An electron back-scattered diffraction study on the microstructure evolution of severely deformed aluminum Al6061 alloy

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Abstract. In this paper dynamic strain ageing behavior in an Al–Mg–Si alloy related to equal channel angular pressing (ECAP) was investigated. In order to examine the combined plastic deformation and ageing effects on microstructure evolutions and strengthening characteristics, the Al6061 alloy were subjected to Φ=90° ECAP die for up to 4 passes via route Bc at high temperatures. For investigating the effects of ageing temperature and strain rate in ECAP, Vickers hardness tests were performed. The combination of the ECAP process with dynamic ageing at higher temperatures resulted in a significant increase in hardness. The microstructural evolution of the samples was studied using electron back-scattering diffraction (EBSD). The grains of Al6061 aluminum alloy were refined significantly at 100 and 150 °C with greater pass numbers and the distributions of grain size tended to be more uniform with pass number increasing. Frequency of sub-boundaries and low angle grain boundaries (LAGBs) increased at initial stage of deformation, and sub-boundaries and LAGBs evolved into high-angle grain boundaries (HAGBs) with further deformation, which resulted in the high frequency of HAGBs in the alloy after ECAP 4 passes.

Keywords: EBSD, Equal channel angular processing, Microstructure evolution, ageing, Aluminum alloy.

1. Introduction

Equal channel angular pressing (ECAP) is a promising process for the production of ultrafine-grained (UFG) structure in bulk materials. There has been a great interest over the last two decades [1–3] in the development of UFG structures through investigating various ECAP parameters (number of pressings, die channel angles, die profile, processing routes, back pressure, friction, pressing speeds and ECAP processing temperature) [4]. When the sample is pressed through several sequential passes, the shearing characteristics may be changed by rotating the sample between each pass. Thus, the route with which the sample was re-entered to the ECAP die in each pass has an influence on the microstructure achieved due to successive change of the shear plane [5]. Compared to conventional techniques, ECAP
offers a large variety of parameters for controlling the microstructure and otherwise the initial microstructure has played a limited role on microstructure orientations [6]. Iwahashi et al. have investigated the process of grain refinement and development of subgrain misorientation, using transmission electron microscopy and selected area electron diffraction, on 99.99 p.c. aluminum processed from 1 to 4 passes in ECAP using different processing routes [7]. Gholini et al. have used scanning electron microscope equipped with electron backscattered diffraction (EBSD) analysis system and provided different conclusions in regard to the superiority of a certain route in which the subgrains evolve most rapidly to high-angle grain boundaries [8]. Lapovok et al. investigated ECAP of AA6111 sheets and determined that ECAP is able to refine the grain size of the sheet and diminish the detrimental as-rolled texture components in the sheet [9]. In this paper, the evolution of microstructure is examined on the Al6061 alloy processed by ECAP in the elevated temperatures using electron backscattered diffraction (EBSD) studies.

2. Experimental procedure
A commercial Al6061 alloy, with a composition of 1.001 wt.% Mg, 0.693 wt.% Si, 0.088 wt.% Mn, 0.211 wt.% Cu, 0.478 wt.% Fe, and balance Al, was used in this study. All bar type samples were annealed for 3 h at 530 ºC, and then water quenched prior to ECAP. The samples were extruded at 100, 150, and 200 ºC through route Bc using ECAP die with tool angles of Φ =90º and Ψ =10º using a hydraulic press of 200 ton capacity, with pressing speeds of 10 mm/min. These die angles were chosen based on an earlier finite element study [10] which showed they induce optimal strain homogeneity in the processed materials. In order to verify the mechanical properties changes, individual values of the hardness were measured on two orthogonal diameters of the billet. All samples were examined by electron backscatter diffraction (EBSD). For this purpose the cross sections were stuck on the glassy blocks and then ground and polished using standard routines. In order to minimize the strain caused by the grinding and polishing processes the specimens were electropolished and then ion milled, respectively. EBSD measurements and analyses were performed using an EBSD detector attached to a FESEM at the magnification of 8000× and primary electron energy of 30 keV. An area of 6×18 µm was scanned with a step size of 20 nm. Grain boundaries were characterized by a misorientation larger than 15º between the neighboring measurement points.

3. Results and discussion
3.1. Microstructure
Fig. 1 shows the polarized light micrograph of the initial sample. The initial grain size measured by the linear intercept method was 47 µm.

