Friction Stir Processing of the Magnesium alloy AZ61: Grain Size Refinement and Mechanical Properties

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Abstract. The effect of friction stir processing (FSP), on the microstructure and mechanical properties of a magnesium alloy AZ61 has been analyzed. This is a widely used wrought magnesium alloy provided in the form of rolled and annealed sheets with a grain size of 45 μm . The FSP was performed with an adequate cooling device in order to increase the heat extraction and reduce the processing temperature. The final microstructure showed a noticeable grain size refinement down to values close to 1.8 μm and an important change in texture. The change in texture favors basal slip during tensile testing leading to an increase of ductility and a decrease in yield stress. The stability of the grain size and the creep behavior at high temperatures were investigated. The optimum conditions for superplastic forming were determined; however, the presence of a large amount of cavities precludes the achievement of high superplastic elongations. Additionally, these results are compared with those obtained by severe hot rolling.

Introduction

Finding materials with high strength and high ductility is an extremely relevant issue and an important goal in the field of materials science due to its industrial applications. This goal can be reached by producing materials having a fine and homogenous microstructure. This results in an increase of the room temperature yield strength (Hall-Petch) [1], an increase of the room temperature ductility by an increase of the strain rate sensitivity [2], and better formability at warm/high temperatures due to the enhancement of the grain boundary sliding mechanism which has the mechanical effect of generating superplasticity [3]. Enhanced elongations are indeed observed in Mg alloys for grain sizes as high as 20 microns but finer grain sizes could lead to obtain superplasticity at higher strain rates and lower forming temperatures [4]. There are various processing techniques that allow reaching this desirable microstructure. These techniques usually involve severe plastic deformation of the materials and the most well known are Equal Channel Angular Extrusion (ECAE)[5], Large Strain Hot Rolling (LSHR) [6], Accumulative Roll Bonding (ARB) [7], Friction Stir Processing (FSP) [8], and High Pressure Torsion (HPT) [9]. However, ECAE and HPT are not adapted to producing ultrafine grained material in the sheet form, while LSHR and ARB are processes having a limitation on the grain size usually of few microns [10]. Therefore, one of the potential applications of FSP is its grain refinement capability, particularly in the case of materials in the form of sheets.

In this work, the mechanical properties at room and high temperatures, of the AZ61 alloy refined by FSP, were analyzed. At room temperature, factors such as the effect of texture and grain size on the work hardening (WH) behavior and the strain rate sensitivity (SRS) were analyzed. At high temperature, the stability of the microstructure was evaluated together with the stress-strain rate

behavior and the ductility in the superplastic regime. These data were compared with previously published data on the material processed by LSHR [11-13].

Experimental procedure

The material used in this study was a commercial Mg alloy AZ61 with the following composition: 6%Al, 1%Zn and Mg (balance) provided by Magnesium Elektron in the form of a rolled sheet, 3 mm in thickness. The alloy AZ61 was received in the condition O (rolled and annealed). The FSP was carried out on the AZ61 sheets in the as-received form.

Figure 1. Schematic drawing of the cooling system used in this work.

In order to increase the heat sink during FSP an experimental set-up was developed, Fig. 1. Usually the FSP is carried out on a steel backing plate. However, it is well known that, as the temperature of the plastic deformation decreases, the grain size of the recrystallized grains decreases [14]. Based on previous works of Chang et al [15] and Du and Wu [16] a backing copper plate with two machined tunnels for liquid nitrogen refrigeration was designed, as shown in Figure 1b. In this way a transfer of the heat generated between the tool and the sample during FSP occurs as quickly as possible. Several FSP tests, under different processing conditions, were carried out on the AZ61 alloy. A comparative analysis of these tests will be reported elsewhere. In the present work, the AZ61 was processed with a two FSP-passes using the cooling system of Fig.1, performed on a MTS PDS-4 Intelligent-Stir using a 1.95 mm thick pin tool. The advancing speed of the rotating pin was kept 50 mm/min, with a rotation speed of the pin of 1000 rpm. The tilt angle was -1.5° in all passes.

The grain structures on cross-sectional planes of the etched specimens were examined by optical microscopy (OM) scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The sample preparation for OM was described previously [13]. For TEM observations the material was prepared by electrolytic polishing using a mixture of 50% nitric acid and 50% methanol. Ion milling at liquid nitrogen temperature was used to remove a fine oxide film forming in the surface. The samples were examined in a JEOL JEM-2032 microscope operating at 200 kV. Tensile samples were spark-machined with the gage length parallel to the FSP direction and a cross section of 3x1.2 mm in coincidence with the nugget zone.

Results and discussion

Microstructure and texture of the processed AZ61 alloy.

In Figure 2a, a cross section of the stir zone (SZ) for the AZ61 is shown. It can be seen that the sample shows a section with no cavities or porosity. It is also highlighted the excellent surface quality of the SZ. Fig. 2b and 2c show TEM and optical micrographs of processed materials respectively. The grain size of the SZ present a small gradient through the thickness with a mean value of 1.8 μm . Figure 2d and 2e shows the effect of performing annealing treatments of 1 hour at

250 and 300°C respectively. Figure 2f shows the evolution of the grain size with the annealing temperature. The data were compared against literature results of the AZ alloys [17-23].

