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THE EFFECT OF FRICTION STIR PROCESSING ON MICROSTRUCTURE AND TENSILE BEHAVIOR OF THIXOMOLDED AZ91 MAGNESIUM ALLOY

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Abstract

Friction Stir Processing (FSP) to partial sheet thickness can be utilized to engineer unique microstructures in metallic alloys. These composite microstructures consist of three distinct layers associated with stirred, transition and core microstructural regions. The stirred region is of particular interest where severe plastic deformation imparted by the rotating and translating FSP tool under frictional heat leads to grain refinement down to ~ 1 μm grain size. In this work, partial depth penetration into thixomolded AZ91 Mg plate from the top and bottom surfaces by friction stir processing is explored. Furthermore, low temperature aging treatments are applied to the processed material. The present results with AZ91 Mg show that FSP processed material exhibits higher strength (> 300 MPa), and improvement in ductility (> 7 % tensile elongation). It is found that in addition to Hall-Petch strengthening produced by ~ 1 μm grain size in the stirred region, the enhanced strength levels and ductility are strongly influenced by dispersoids of the intermetallic precipitates found in this alloy.

Introduction

Magnesium (Mg) and Mg-based alloys have drawn significant interest as attractive lightweight structural materials with high specific mechanical properties. However, magnesium alloys exhibit moderate strength and limited ductility at room temperature due to their hexagonal close-packed (HCP) crystal structure [1-3]. Presently, most of the magnesium based structural parts are produced by casting processes and despite alloy additions; they have lower strength and formability as compared to their wrought alloy counterparts. Therefore, development of several different high strength wrought Mg alloys with enhanced ductility is imperative for fabricating a variety of parts and products to increase the consumption of Mg. Earlier studies on several metallic alloys have demonstrated that grain refinement and control of precipitates, attained by thermo-mechanical processing can significantly enhance strength and ductility [4-7]. Severe plastic deformation is an effective method for grain refinement of bulk metallic materials. Some noteworthy severe deformation processes include: Equal Channel Angular Extrusion ECAE [8], High Pressure Torsion HPT [9], Accumulated Roll Bonding [10] and Friction Stir Processing [11]. Ultra-fine grained microstructure with < 1 μm grain size can be produced by such methods.

AZ91D is a commercial Mg alloy which is typically available in die-cast or thixomolded condition. Thixomolding is a semisolid injection molding process that was derived from semisolid metal casting techniques developed in the 1970s [12]. The details of the thixomolding process have been reported extensively in the past [13-14]. In addition to near-net shape parts, thixomolding routinely produces low porosity Mg plates. Microstructure of thixomolded alloys typically consists of 3-15 vol.% α_p-Mg globules (~ 30-100 μm) in a matrix of homogenous and fine, secondary α_s-Mg grains (4-5 μm). Furthermore, the α_s-Mg grains are surrounded by a eutectic aggregate (1-2 μm) that consists of α_e-Mg and an intermetallic compound β ($Mg_{17}Al_{12}$) [15]. Commercially available, wrought Mg alloys in hot-rolled or extruded condition are characterized by coarse and highly bimodal microstructures [15]. In comparison, thixomolded alloys with fine and relatively homogeneous microstructure are therefore, suitable candidates for secondary thermomechanical processing to enhance mechanical properties by further grain refinement and homogenization. In this work, severe plastic deformation was applied to thixomolded AZ91 Mg by friction stir processing to homogenize and refine as-molded microstructure. Friction-stir processing is an innovative, emerging technology that is based on friction stir welding (FSW) and many recent studies have been reported on FSW/P of magnesium alloys [16-19]. FSP involves plunging a rapidly rotating tool with a profiled probe and larger

