Intermetallic Particle Evolution during ECAP Processing of a 6082 Alloy

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Intermetallics evolution in a commercial 6082 aluminium alloy severely deformed by Equal Channel Angular Pressing was investigated. Chemical electron probe microanalyses allowed to state that in the severely deformed alloy, Si-rich phases were progressively dissolved whereas the amount and composition of the Fe-Mn-Si containing intermetallics remained substantially unchanged. The moderate hardening effect measured on isothermal aging at 130°C of the ECAP processed samples was accounted for by the reprecipitation of the phases dissolved during severe plastic deformation. Tensile tests and fractographic analyses on the broken specimens showed that the 6082 alloy featured a relatively high ductility and a substantially unaltered fracture mode even after several ECAP passes.

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1. Introduction

In recent years, considerable research efforts have been devoted to the development of techniques to apply severe plastic deformation (SPD) to ductile metals using methods such as equal channel angular pressing (ECAP), accumulative roll bonding (ARB), high pressure torsion straining (HPTS). The principles of ultrafine microstructure generation during SPD have been well documented in a number of original works and review papers, especially for pure Al and Al alloys.

Recent literature works also highlighted the role played by second phase particles on the deformation behaviour and on the development of ultrafine structures and related properties. In a work on an Al 6061 alloy, Moon and co-workers recorded a marked difference in plastic flow behaviour during ECAP in underaged and overaged alloys. Deformation banding was favoured in the material with the highest workhardening rate such as the underaged alloy while the coarse precipitates formed in the overaged alloy promoted a more homogeneous deformation pattern in the billets. Similar conclusion were also drawn by Barlow et al. in a study on the effects of small alumina particles in a severely deformed pure Al matrix. It was stated that particles in the size range from 50 to 100 nm were able to promote homogeneous slip by dislocation nucleation as well as to hinder dislocation movement within the structure.

A notable feature of SPD processes is the ability to decompose fine second phase particles and to enhance homogeneity of the Al matrix. A commercial Al alloy supplied in the form of extruded bars of diameter 10 mm was investigated. The alloy chemical composition is given in Table 1. Samples having a length of 100 mm were cut from the bars and fully annealed in a muffle furnace at 530°C for 2 hours followed by furnace cooling.

ECAP pressing was carried out using a die with channels intersecting at an angle $\Phi = 90^\circ$ and with an external curvature angle $\Psi = 20^\circ$, corresponding to a theoretical strain of 1.05 for each pass. Samples were processed at room temperature by the so-called route C (rotation by 180° of the billets at each pass) to accumulate up to six passes. The experimental results of the ECAP facility and of material processing are described elsewhere.

Table 1 Chemical composition (mass%) of the 6082 alloy investigated.

<table>
<thead>
<tr>
<th>Element</th>
<th>Mg</th>
<th>Si</th>
<th>Mn</th>
<th>Fe</th>
<th>Cu</th>
<th>Cr</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass %</td>
<td>1.193</td>
<td>1.019</td>
<td>0.650</td>
<td>0.267</td>
<td>0.005</td>
<td>0.010</td>
<td>0.015</td>
<td>balance</td>
</tr>
</tbody>
</table>
Specimens cut from the processed billets were prepared by standard metallographic techniques and etched in a solution of 0.5% HF in water to identify intermetallic particles constituting the alloy. The starting grain size was evaluated by electrolytic etching with a solution of 2% HBF$_4$ in water (Barker’s reagent) at 25 V. Optical and Scanning Electron Microscope (SEM) analyses were carried out to identify morphology and composition of the intermetallics. In particular, systematic electron probe microanalyses (EPMA) were performed to collect the chemical composition of the second phase particles found in the alloy microstructure. The approach suggested by Quian et al. was adopted in order to numerically treat the unwanted modification of X-ray signals caused by electron excitation of the surrounding matrix when measuring small intermetallic particles.

The mechanical behaviour of the ECAP processed samples was evaluated by Vickers microhardness tests with a indenter load of 500 g, and by tensile tests at room temperature, at an initial engineering strain rate of $6.7 \cdot 10^{-4}$ s$^{-1}$. The tensile specimens were machined from the processed billets with a gauge length of 30 mm and a diameter of 6 mm. Fractographic analyses of the broken tensile samples allowed to elucidate the fracture mechanisms of the materials as a function of the amount of SPD experienced. Representative fracture surfaces were observed in the central regions of the specimens, so as to avoid misinterpretation of the fracture mechanisms owing to larger shear stress effects acting close to specimens surfaces.

3. Results and Discussion

3.1 Microstructure

In Fig. 1(a) the representative grain structure of the starting alloy is depicted. The average grain size was relatively uniform throughout the billet diameter and corresponded to grade 6 according to the ASTM E112 standard. Fig. 1(b) and Fig. 1(c) show the evolution of the grain structure obtained after one and two ECAP passes. It can be readily seen that after a single pass through the ECAP die, individual grains became slightly elongated and shear bands appeared within the largest grains. From simple geometrical consideration, it is known that when using the route C, the shear strain is reversed after two passes and therefore the total strain becomes redundant. Accordingly, inspection of Fig. 1(c) shows that the grains appear less elongated and similar in shape to those found in the starting sample, although there is some evidence of the formation of a substructure within the grains and the grain boundaries are not well distinct as in the starting microstructure. After three or more passes, the structure developed by the severe plastic deformation became more complex and less resolved to the optical microscope. The achievement of the expected ultrafine structure, as observed by TEM analyses, was already described elsewhere. For the present case, it can be recalled that parallel bands of subgrains separated by small-angle boundaries formed after the first ECAP pass. This substructure rapidly evolved into submicrometer-size high-angle grains and led to the development of a fully equiaxed microstructure after about four passes, in full agreement with published works.

