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Microstructure stability of ultra-fine grained magnesium alloy AZ31 processed by extrusion and equal-channel angular pressing (EX–ECAP)

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ABSTRACT

Thermal stability of the ultra-fine grained (UFG) microstructure of magnesium AZ31 alloy was investigated. UFG microstructure was achieved by a combined two-step severe plastic deformation process: the extrusion (EX) and subsequent equal-channel angular pressing (ECAP). This combined process leads to refined microstructure and enhanced microhardness. Specimens with UFG microstructure were annealed isochronally at temperatures 150–500 °C for 1 h. The evolution of microstructure, mechanical properties and dislocation density was studied by electron backscatter diffraction (EBSD), microhardness measurements and positron annihilation spectroscopy (PAS). The coarsening of the fine-grained structure at higher temperatures was accompanied by a gradual decrease of the microhardness and decrease of dislocation density. Mechanism of grain growth was studied by general equation for grain growth and Arrhenius equation. Activation energies for grain growth were calculated to be 115, 33 and 164 kJ/mol in temperature ranges of 170–210 °C, 210–400 °C and 400–500 °C (443–483 K, 483–673 K and 673–773 K), respectively.

1. Introduction

Magnesium alloys belong to materials with potential to replace some conventional structural materials in automotive, aircraft and other industry branches. Magnesium is a very light metal with relatively good mechanical properties which result in its expanding use in weight-critical applications. Interest in magnesium-based materials has recently been revived primarily

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due to its gradually decreasing cost and the determination of scientists, researchers and engineers to cut down energy consumption and greenhouse gas emissions [1].

Ultra-fine grained (UFG) materials with submicrometer or even nano-scale grain sizes can be produced by severe plastic deformation (SPD) techniques. These methods are very efficient in achieving significant grain refinement in polycrystalline metals. These UFG materials have usually superior mechanical properties including high strength and, if the UFG microstructure is sufficiently stable, a superplastic capability at elevated temperatures [2,3]. Nowadays, the most attractive procedures for processing by SPD are equal-channel angular pressing (ECAP) [4] or combined process of extrusion followed by ECAP (EX–ECAP) [5], high-pressure torsion (HPT) [6] and accumulative roll-bonding (ARB) [7]. In practice, ECAP or EX–ECAP processes are especially attractive because of their simplicity in laboratory operation. Moreover, these processes can produce relatively large billets for industrial applications [8]. There are several reports to date of the successful processing of magnesium AZ31 alloy using ECAP at elevated temperatures and employing different processing procedures [9–13].

Extremely small grain sizes of polycrystalline materials usually lead to enhanced mechanical properties. However, grain size refinement achieved by ECAP or EX–ECAP may not play a major role in the yield stress of the material. H.K. Kim [14] claims that the AZ31 alloy after 1 and 2 ECAP passes with smaller grain sizes shows a lower yield stress compared to the extruded only (no ECAP passes) material with large grains due to texture modification during the ECAP process. According to his predictions of flow stress, the strain hardening is the largest contributor to flow stress whereas grain size refinement plays a relatively minor role in ECAPed specimens. Masoudpanah et al. [15] also reported lower yield stress of the AZ31 alloy after 4 ECAP passes compared to the extruded state. Jin et al. [16] studied in detail the microstructure evolution during ECAP, proving the rearranging of dislocations induced in the initial stage of ECAP and their formation into sub-grain boundaries. The newly created dislocations in sub-grains are absorbed by the sub-boundaries with the increasing strain induced by additional ECAP passes. The misorientations between sub-boundaries gradually increase and evolve to low and high-angle grain boundaries. Nevertheless, functional properties like biocompatibility, special magnetic properties or corrosion resistance (investigated in our previous paper [17]) should be also considered.

The practical applications of the UFG materials are limited due to low microstructure stability at elevated temperatures that complicates the processing of final products. Thermal stability depends on many variables, such as stacking fault energy of the material, processing or properties of grain boundaries [18]. Microstructure stability can be improved by various alloying elements or composite reinforcements. Microstructure stability of the AZ31 alloy after ECAP was studied by Kim [19] or by Radi and Mahmudi [20], who investigated the AZ31 alloy reinforced by alumina nano-particles. Both papers present calculations of activation energies for grain growth which identified two or three temperature regimes with significantly different values of activation energy.

The main objective of this work is the investigation of microstructure stability during annealing of the UFG AZ31 magnesium alloy prepared by extrusion and 4 passes of ECAP.

2. Material and Methods

As-cast commercial AZ31 alloy (nominal composition of Mg–3%Al–1%Zn) was first extruded at 350 °C with an extrusion ratio of 22. Subsequently, it was processed by equal channel angular pressing. ECAP pressing was performed at 180 °C following route B_C, i.e. rotating the sample 90° between the individual passes, with the velocity of 50 mm/min. The angle Φ between two intersecting channels and the corner angle Ψ were 90° and 0°, respectively. Both channels have a square cross section of 10 mm × 10 mm. The ECAP die was equipped with an ejector that allows pushing the sample out of the die immediately after pressing from the feed-in channel to the exit channel. Molybdenum disulfide grease was used as a lubricant. All the specimens were pressed four-times through the ECAP die.

Investigated flat specimens with approx. 2 mm thickness were cut from the middle part of the billets perpendicular to the pressing direction. Microstructural observations and microhardness measurements on planes parallel to the pressing direction are very similar to the perpendicular one [21] and they are not studied in this work.

Series of specimens for thermal-stability investigation were prepared by isochronal annealing at the temperatures 150–500 °C for 1 h followed by water-quench.

Specimens were mechanically grinded on watered abrasive papers, and then polished with polishing diamond suspension of grades 3, 1 and 1/4 μm and alumina suspension of grade 0.05 μm. Using this procedure, flat samples for Vickers microhardness measurements (load 100 g, 10 s) with minimum surface scratches were obtained. Finally, the specimens' surface was polished by argon ions (Gatan PIPS™) which enables EBSD measurements. Specimens for positron annihilation spectroscopy measurements were additionally etched for 30 s in the mixture of nitric acid, water and ethylene glycol.

Microhardness was measured by LECO M-400-A microhardness meter. A FEI Quanta 200 FX scanning electron microscope equipped with EDAX EBSD camera and OIM software was utilized for EBSD observations.

A $^{22}Na_2CO_3$ positron source with the activity of 1.5 MBq was used in positron lifetime measurements. The source spot with diameter of 1 mm was deposited on a 2 μm thick Mylar foil and sealed between two identical specimens of the studied material. The source contribution consists of two components with lifetimes of 368 ps (intensity 8%) and 1.5 ns (intensity 1%) which come from positrons annihilated in the $^{22}Na_2CO_3$ source spot and in the covering Mylar foil, respectively.

A fast–fast spectrometer [22] with the time resolution of 150 ps (FWHM ^{22}Na) was employed for positron lifetime measurements. At least 10^7 positron annihilation events were accumulated in each positron lifetime spectrum which was subsequently decomposed into individual exponential components by a maximum likelihood procedure [23].