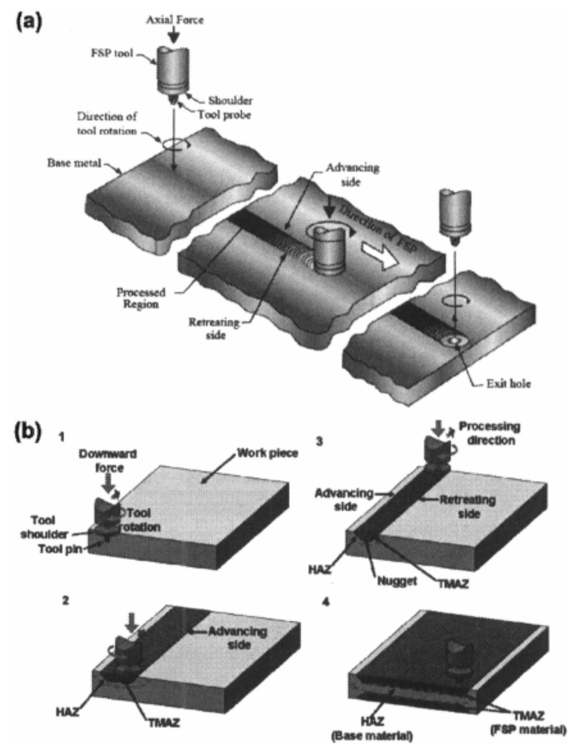

Figure 1. (a) Schematic of FSW/P process, adapted from the *Welding Handbook*, Vol. 3, 7th Ed. [20]. (b) Schematic shows several stages of overlapping FSP. Also, stirred and HAZ (base material) have been identified.

diameter shoulder beneath material surface and then traversing it across (Figure 1 (b)). Frictional heating and intense plastic deformation causes plasticized material (constrained by the shoulder) to flow around the tool and consolidate thereby improving mechanical properties.

The aim of the present study is to reveal the microstructural evolution during partial depth FSP of AZ91 Mg alloy and study the effect of fine grain size and aging treatments on room temperature mechanical properties. The effect of texture on mechanical properties is not investigated at present and will be the subject of a later study.

Experimental

Thixomolded AZ91D Mg alloy was provided by Thixomat Inc. in the form of 3.25 mm thick plate. The nominal composition is 9.0 wt. % Al, 0.75 wt. % Zn, 0.3 wt. % Mn, Fe and Ni < 0.001 wt. % and Mg as balance. In order to achieve grain refinement in thixomolded AZ91 Mg alloy, severe plastic deformation was applied by FSP.

Processing

Friction stir processing to partial depth was performed on top and bottom faces of thixomolded AZ91 Mg plate by inserting a rotating FSP tool into the metal to homogenize and refine as-molded microstructure. A schematic illustration of the FSP process and FSP tool is shown in Figure 1 (a). Important stages of overlapping, multi-pass FSP are shown in Figure 1 (b); stirred zone and Heat-Affected Zone (HAZ) are also identified. Overlapping steps were performed such that the retreating side from the previous pass was overlapped by the advancing side of the subsequent pass. Therefore, the processed region is a collection of advancing sides of multiple overlapping passes. In this work a 100 mm x 100 mm work-piece was processed by FSP. A total of 25 overlapping FSP passes with a 1.5 mm overlap were carried out. FSP was performed on a Doosan milling machine using a friction stir tool machined from H13 tool steel, heat-treated to a hardness of 52-55 Rc. The tool used in the present study had a shoulder diameter of 25.4 mm and the tool pin is 3 mm in dia and 1 mm in length. Dwell periods of 2-3 mins were applied prior to the initial FSP step to reach sufficiently high temperature (300-350 °C). The dwell periods were progressively decreased for the subsequent steps. The FSP processing parameters were empirically determined. The FSP was performed at a rotational speed of 2100 rpm and a linear speed of 101.4 mm/min. A post FSP milling step was performed on the processed sheet to remove the roughness and thickness variations. In this manner, a FSP processed AZ91 Mg sheet with 3 mm uniform thickness was obtained.

Microstructure

Microstructure examination was carried out on as-molded, FSP processed and aged Mg alloys. The as-molded samples were sectioned in a plane containing the normal direction (ND) and molding/rolling direction (RD). FSP samples were sectioned in a plane containing plate normal direction (ND) and transverse direction (TD). The metallographic samples were mechanically polished by the standard methods and etched by acetic-picral solution (4.2 g picric acid, 70 ml ethanol, 10 ml acetic acid and 10 ml distilled water). A Quanta FEG 250 SEM operating at 20-30 KeV was employed to observe the very fine microstructures. Grain sizes were measured from the micrographs by an area-based approximation. In order to determine the optimum conditions for aging; several low temperature aging treatment were performed at 110°C, 150°C and 180°C for 1, 5, 10 and 24 hours. The aging treatments were carried out in air; inside a Nabertherm box electric furnace.

