Grain refinement and Lattice Imperfections in Commercial Aluminum Alloy Processed by Severe Plastic Deformation

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Abstract. In the present work, investigations on the microstructure of an aluminum alloy that had been subjected to severe plastic deformation (SPD) by equal channel angular pressing (ECAP), filing and ball milling, were carried out using X-ray diffraction and scanning electron microscopy. SPD leads to lattice distortions, increased dislocation density and an intensive refinement of the microstructure. The refinement and lattice imperfections of the material are greatly affected by the deformation modes and loading performance occurring during SPD. During the milling, the dislocation annihilation increases at higher strains thereby resulting in a smaller crystallite size. After ECAP, the material manifests a strong shear texture and anisotropy of the deformation behavior. Strain anisotropy is less pronounced in filed and ball milled powder particles.

1. Introduction

In recent years, various techniques of severe plastic deformation (SPD) are being developed to achieve high strains at a relatively low temperature. High strains leads to enhanced grain refinement to the submicrometer (100-1000 nm) or nanometer (<100 nm) levels. Ultrafine grained materials (UFG) obtained by SPD have attracted considerable scientific interest in the last decade because of their unusual physical, chemical and mechanical properties. For example, the intensive grain refinement is responsible for the enhancement of physical and mechanical properties of the processed materials, such as highly enhanced flow stress [1] and Vickers microhardness [2,3].

SPD techniques include, among others, torsion under high pressure [4], accumulative roll bonding (ARB) [5], high energy milling [6] and equal channel angular pressing (ECAP) [7,8]. The latter procedure, introduced almost 30 years ago to achieve enhanced plastic strains, is especially attractive for many near superplastic applications. Since the cross section of the sample remains unchanged during ECAP, repetitive pressings on the same sample are possible to achieve the required strains and to invoke different slip systems by rotating differently the sample between consecutive passes.

In high energy milling, powder particles are subjected to SPD through repetitive compressive loads arising from the impacts between the balls and the powder particles, which lead to their fracturing and welding. As a result, new crystalline and amorphous materials could be produced with crystallite sizes at the nanometer scale [9]. Furthermore, the mechanical milling kinetics depends on the energy transferred to the powder from the balls during milling [10]. The energy transfer is governed by many
parameters such as milling speed, size and size distribution of the balls, dry or wet milling and temperature and duration of milling [11]. High energy milling has advantages of being simple, relatively inexpensive to produce [12], applicable to any class of materials and easily scaled up to large quantities [13].

Several techniques of analysis are used to examine the microstructure of ultrafine grained (UFG) materials obtained by SPD. Transmission electron microscopy (TEM), scanning electron microscopy (SEM) and X-ray diffraction (XRD), considered as complementary tools, are the most powerful methods used. TEM and SEM provide a direct image of the grain sizes and distribution and XRD gives quantitative estimations on the lattice imperfections, i.e. lattice distortions and dislocation density, lattice parameter, texture, etc.

The present work aims, essentially, to examine the microstructure of aluminum alloy processed by various SPD techniques, that are, ECAP, filing and ball milling using XRD and SEM. Also, mechanical tests were performed using tension and Vickers microhardness measurements. From XRD line profile analysis, the coherent domain size, lattice distortions, dislocation density and lattice parameter were calculated. The estimated values obtained from the different severe plastic deformation techniques used were compared.

2. Material and experimental procedures

2.1. Material and severe plastic deformation processes

The experiments were carried out on an aluminum (99.1%) material received in the form of cast ingots. The chemical composition, determined by inductively coupled plasma optical emission spectrometry (ICPOES), is given in Table 1. The material contains a low volume fraction of $\alpha$-AlFeSi phase [2]. The material was annealed for 24 hours at 500°C and quenched in iced water. After annealing, the alloy was formed by equiaxed grains of about 85 $\mu$m in size.

