Using ball-indentation to evaluate the properties of an ultrafine-grained Al–2% Si alloy processed by ECAP

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Abstract

As-cast Al–Si alloys exhibit excellent mechanical properties but these properties may be further improved through processing by equal-channel angular pressing (ECAP). In this investigation, specimens of a hypo-eutectic Al–2% Si alloy were successfully pressed up to a maximum of four passes at room temperature and the microstructures and mechanical properties were evaluated. The results show that pressing through four passes reduces the grain size from ~40 to ~0.7 μm, reduces the size of the Si particles from ~12.0 to ~1.08 μm and increases the yield stress and the ultimate tensile strength by a factor of ~2 by comparison with the as-cast alloy. The mechanical properties were examined using a ball-indentation technique (BIT). It is suggested that, since BIT provides the capability of obtaining extensive mechanical data using only a very small sample, it is an ideal technique for evaluating the properties of samples processed by ECAP.

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1. Introduction

Considerable attention has focused recently on the fabrication of ultrafine-grained materials through the application of severe plastic deformation (SPD) [1]. Generally, metals processed by SPD have grain sizes in the nanometer or submicrometer range and they exhibit high strength at ambient temperatures [2]. Although several different SPD processing procedures are now available, the most promising technique appears to be equal-channel angular pressing (ECAP). In this investigation, specimens of a hypo-eutectic Al–2% Si alloy were successfully pressed up to a maximum of four passes at room temperature and the microstructures and mechanical properties were evaluated. The results show that pressing through four passes reduces the grain size from ~40 to ~0.7 μm, reduces the size of the Si particles from ~12.0 to ~1.08 μm and increases the yield stress and the ultimate tensile strength by a factor of ~2 by comparison with the as-cast alloy. The mechanical properties were examined using a ball-indentation technique (BIT). It is suggested that, since BIT provides the capability of obtaining extensive mechanical data using only a very small sample, it is an ideal technique for evaluating the properties of samples processed by ECAP.

This paper describes experiments conducted to evaluate the properties of a hypo-eutectic Al–2% Si alloy after processing by ECAP, where the composition of this alloy and all subsequent alloys are given in wt.%. It is well-known that Al–Si alloys can be produced by casting and they have a wide range of applications, especially in the automotive and aerospace industries, due to their high strengths at elevated temperatures, their good wear resistance and their low coefficients of thermal expansion [7–9]. However, the cast alloys generally exhibit a low fracture toughness. Several experiments have been conducted to determine the microstructures and properties of eutectic and hyper-eutectic Al–Si alloys after processing by ECAP. Thus, there have been investigations of the Al–7% Si–0.35% Mg alloy [10–13] and the Al–11% Si alloy [14–16] and, more recently, tests were conducted using the hyper-eutectic Al–23% Si alloy [17]. Without exception, all of these investigations were performed using a
special rotary-die ECAP in which the sample is inserted into the die, pressed through an angle of 90° and the die is then rotated for the second pressing. This procedure has an advantage because it is simple to perform repetitive pressings and thus the samples may be pressed through a large number of passes (for example, through up to 32 passes [14,16,17]). Nevertheless, the procedure has some disadvantages because the pressing operation corresponds to route A which is not the optimum procedure for achieving a homogeneous microstructure and the samples often have small aspect ratios so that there are significant regions of non-uniformity at either end of the billet.

The present investigation was undertaken with four specific objectives. First, to extend the application of ECAP to evaluate the potential for using this procedure with a hypo-eutectic Al–Si alloy. Second, to make use of a conventional ECAP facility instead of a rotary-die ECAP and to thereby have the capability of using the optimum procedure of route BC. Third, to examine whether it is feasible to successfully press the Al-Si alloy at room temperature. Thus, all of the earlier investigations used pressing temperatures in the range from 543 to 673 K [10–17] but processing at room temperature is an easier operation especially when using a conventional ECAP die where the sample is removed between each pass. Fourth, to evaluate the mechanical properties after ECAP using conventional tensile testing and a ball-indentation technique (BIT) where BIT has the potential of providing very extensive mechanical information through the utilization of only a very small amount of test material [18,19]. As will be demonstrated, the Al–2% Si alloy was successfully processed by ECAP at room temperature and BIT provided a simple and expedient method of evaluating the mechanical properties in the as-pressed condition.