Mechanical Properties

Room-temperature tensile tests were performed on samples with 12.5 mm long × 3 mm wide gauge sections in a computer-controlled, 5982 Instron machine at a constant crosshead speed of 0.5 mm/min. Vickers hardness was measured at an indentation load of 300g for 30 seconds loading time.

Results and Discussions

Microstructure of Thixomolded AZ91 Mg

The typical microstructural features of as-molded AZ91 Mg alloy are shown in Figure 2. The microstructure can be described as equiaxed primary particles of (un-melted) α_p-Mg, dispersed in a continuous matrix of solidified, fine, secondary eutectic α_s-Mg.

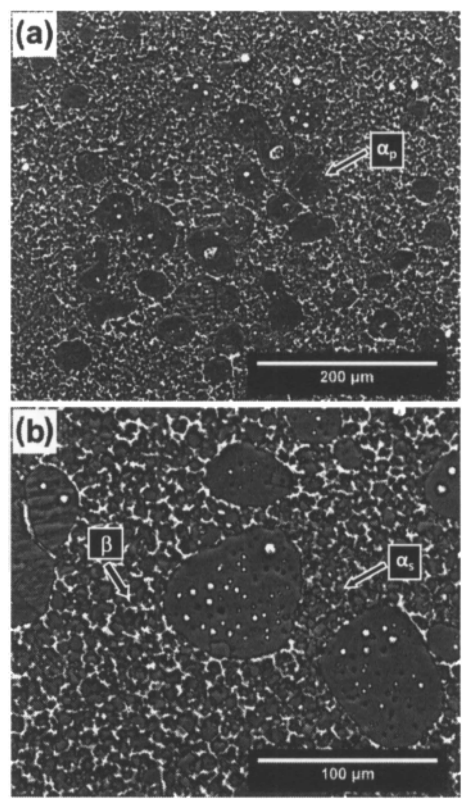

Figure 2. Typical microstructure of as-molded AZ91 Mg alloy. SEM image in (a) shows the overall structure and different morphologies of primary solid phase α_p-Mg particles. (b) shows a matrix of equiaxed secondary α_s-Mg grains with the eutectic (β) intermetallic network distributed in the spaces around the secondary α_s-Mg grains (indicated by the arrow).

The secondary α-Mg grains are surrounded by a fine eutectic β phase network. This corresponds well with the microstructure of a rapidly cooled liquid (~100 °C/s) as no long dendrites (typical of casting microstructure) are visible. Several distinct morphologies of the primary solid particles; spheroidized during thixomolding are found in the micrograph as shown in Figure 2(a), e.g. one is essentially un-modified, solid while another one with entrapped liquid particles is also visible. Figure 2(b), shows morphologically equiaxed, fine secondary α_s-Mg grains; delineated by the network of eutectic component i.e. an intermetallic compound β ($Mg_{17}Al_{12}$). The average grain size of α_s-Mg grains is ~ 5 μm while the eutectic intermetallic film, distributed between the spaces of α_s-Mg grains has a thickness of ~1 μm. In this work, no evidence of coupled eutectic growth was found. it was in fact, difficult to distinguish (both morphologically and chemically) between the fine α_s-Mg grains and the eutectic α_e-Mg phase [21].

Microstructural Evolution During FSP Processing

A low magnification overview of partial depth FSP processed AZ91 Mg is shown in Figure 3. Since the FSP tool pin partially penetrates the depth of work-piece during processing, sheet thickness can be divided in to three distinct and easily identifiable zones.