Samples with dimensions $\sim$10×10×70 mm$^3$ were cut for ECA pressing. The die used for ECA pressing has two square channels of 10×10 mm$^2$ cross sectional area intersecting at an angle $\phi = 90^\circ$. The arc of curvature at the outer point of the intersection of the two channels delineated an angle $\Psi = 16^\circ$ (figure 1). According to the equation given by Iwahashi et al. [14], the total equivalent strain, $\varepsilon_N$, accumulated by N passes through the die was equal to 1.07N. In the present work, repetitive pressings of the same sample were conducted to achieve an equivalent strain up to 4.28 (N=4) without rotation of the sample between two successive passes (the so called route A [15]). ECAP was performed at room temperature.

| Table 1. Chemical composition (weight %) of the investigated aluminium alloy. |
|---------------------|---------------------|---------------------|---------------------|
| Ca                  | 0.0014              | Ni                  | 0.0036              |
| Co                  | 0.0002              | Pb                  | <0.0001             |
| Cr                  | 0.0056              | Si                  | 0.1071              |
| Cu                  | 0.0558              | Ti                  | 0.0060              |
| Fe                  | 0.2500              | Zn                  | 0.1660              |
| Mg                  | 0.2190              | Al                  | balance             |
| Mn                  | 0.0600              |                     |                     |
The same material was also filed and ball milled up to 20 h. The hand filing was carried out using a steel file. The collected powders were then milled in P7 planetary mill up to 20 h. Ball to powder ratio was about 15:1 and the mill speed maintained at 300 rpm. Milling proceeded with a stationary speed of rotation. The duration of milling was automatically fixed by an electronic regulator and fixed on 15 min milling 15 min breather. Ethanol (2 weight %) was added to retard excessive welding. The milling atmosphere was argon that was purged into cups before milling to prevent oxidation.

For XRD and SEM investigations, specimens of ∼5×5×10 mm3 were cut from the central part of each ECA pressed sample. Their surfaces were mechanically ground and electrolytically polished for 30 min at a temperature below 10°C under an operating voltage of 30V, using a mixture of 66% (CH3CO)2O and 34% HClO4. The scanned area was 80 × 80 μm² with a step size of 0.1 μm for each processed sample. XRD analysis of the samples deformed by ECAP and the hand filed and ball milled powders was carried out at room temperature with a diffractometer in step scanning mode using Cu-Kα radiation. The XRD patterns were recorded with a scan rate of 2×10⁻³ deg./s in the scanning range 2θ = 20-120°.

2.2. Mechanical tests

Mechanical tests were performed using tension and Vickers microhardness measurements. The Vickers microhardness,HV, was measured using a DURIMET microhardness tester with a diamond pyramidal indenter. At least eight measurements were taken at randomly selected points with a load of 100 g applied for 15 s. For compression tests, cylindrical specimens of ∼6 mm in diameter and ∼9 mm in length were machined from ECA pressed samples, so that the compression direction was parallel to the X-axis (Figure1). A maximum compression stress of 15 kN was applied to the specimens. Compression tests were performed at room temperature, using an Instron testing machine, at a cross-head displacement speed of 0.05 mm/min corresponding to initial strain rate of 5.5 × 10⁻⁵ s⁻¹.

2.3. X-ray diffraction analysis

The broadening of the experimental reflection is caused by the small size of crystallites (or coherent length), internal lattice distortions (or microstrains) and the instrumental broadening [16]. In principle, the experimental broadening of reflections is the convolution of the instrumental broadening and the intrinsic broadening. The instrumental effects vary with each diffractometer and are caused by imperfect measurement capabilities, such as unfocused beams and unresolved α1 and α2 reflections. It is necessary to quantify the instrumental broadening of the diffractometer so that the effects of crystallite size and lattice strain can be individually identified in the diffraction patterns of experimental samples. A correction for the contribution from the instrumental effects to the experimental full-width at half maximum (FWHM), βexp, for each peak was obtained using the following expression:

![Figure 1. Schematic illustration of the die used for ECAP showing the three directions X, Y and Z.](image-url)
where $\beta_{\text{ins}}$ is the instrumental FWHM, which was determined using the undeformed aluminium as a standard reference material. However, subtracting the instrumental broadening effect from the diffraction pattern of a material sample, the remaining widths of the diffraction peaks are attributed to the small crystallite sizes and lattice strains. A decrease in crystallite size or enhanced microstrains leads to much broader reflections [16,17]. The Williamson–Hall (WH) and Halder–Wagner (HW) methods [16], among others, are used extensively for determining the crystallite size and the lattice microstrains of SPD materials. The WH equation used in the present work, in which the FWHM, $\beta$, due to lattice imperfections is related to the crystallite size, $D$, and the lattice distortions, $\varepsilon$, is expressed as follows:

$$
\beta^* = d^* \varepsilon + \frac{1}{D}
$$

where $\beta^* = \beta \cos \theta / \lambda$ and $d^* = 2 \sin \theta / \lambda$; $\theta$ the Bragg angle and $\lambda$ is the wavelength used. From Eq. (2), the intercept and the slope of the plot of $\beta^*$ against $d^*$ give the crystallite size ($1/D$) and the microstrains $\varepsilon$, respectively. The WH plot shows whether the breadth depends on $d^*$ and the nature of any hkl dependence. In the HW method, which assumes a Voigt peak shape, the crystallite size, $D$, and the strain $\varepsilon$, are related to $\beta^*$ by the following equation:

$$
(\frac{\beta^*}{d^*})^2 = \frac{1}{D} \left(\frac{\beta^*}{(d^*)^2}\right) + (\frac{\varepsilon}{2})^2
$$

From Eq. (3), the intercept of the plot of $(\beta^*/d^*)^2$ versus $(\beta^*/(d^*)^2$ provides the value of the microstrains and the slope gives the crystallite size. In the present work, the FWHM and the position of each peak were obtained using the X’Pert HighScore Plus software. In order to estimate the lattice distortions and the crystallite size in the SPD samples, the Williamson–Hall (WH) and Halder–Wagner (HW) methods were used.

The lattice parameter of the material before and after SPD was obtained from a linear regression analysis of the measured lattice parameter, $a_{\text{app}}$, obtained from each peak, plotted against the Nilson–Reley (NR) function given by the following equation [18]:

$$
\text{NR} = \cos^2 \frac{\theta}{2} \times \left(\frac{1}{\sin \theta} + \frac{1}{\theta}\right)
$$

and extrapolated to NR = 0, that is $2\theta = 180^\circ$.

For severe plastic deformed samples, dislocations are the main defects of which density, $\rho$, can be expressed in terms of crystallite size $D$ and the mean lattice microstrains $\langle \varepsilon^2 \rangle^{1/2}$ by [19-21]:

$$
\rho = \frac{2\sqrt{3} \langle \varepsilon^2 \rangle^{1/2}}{Db}
$$

where $b$ is the magnitude of the Burgers vector of dislocation equal to $a/\sqrt{2}$ for a fcc aluminum alloy; $a$ is the lattice parameter of the material.

3. Experimental results and discussion

Microstructure examination by SEM

The micrograph in figure 2 shows typical SEM image of the microstructure of the undeformed aluminum alloy and in the Y plane of the samples processed by ECAP after two and four passes using route A. The microstructure of the undeformed alloy consists of equiaxed coarse grains with an average size of ~85 $\mu$m. The aluminum matrix contains precipitates located essentially at grain boundaries, even though some precipitates were also observed in the grain interior. The microstructure
of the ECAP’ed (N=2) sample consists mainly of elongated grains and few equiaxed grains are also observed. After four passes, besides the elongated grains, the formation of equiaxed grains of about 0.2 µm is depicted in the ECAP’ed samples. Therefore, ECAP leads to intensive grain refinement of the aluminum alloy. Similar trend has been reported in microstructure investigation by TEM on high purity aluminum processed by ECAP [22,23]. In previous works [24,25], it was shown that the presence of the α-AlFeSi precipitates essentially at grain boundaries allowed to reveal the initial shape of the grains and to follow their evolution during ECA pressing, i.e. the grains appeared to be elongated in the side face of the sample (Y plane) [24]. In addition, the fraction of submicrometer grains (<1 µm) increased with the number of ECAP passes. However, the grain boundaries misorientation depended weakly on the number of passes and most subgrains were separated by low angle grain boundaries (< 10°) [25].

Figure 2. SEM micrographs of the (a-b) undeformed aluminum alloy and in the Y plane of the ECAP’ed samples for (c) N=2 and (d) N = 4. EqG: equiaxed grains, ElG: elongated grains.

