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TMS (The Minerals, Metals & Materials Society), 2013

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Nanostructured Materials

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THERMAL STABILITY OF ULTRA-FINE GRAINED MAGNESIUM ALLOY PROCESSED BY EXTRUSION AND ECAP

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Keywords: AZ31 magnesium alloy, Severe plastic deformation (SPD), Equal channel angular pressing (ECAP), Thermal stability

Abstract

The mechanical properties and thermal stability of ultra-fine grained (UFG) structure of magnesium alloy AZ31 during annealing was investigated. UFG specimens were prepared by a combined two-step severe plastic deformation process: the extrusion (EX) and the equal-channel angular pressing (ECAP). This combined process leads to microstructure refinement and enhanced microhardness. Specimens were annealed isochronally at temperatures 150 - 400 °C for 1 hour and isothermally at temperatures 170 - 210 °C. The evolution of microstructure and mechanical properties were studied by light and scanning electron microscopy and microhardness measurements. The coarsening of a fine-grained structure at higher temperatures was accompanied by a gradual decrease of the microhardness.

Introduction

Magnesium alloys are energy-efficient materials having the potential to replace some conventional structural materials, e.g. steel or aluminum alloys. Magnesium is a very light metal (its density is 1.74 g/cm^3) with relatively high specific mechanical properties which results in expanding use of magnesium-based materials in weight-critical applications [1].
The mechanical and other physical properties may be improved by refining the grain size to submicrometer or even nanometer level. It has been known for many decades, going back to the 1950s, that the structure of deformed metals can change with increasing plastic deformation such that random dislocation arrays can lower the energy of the system by "self-assembling" into "cells" or "subgrains" such that there is a high dislocation density in the cell walls and lower dislocation density within the cells [2].
A variety of special techniques are used for the production of bulk ultra-fine grained (UFG) materials, e.g. equal channel angular pressing (ECAP) [3], high pressure torsion (HPT) [4], accumulative roll-bonding (ARB) [5], twist extrusion [6] or multi-directional forging [7].
Among these techniques, which introduce the severe plastic deformation (SPD) in the material, ECAP is quite an easy and widely-used method of preparation of very fine grain structure. A combined two-step process involving an initial extrusion step and subsequent processing by ECAP (so called EX-ECAP) leads to the enhanced mechanical properties and homogeneous ultra-fine grain microstructure [8, 9].
However, the application of UFG materials is limited due to the structure stability at elevated temperatures. Thermal stability depends on many variables (e.g. stacking fault energy of the material, preparation process and its parameters or properties of grain boundaries) [10].
This work is therefore motivated by this fact and its main objective is to investigate thermal stability - microstructure and microhardness evolution during isochronal and isothermal annealing of the UFG AZ31 magnesium alloy prepared by extrusion and 4 passes of ECAP.

Experimental procedures

Commercial AZ31 magnesium alloy, with a nominal composition of Mg-3%Al-1%Zn in the initial as cast condition was used in this investigation. The material was first extruded at T = 350 °C with an extrusion ratio of ER = 22 using a 630 t direct extrusion press. Billets of the dimensions 10 × 10 × 120 mm were machined from the extruded bar. ECAP pressing was performed at 180°C to reduce grain growth during pressing [11] following route B_c at the speed of 50 mm/min for 4 passes.
Specimens for light (LM) and scanning electron microscopy (SEM) observation of the microstructure were taken from the middle part of the billets perpendicular to the pressing direction. Specimens were first mechanically grinded on watered abrasive papers, and then polished with polishing diamond suspension of grade 3, 1 and ¼ μm and alumina suspension of grade 0.05 μm. Using this procedure, flat samples for Vickers microhardness (load 100 g, 10 s) measurements with minimum surface scratches were obtained. Finally, the specimens´ surface was etched with picric acid for 1 second which enables observations in LM and SEM.