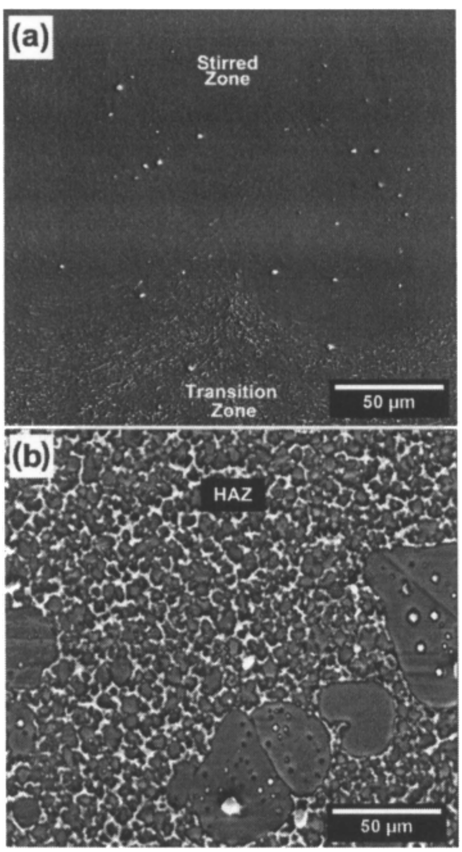

Figure 3. The microstructure AZ91 Mg alloy processed by overlapping multi-step partial depth friction stir process. A unique layered microstructure comprising of (a) stirred zone, transition zone and (b) heat affected zone is created.

The stirred zone, HAZ and transition zone have been identified in Figure 3. The HAZ is located in the middle and it retains the grain structure similar to the as-molded condition. FSP process has no apparent effect on microstructure of HAZ. It has both the as-molded microstructure and the spherical coarse particles (~ 50 μm in diameter). These are the spherical, coarse primary solid particles of nearly pure Mg. However, the continued exposure to elevated temperature leads to solutioninzing and a reduction in primary solid particle size and distribution as compared to as-molded condition.

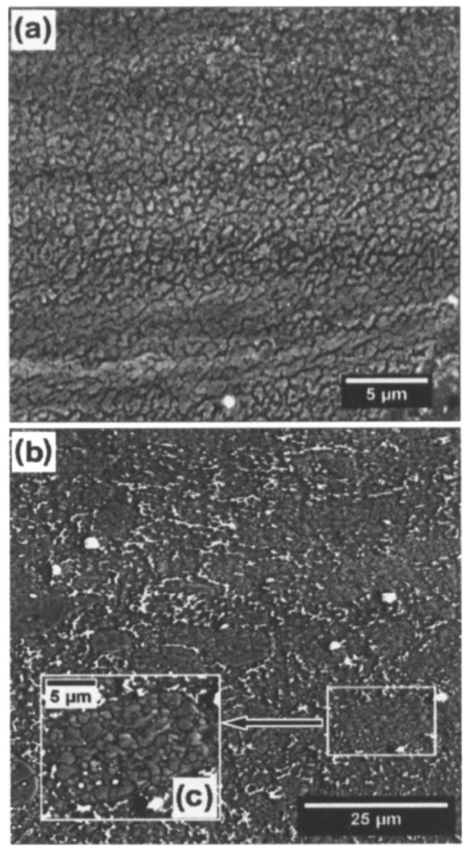

Figure 4. SEM image of FSP processed AZ91 Mg alloy. (a) Shows UFG microstructure with 0.85 μm average grain size in stirred zone, (b) transition zone (near root of tool pin) between FSP processed heat affected zone, (c) in the inset shows the subdivision of a primary solid particle in to several small grains.

