THERMAL STABILITY OF MAGNESIUM ALLOY AZ91 PREPARED BY SEVERE PLASTIC DEFORMATION

Roman Štěpánek [1,*], Libor Pantělejev [1], Ondřej Man [1]

[1] Institute of Materials Science and Engineering, Faculty of Mechanical Engineering, Brno University of Technology, Technická 2896/2, Brno, 616 69, Czech Republic

[*] corresponding author: e-mail: y107620@stud.fme.vutbr.cz

Resume

This paper deals with the thermal stability of ultrafine-grained alloy AZ91 prepared by means of ECAP (Equal Channel Angular Pressing) method. Annealing experiments were conducted isochronally for 30 minutes in the temperature range of 220 to 400 °C in argon atmosphere. EBSD (Electron Backscatter Diffraction) method was used to image the changes in microstructure due to increased temperature.

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1. Introduction

The microstructure of ultrafine-grained materials prepared by any method utilizing severe plastic deformation (SPD) is inherently unstable [1]. Either a specific bimodal structure creation, or heavy grain coarsening throughout the whole volume of the material, takes place during thermal exposure. The scale of changes in microstructure depends not only on the temperature but also on the time of exposure. The changes in microstructure are characterized not only by grain coarsening but also by misorientation variations at low angle boundaries (LAB) and high angle boundaries (HAB). Changes in dislocation density and crystal lattice distortions also take place; the latter can be assessed by kernel average misorientation (KAM). Despite the relatively broad industrial exploitation of magnesium alloys nowadays, only a small number of papers deal with the stability and potential use of UFG magnesium alloys [2, 3] in contrast to other metals and alloys, like copper and aluminum alloys, whose stability and property development is sufficiently documented [4–6]. Among the most widespread magnesium alloys in industry are those alloyed with aluminum and zinc as the members of the AZ group. The papers published so far concentrated on the AZ31 alloy, which is the one with the lowest amount of aluminum and so of the intermediary phase $Al_{12}Mg_{17}$ [7–9]. Thermal stability data of the most widespread alloy AZ91 are rather sparse and incomplete. This is the reason, why this work focuses on microstructural stability of AZ91 alloy after SPD process.

2. Material and experimental methods

AZ91 magnesium alloy treated by ECAP in Ufa State University laboratory, Russia, was used for the experiments. The material was processed by six passes through the ECAP die following route B_c at 300 °C. The angle between the die channels Φ was 120°.

The billets were then machined into cylindrical raw products, 15 mm in diameter and 100 mm long. The beam-like specimens for annealing experiments were made from 5 mm thick "pills" cut from the billet. The faces selected for examination were prepared conventionally by metallographic grinding and polishing and then electrolytically polished in a solution of hydrochloric acid and ethyl-cellosolve.

The annealing experiments were performed in a tube furnace Heraeus ROS 4/50 in argon atmosphere. The specimens were held for 30 min at selected temperature levels (220 °C to 400 °C); the temperature was sensed directly on the specimen by a Pt100 probe and regulated within ±0.3 °C. The microstructure was observed before and after the thermal exposure utilizing partially the "site specific" approach, which means that identical place is analyzed before and after exposure, and which allows for direct comparison of the changes in microstructure [10]. It was found by the analysis of microstructure that the material after ECAP process exhibits bimodal microstructure that is areas of large grains were surrounded by areas of small grains; particles of $Al_{12}Mg_{17}$ phase were observed rather in the fine-grained areas than in the coarse-grained ones, there was also found high amount of relatively large manganese particles uniformly spread in matrix (Fig. 1). Chemical composition of matrix and particles measured by EDS method is given in Table 1. It is advisable to map more than one area in the particular case of material of bimodal microstructure since otherwise the coarse and fine-grained areas can be confused when making conclusions on the influence of thermal exposure. It is more convenient to use the "site specific" approach when relevant results are to be obtained in relatively short time. Considering the reactivity of Mg alloys even the "site specific" approach has its limitations, because there is a risk of humidity-induced contamination of the specimens during their transport from the inert atmosphere into the electron microscope chamber. The results presented in this work therefore combine both the "site specific" approach and observation of distinct areas. The changes in microstructure were traced by EBSD analyses performed on a scanning electron microscope Zeiss Ultra Plus 50. Changes in microstructure are quantified by change in average grain size and proportion of LAB and HAB.

