Fine And Ultra-Fine Grained AZ61 And AZ91 Magnesium Alloys Obtained By Friction Stir Processing

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Abstract. Friction Stir Processing (FSP) has attracted much interest as a tool for refining grain size and achieving high angle boundary misorientation in magnesium alloys. These characteristics have a great influence in key engineered properties such as strength and ductility, which could be markedly improved by means of this technique. The main objective of this work is to study the microstructural modifications produced when FSP is applied to homogenized cast AZ91 and wrought AZ61 magnesium alloys. Several attempts were made for achieving a homogenous microstructure without defects and enhancing the refinement of the grain size in the stir zone.

It was revealed that is of great importance to break the initial microstructure, of coarse grains unfavourably oriented for deformation, in order to facilitate the process, particularly in the case of cast AZ91 alloy. It is highlighted that, after breaking up the initial microstructure, is possible to process the material, in subsequent passes. Furthermore, the use of different backing materials as heat sink and a previous heating treatment of the sample were evaluated. Changing the backing plate can improve more the reduction of the grain size during a second pass. Using a copper plate instead of a steel one can promote a refinement up to 700 nm in AZ91 and 1 μ m in AZ61. A coolant agent can be used for inhibiting the grain growth causing a little more reduction of the grain size.

Introduction

Recent investigations have shown that there is a great potential to improve extraordinarily the physical and mechanical properties of alloys by refining their grain structure to submicrometric or nanometric scale. Grain refinement results in an increase of the room temperature yield strength (Hall-Petch) [1], an increase of the room temperature ductility by an increase on strain rate sensitivity [2], and on the formability at warm/high temperatures due to the enhancement of the grain boundary sliding mechanism which has the mechanical effect of generating large tensile deformations (*superplasticity*) [3]. Enhanced superplastic 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 involve severe plastic deformation of the materials and the most well-known are Equal Channel Angular Extrusion (ECAE), Large Strain Hot Rolling (LSHR), Accumulative Roll Bonding (ARB), Friction Stir Processing (FSP) and High Pressure Torsion (HPT) [5-9].

Superplastic forming procedures improve the formability of light alloys due to the extremely fine grain size. However, ECAE and HPT are not adapted to producing ultrafine grained material in the sheet form, while LSHR and ARB are processes which have a limitation on the grain size obtained to values close to the micron. In addition these conventional grain refinement techniques usually involve thermo-mechanical processing which is costly, time consuming, and negatively affects the environment due to high energy consumption. FSP is an alternative effective method to produce

very fine-grained microstructures at relatively low temperatures for aerospace and automotive applications. The advantage is that the materials can be processed in sheet form.

This work focuses on FSP induced microstructural modifications in both cast and wrought magnesium alloys. Particular attention was paid to the level of grain refinement related to the heat release efficiency together with the achievement of a stirring zone with a good quality. The effect of composition on the feasibility of the process is also highlighted

Experimental

Two commercial Mg alloys provided by Magnesium Elektron were studied: AZ61 rolled sheet and AZ91 as-cast ingot with the following composition: 6%Al, 1%Zn and Mg (balance) and 9%Al, 1%Zn, and Mg (balance) respectively. The FSP was carried out on the AZ61 sheets in the as received form (condition O, rolled and annealing). The AZ91 ingot was homogenized at 415-420 C° for 24 hours and air quenched. From the ingot, 2 mm in thickness plates were cut by wire electrical discharge machine. Successively, the plates were homogenized at 415-420 C° for 1 hour and quenched in a press under pressure in order to obtain a flat plate of material in a solid solution state.

The FSP passes were performed on a MTS PDS-4 Intelligent-Stir using a whole MP159 tool with a scrolled shoulder diameter of 9,5 mm and a threaded pin length slightly shorter than 2 mm. The rotation rate was 700 rpm in clockwise direction and traverse speed of the tool was 120 mm/min. The tilt angle of the tool was 1.5°. In order to controls the heat sink during the FSP processing two experimental set-ups were developed (Fig 1). As it is well known, as the temperature of the plastic deformation decreases, the grain size of the recrystallized grains decreases. Based on previous works of Chang et al [10] a backing copper plate with two channels for refrigeration with liquid nitrogen (LN) was designed (Fig. 1-b). In this way a transfer of the heat generated between the tool and the sample during FSP occurs as quickly as possible. Several PSP test were carried out on the AZ91 and AZ61 alloys (Table 1).