Results and discussion

The first set of specimens was annealed isochronally at temperatures 150 – 400 °C for 1 hour and quenched in water. Subsequently, the Vickers microhardness HV0.1 was measured at the specimens (at least 15 indents were made in each sample). The results are shown in Fig.1a. The first data point (HV0.1 = 86) corresponds to the initial non-annealed specimen. One can see that microhardness values after annealing at 150 and 170 °C differ within the statistical error only. However, the microhardness declines abruptly in the range of 170 - 230 °C and then continuously up to 400 °C.
Results of isothermal annealing at the temperatures 170, 190 and 210 °C are depicted in Fig.1b and 1c. The HV0.1 values decline more rapidly at higher temperatures after short annealing times. The final microhardness of the specimen annealed at 170 °C for 64 hours is approximately equal to 76, at 190 °C 72 and at 210 °C 67 HV0.1.

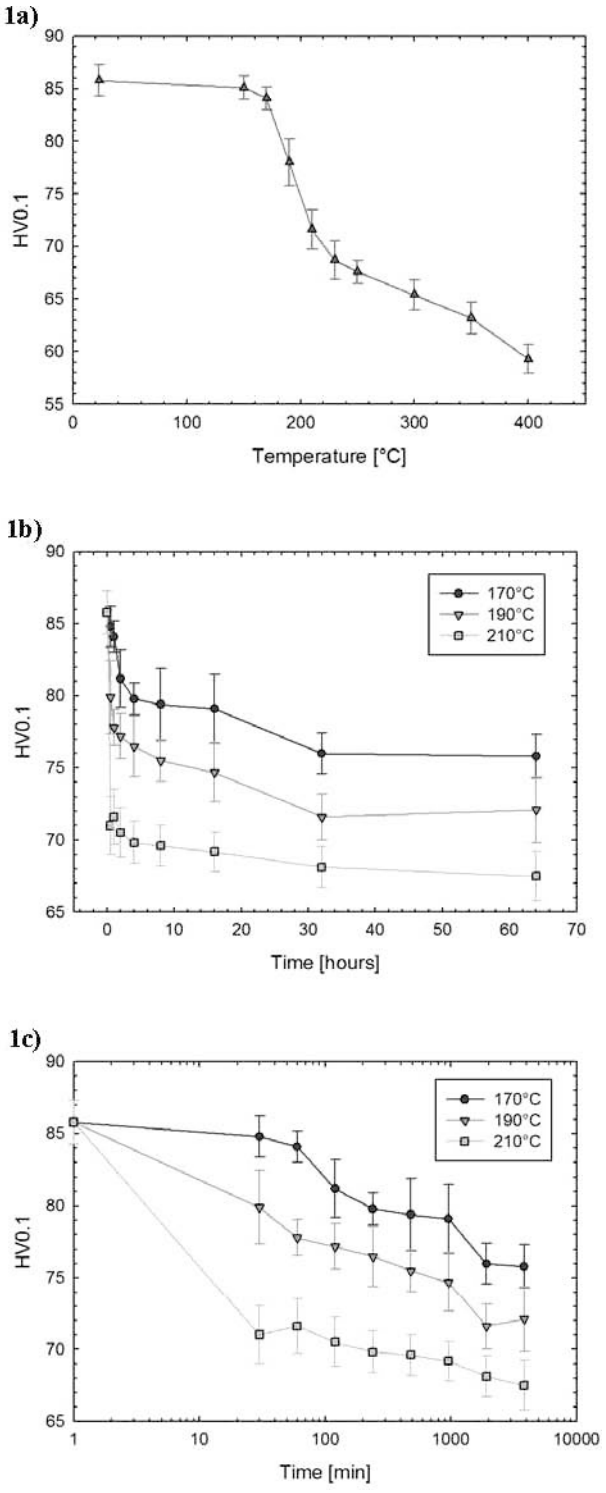


Figure 1: Measurements of thermal stability of AZ31 alloy after extrusion and 4 passes of ECAP: a) microhardness after isochronal annealing, b) and c) microhardness after isothermal annealing at temperatures 170, 190 and 210 °C in a linear and logarithmic scale, respectively.