3.2. EBSD
ECAP specimens were examined by EBSD in the transverse section of the ECAPed samples. The EBSD patterns of the processed samples after 2 and 4 passes at varying temperatures using route Bc are shown in Fig. 2. The strong dependence on the number of passes is obvious. The gradual refinement of the deformed structure and the generation of higher angle boundaries observed with increasing number of passes are consistent with what has been observed in many other studies [5-8]. At temperature of 100 ºC and two passes, the long grains in the direction of 45º are detectable. By increasing the number of passes to four, the long grains are refined to granular grains. A further increase in temperature (150 ºC) results in a higher fraction of refined grains. Moreover, these grains are more granular and homogenous. At 200 ºC some other mechanisms like dynamic ageing restricts the grain refining by impeding the dislocations movement [5].
The typical orientation imaging microscopy (OIM) patterns and boundary maps of the grains relating to the forgoing conditions are shown in Fig. 3.

Fig. 1. Polarized light micrograph of the as-received Al6061 alloy.

Fig. 2. EBSD patterns of the processed samples at varying temperatures using route Bc; (a) 100 °C-two pass, (b) 100 °C-four pass, (c) 150 °C-four pass, (d) 200 °C-four pass.
According to Figs. 3(a) and 3(b) the subgrain evolved more rapidly into arrays of high-angle boundaries when the sample was undergone two more passes. In newly evolved grains, further deformation may trigger subgrain formation by slip and lattice rotation, revealed as a distinct local orientation gradient [5].

The microstructures shown in Fig. 3 provide evidence for grain refinement and consist of both elongated and equiaxed grains. This inhomogeneity in deformation may be related to misorientation between grains. The grains with heavy dislocation density break down to finer grains while the ones that are relatively low in dislocation density remain less affected [11]. This could be the reason why sometimes the final ECAP processed microstructure shows an inhomogeneous distribution of grain size.

The temperature being as important as the number of pass may influences on the microstructure evolution. At all temperatures after four passes of ECAP with route Bc the microstructure mainly consists of high-angle grain boundaries, i.e. at 100, 150 and 200 °C the volume fraction of HAGBs are 83.8%, 77.9% and 68.7%, respectively. A rise in the temperature results in an increase in dynamic precipitation/ageing which affect the grain refining mechanisms. Additionally, the OIM images show that increasing temperature changes the texture of the material. Referring to Figs. 3(c) and 3(d), at 200 °C the dominant texture is between <111> and <101> while at 150 °C there is a random texture. Further deformation leads to increasing the misorientation and formation of new high-angle grain boundaries and grain fragmentation depend on grain orientation [5]. It has been suggested that combination of dynamic ageing and ECAP promotes grain refinement because the more heavily strained grains are constrained by precipitates nucleating between and inside the grains after each pass [12].
3.3. Mechanical properties
The average microhardness of the alloy after ECAP processing is plotted against the number of passes in Fig. 4. The initial sample was solution-annealed and its microhardness is reported as a reference. The average hardness was progressively increased in the pressed material at 100 ºC indicating that the age hardening effect was more dominant than the softening effect. A side-effect of the processing at elevated temperature is grain growth which coarsens the UFG microstructure and contributes to a reduction in hardness during the prolonged dynamic ageing.

The results confirm that at 150 and 200 ºC the effect of the dynamic recovery (DRV) and/or grain coarsening of heavily deformed substructure overcomes the effect of precipitation hardening by dynamic ageing. Grain refinement during ECAP may be attributed to the precipitations that inevitably nucleate during the process and has two possible models. According to the first model tiny precipitates which are thermodynamically unstable dissolve in the matrix result an enhancing strength due to solid solution strengthening. According to the second model, the stable precipitates enhance the strength by decreasing the mean free pass of mobile dislocations [12].

![Fig. 4. Average Vickers hardness versus the number of passes in Al6061 ECAP processed using route Bc.](image)

4. Conclusions
The microstructure evolution and misorientation were studied on an ECAP processed Al6061. The main results are summarized as follows:

Combining ECAP with ageing phenomena, in this research dynamic ageing which accelerates the precipitation and grain refining, can be an effective technique to control the misorientation as well as the production of UFG Al6061 alloy. The alloy sample ECAPed to two passes exhibited a marked increase in its low-angle grain boundaries and the sample ECAPed to four passes showed a noticeable decrease in its the grain size. This was found to be related to the initial texture acquired by the ECAP process. Development of a large number of low-angle boundaries in the primary passes precedes the formation of nano/submicron grains.
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