Figure 3 shows SEM micrographs of the filed and 20 h milled powders. After filing, the powder particles are coarse and non-uniform in shape and size (figure 3a). After 20 h of milling, the particles became more regular and one can easily observe a significant change in the particle size and morphology (figure 3b). The particle size distribution became at least bimodal since very fine particles and coarser ones are examined. Some of the very fine particles welded to each other or to the surface of coarser ones.
Microstructure investigation by X-ray diffraction

The XRD patterns of SPD samples compared to that of the undeformed sample are given in figure 4. The intensities of the undeformed and ECAP’ed samples reflections are quite different from tabulated ones [26]. For example, for both samples, the 311 reflection is more intense than the 200 reflection and the 222 and 400 reflections are not observed in the undeformed material because of their very low intensities. Moreover, a significant decrease of the 200 reflection intensity is seen after ECAP. The change of the reflections intensities manifests a strong shear texture in the material. The SPD through filing and ball milling removes this texture since all the fundamental aluminum reflections in the range $2 \theta = 20$-120° are detected and their intensities are comparable to tabulated data [26].

Figure 4. XRD patterns of SPD samples compared to that of the undeformed sample.

Figure 5 gives the enlarged view of the 111 and 200 reflections of SPD samples compared to that of the undeformed sample. One can easily observe a significant enlargement of these reflections accompanied with a slight shift. The $K_{\alpha 2}$ reflection is well resolved for the undeformed sample but not after severe plastic deformation because of the reflection broadening. The 111 reflection of the ECAP’ed sample shifts to higher 20 angle (Figure 5a), however the 200 reflection of the same sample shifts to lower 20 angle (Figure 5b). This indicates a strong anisotropy of the deformation behavior of the ECAP’ed sample, i.e, the sample is subjected to compression stress along the [111] direction which, considering a constant volume of the pressed sample, provokes tensile behavior along different crystallographic directions, in particular along the [200] direction. On the other hand, all
reflections of filed and ball milled samples shift towards higher 2θ angle, as depicted for the 111 (Figure 5c) and 200 (Figure 5d) reflections, indicating that these two deformation processes lead to isotropic compression of the aluminum powders. The reflection shift implies the variation of the lattice parameter. The lattice parameters of the undeformed and SPD samples calculated by the Nilson-Reley function presented in equation 4 are given in Table 2. The lattice parameter of the undeformed aluminum is equal to 4.05097 Å. The SPD leads to a small decrease of the material lattice parameter. The lowest lattice parameter value (4.04418 Å), corresponding to a relative variation of about 0.16 %, was obtained after filing. The reflection shift could be attributed to planar faults [27], internal stresses, residual compressive or tensile stresses [28] and dissolution of a second phase containing elements with negative effect on the aluminum matrix (i.e. provoking the decrease of the lattice parameter) such as Fe and Si [28]. The enlargement of XRD reflections depicted in Fig. 5 is due mainly to small crystallite sizes and lattice microstrains.

![Figure 5](image)

**Figure 5.** Enlarged view of the (a,c) 111 and (b,d) 200 reflections of SPD samples compared to those of the undeformed sample.

Figures 6a and b give the WH plot of the ECAP’ed sample and filed and 20 h milled samples, respectively. The WH plot shows the degree of isotropy of lattice strains. It is easy to note that the WH plot of the ECAP’ed sample manifests slightly higher scatter when compared to the filed and milled samples. The enhancement of scatter is also revealed by the increase of the adjust $R^2$ values of fitted linear plots from 0.7525 for ECAP’ed sample to 0.9206 and 0.9248, respectively, for filed and 20 h milled samples. Therefore, the strain is more isotropic after filing and milling than when using ECAP process. The HW method, using pseudo-Voigt approximation, was used in the present work to determine lattice distortions and crystallite sizes of the SPD alloy. The corresponding plots are shown in Figures 6c and d. The values obtained, depicted in Table 2, show that SPD leads to lattice distortions (between 0.2 and 0.4%) and an intensive refinement of the microstructure (<200 nm). Also, both filing and milling lead to nanometer crystallite sizes (<100 nm). Comparable results were obtained for Al-7075 milled for 20 h [21]. The crystallite size determined by HW method is lower than that
deduced from direct observations by SEM analysis. The latter technique provides the size of grains that have well-defined, high-angle boundaries. Theses grains sometimes contain a substructure characterized by low-angle boundaries (crystallite). XRD allowed determining mean size values of these coherent domains or crystallites.

Figure 6. WH (a,b) and HW (c,d) plots of the ECAP’ed sample and filed and 20 h milled samples.