Microstructure of the AZ31 alloy after extrusion and 4 passes of ECAP is nearly homogeneous with very fine grains of the average size 1 μm and few remaining larger grains (see Fig. 2 and details in Janeček et al [8]).

In order to characterize microstructure changes during annealing we will compare the final conditions of the isothermal annealing (after 64 hours) in the temperature range of 170 - 210 °C in which microhardness drop was found and therefore grain structure variations are expected to occur due to static recovery and grain growth. The grain structure after long term exposure (64 hours) in this temperature range will be characterized both by light microscopy using Nomarski contrast and by scanning electron microscopy using secondary electron signal.

The microstructure of sample annealed at 170 °C for 64 hours is shown in Figs. 3a (LM) and 4a, 5a (SEM). It is seen that in this condition the ultra-fine grained character of microstructure remains with the majority of very fine grains (approximately 80% of the volume). However, first larger grains of the sizes ~ 5 - 10 μm started to be formed.

The bimodal character of grain structure is also observed at long term annealing at 190 °C, see Figs. 3b (LM) and 4b, 5b (SEM) consisting of agglomerates of ultra-fine grains and large grains of the sizes ~ 5 – 15 μm. However, the fraction of corse grains significantly increases as compared to annealing at lower temperature. Approximately the same fraction of coarse and fine grains was found in this specimen.

Annealing at the highest temperature from the range of microhardness drop (210 °C) results in much more homogeneous and coarser grain structure as compared to previous conditions as depicted in Figs. 3c (LM) and 4c - 5c (SEM). Coarse grains of the average size of ~ 5 – 15 μm prevail in the microstructure. However, a certain fraction of fine grains still remains.

Fig. 3d (LM) and 4d - 5d (SEM) present the microstructure of the condition upon annealing at 400 °C for 1 hour, where the processes of recovery and grain growth are expected to be mostly developed due to very low values of microhardness. The microstructure in this condition is much more homogeneous in comparison with the isothermally annealed specimens. It consists of larger grains of the average sizes ~ 5 – 20 μm only.

Microstructure observations confirmed that different microstructures correspond well to the different microhardness values measured at isochronally and isothermally annealed specimens.