3. Results

It was possible to apply the "site specific" approach for annealing experiments at temperatures up to 300 °C. It was found that annealing at 220 °C for 30 min led to a change in average grain diameter from 8.02 µm (initial state) to 7.08 µm. Similar results were obtained by increasing the annealing temperature up to 300 °C, i.e. small increase in HAB amount on the account of LAB and minute changes in grain size distribution (in class occupancy). Pronounced coarsening of the microstructure was not observed (Fig. 2 a, d) – see Table 2.

Table 1

Chemical composition of AZ91 matrix and particles

	Mg [wt.%]	**Al [wt.%]**	**Ti [wt.%]**	**Cr [wt.%]**	**Mn [wt.%]**	**Fe [wt.%]**	**Zn [wt.%]**
Particles	0.582	40.409	0.420	0.344	49.865	8.380	-
Matrix	90.065	9.043	-	-	0.144	-	0.748

Table 2

Fraction of low angle and high angle boundaries in green and annealed state as a function of annealing temperature

Annealing time 30 min	**Temperature**	
	220 °C	300 °C
	LAB/HAB [%]	LAB/HAB [%]
As received	91.7/8.3	97.0/3.0
Annealed	87.2/12.8	95.5/4.5

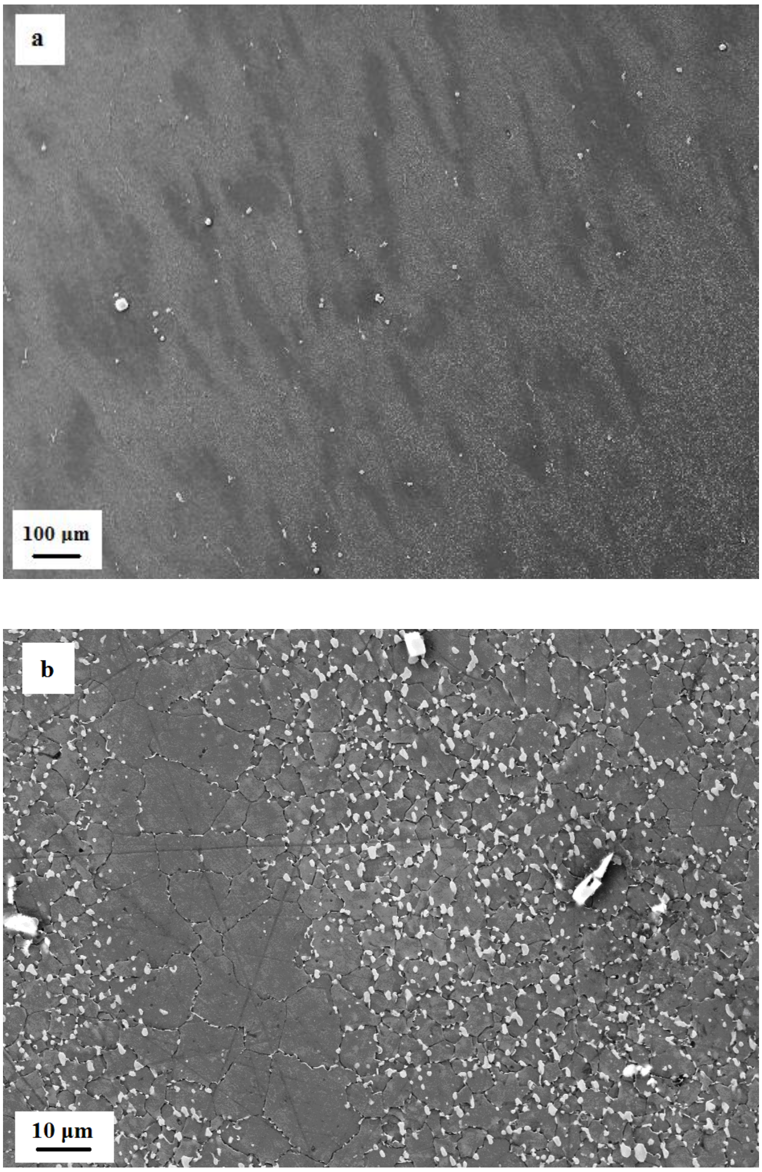


Fig. 1. Initial microstructure of AZ91 alloy (a), detail of bimodal area (b).

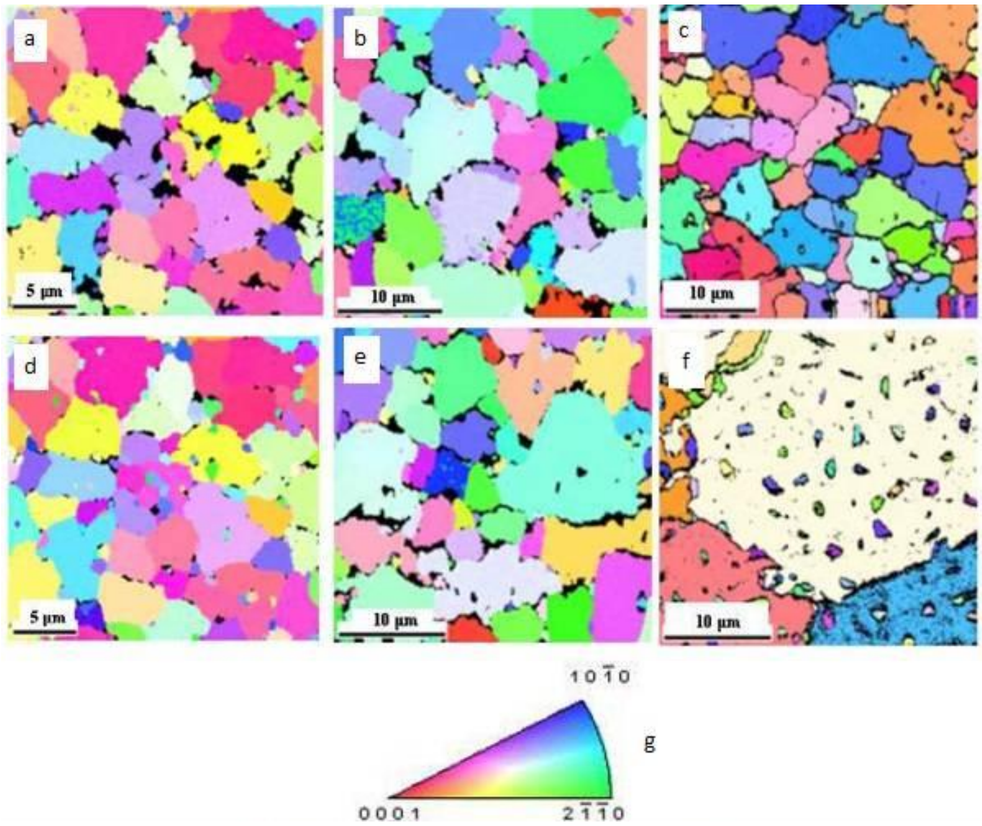


Fig. 2. Inverse pole figure maps of AZ91 alloy's microstructure: site 1 – as received (a), annealed – 300 °C/30 min (d), site 2 – as received (b), annealed – 350 °C/30 min (e), site 3 –as received (c), annealed – 400 °C/30 min (f), color code in a stereographic triangle (g).