Figure 3 shows the influence of FSP processing on texture. The initial texture corresponds to the well known basal texture of the rolled AZ61 alloy. The FSP produces a noticeable change in texture. The prismatic planes rotate to a position nearly parallel to the sheet plane, the $\langle 10\text{-}10 \rangle$ directions concentrates in a pole with a intensity of 6 and a tilt of approximately 30° towards the FSP direction takes place. The c-axis is distributed in a radial form around this pole. This texture yields the basal planes rotated 60° with respect to the FSP direction. These results are in agreement with other of literature [24, 25]

Figure 2: (a) Macrograph of the cross section of the SZ (b) TEM micrograph (c) OM micrograph. (d) SEM micrograph of the microstructure after annealing 1 hour at 250 °C (e) OM of the microstructure after annealing 1 hour at 300 °C (f) Grain size as a function of the annealing temperature, compared with data from the literature.

Figure 4a shows the mechanical behavior at room temperature during tensile test, in contrast with the data of the rolled material published elsewhere [11, 12]. In comparison with the rolled alloy, an increase in ductility with a decrease in yield stress are the most noticeable findings. The

decrease in yield stress, despite the finer grain size of the FSP sample, can be attributed to its much favorable Schmid factor for basal slip in the FSP samples in rolled samples.

Figure 3: (0002) pole figures of (a) as-received AZ61 maximum intensity of 11, (b) FSP samples of AZ61. (c) and (d) AZ61 rolled samples, data from ref. [11].

Figure 4: (a)(b)(c) Tensile curve, work hardening rate and SRS of FSP samples compared against the rolled alloy. (d) Creep behavior at 225°C and 275°C.

It is interesting to compare the work hardening rate of FSP and rolled materials, Fig 4b. The large ductility of the FSP material is related to a WH stage with constant low hardening rate. A similar behavior was reported in samples processed by ECAP having the basal planes rotated 45°C with respect to the tensile direction [26]. This stage of constant hardening is related to the

predominance of basal slip, due to the favorable orientation of basal planes under tensile stress. In both case tensile plasticity is stable while the hardening rate is beyond the Considere's limit $d\sigma/d\varepsilon = \sigma$. The evolution of the strain rate sensitivity against flow stress was measured by means of strain rate jumps, described in ref. [12]. Figure 4c shows that the sensitivity is lower than for rolled samples of similar grain size, during most of the tensile test. High strain rate sensitivities were previously related [12] to the activity of prismatic slip. Congruently, the low strain rate sensitivity of FSP samples is close related to the basal slip activity.

Figure 4d shows the flow properties at high temperature. Strain rate change tests [27] were carried out to determine the flow stress vs. strain rate behavior, Fig 4d. As reported in fine-grained magnesium alloys, the creep behavior can be described by a power law [26]. In Fig 4d, a regime with stress-exponent, n , close to two was determined below of 10^{-2} s^{-1} . Data shown in Fig. 4d indicates that, due to the fine grain size, higher superplastic strain rates can be achieved at lower temperatures in comparison with the material processed by severe hot rolling [13]. In theory, a strain rate of 10^{-2} s^{-1} is possible for superplastic forming using a temperature of 275°C . Higher temperatures are forbidden due to the undesirable grain growth, as shown in Fig. 2f. Unfortunately, tensile test performed at 10^{-2} s^{-1} and $T = 275^\circ\text{C}$ has shown an unexpected low ductility of 150%. A detailed study of the microstructure after the tensile test showed a large amount of cavities, Fig 5. This cavity formation follows a circular pattern related to deformation during prior friction stir processing, giving a clue about the existence of microstructural damage in the FSP SZ. In addition, a noticeable grain growth occurs in the gauge length of the sample, i.e. induced by deformation, leading to a grain size of $6 \mu\text{m}$.

Figure 5: Tensile test performed at 10^{-2} s^{-1} , $T = 275^\circ\text{C}$, (a) region of the sample near the fracture (b-c) cavity formation following a circular pattern related to the deformation during prior FSP.

Summary

1) FSP processing was successfully performed on the AZ61 alloy with a cooling device in order to reduce the processing temperature. The final microstructure showed a noticeable grain size refinement down to values close to $1.8 \mu\text{m}$ and an important change in texture.

2) The texture developed during FSP favors basal slip during the tensile test, leading to an increase of ductility, a decrease in yield stress and a decrease in strain rate sensitivity in comparison with rolled AZ61 alloy having a basal texture.

3) The stability of the grain size and the creep behavior at high temperatures were investigated. The optimum conditions for superplastic forming are given by temperatures lower than 300°C. In theory, high forming strain rates are possible; however the presence of a large amount of cavities, related to prior FSP, precludes the achievement of high superplastic elongations. Therefore, the routes of FSP should be further improved for superplastic forming applications.

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