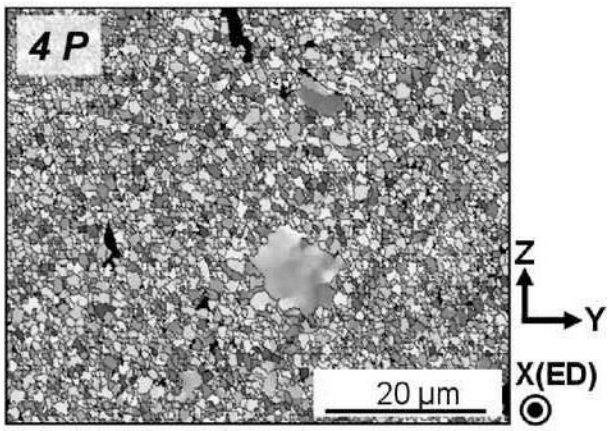
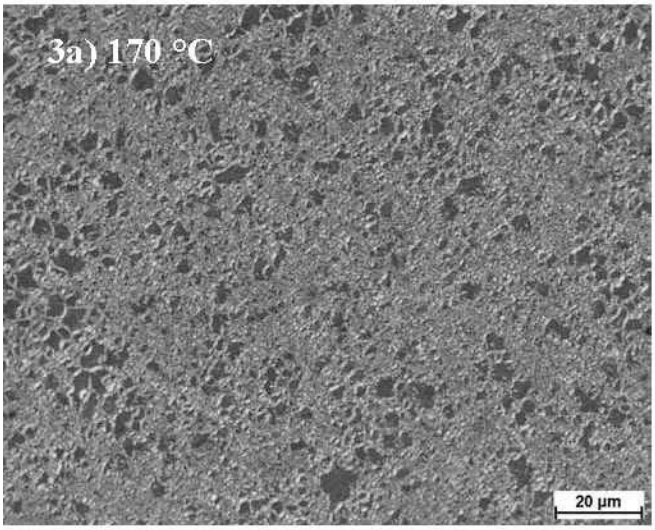
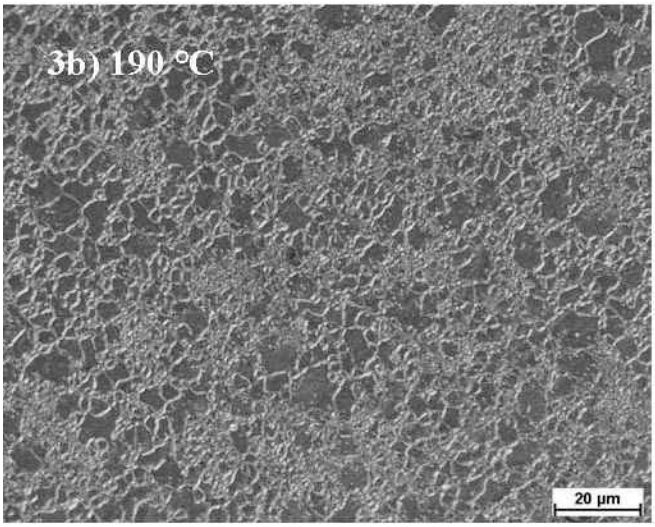
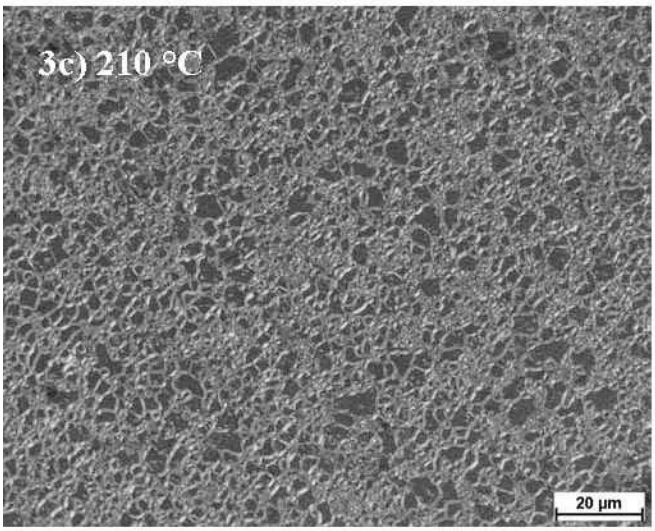


Figure 2: Microstructure of the AZ31 alloy after extrusion and 4 passes of ECAP (EBSD) [8].







