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Microstructure evolution of 7003 Al alloy by equal channel angular extrusion process

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Abstract

The 7003 Al alloy was prepared by equal channel angular extrusion process (ECAP), which microstructures were characterized by transmission electron microscope (TEM). Results showed that dislocation density increases obviously and the average grain size decreases with increasing passes of ECAP. However, after the fourth pass of ECAP, average grain size fails to decrease remarkably and the microstructure is still inhomogeneous. Some parallel micro-bands and the second phase MgZn2 are present in the alloy during annealing.

Introduction

The homogenization and refinement of microstructure by deformation processing is beneficial to the mechanical properties of engineering materials. Highly refined grains contribute to ambient strength and may lead also to low-temperature superplasticity[1,2]. Many studies have shown that ultra-fine grain sizes, in the sub-micrometer or even nanometer range, may be achieved by imposing extremely large plastic strains through processing by severe plastic deformation (SPD) [3,4]. Equal channel angular pressing (ECAP) has been widely studied as a promising SPD method. However, the dependence of high-angle boundaries development on ECAP process parameters (e.g., die geometry) and material properties [5–6] has not been clarified and there is little information on the mechanisms by which the ultra-fine grains and high-angle boundaries evolve in the deformation-induced microstructures.

Typically, ECAP is carried out at low homologous temperatures and it has been shown that the resulting deformation induced microstructures evolve in a manner similar to those produced by cold-working [7]. Recent overviews [8,9] have described cold-worked microstructures and provided catalogs of various features that evolve during cold-rolling. The processes of grain subdivision include the formation and evolution of lamellar boundaries (LBs), microbands (MBs) and dense dislocation walls (DDWs) as well as subgrains and cellular substructures. The LBs, MBs and DDWs elongate as the prior grains deform and the LBs and MBs become band-like features with high disorientation boundaries that eventually align with the rolling direction. These features generally separate regions within prior grains that have experienced lattice rotation in opposite senses away from the original grain orientation. Deformation banding (DB) is also a process of grain subdivision wherein the lattice rotates in different senses in adjacent regions of the prior grains but towards symmetrically-related end orientations in the texture[10]. Processes of grain subdivision and high-angle boundary formation in ECAP materials have been cited recently [9,10], analogous to processes in rolled materials [11,12]. The differences in the relative populations of low- and high-angle boundaries reflect, in part, different experimental techniques. Most investigations have used transmission electron microscopy (TEM) and there have been few meso-scale studies of texture and DB in association with ECAP [9].

The present report documents the results of such an investigation of microstructure and grain boundary statistics during the repetitive ECAP of 7003 Al alloy. The TEM data show that the band–band interfaces are well-defined boundaries. The results lead to a proposed mechanism for the development of high-angle boundaries where this mechanism is consistent with proposals in the literature [10].