Typical intermetallics distribution of the 6082 alloy in the annealed condition and after ECAP pressing to 6 passes is depicted in Fig. 2. The quantitative EPMA results allowed to classify two main kinds of secondary phases in the alloy structure: a first series of particles containing Si and/or Si + Mg (dark phases), a second series of particles containing Al, Si, Fe and Mn (light phases). In the starting condition, this latter phase was roughly aligned along the extrusion direction of the bars (horizontal direction in the micrographs). After ECAP, the light precipitates lost their preferential alignment and appeared somewhat more fragmented and therefore of smaller average size.
3.2 Composition of intermetallics

In Fig. 3, a summary of the analysed second phase particles is given as a function of their composition expressed in terms of Si and Fe + Mn content. Fe and Mn were considered together because in Mn containing Al-Mg-Si alloys, many of the Al$_x$Fe$_y$Si$_z$ intermetallics are replaced by Al$_x$(FeMn)$_2$Si$_z$ phases. In the graph, straight lines joining the matrix composition to the theoretical composition of several known intermetallic phases are also drawn. Depending on the relative influence of the surrounding matrix and of the analysed particle, the data point of each particle can be located at different positions on the straight lines: close to the lower edge of the line if the matrix effects prevail, at the upper edge of the line if the particle is large enough to fully contain the electron excitation volume, thus allowing to neglect the disturbing effects of the matrix. The measurements clearly confirmed that the Fe-rich intermetallics were mainly of the Al$_{15}$(FeMn)$_3$Si$_2$ type, as expected for this type of alloy. No significant changes were measured when considering the possible modifications induced by SPD on the composition of these intermetallics. The Si-rich phases were better analysed by plotting their composition as a function of Mg and Si content, as shown in Fig. 4. Here again, a straight line representing the theoretical slope of the Mg$_2$Si phase was drawn as a reference and the influence of ECAP passes was highlighted by using different symbols. Despite the expected data scatter, it is apparent that the Si content of these intermetallics systematically exceeds that of the stoichiometric Mg$_2$Si composition. It is also suggested that the almost pure Si particles (data points lying near the X-axis) found in the annealed alloy and in smaller amounts in the samples subjected to 2 ECAP passes, disappeared in the heavily deformed alloy, namely after 6 ECAP passes.

Finally, considering the observed dissolution of Si-rich intermetallics during severe plastic deformation, the possible aging response of the samples ECAP processed to six passes was evaluated by microhardness testing after isothermal aging at 130°C. The obtained microhardness profile is reported in Fig. 5. It is highlighted that a limited gain in strength can be achieved, although the hardness improvement induced by aging is of the same order of magnitude of the experimental data scatter. Accurate TEM analyses aimed at demonstrating the nature of the precipitates are currently in progress and will be the subject of a future paper.

3.3 Mechanical properties

The strength and ductility of the billets as a function of the number of ECAP passes experienced is depicted in Fig. 6. The strengthening achieved by the ECAP processed samples followed the expected trend found also for other severely deformed Al alloys. A steep increase is noticed on the first
ECAP pass while a substantial saturation of properties followed on further straining. Concurrently, fracture elongation underwent a drop to about one half of the original value on the first pass but remained constant on further passes, keeping a satisfactory level of about 8%.

SEM fractographs taken on broken tensile samples showed that in all of the investigated alloy conditions, fracture occurred by growth of microdimples that nucleated at intermetallic particles, as depicted in Fig. 7. The average size and depth of the main dimples revealed to be comparable for all the samples, thus suggesting that the investigated alloy maintains a notable intrinsic plasticity even after the large amount of deformation imparted by ECAP. From Fig. 7(b) it can also be observed that secondary dimples of smaller size are traceable on specific flat regions of the fracture surface of the samples processed to a large number of ECAP passes. These features were indeed noticed only occasionally. To the authors knowledge, similar evidences toward an incipient transition in fracture mechanism in ECAP processed alloys were never commented by other researchers. It can be supposed that the appearance of these first fine-dimpled surfaces is the result of the increasing sensitivity toward plastic flow localisation onto specific planes of the severely deformed alloys, as opposed to second-phase induced void growth, typically found in the standard 6082 alloy.

4. Conclusions

The investigation carried out on annealed billets severely deformed by the ECAP process, allowed to draw the following conclusions.

(1) A bimodal distribution of intermetallic particles originally aligned along the longitudinal billet direction was found in the alloy structure. Almost pure Si crystals and Mg₂Si precipitates with excess of Si as well as Al₁₅(FeMn)₃Si₂ type intermetallics were detected.

(2) Accurate EPMA analyses performed on the ECAP processed samples allowed to state that in the severely deformed alloy, Si-rich phases were progressively dissolved whereas the amount and composition of the other secondary phases remained substantially unchanged.

(3) Microhardness measurements after isothermal aging of the annealed and ECAP processed samples suggested that a moderate aging effect could be achieved, presumably owing to reprecipitation of phases dissolved during ECAP processing.

(4) Tensile tests carried out on samples processed to different ECAP passes showed that the alloy strength underwent a steep increase on the first ECAP pass and a near-saturation of properties on further straining.
Fracture elongation dropped to about 50% of the starting value but kept a satisfactory level with further ECAP passes.

(5) Fracture in tension of the ECAP processed samples occurred by a fully ductile mode, by the nucleation of voids at intermetallics. In samples subjected to a large number of ECAP passes, smaller secondary dimples appeared on specific flat regions of the fracture surface. It was suggested that these fine-dimpled surfaces are the result of the increasing sensitivity toward plastic flow localisation onto specific planes of the severely deformed alloys.

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REFERENCES