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Characterization of microstructure and deformation texture during equal channel Angular pressing of Al–Zn–Mg–Cu alloy

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ABSTRACT

The aim of this paper was to examine the microstructure and texture that develops during equal channel angular pressing (ECAP). Al-7075 alloy was subjected to 4 passes of ECAP deformation at room temperature by using Route B_C and A. The texture was investigated by X-ray diffractometer (XRD) in ED-plane (plane perpendicular to extrusion direction) as well as TD-plane (plane parallel to extrusion direction). The texture calculation by Labotex software reveals that in 1 pass ECAPed specimens, C_θ is the strongest texture component, while after 4 passes B_θ/B'_θ and $A^*_{1\theta}$ are the strongest components in route A and B_C, respectively, and also the results in both TD (z) and ED (x) planes are in good agreement. Texture strengthening was observed after the initial pass but there was evidence for texture weakening after 4 passes. Microstructure investigation by transmission electron microscopy (TEM) shows that ultrafine-grained materials with grain size less than 500 nm could be obtained after 4 passes of ECAP process.

1. Introduction

Equal channel angular pressing (ECAP) or extrusion (ECAE) is known as the most effective tool among the potential severe plastic deformation (SPD) processes, for obtaining ultrafine-grained bulk materials with extraordinary mechanical properties [1]. In Equal channel angular pressing, grain refinement is achieved by severe plastic deformation during multiple extrusions of the billet through a die with two channels of equal cross section intersect at an abrupt angle, Φ, and with a corner curvature angle, Ψ, [2,3]. Due to the frequent strain path changes and severe plastic deformation involved in multi-pass ECAP, microstructure and texture evolution has proven to be most sensitive to rotation of the billet between successive passes (route) and pass number (N). The most common processing routes to date are termed A, C, B_A and B_C according to the rotation of the ECAP billet around the specimen's longitudinal axis. [3–10].

Since the fragmentation of grains is orientation dependent, investigation into the texture evolution in the material is essential for understanding mechanisms of plastic deformation and grain refinement during ECAP [11]. The large plastic deformation and strain-path changes during ECAP process leads to significant and complex changes of crystallographic texture [12]. To date, many theoretical (analytical or numerical) and experimental studies on polycrystalline materials have been conducted to investigate the effects of material properties and processing variables on ECAP texture development [7–26]. For a given material, the texture evolution mainly depends on applied deformation (such as processing route, number of passes (N) and die angle (Φ)), deformation mechanism (e.g. slip and twinning system) and initial texture [23–27]. As the ECAP deformation is near simple shear in the intersection plane of the two channels, most studies have shown that textures developed in ECAP deformation are often compared with those after simple shear tests, such as torsion. [28–30].

Studies on aluminum alloys have indicated that ECAP can be an effective processing technique to control texture evolution because of many different processing parameters of the method. Many different texture orientations with all types of texture strength can be produced at different numbers of passes and various processing routes and temperatures. Gholinia et al. [15] studied the deformation texture of pure Al by ECAP and found that the main texture components are $\{001\}\langle 110\rangle$ and $\{112\}\langle 110\rangle$. Texture investigations by Cepeda et al. [18] demonstrates that the ECAPed Al 7075–O sample at 403 K shows a typical FCC shear texture, with multiple orientations through the A ($\{111\}\langle uvw\rangle$) and B ($\{hkl\}\langle 110\rangle$) fibers. Additionally, Suwas et al. [8] reported that ECAP processing of pure Al through different routes leads to different type of texture, in both qualitative as well as quantitative sense. Texture evolution of Al-6061 alloy by jinging et al. [22] shown that The ECA pressing temperature strongly affects the texture formation in 6061 aluminum alloy.

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It is very beneficial to investigate the influence of ECAP processing strain on the texture evolution in the ultrafine-grained microstructures produced in bulk Al alloys (7075) for its potential applications in aerospace industries. There are very limited reports in literature [18] on the effect of induced plastic strain on texture evolution in the ultrafine-grained Al-7075 alloy produced by ECAP. The production of ultrafine-grained Al-7075 alloys with excellent mechanical properties is heavily influenced by crystallographic textures and morphology of the grains and precipitates [12].

The objective of the current investigation is to study the texture and microstructure of Al-7075 alloys processed by equal channel angular pressing by route A and B_C. For this purpose the texture of aluminum specimens were analyzed by XRD in 2 directions (planes parallel and perpendicular to extrusion direction) and then the results were compared. Furthermore, transmission electron microscope (TEM) was applied to study the microstructure.

2. Experimental

The experiments were conducted using commercial Al 7075 alloy with the chemical composition of 5.7 Zn, 2.65 Mg, 1.5 Cu, 0.21 Cr, as the main ECAP material. Prior to ECAP the as-received extruded Al 7075 rods were annealed for 1 h at 415 °C and then cooled in the furnace. The Al-7075 alloy was annealed in order to increase the formability of the alloy and avoid crack formation during ECAP process [31]. Aluminum rods were machined into cylindrical specimens, 140 mm in length and 19.1 mm in diameter, and then pressed through an ECAP solid die having a channel angle of $\Phi = 90°$ and an outer curvature angle of $\Psi = 20°$. The image of applied die was presented in our previous paper [31]. All of the ECAP processing was performed using a hydraulic press of 400 ton capacity operating at a ram speed of ~1 mm s^{-1}. Billets were pressed at room temperature for either 1 or 4 passes, equivalent to imposed strains of about 1 and 4, respectively [32], using processing route A (no bar axis rotation) and route B_C (90° rotation around longitudinal axis after every pass).