1. Experimental procedures

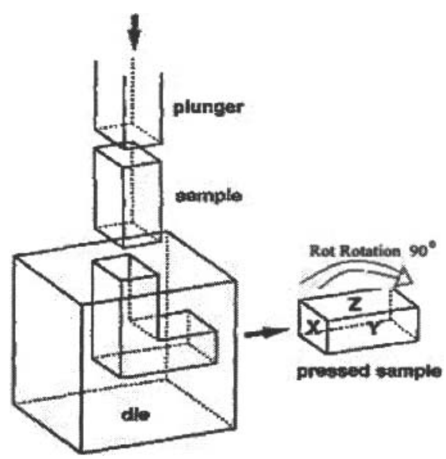


Fig.1 Diagram of Equal Channel

Table 1 – Chemical composition of the 7003 Al alloy samples studied

Table 1 – Chemical composition of the 7003 Al alloy samples studied

Elem.	Zn	Mg	Cu	Fe	Si	Mn	Ti	Al
Wt.%	5.67	0.76	0.08	0.11	0.04	0. 26	0. 05	Bal

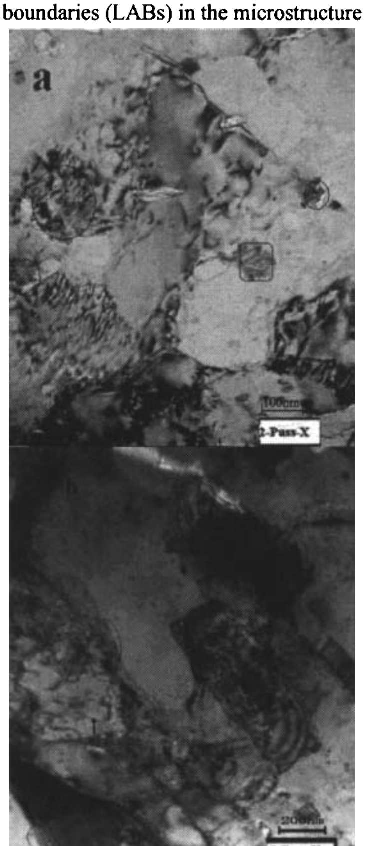
The experiments were conducted using commercially the 7003 Aluminum alloy. Table 1 presents the chemical composition of the Al alloy used in this study. The material was cut to provide billets with dimensions of 10mm×10mm×30 mm. Before pressing, samples were annealed at 400°C for 2 h and a fully recrystallized microstructure with an approximate grain size of 50 μm was obtained. The ECAP process was carried out at room temperature through a die having an intersecting channel angle of 90° and a sharp outer corner (see to figure 1). Mixture of MoS_2 and lube oil was used as a lubricant. Samples were pressed up to 5 passes using the processing route of B_C denoting a 90° rotation in the same direction between each pass. The microstructures of the

ECAP-processed materials were characterized by a Philips EM-420 TEM without Kikuchi-line analysis. For the TEM analysis, after mechanical polishing, thin foils were made by twin-jet electro-polishing in100 mL $HClO_4$+900 mL CH_3OH solution at room temperature and a voltage of 50 V.

2. Experimental results

2.1.Microstructure in 7003 aluminum alloy processed by ECAP

After subjected to 2-pass and 3-pass ECAP respectively, samples were sectioned along a plane parallel to the X-plane (i.e. normal to the X-direction,see to Fig. 1) and thin discs were extracted from the central zone parallel to the X-plane. TEM micrographs of fig.2 (a) and (b) revealed a fine subgrain structure and inhomogeneous microtextures and microstructures in the alloy after 2-pass and 3-pass ECAP. After subjected to 4-pass and 5-pass ECAP respectively, samples were sectioned along a plane parallel to the Y-plane (see to Fig. 1) and thin discs were extracted from the central zone parallel to the Y-plane. TEM micrographs of fig.2 (c) and (d) revealed some tilting deformation bands besides a further fine subgrain structure and distinct inhomogeneous microtextures and microstructures in the alloy after 4-pass and 5-pass ECAP. There are some micro-bands (MBs) and dense dislocation walls (DDWs) assembling together in the selected zone of figure 2. The fraction of high-angle boundaries (HABs) and low-angle boundaries (LABs) can be easily estimated because contrast at high-angle boundaries is distinct higher than that at low-angle boundaries according to Kikuchi patterns's effect. It is shown from fig.2 that high-angle boundaries (HABs) are present as well as low-angle boundaries (LABs) in the microstructure of the alloy.



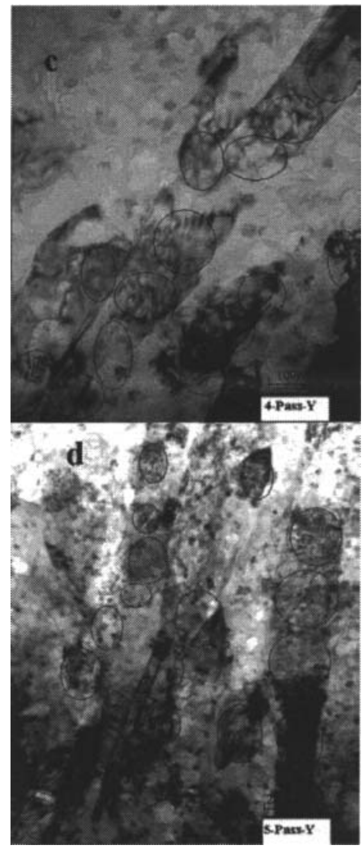


Fig. 2 The TEM micrographs of the 7003 Al alloy samples subjected to (a)2-pass ECAP,(b)3-pass ECAP,(c) 4-pass ECAP and (d) 5-pass ECAP

2.2.Influence of anneal conditions on microstructure in 7003 aluminum alloy after 4-pass ECAP

