$ β (Mg5Gd, cubic) [2, 5]. The β'' and β' are metastable phases, while β formed at higher temperatures is a stable phase. The PL spectra of isochronally annealed UFG Mg10Gd consisted of two components with lifetimes τ_1 (free positrons) and τ_2 (positrons trapped at dislocations). The lifetime τ_2 = 256 ps did not change with temperature (except of statistical fluctuations) indicating that the nature of positron traps remains unchanged. Temperature dependence of the intensity I_2 is plotted in Fig. 1b, while Fig. 2b shows HV as a function of the annealing temperature. A radical decrease of I_2 in the temperature interval (100-240) ^{o}C directly indicates a significant recovery of dislocations. A slight local increase of I_2 at 100 ^{o}C is likely due to formation of the β'' phase as confirmed by coincidence Doppler broadening [5]. However, the β'' phase particles are very fine ($\leq$ 10 nm) because they were not observed by TEM. One can see in Fig. 2b that HV exhibits an abrupt decrease after annealing to 80 ^{o}C, i.e. there is a substantial softening, which takes place prior to the recovery of dislocations. It occurs likely due to a relaxation of the stresses introduced into the sample by HPT or a rearrangement of dislocations. The TEM observations confirmed a decrease of dislocation density in the temperature range (100-240) ^{o}C, but no grain growth was seen. Thus, the recovery of dislocations is not accompanied by recrystallization. A TEM image of the sample annealed up to 300 ^{o}C is shown in Fig. 3a. The grains are almost free of dislocations, but the grain size remains still around 100 nm. Grain growth was observed only after annealing at 380 ^{o}C, see Fig. 3b. It demonstrates exceptionally good thermal stability of UFG structure in the HPT deformed Mg10Gd. A local maximum of I_2 seen at 300 ^{o}C (Fig. 1b) is due to precipitation of the equilibrium β phase which was identified by TEM. Positrons are trapped at the misfit defects between the incoherent β phase particles and the matrix. Contrary to the coarse-grained sample [5], precipitation of the metastable β' phase is omitted in UFG Mg10Gd and the stable β phase is formed at significantly lower temperatures. Coarsening of the β phase particles causes subsequent decrease of I_2. Above 450 ^{o}C, the β phase particles dissolve and the solid solution is restored.

4 Conclusions Defects in HPT deformed Mg10Gd and their recovery with temperature were investigated by PL spectroscopy combined with other techniques. The HPT deformed specimen exhibits UFG microstructure with high density of dislocations and grain size $\approx$100 nm. Recovery of dislocations occurs in the temperature range (100-240) ^{o}C, but is not accompanied by grain growth. The grain size remains around 100 nm up to $\approx$ 350 ^{o}C. Contrary to the coarse grained alloy, metastable β' phase does not form in UFG Mg10Gd and the precipitation of the stable β phase is shifted to significantly lower temperatures.

Acknowledgements The work was financially supported by The Czech Science Foundation (contract 106/05/0073) and The Ministry of Education, Youth and Sports of Czech Republics (project MS 0021620834).

References

[1] B. L. Mordike, Mater. Sci. Eng. A **324**, 103 (2002).
[2] P. Vostrý, B. Smola, I. Stulíková, F. von Buch, and B. L. Mordike, phys. stat. sol. (a) **175**, 491 (1999).
[3] R. Z. Valiev, R. K. Islamgaliev, and I. V. Alexandrov, Prog. Mater. Sci. **45**, 103 (2000).
[4] F. Bečvář, J. Čížek, L. Lešťak, I. Novotný, I. Procházka, and F. Šebesta, Nucl. Instrum. Methods A **443**, 557 (2000).
[5] J. Čížek, I. Procházka, F. Bečvář, I. Stulíková, B. Smola, R. Kužel, V. Cherkaska, R. K. Islamgaliev, and O. Kulyasova, phys. stat. sol. (a) **203**, 466 (2006).
[6] J. Čížek, I. Procházka, B. Smola, I. Stulíková, R. Kužel, Z. Matěj, V. Cherkaska, R. K. Islamgaliev, and O. Kulyasova, Mater. Sci. Eng. A (2007), in press.