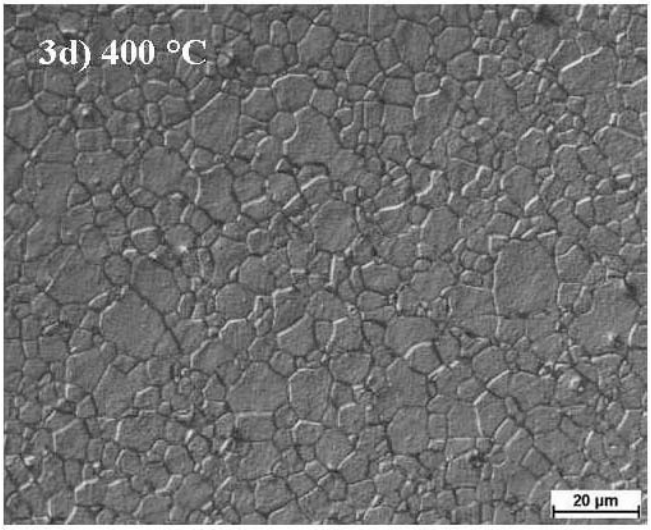
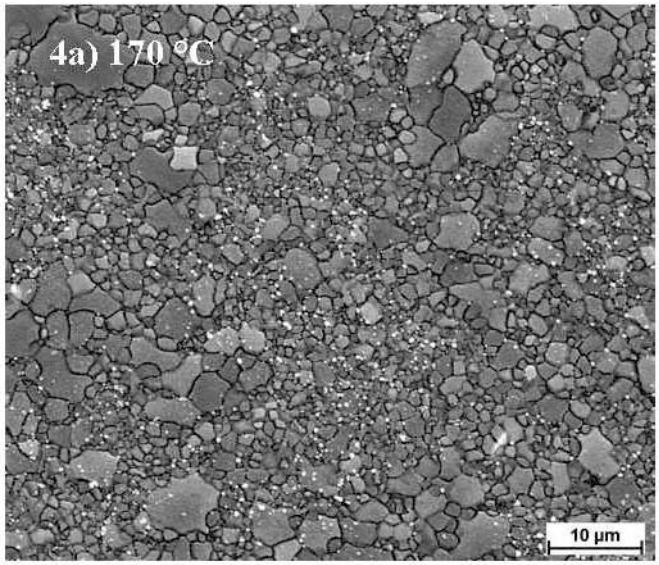
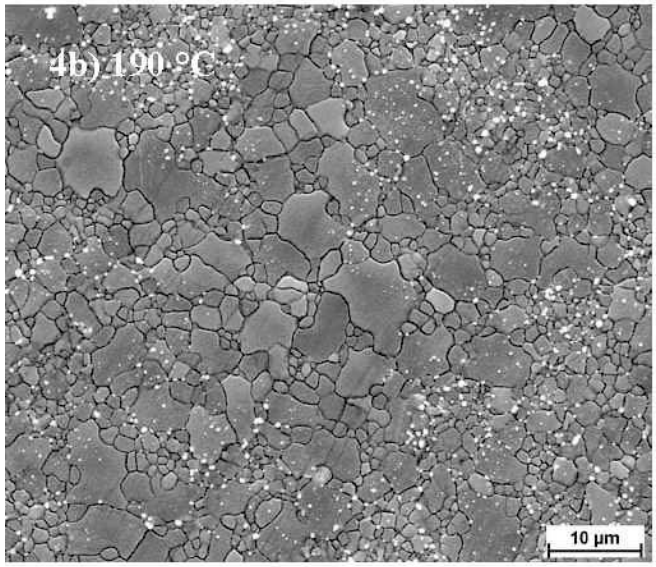
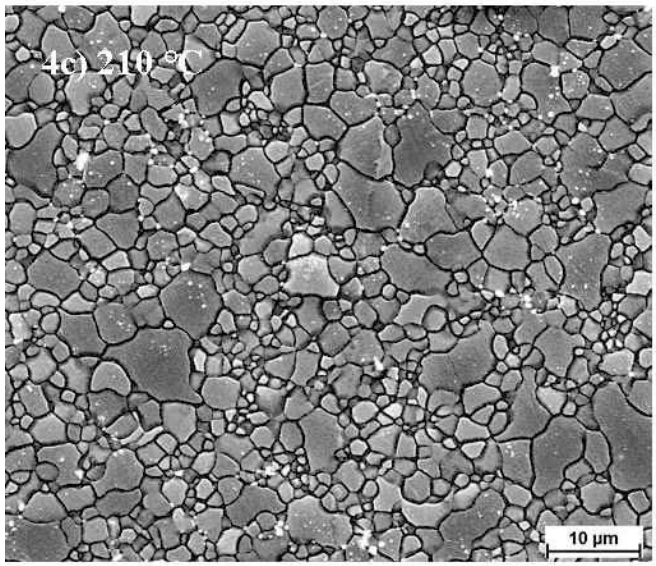