(full colour version available online)

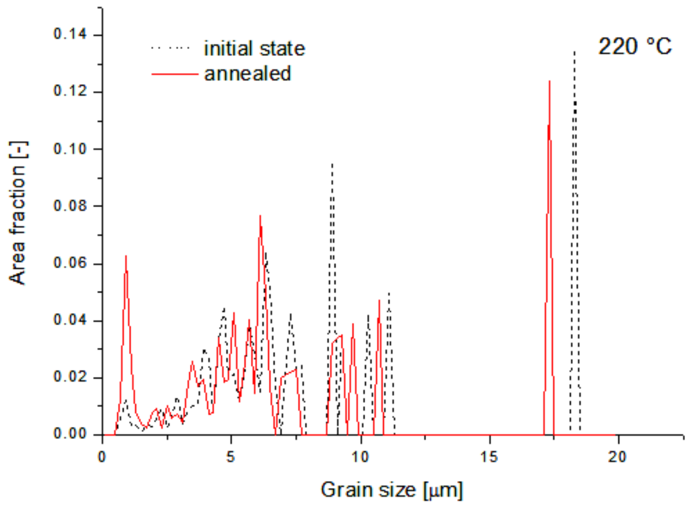
Minor contamination of the area mapped in green state took place during exposure at 350 °C (Fig. 2 b, e), which rendered "site specific" post-anneal EBSD analysis undoable. Therefore another area, lying in the same band of fine grains of the bimodal structure, was selected as a representative of the annealed state. Comparison based on misorientation angles was thus inapplicable; nonetheless, slight tendency to grain coarsening was detected (5.7 µm vs. 7.14 µm). Enormous grain coarsening occurred as a result of increase in temperature to 400 °C (Fig. 2 c, f) – analyzed area virtually encompassed just one single grain. Due to this reason, a larger area was analyzed to confirm grain coarsening in the whole volume, which resulted in a finding that the average grain size increased from 5.9 µm to 20 µm.

4. Discussion

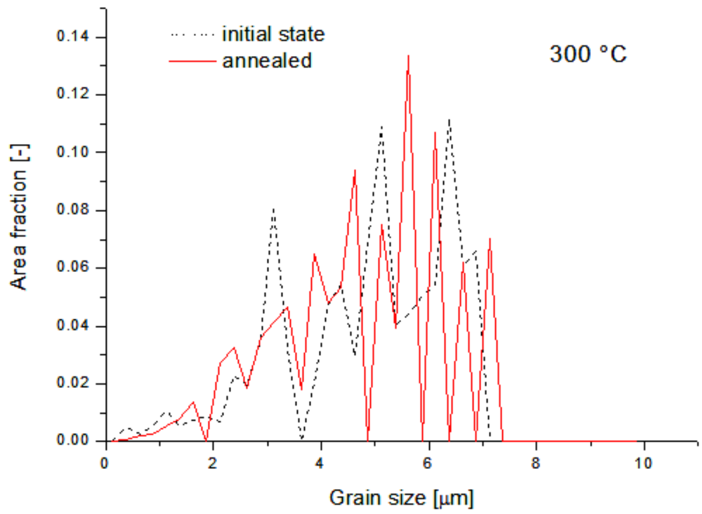
Stability of microstructure of ultrafine-grained materials is a key prerequisite for conservation of their unique properties like higher strength compared to coarse-grained materials. It was discovered by the annealing experiments that the ratio between LAB and HAB moderately changes in favor of HAB (table 2), which holds true for annealing at 220 °C and 300 °C. Although it was not possible to use the "site specific" approach for the experiments at temperatures above 300 °C, the misorientation angle distributions were acquired and interpreted in a way that the fraction of LAB is still high compared to HAB. This finding is completely different from that obtained with UFG Cu or Al alloys, which exhibit the LAB to HAB ratio of approximately

1:1 after SPD process [4, 5] and vastly increased amount of HAB on account of LAB after exposure to critical temperature [6].