2. Experimental material and procedures

The material used in this investigation was an Al–Si alloy prepared by melting aluminum and silicon in an electric resistance furnace at 850 °C and taking care to remove the gases by the addition of a commercial degasser of 1% hexa-chlororothene. The melt was cast into an iron mould to form a bar having a diameter of 12.5 mm and length of 80 mm. The chemical composition of the Al–Si alloy, recorded using a flame-emission spectrometer, was 2.0% Si, 0.15% Fe, 0.18% Ti with the balance as Al. It should be noted that higher levels of Si give improved tribological and mechanical properties but the present experiments were conducted specifically to evaluate the mechanical properties after ECAP of an aluminum-based alloy having a low Si content. Finally, samples from the as-cast alloy were machined for processing by ECAP with diameters of 10 mm and lengths of 60 mm.

The ECAP was conducted using a solid die with an intersection of the two parts of the channel of $\Psi = 90^\circ$. It can be shown from first principles that these values of $\phi$ and $\Psi$ lead to an imposed strain of $\sim 1$ on each pass through the die [20]. Repetitive pressings were conducted at room temperature up to a total of 4 passes, equivalent to an imposed strain of $\sim 4$, using route BC where the sample is rotated by 90° in the same sense between each pass [5]. Separate samples were pressed through totals, $N$, of 1–4 passes for subsequent examination of the mechanical properties.

After pressing, the mechanical properties of the as-pressed billets were evaluated in two different ways. First, conventional tensile specimens were machined from the billets with gauge lengths of 4 mm lying parallel to the pressing direction and cross-sectional areas of 3 mm × 2 mm. For comparison purposes, similar tensile specimens were also machined from the as-cast material. All of the tensile tests were conducted at 298 K using a testing machine operating with a constant rate of cross-head displacement and with initial strain rates in the range from $1.0 \times 10^{-3}$ to $1.0 \times 10^{-1}$. Second, the mechanical properties were also evaluated using a new ball-indentation technique (BIT) in which a spherical indenter is pressed repetitively onto the same point on the sample surface with intermediate partial unloading. A full description of the BIT testing procedure is given elsewhere [18,19] and there are recent reports of the results obtained in applying this procedure to a range of materials [21,22]. Briefly, very small samples were used for BIT with typical dimensions of 5 mm × 5 mm × 2 mm, the load was applied from a selected height and repetitive loadings were performed through a loading-unloading-reloading sequence in order to establish multiple load-deflection curves. These curves were then converted into plots of true stress versus true plastic strain using appropriate software [21], thereby providing a comprehensive set of information on the engineering properties of the material. In the present investigation, the indenter was in the form of a tungsten carbide ball having a diameter of 1.57 mm, the loadings were conducted using a load cell of 2500 N capacity, and a repetitive sequence of loading and unloading was applied to the sample at a velocity of 0.5 mm min$^{-1}$.

To evaluate the microstructure before and after ECAP, several samples were prepared for metallographic examination using standard techniques and they were examined using optical microscopy, scanning electron microscopy (SEM) and atomic-force microscopy (AFM). For observations using AFM, samples were carefully prepared with a final polish using 1 μm diamond paste. For grain size measurements, samples were etched with nitric acid and methanol in a 1:3 solution. The AFM observations were undertaken using an SPA-400 SIEK microscope operating in contact-force mode with a force reference value during scanning of 1.465 nm for all samples. This procedure ensured good approaching distances between the sample surfaces and the tip and hence the topographic images provide a good representation of the true surface features. Average grain sizes were measured using SEM or AFM.

3. Experimental results

3.1. Microstructural observations

Typical microstructures of the Al–2% Si alloy are shown in Fig. 1 in the as-cast condition without ECAP processing using (a) optical microscopy, (b) SEM and (c) AFM. From the appearance in optical microscopy, and from similar photomi-
crographs recorded using SEM, it was concluded that the Si is distributed reasonably uniformly within the Al matrix after casting. In Fig. 1(c), the topographic image obtained in AFM shows that, due to the etching, the Si particles protrude in the z-direction from the specimen surface where these protrusions are revealed as bright contrast. The measured average grain size in this condition was \( \sim 40 \mu m \). Using Fig. 1(c), the topography was recorded in one of the Al grains and the distribution of Si was scanned at various selected positions. The results confirmed the uniform distribution of Si. For this as-cast condition, detailed measurements from several micrographs gave an average size for the Si particles of \( \sim 12.0 \mu m \).