Fig. 1. Schematic drawing of the heat insulation made previously of the using the cooling system (a) and the cooling system used in this work (b).

Table 1: Processing conditions applied to the alloys AZ61 and AZ91

FSP on AZ61	FSP on AZ91
FSP over steel backing plate (AZ61/1)	FSP over steel backing plate (AZ91/1)
FSP over copper cooler (without LN) (AZ61/2)	FSP over copper cooler (without LN)(AZ91/2)
FSP with pre-heating to 200 °C, over a heat insulator (AZ61/3)	FSP with pre-heating to 200 °C, over a heat insulator (AZ91/3)
FSP first pass as AZ61/3 plus a second pass over copper cooler (without LN) (AZ61/4)	FSP first pass as AZ91/3, plus a second pass over copper cooler (without LN)(AZ91/4)
FSP first pass with preheating to 200 °C, plus a second pass over copper cooler (with LN) (AZ61/5)	FSP first pass with preheating to 200 °C, plus a second pass over copper cooler(with LN) (AZ91/5)
FSP first pass with preheating to 200 °C, plus a second and a third pass over copper cooler (with LN)(AZ61/6)	FSP first pass with preheating to 200 °C, plus a second and a third pass over copper cooler (with LN)(AZ91/6)

The effect of using a backing plate of steel, or alternatively cooper (without LN flux) was first investigated, following the procedure of using the lowest temperature possible to make the friction

stir in one pass. This procedure is relatively successful in the case of the alloy AZ61 even using the fastest heat extraction provided by the use of copper plate with a flow of nitrogen. On the other hand, in the case of AZ91 alloy was not possible to obtain FSP passes of good quality even with the slower heat extraction (steel backing plate). Therefore, we designed a two-pass process, with the first conducted with a sample preheat to 200 degrees and a Sindanyo® 5 mm plate was used as a backing plate heat insulator during the first pass as it is shown in fig.1-a., and a second under refrigerated conditions. To compare with the AZ61 microstructures, two-pass process also was applied to AZ61. The influence of the cooling rates on the quality of the stirring zone and the refinement of the grain size were therefore determined.

The grain structures on cross-sectional planes were examined by optical microscopy (OM) and scanning electron microscopy (SEM). The sample preparation for OM consists of grinding with a SiC paper, followed by mechanical polishing with 6 μ m and 1 μ m diamond paste and short final polishing using colloidal silica. Subsequent etching using a solution of ethanol (100 ml), picric acid (5 g), acetic acid (5 ml) and water (10 ml) revealed the grain structure. For SEM observation, the material was prepared initially by mechanical polishing with 600 and 1200 SiC papers followed by electropolishing in phosphoric acid and ethanol (5/1) at -8°C and at a potential of 5 V during 2 s.

Results and discussion

Microstructural analysis of the AZ91 and AZ61 alloys previous to FSP.

Fig. 2.A shows a polarized optical micrograph with the microstructure of the original AZ91D. A fully developed dendritic structure with a typical eutectic phase between dendritic arms is observed. The microstructure of this alloy is characterized by α -Mg primary dendrites with a eutectic Al-rich solid solution and the intermetallic β -Mg₁₇Al₁₂ in between. It is necessary to change the morphology of this phase by means of thermal treatments for making the FSP tests. Fig. 2-B shows micrographs of AZ91 samples in the as-cast condition and after annealing at 413°C for 24 hours (2-C). Micrographs reveal that the thermal treatment is effective for the partial dissolution of almost coarse eutectic Mg₁₇Al₁₂. The grain size of the AZ91 plates is close to 2-4 mm after annealing and the texture of these samples is close to random, as usual in cast magnesium alloys.

Fig. 2. Macrograph of as-cast AZ91 (a) and micrograph of as-cast (b) and after 24h annealing 415-420 C° showing the modification of initial coarse microstructure (c)

On the other hand, the AZ61 sheets present a quite different initial microstructure. The mean grain size was determined to be 60 μ m (Fig. 3a), and the sheet presents a strong basal texture as expected in rolled magnesium alloys (Fig. 3b).

Fig. 3. Microstructure of as received AZ61 sheet (a) and (0002) pole figures of as-received AZ61 maximum intensity of 11 (b).

Feasibility of the FSP on the AZ91 alloy

Fig. 4 shows the result of the FSP corresponding to the sample AZ91/1. Fig. 4-a shows a good quality in the stirring zone. However the microstructure in this zone observed by SEM shows a grain size not suitable for achieving superplastic properties (Fig. 4-b). Final grain average size of 8μm was observed.

