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Annealing response of binary Al–7Mg alloy deformed by equal channel angular pressing

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ABSTRACT

The annealing response in a binary Al–7Mg alloy processed at room temperature by equal channel angular pressing (ECAP) has been investigated via X-ray diffraction (XRD), electron-probe micro analysis (EPMA) and electron backscattering diffraction (EBSD). After ECAP and subsequent annealing, Mg remains mainly homogeneously distributed in solid solution. A bimodal structure with ultrafine grains accompanied by micrometer-sized crystallites was developed after 3 passes. Upon annealing at ~275 °C for 96 h, extensive recovery was observed in the as-deformed material, leading to a relatively uniform microstructure; at ~300 °C a discontinuous recrystallization initiated in less than 30 s with subsequent grain growth clearly evident. Such remarkable thermal stability, i.e., slower recovery and recrystallization kinetics, of the present material, in contrast to other severely deformed commercial pure Al and Al–Mg alloys, is due mainly to the presence of high Mg solid solution contents, the formation of the bimodal structure consisting of both coarse crystallites and ultrafine grains. In addition, the possible Mg-containing precipitates during annealing might also retard the recrystallization kinetics.

1. Introduction

Quite a few studies have been reported on equal channel angular pressing (ECAP) processing of work hardening Al–Mg alloys [1–7]. The role of Mg-alloying in obtaining an ultrafine grained (UFG) Al-matrix structure during severe plastic deformation (SPD) is quite well understood [1,8]. So far, focus has been devoted mainly to deformed microstructures, annealing responses and mechanical behavior of ECAPed Al–Mg alloys with Mg-contents up to 3% [1–4,6,7]. After ECAP the material appears to retain high levels of internal strain and a majority of the grain boundaries in non-equilibrium configurations, necessitating further evaluation of their thermal stability [1,2]. Systematic investigations of the annealing response have been reported for Al–3Mg processed by 8 passes of room-temperature ECAP via route A [1,2,7].

Generally, increase of the Mg content leads to a decrease in the stacking fault energy, causing further grain size refinement during ECAP [9]. It is therefore of interest to investigate Al–Mg alloys with higher Mg alloying, mainly as candidates for the production of ultrafine microstructures leading to high mechanical hardness and strength. However, cracks and failure have been reported as a reoccurring problem in room-temperature ECAP of Al–Mg alloys with >4 wt% Mg [9,10].

To the best of our knowledge, up till now, studies have not been reported on the annealing response in room temperature-ECAPed Al–Mg alloys with >5 wt% Mg. The present investigation was therefore initiated to reveal the microstructural evolution upon annealing in an Al–7Mg alloy subjected to 3 passes of ECAP, and in particular to study the recovery and recrystallization behavior in such a severely deformed, high solid solution alloy.

2. Experimental

Materials used for the present work were taken from Al–7Mg cast ingots supplied by Hydro Aluminum having chemical composition (in wt%): Mg 7.0, Fe 0.05, Si 0.06 and Al in balance. Before ECAP, samples dimensioned $19.5 \times 19.5 \times 100$ mm^3 were homogenized in an air circulation furnace for 3 h at 500 °C, followed by water quenching. The as-homogenized billets were deformed in a 90° ECAP die giving a strain of $\varepsilon = 1$ in each pass [11], applying the Bc route at room temperatures. To lower the friction during pressing, samples were coated with a graphite lubricant. The ECAP process was interrupted after 3 passes, as severe cracks had extended to the center thickness of the billets. Samples for subsequent hardness testing and annealing experiments were cut from the uniformly deformed central region of the billets. Depending on the selected

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temperature, isothermal annealing treatments were carried out with an air furnace (~275 °C) or molten salt baths (~300–400 °C), and with annealing times varying with the temperature. The temperature was controlled within ±3 °C in all cases. Immediately after annealing, samples were water-quenched to room temperature. Samples for X-Ray diffraction analysis (XRD), electron probe micro analysis (EPMA) and electron backscatter diffraction (EBSD) studies were prepared by standard metallographic techniques, i.e., grinding on successively finer grained SiC papers down to 1200 ASTM mesh, followed by mechanical polishing through 3- and 1-μm diamond paste, and thereafter, electro polishing with a solution of 80 pct C_2H_5OH + 20 pct $HClO_4$ at 20 V for 15–25 s at −30 °C. The TEM foils were prepared by twin-jet electro polishing in a solution of 33% nitric acid-methanol at −30 °C.

XRD was undertaken with a Siemens D5000 at 40 kV, Cu K_α radiation, with 0.02 step size and 20 s times/step. EPMA was carried out in a JXA-8500F at 15 kV with 1 μm scan steps. TEM analysis was performed with a Philips CM30 operating at 150 KV. EBSD analysis was carried out with a Zeiss 55VP FEG-SEM equipped with a Nordif EBSD detector and TSL OIM software [12], at 20 kV, 20 mm working distance, 70 deg tilt, and 0.05–0.1 μm scan steps. The grain construction has been done by using a grain tolerance angle of 5 deg with a minimum grain size of 5 and a minimum confidence index of 0.1. The minimum grain size defines the number of scan points needed to identify a group of neighboring and similarly oriented points as a grain in the OIM software [13]. It should be noted that for the as-ECAPed sample, the quality of the Kikuchi patterns obtained from EBSD was too low for proper indexing by the OIM software. EBSD analysis was therefore done on as-ECAPed sample after low-temperature annealing (~200 °C for 30 min). As only a slight decrease of ~10 Hv in hardness is detected, it is assumed that only very weak recovery occurred and the obtained EBSD information roughly represents the as-deformed structure.