Figure 3: Microstructure of the AZ31 alloy after annealing: a) 64 hours at 170 °C, b) 64 hours at 190 °C, c) 64 hours at 210 °C and d) 1 hour at 400 °C. (Light microscopy, magnification 1000×, etchant picric acid, Nomarski contrast)







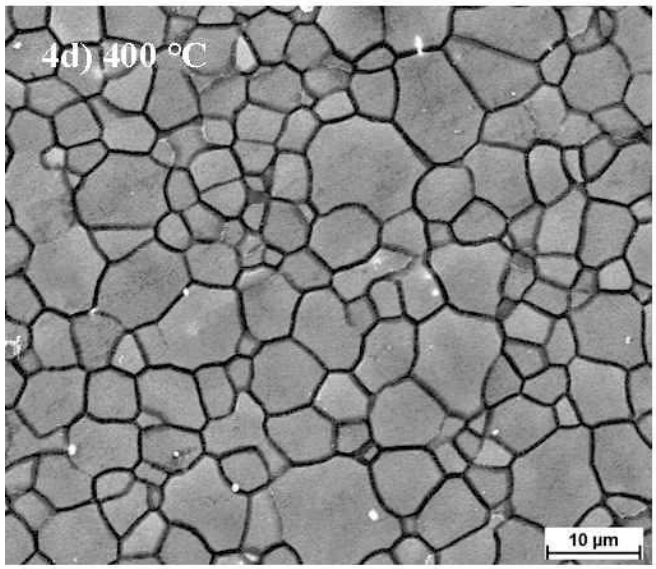
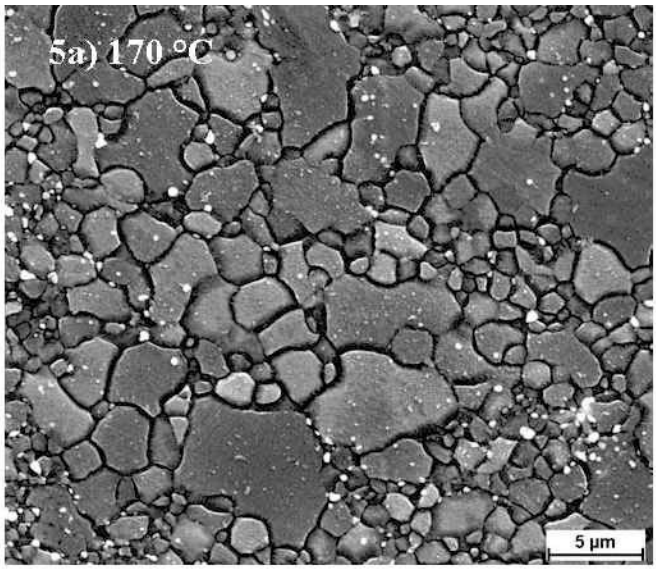
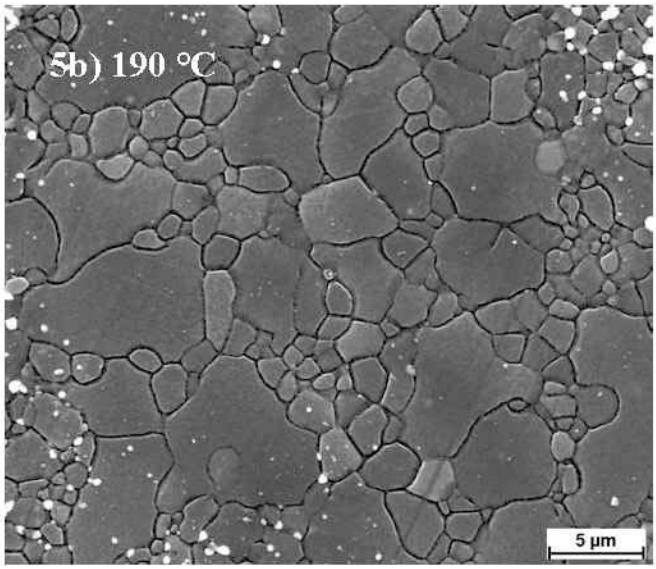
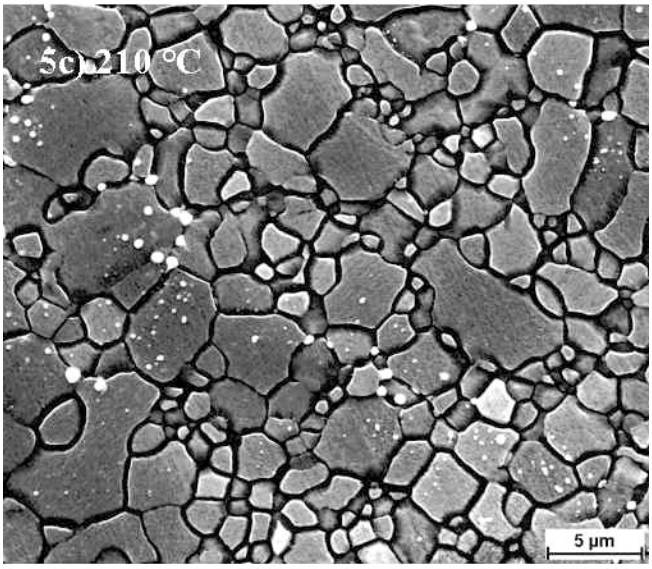