Inspection of samples after ECAP revealed the occurrence of several significant changes. Examples of these changes are visible in the SEM micrographs recorded in Fig. 2 after (a) 2 passes, (b) 3 passes and (c) 4 passes and in the AFM image recorded after 4 passes in Fig. 3. An important observation after processing by ECAP was that the average size of the Si particles was significantly reduced. Thus, the Si particles were distributed along the grain boundaries and within the crystalline matrix with typical sizes in the range of \( \sim 5-15 \mu m \) in the as-cast alloy whereas the sizes were reduced to \( \sim 1.0-1.5 \mu m \) in the alloys processed by ECAP. Table 1 records the measured average particle sizes before and after ECAP and it is apparent the average particle size drops significantly in the first pass of ECAP but thereafter there is only a minor reduction with additional pressings up to the maximum of 4 passes. As anticipated, processing by ECAP leads also to very significant grain refinement and the grain (or subgrain) size after 4 passes was measured as \( \sim 0.70 \mu m \). It was also concluded from detailed inspection of large areas that the Si particles were primarily situated along the grain boundaries after 2 or more passes of ECAP whereas in the as-cast structure the Si was more uniformly distributed both along the boundaries and within the matrix. This change in particle location is due to the very significant grain refinement introduced by ECAP.

3.2. Conventional tensile testing

Tensile testing was conducted at 298 K and Fig. 4 plots the values of (a) the yield stress (YS) and (b) the ultimate tensile stress (UTS) for samples taken through 1–4 passes of ECAP. By comparison, the measured values for YS and UTS in the as-cast and unpressed alloy were \( \sim 47 \) and \( \sim 100 \) MPa, respectively. It is apparent, therefore, that processing by ECAP produces a
significant increase in the strength of the alloy even after a single pass. For \(N=1\) pass, the values of the yield stress and UTS were \(\sim 192\) and \(\sim 210\) MPa at the fastest testing strain rate of \(1.0 \times 10^{-1}\) s\(^{-1}\) and this increase in strength by a factor of \(\sim 2\) is consistent with a wide range of commercial aluminum alloys [2]. Furthermore, it is apparent from Fig. 4 that the values of YS and UTS systematically increase with increasing numbers of passes and ultimately, after \(N=4\) passes, the YS and UTS at the fastest strain rate are \(\sim 250\) and \(\sim 271\) MPa, respectively. There is also a clear decrease in the values of YS and UTS with decreasing strain rate.

3.3. Mechanical properties using the ball-indentation technique (BIT)

All indentations were performed using a WC ball having a diameter of 1.57 mm and with an indentation speed of 0.5 mm min\(^{-1}\). In practice, it is possible to convert the speed of indentation into an effective strain rate, \(\dot{\varepsilon}\), using the relationship

\[
\dot{\varepsilon} = \frac{2V_i}{5d_i}\]  

(1)

<table>
<thead>
<tr>
<th>Condition</th>
<th>Size of Si particles (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>(\sim 12.0)</td>
</tr>
<tr>
<td>1 pass</td>
<td>(\sim 1.40)</td>
</tr>
<tr>
<td>2 passes</td>
<td>(\sim 1.34)</td>
</tr>
<tr>
<td>3 passes</td>
<td>(\sim 1.25)</td>
</tr>
<tr>
<td>4 passes</td>
<td>(\sim 1.08)</td>
</tr>
</tbody>
</table>
Fig. 4. Values of (a) yield stress and (b) ultimate tensile stress obtained from conventional tensile testing after 1–4 passes at room temperature.

where \( V \) is the speed of the indenter and \( d_p \) is the diameter of the plastic indentation [23]. As the value of \( d_p \) varied with the indentation depth in the range from \( \sim 0.3 \) to \( \sim 1.0 \) mm, it follows from Eq. (1) that the effective strain rate varied from \( \sim 0.66 \) to \( \sim 0.20 \) min\(^{-1}\).

Typical load-deflection curves are shown in Fig. 5 for the Al–2% Si alloy both in the as-cast condition labeled A and after 1–4 passes of ECAP labeled B–E, respectively, where these curves document the sequential loading/unloading/reloading cycles of the indentation process. Thus, the load-deflection curves for the as-cast alloy are significantly lower than after processing by ECAP and the other curves reveal a sharp jump in the recorded loads after a single pass (curve B) and then smaller incremental increases in the load after 2–4 passes (curves C–E).