Fig. 4. Macrograph of the AZ91/1 (a) and micrograph for the stirring zone (b)

When a copper backing plate (AZ91/2) is used, the quality of the stirring zone decreased as a consequence of the faster heat extraction as can be shown in Fig. 5-a. For improving the quality of this zone, a preheating of 200°C was carried out and a first pass over an insulator was made for breaking the initial grain (AZ91/3). When a second pass over the copper cooler was made with the previous pass (AZ91/4), the stirring zone results without defects as can be seen in Fig. 5-b.

Fig. 5. Macrographs corresponding to the samples AZ91/2 (a) and AZ91/4 (b)

When LN is used with the copper plate after the first pass over the heat insulator (AZ91/5), the results are also good with only a little void found on the advancing side (Fig. 6-a). When a second pass over Cu with LN is done (AZ91/6), any void was found in the stirring zone (Fig. 6-b).

Fig. 6. Macrographs corresponding to the samples AZ91/5 (a) and AZ91/6 (b).

A fine microstructure in the nugget region was observed which contrasts with the dendrite structure that is present in the base material. However, the microstructures in the nugget of the copper samples are significantly finer than those without the copper plate which proves the contribution of this plate as rapid heat sink. Fig. 7 shows the SEM micrographs of the stirred zone for the specimens AZ91/4 and AZ91/5. The mean grain size for AZ91/4 in the stirring zone is 700 nm. The use of a cooling agent seems to exert some influence in the final grain size. As a consequence of using liquid nitrogen, a lower grain size is produced because of a faster heat sink and a lower initial temperature. Lower temperatures avoid the grain growth in the stirred zone. When LN is used a final grain size of 400 nm is achieved. However, the effect of the coolant is less accused than when we use the copper cooler compared with the steel backing plate. Moreover, the effect of doing a second pass over the copper with LN (AZ91/6) not produced any further refinement in the grain size.

Fig. 7. Micrographs showing the grain size in the stirring zone in AZ91/4 (a) and AZ91/5 (b)

Feasibility of the FSP on the AZ61 alloy

For comparison purposes, the same FSP passes were made in AZ61. The macrographs reveal consolidated stirring zones for all samples with only little voids in AZ61/4. This reveals that AZ61 is more feasible for doing FSP and that any previous thermal treatment must be done as in AZ91. Fig. 8 shows the macrographs for the FSP made directly over the copper cooler without LN (AZ61/2) and over the copper cooler using LN (AZ61/5).

Fig. 8. Macrographs corresponding to samples AZ61/2 (a) and AZ61/5 (B)

Compared to AZ91, any void is found in the stirring zone. However the grain size for AZ61 is quite different. When FSP is made over the steel backing the grain size is smaller than for AZ91

(Fig. 9-a) but when a copper backing plate with LN flux is used, the resultant grain size is larger than for the AZ91 sample processed in the same conditions (Fig. 9-b). An average size of 6 μ was found in the stirring zone for AZ61/1 whereas a mean grain size of 800 nm was found for the AZ61/5. Any further refinement is achieved when a second pass is made. The lower grain achieved in AZ91 can be a consequence of the massive precipitation of β -phase which makes a pinning effect inhibiting the grain growth.

Fig. 9. SEM micrographs for the stirring zone of AZ61/1 (a) and AZ61/5 (b)

Summary

1) The effect of the thermal conductivity of the backing plate in the quality of the stirring zone and in the final grain size was studied independently of the welding and rotation speed.

2) The processing feasibility of the AZ61 alloy is higher than AZ91 and it can be processed with a high heat extraction in the first pass without doing a previous thermal treatment or pass to break the microstructure. However, AZ91 can not be processed in a unique pass with a small grain size unless it was pre-heated. This problem can be attributed to an inadequate initial microstructure because of the big grain size and random texture. Doing a previous pass over a heat insulator, promotes a favourable microstructural refinement and texture for further FSP passes.

4) For the same FSP conditions, the final grain size is lower for the AZ91 alloy than for the AZ61 alloy due to a massive precipitation of β -phase which makes a Zenner pinning effect on the grain boundaries. Mean grain size founded in the stirring zone for AZ91/5 and AZ61/ 5 was 400 and 800 nm respectively

5) FSP process has been optimized obtaining stirring zones with high quality and fine microstructures for both AZ91 and AZ61 alloys.

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