Figure 4: Microstructure of the AZ31 alloy after annealing: a) 64 hours at 170 °C, b) 64 hours at 190 °C, c) 64 hours at 210 °C and d) 1 hour at 400 °C. (Scanning electron microscopy, secondary electron signal, magnification 4000×, etchant picric acid)







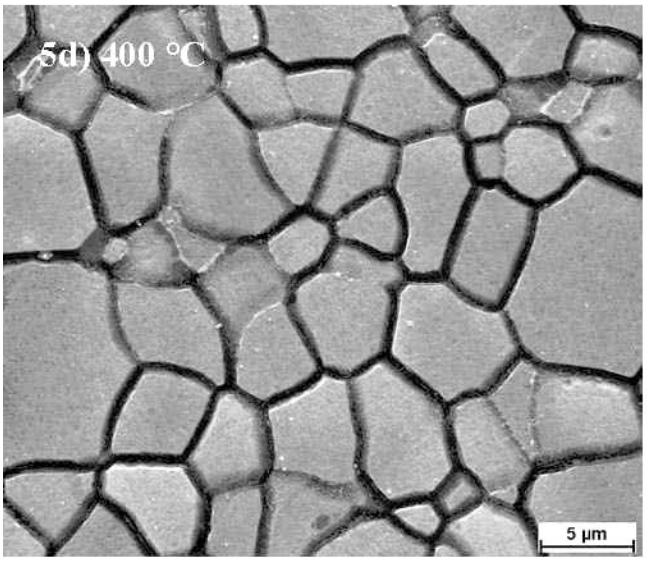


Figure 5: Microstructure of the AZ31 alloy after annealing: a) 64 hours at 170 °C, b) 64 hours at 190 °C, c) 64 hours at 210 °C and d) 1 hour at 400 °C. (Scanning electron microscopy, secondary electron signal, magnification 8000×, etchant picric acid)

Conclusions

The microstructure and microhardness of ultra-fine grained (UFG) magnesium alloy AZ31 and its evolution with annealing temperature and time were investigated. The following conclusions may be drawn from this work:

- The isochronal annealing leads to decrease of the microhardness (from 86 to 59 HV0.1) and significant coarsening of the initial UFG microstructure.
- The isothermal annealing measured at 170, 190 and 210 °C results in microhardness decrease to the values of 76, 72 and 67 HV0.1 and formation of inhomogeneous microstructure.

Acknowledgements

This work was financially supported by the Czech Grant Agency (GACR) under the Grant 106/09/0482. J. Vrátná acknowledges finacial support by GAUK 530712/2012 and SVV 2012-265303.

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