It follows from the grain size analysis, considering the boundary misorientation of 15°, that thermal exposure below 300 °C does not affect the grain size distribution markedly, which can be interpreted as no grain coarsening (the decrease of average grain diameter at lower temperatures should not be interpreted as "grain refinement" because, in fact, there, in the grain size histogram, some classes just emptied and some became more occupied as a result of modest changes in misorientation angles). On the other hand, at the temperatures of 350 °C and 400 °C the whole grain size distribution becomes shifted towards higher values (Fig. 3), therefore the grains coarsen. Similar results can be found in literature for AZ31 alloy [7].

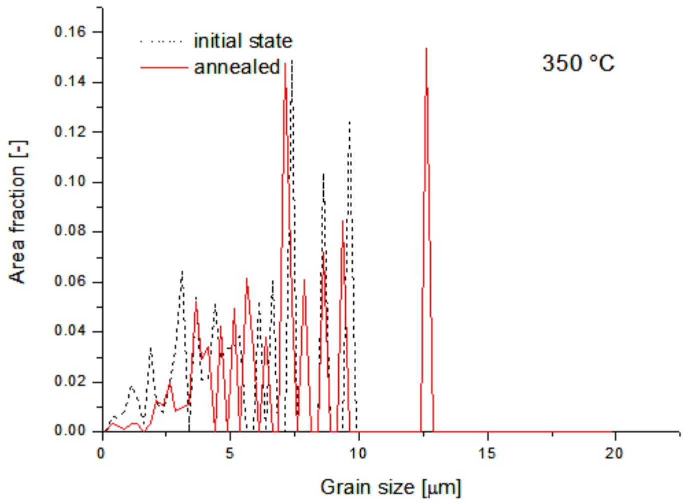


a) 220 °C

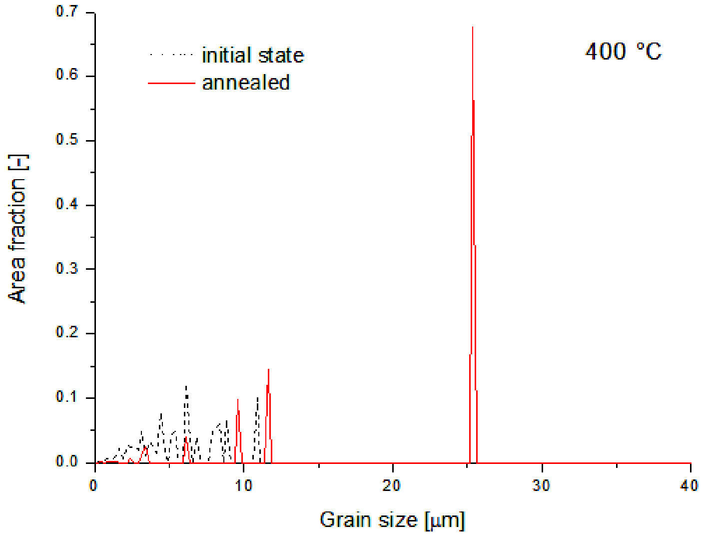


b) 300 °C

Fig. 3. Change in grain size distribution after annealing at different temperatures.
(full colour version available online)



c) 350 °C



d) 400 °C

continue of Fig. 3. Change in grain size distribution after annealing at different temperatures.

(full colour version available online)

5. Conclusions

The microstructure of the alloy AZ91 processed by ECAP is bimodal, being composed of areas of coarse grains (average diameter of 20 µm) and fine ones (average diameter of 6 µm), fine-grained areas containing considerably higher fraction of $Al_{12}Mg_{17}$ intermediary phase.

It was found by the annealing experiments with constant dwell at each temperature (30 min) that the microstructure of the alloy under examination is stable below 300 °C; increased temperatures (350 °C and 400 °C) led to gradual grain coarsening, the average grain size increasing from 6.07 µm to approx. 25 µm.

It was found by the EBSD analysis of misorientation angles that the boundaries contained therein are prevalently low angle ones; thermal exposure leads only to minor increase in HAB fraction even at temperatures above 300 °C.

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