Indeed, the curves for 3 and 4 passes are clearly close to a saturation condition which is consistent with the values of the yield stress and UTS shown in Fig. 4. Thus, it is immediately apparent there is a similarity between the information obtained using conventional tensile testing and the results from the BIT technique. Fig. 5 shows there is a maximum deflection of 0.2 mm for the as-cast alloy and the corresponding load was \( \sim 500 \) N. This maximum load increased to \( \sim 700 \) N after 1 pass and then to close to \( \sim 800 \) N after 3 or 4 passes.

Fig. 6 shows the calculated values of true stress versus true strain derived from the BIT data at room temperature for the as-cast condition and after 1–4 passes.

A complete summary of the experimental data is given in Table 2 for the sample taken through \( N = 4 \) passes: thus, the various columns denote the total depth of the indentation, \( h_t \), measured with the indenter applied to the sample, the estimated...
plastic depth, $h_p$, measured after unloading the indenter, the total diameter of the indentation with the indenter in place, $d_t$, the diameter of the plastic zone after removing the indenter, $d_p$, and the final two columns show the estimated values for the true stress, $\sigma_t$, and the true plastic strain, $\varepsilon_p$. In these measurements, as described in detail elsewhere [19], the total indentation depth includes both the elastic and the plastic portions of the indentation and the experiment was conducted so that the total indentation depth was measured during loading and the plastic indentation depth was measured during unloading. It is apparent from Table 2 that an increase in the load leads to an increase in the total depth and thus the trends visible in the BIT data are consistent with those obtained from conventional tensile testing. However, there is a significant advantage in using the BIT procedure because it utilizes only a very small amount of test material. It is anticipated this will often be an important consideration when evaluating samples after processing by ECAP where the total quantities of the as-pressed materials may be severely limited.

### 4. Discussion

Several important conclusions may be reached from this investigation.

First, it is feasible to process the Al–2% Si alloy by ECAP at room temperature. In the present experiments, the Al–2% Si alloy was successfully processed using a conventional die and the optimum processing route BC.

Second, processing by ECAP leads to significant grain refinement, with a reduction in grain size from ~40 μm in the as-cast alloy to ~0.7 μm after ECAP through 4 passes, and it leads in addition to a significant breaking of the Si particles. Thus, the average particle size was reduced from ~12.0 μm in the as-cast condition to ~1.08 μm after 4 passes. The reduction in grain size increased the yield stress and the ultimate tensile strength by factors of ~2 with reference to the as-cast alloy.

Third, and most important, it is feasible to obtain critical information on the mechanical properties of the material using a ball-indentation technique (BIT) which requires the use of only a very small amount of test material. By contrast, conventional tensile testing requires the processing of a large number of billets and the machining of a set of individual tensile specimens from these as-pressed billets. The alternative BIT procedure used in this investigation is especially attractive for use with samples processed by ECAP because it is simple to execute and requires only the preparation of a polished surface.

Finally, the present results documented in Table 1 provide a very clear demonstration that the Si particles are fractured during processing by ECAP because of the imposition of an extremely high stress during the pressing operation. The potential for precipitate fragmentation in ECAP was first reported for the $\theta^\prime$-precipitates in an Al–Cu alloy [24] and subsequently there were similar reports for several other aluminum-based alloys [25–28]. The results obtained for the Al–2% Si alloy in this investigation are consistent with these other reports and provide further confirmation that SPD processing may introduce additional significant changes that necessitate the use of careful microstructural characterization following the processing operation.

### 5. Summary and Conclusions

1. A cast Al–2% Si alloy was successfully processed using ECAP for up to a maximum of 4 passes at 298 K. The ECAP was conducted using a conventional die and processing route BC.

2. Processing by ECAP reduced the grain size from ~40 to ~0.7 μm after 4 passes and there was also a concomitant reduction in the average size of the Si particles from ~12.0 μm in the as-cast condition to ~1.08 μm after 4 passes. The reduction in grain size increased the yield stress and the ultimate tensile strength by factors of ~2 with reference to the as-cast alloy.

3. A ball-indentation technique (BIT) was used successfully to evaluate the mechanical characteristics of the samples processed by ECAP. Since this is a non-destructive technique requiring only a very small sample, it appears to be an ideal procedure for evaluating the properties of samples processed by ECAP.

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