Table 2. Crystallite size and microstrains determined by the Halder-Wagner method and dislocation density of the SPD aluminium alloy.

|              | Crystallite size (nm) | Microstrains (%) | Lattice parameter (Å) | Dislocation density $(10^{15} \text{ m}^{-2})$ |
|--------------|-----------------------|------------------|-----------------------|---------------------------------------------|
| ECAP’ed (N=4)| 192                   | 0.28             | 4.05057               | 0.17                                        |
| Filed        | 80                    | 0.28             | 4.04418               | 0.42                                        |
| 20h milled   | 68                    | 0.34             | 4.04589               | 0.61                                        |

Figure 7. Evolutions of the Vickers microhardness ($H_V$), yield strength at 0.2 % of reduction ($\sigma_{0.2}$) and dislocation density during strain accumulation by ECAP.
3.1. Mechanical properties

The hardness could not be directly determined from filed and milled powders. It was shown that mounting powder in epoxy and forming discs of powder in a compaction die proved unsuccessful because the localized pressure of the hardness indenter displaced surrounding material, leading to inaccurate measurements [30]. Therefore, hardness and compression tests were only performed on the ECAP’ed samples. The evolutions of the Vickers microhardness (HV) and yield strength at 0.2 % of reduction (σ0.2) and dislocation density during strain accumulation by ECAP are given in Figure 7. It is clearly seen that HV and σ0.2 increased significantly with the accumulated strain. For example, HV increased from 33 for the undeformed sample to ~77 after four passes through the die and σ0.2 became approximately five times higher after four passes (σ0.2 =211 MPa) compared to that of the undeformed material (σ0.2 =43 MPa). This increase is also associated with an increase in the dislocation density produced by ECAP. It should be noted that during SPD of polycrystalline materials, geometrically necessary dislocations (GNDs) and statistically stored dislocations (SSDs) are produced [29,31]. GNDs are related to strain gradient whereas SSDs are generated by the interaction of dislocations with lattice imperfections such as grain boundaries, twin boundaries, defects and tangled dislocations. The improvement of the mechanical properties is due to grain refinement and lattice dislocations accumulation during ECAP. We showed in a previous work [25] that, according to the Hall-Petch relationship, which predicts that the strength or hardness should increase when the grain size decreases, the dependence of the microhardness on the grain size, D, is consistent with a linear function: HV = 413 + 118D^{1/2} and σ0.2 = 35 + 76D^{1/2}. Transmission electron microscopy observations carried out on the same aluminum alloy indicated a high dislocation density generated after ECAP and revealed the formation of dislocation loops and dislocation walls [17]. The calculation of the dislocation density of the ECAP’ed alloy using equation 5 showed that a high dislocation density was obtained after two passes (ρ = ~ 0.17 × 10^{15} m^{-2}). Thereafter, only a slight increase was observed. The low variation of the dislocation density over N =2 could be explained by too many microstructure features, such a dynamic recovery which balances the creation of dislocations during ECA pressing [28], cell and grain boundary evolution, low- and high-angle misorientation evolution with strain [25] and tangled dislocation pinning by the existing fine particles.

4. Conclusion

In the present work, the experiments were carried out on an aluminum alloy that had been subject to SPD through ECAP, filing and ball milling. The microstructure of the alloy was examined by XRD and SEM. After annealing of the as-cast aluminum at 500 °C, the microstructure consisted of equiaxed coarse grains with an average size of ~85 μm. Independently of the deformation method used in the present work, lattice distortions, increased dislocation density and an intensive refinement of the microstructure were obtained after SPD. SEM analysis showed that the microstructure of the ECAP’ed sample consists of elongated grains and equiaxed grains of about 0.2 μm. The ECAP’ed material manifests a strong shear texture and anisotropy of the deformation behavior. Strain anisotropy was less pronounced in filed and ball milled powder particles. The highest dislocation density (0.61 × 10^{15} m^{-2}) and the lowest crystallite size (68 nm) calculated from X-ray reflections profiles were obtained after 20 h of ball milling. This was due to increased dislocation annihilation at higher strains. The SPD led to a small decrease of the material lattice parameter. The lowest value was obtained after filing. The decrease of the lattice parameter was attributed to lattice imperfections provoked by SPD.

Acknowledgment

The authors would like to acknowledge Professor N. Njah from the Laboratory of Applied Metallurgy -FSS (Tunisia) for useful helps.
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