Thermal stability was studied by annealing the alloy at 80℃, 120℃,160℃ and 200℃ for 1 h and by annealing the alloy at 160℃ for 30min, 60min and 90min after 4-pass ECAP. It is shown from fig.3 a,b,c and fig.4 a,b that, the TEM micrographs consist of inhomogeneous microtextures and microstructures, and dense dislocation zones are still present and fraction of high-angle boundaries is still high in the alloy. However, it is well known from fig.3 d that fraction of low-angle boundaries remarkably increases, dense dislocation zones distinctly decrease and some parallel micro-bands are present in selected area, and that the grains are non-uniform in size throughout the sample. Besides dense dislocation zones distinctly decreasing, relatively homogeneous equiaxial grains are present in fig.4 c. The selected black area in fig.4 b consists of $MgZn_2$ and Al phases according to electron diffraction pattern of fig.4 d.

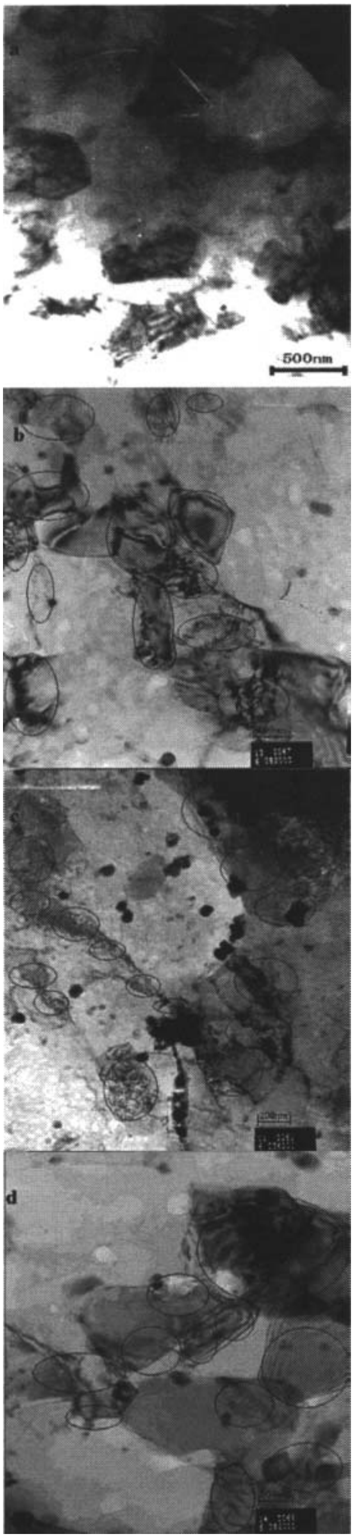


Fig.3 The TEM micrographs of the 7003 Al alloy samples subjected to 4-pass ECAP after annealing at (a)80℃,(b) 120℃, (c)160℃ and (d)200℃ respectively for 1h.

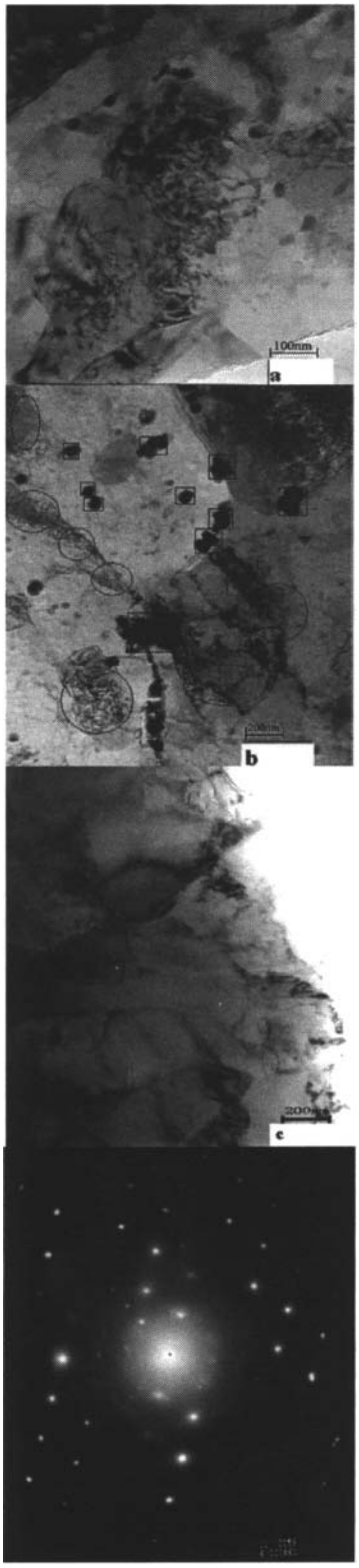


Fig.4 The TEM micrographs of the 7003 Al alloy samples subjected to 4-pass ECAP after annealing at 160℃for (a)30min,