Measurements of crystallographic texture were performed employing a RIGAKU, D/MAX-2500 X-ray diffractometer (Cu Kα radiation, $\lambda = 1.5406$ Å) on the TD and ED planes equivalent to the side plane at the point of exit from the ECAP die and plane perpendicular to extrusion direction, respectively (Fig. 1[25]). The measurements in both TD (z) and ED (x) planes were taken at approximately the midpoint on each longitudinal and cross sections by preparing a flat surface through mechanical polishing and grinding and with the textures recorded over an area having dimensions of 19 mm × 19 mm. X-ray pole figure measurement was carry out using Schulz back reflection method. The Labotex 2.1 software was used to process the raw data and calculate volume fraction of the texture components.

The microstructure of processed specimens was analyzed by transmission electron microscopy (TEM) using a JEOL JEM 3010 equipment operating at accelerating voltage of 300 kV. For TEM characterization, thin foils of the material were cut from the center of the cross-section of each billet (ED (x) plane which is perpendicular to the pressing direction). The foils were ground mechanically to a thickness of about 15 μm. Then 3 mm discs were punched from the specimens and subsequently polished to perforation using a twin-jet electropolishing facility with a solution of 30% nitric acid in methanol at −25 °C and 15 V. Selected area electron diffraction (SAED) patterns were obtained from regions having a reasonably homogeneous microstructure consisting of diameters of 2 μm.

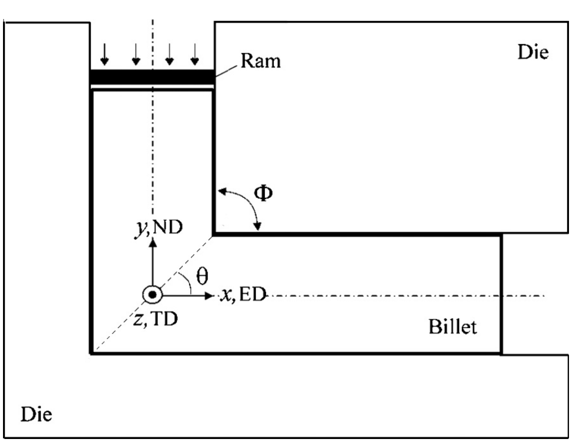

Fig. 1. ECAP die geometry and coordinate system employed in this paper.

3. Results and discussion

3.1. microstructure

Fig. 2 shows optical image of the starting materials etched with Weck's color reagent (4 g Permanganate potassium, 1 g NaOH, 100 ml distilled water) [33] and TEM micrograph of specimens subjected to four passes of ECAP process by route A and B_C. As can be seen in Fig. 2a as well as EBSD results reported in our previous work [34], the microstructure of starting material consists of grains with the grain size in the range of 10–80 μm, and sub-grains with a grain size of less than 5 μm. TEM images of ECAP processed specimens reveal that, the grains of initial material with average grain size of about 40 μm refine to grains with average grain size less than 500 nm after 4 passes of ECAP. It is also apparent that the grains after four passes by route B_C are essentially equiaxed, and the microstructure is homogeneous; whereas, the grains tend to be elongated when using route A.

The corresponding SAED patterns are shown at the bottom of each TEM micrograph. It is apparent that the SAED pattern of the specimen processed by route B_C consists of rings of diffraction spots showing the grain boundaries have high angles of misorientation, while, the presence of some discrete spots in the SAED pattern of the specimen processed by route A suggests the presence of a reasonable fraction of low angle or sub-grain boundaries. Based on TEM observations, it can be concluded that, route B_C represents the optimum procedure for attaining a homogeneous equiaxed grain with high angle boundaries [35–39]. The reason lies in different shear system in route A and B_C. Route B_C has two shearing directions lie on planes which intersect at 120°, while route A has two shearing planes intersecting at 90° [40–43].

3.2. Texture evolution

The (1 1 1) and (2 0 0) pole figures of the starting material are shown in Figs. 3 and 4 in the laboratory reference system projected onto the TD (z) and ED (x) planes, which are parallel and perpendicular to the specimen longitudinal axis, respectively. The pole figures clearly indicate the texture of the starting material is consist of strong ⟨1 0 0⟩ fiber together with a weak ⟨1 1 1⟩ fiber, with the fiber axis parallel to the billet longitudinal axis (ED (x) direction). The XRD results calculation by Labotex software demonstrates that the volume fraction of ⟨1 0 0⟩ fiber in starting material is about 68%, while the volume fraction of ⟨1 1 1⟩ fiber is about 20% (Table 2).

For ECAP textures, the Miller indice $\{hkl\}\langle uvw\rangle$ is used to denote an orientation that has an $\{hkl\}$ plane parallel to the ND-plane and a $\langle uvw\rangle$ direction parallel to ED-direction [9]. The Euler angles and Miller indices of the main ideal orientations of ECAP processed face-centered cubic (fcc) materials for $\Phi = 90°$ are listed in Table 1, based on the calculations by Li et al. [7,12,44].As shown in Ref. [7,9,12], these ECAP specific orientations can be derived from those for negative simple shear, by increasing the φ_1 of a simple shear ideal orientation by θ (=$\Phi/2$), while the two other Euler angles remain the same. So the ideal ECAP texture components in pole figures are the same as those known already for simple shear; they are just rotated 45° around TD axis [8,45]. To aid interpretations of experimental ECAP pole figures, the (1 1 1) and (2 0 0) key pole figures with the locations of the ideal ECAP orientations on the TD (z) and ED (x) planes are shown in Figs. 5 and 6, respectively [7,27].

The experimental (1 1 1) and (2 0 0) pole figures of ECAP processed materials for 1 and 4 passes by route A and B_C are shown in Figs. 7–12. The same iso-intensity contour map color bars were used for all pole figures in Figs. 7–12. The pole figures were