The volume fraction of recrystallized grains in Fig. 6 was measured by a simple point counting technique (ASTM E562-02). Two to four digital optical micrographs were taken for each sample at magnifications from 100× to 200×, depending on grain sizes. Next, 11 × 14 point grid was superimposed on the images. The volume fraction of recrystallized grains was determined by calculating the ratio of the number of points located within recrystallized grains to the total number of points. Grain sizes in Fig. 6 were determined from optical micrographs by the linear intercept method for recrystallized grains (counting >300 grains), and by measuring lengths and widths for unrecrystallized ones (counting >50 grains).

Vickers hardness tests were performed on samples prepared for microstructural observations using a load of 500 g. At least six separate measurements were performed on each sample.

3. Results and discussion

3.1. Distribution of Mg atoms

Fig. 1 shows XRD patterns of the Al–7Mg materials at different states. The theoretical diffraction peak positions of pure aluminum (a=4.0412 Å) are presented by the vertical dotted lines for comparison. For all samples only peaks consistent with fcc Al are observed. Appreciable shifts in peak positions towards lower scattering angles are observed for all samples, as compared to the pure Al phase, showing lattice expansion that could be ascribed to solid solution of Mg. A least square refinement has been done to evaluate the lattice constants, showing that the as-ECAP'ed, 200 °C-annealed, 275 °C-annealed and 300 °C-annealed sample has a lattice parameter of 4.0845 ± 0.0002 Å, 4.0800 ± 0.0001 Å, 4.0743 ± 0.00002 Å and

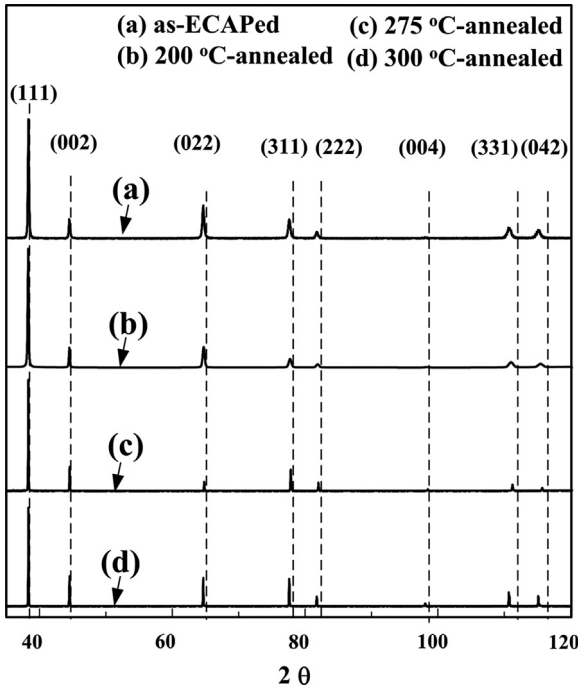

Fig. 1. XRD pattern of Al–7Mg at different state: (a) 3 passes ECAP, (b) annealed at 200 °C for 30 min, (c) annealed at 275 °C for 96 h, and (d) annealed at 300 °C for 120 s.

4.0854 ± 0.00001 Å, respectively. Obviously, the lattice parameter is reduced after annealing at 275 °C for 96 h, but keeps almost invariant after annealing at 300 °C for 2 min. The lattice parameter a of Al–Mg alloys is directly linked to the amount of Mg in solid solution (1 at% Mg resulting in a change of a by 0.0046) [14]. The decrease in the lattice parameter after annealing, i.e., Δa=0.0045 for the 200 °C-annealed sample, Δa=0.0102 for the 275 °C-annealed sample, can partly be related to a loss of Mg in solid solution. Such a feature could be the result of segregation of Mg atoms or precipitation of Mg-containing phase. However, no Mg-containing precipitates have been detected by XRD, due probably to their small volume fraction. Closer examination reveals apparent peak broadening in Fig. 1(a) and (b), indicative of the significant internal lattice strain imposed by ECAP still existed and little dislocation recovery occurred during annealing at 200 °C for 30 min. However, the peaks become much sharper in Fig. 1(c) and (d), indicating a significant dislocation recovery during annealing at 300 °C for 2 min. In addition, comparing integrated intensities, I_{hkl}, extracted by the use of the TOPAS software [14], shows that the ratio I_{200}/I_{111} and I_{200}/I_{220} increased from 0.15 and 0.59 in Fig. 1(a) to 0.44 and 1.06 in Fig. 1(d), respectively, indicating that strong textures from the ECAP process are reduced during annealing at 300 °C for 2 min.

Fig. 2 presents EPMA results from the line scanning (see line AB, CD and EF in Fig. 2) of the as-homogenized, as-ECAP'ed and as-annealed sample, respectively. As can be seen, the Mg distribution is quite homogeneous under all conditions, except for some minor deviations at grain boundaries in the as-homogenized sample (as indicated by arrows in Fig. 1(b)), mainly due to the local etching caused by electro polishing. However, grain boundaries cannot be clearly distinguished in the as-ECAP'ed material mainly due to the fine grain sizes (see Figs. 7 and 8). The EPMA results suggest that there is no obvious segregation and the major part of Mg is kept in solid solution in the Al-matrix. This result seems reasonable for the as-homogenized and as-annealed samples, taking into account the equilibrium solid solubility of Mg in Al is ~7.2 wt% at 300 °C [15], slightly larger than the Mg contents in the present Al–7Mg. However, the segregation of Mg along the grain boundaries in UFG Al alloys processed by SPD has been frequently observed by atomic scale