(b)60min and(c)90min respectively,(d) the selected area electron diffraction pattern

3. Discussion

3.1. Microstructure evolution of 7003 aluminum alloy by ECAP

High-angle boundaries (HABs) and low-angle boundaries (LABs) can be easily distinguished because the contrast at high-angle boundaries is different from that at low-angle boundaries according to Kikuchi patterns's effect. Concentration of dislocations at high-angle boundaries is usually more than that at low-angle boundaries. Fraction of high-angle boundaries and concentration of dislocations in 7003 Al alloy increase during ECAP, and distribution of fine subgrain structure and microtextures is distinct inhomogeneous in Al alloy during ECAP. 7003 Al alloys after 4-pass ECAP are subjected to annealing at 200℃ for 1h or at 160℃ for 1.5h. Besides fraction of high-angle boundaries and concentration of dislocations remarkably decreasing, there are some parallel micro-bands in the selected area of the alloy annealing at 200℃ for 1h (see to fig.3 d) , and relatively homogeneous equiaxial grains in the alloy annealing at 160℃ for 1.5h (see to fig.4 c).

3.2. Grain refinement in 7003 aluminum alloy processed by ECAP

Shear plastic deformation in ECAP occurs through the formation, movement and storage of dislocations. In fact, microstructure evolution during ECAP is directly linked to a complex evolution of dislocation networks and their recombination and annihilation phenomena. Imbalance of dislocations of opposite signs moving in opposite directions (or having some Burgers vector components of opposite sign and sliding direction) leads to accumulation, generally at the grain boundary, or at high-angle boundaries (HABs), of a surplus of dislocations of the same sign. The groups of excess dislocations in fact stimulate crystallographic slip. Therefore, the evolution and accumulation of misorientation across both low-angle boundaries (LABs) and HABs is closely linked to the crystallographic accommodation of each cell, grain and the neighbouring crystallites during the shearing deformation. Route B_C is effective in refining the microstructure, because four sets of shear bands are created after a fully redundant cycle (4n passes) [11].

Under severe plastic deformation (SPD), and for every possible route in ECAP, dislocation boundaries evolve into a regular pattern of grain subdivisions belonging to two scales [12–13]: large-scale long and continuous dislocation boundaries, called geometrically necessary boundaries (GNBs), and small-scale incidental dislocation boundaries (IDBs) [13]. The former include micro-bands (MBs) and dense dislocation walls (DDWs) surrounding groups of equiaxed cells. The groups of cells enclosed by GNBs are called cell blocks (CBs), while IDBs include ordinary cell boundaries. The boundary spacing and misorientation angle distribution of GNBs and IDBs evolve differently as a function of strain; in particular, they exhibit different morphologies at small to medium strains, but similar at high strains [14]. Generally, misorientation axes for IDBs are randomly distributed, whereas GNB orientation distribution clusters on preferred axes and some GNBs show a pattern of rotation around the transverse direction [15]. GNBs play an important role in the deformation process.

4. Conclusions

(1) Processing by ECAP of 7003 Al alloy leads to the development of an inhomogeneous microstructure after two passes. Repetitive ECAP pressing will produce a relative homogenization of the deformation microstructure.
(2) A TEM investigation of route B_C confirm that alternating bands of distinct orientations are formed in the processing of 7003 Al alloy by ECAP. These deformation bands became especially pronounced after 4 passes of ECAP and there is further refinement of the microstructure with increasing number of processing pass.
(3)Study on thermal stability of 7003 Al alloys after 4-pass ECAP confirms that fraction of high-angle boundaries and concentration of dislocations remarkably decrease during annealing, there are some parallel micro-bands in the selected area of the alloy annealing at 200℃ for 1h , and relatively homogeneous equiaxial grains in the alloy annealing at 160℃ for 1.5h.

Acknowledgements
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