The depth of stirred zone corresponds to the FSP pin length ~ 1 mm (not shown here). The general features of the stirred zone microstructure are consistent with earlier reported FSW/P findings for Mg alloys [16-17]. The microstructures for different zones of FSP processed AZ91 Mg are shown in Figure 4. Intense plastic deformation and frictional heating by FSP result in the generation of a ~ 1 μm ultrafine microstructure throughout the stirred zone, as shown in Figure 4(a). These micrographs show that by over-lapping pass FSP, the as-molded microstructure of the thixomolded material can be replaced with a fine and relatively homogeneous α-Mg microstructure. The stirred zone makes up for about 60 vol. % of the processed material. FSP results in complete dispersion and fragmentation of primary solid grains and secondary phases in the stirred zone. No defects, such as cracks and/or porosity, are observed in the stirred zone. According to Al-Mg binary phase diagram, the eutectic structure should completely dissolve into the α-Mg matrix at temperatures > 370 °C. Additionally, the α-Mg matrix should also dissolve when the temperature during FSP exceeds the solidus temperature of AZ91 (~500 °C). This should result in an as-molded microstructure after subsequent cooling. As shown in Figure 3 and Figure 4, as-molded microstructure is not observed in the stirred zones. It is known that this zone is heated to temperatures between 370-500 °C during FSP [21]. Exposure to this temperature range by frictional heating results in a supersaturated solid solution of the α-Mg phase. Also, intense plastic strain at rapid rate is simultaneously introduced into the material. This results in consolidation, mixing and shearing of the intermetallic network during FSP. Thus frictional heating and intense plastic strain at rapid strain rates may cause dynamic recrystallization in FSW/P of Mg alloys [22]. Therefore, the stirred zone experiences some dynamic recrystallization in addition to the dissolution of the eutectic structure. The heating cycles from the subsequent overlapping FSP step may result in recovery and or grain growth in the stirred zone belonging to the previous step. Therefore, a careful selection of dwelling times and processing parameters is absolutely crucial in obtaining a uniform microstructure in the processed regions of the plate. In Figure 4(b), the transition zone is seen below or near the root of the FSP tool pin and is present between the recrystallized stirred zone and the HAZ. It shows a bimodal grain structure with the coarse island shaped primary solid grains, reminiscent of the as-molded microstructure, sub-divided in to several small units. The higher magnification image of the selected area in inset (Figure 4 (c)), shows a large primary α-Mg grain, completely consumed by the smaller grains. Necklace structure, typical of dynamic recrystallization and recovery processes is also observed. The morphology and distribution of eutectic β ($Mg_{17}Al_{12}$) network is significantly altered. It has been fragmented into dispersoids that intermittently exists in the form of a much finer film. Some twinning is also observed in the sub-divided, primary α-Mg grains. This unique combination of layered structure and with UFG grain structure in stirred zone and two phase grain structure in the HAZ is likely to affect strength and ductility. A TEM analysis to understand the size, distribution and orientation of second phase particles and precipitates after FSP and aging will also be the subject of a later study.

Mechanical Property Improvement

The variation in Vicker's hardness measured along the sheet thickness for the FSP and aged conditions is presented in Figure 5(a). The regions corresponding to the stirred zone exhibit the highest hardness while the un-processed heat affected core region retains the hardness of the base material. Over-all there is a slight, through the thickness variation in hardness and it corresponds well with the corresponding grain sizes and phase distributions. Conventionally, AZ91 alloy processed by gravity or low-pressure casting techniques is subjected to a T6 heat treatment. This treatment involves two separate steps, solution treatment at (430 °C for 16–24 hrs) and subsequent aging at 168 °C for 16 hrs or alternatively 216 °C for 6 hrs [23]. In this work, peak hardness (stirred zone) is achieved by aging alone, without a solutioninzing treatment because FSP results in a super-saturated solid solution of the α-Mg phase. Also, aging is done at a much lower temperature of 150°C as compared to the relatively higher aging temperature typically used for this alloy. Figure 5(b). Values of Yield Strength (YS), Ultimate Tensile Strength (UTS), percent elongation and fracture-strain are summarized in Table I. In FSP condition, the material exhibits highest strength; accompanied with some improvement in tensile ductility. However, after suitable low temperature aging treatment, further improvement in strength (>300 MPa) with a combination of good ductility (>7 %) is observed. The UTS of the FSP processed material is approximately 1.5 times that of the as-molded condition and the UTS of the aged material is approximately 1.4 times that of the as-molded condition. On the other hand, the tensile elongation of the as-molded and FSP condition are similar but the aged alloy shows a significant improvement in ductility. This could be explained by the fact that the aging results in a lower dislocation density compared to the FSP condition. This can also explain the lower strength exhibited by the aged alloy as compared to the FSP material. The stress strain curves also show different strain hardening behavior for the three conditions. The FSP condition exhibits the highest strain hardening and the greatest UTS while the as- molded condition exhibits the lowest strain hardening and the lowest UTS.

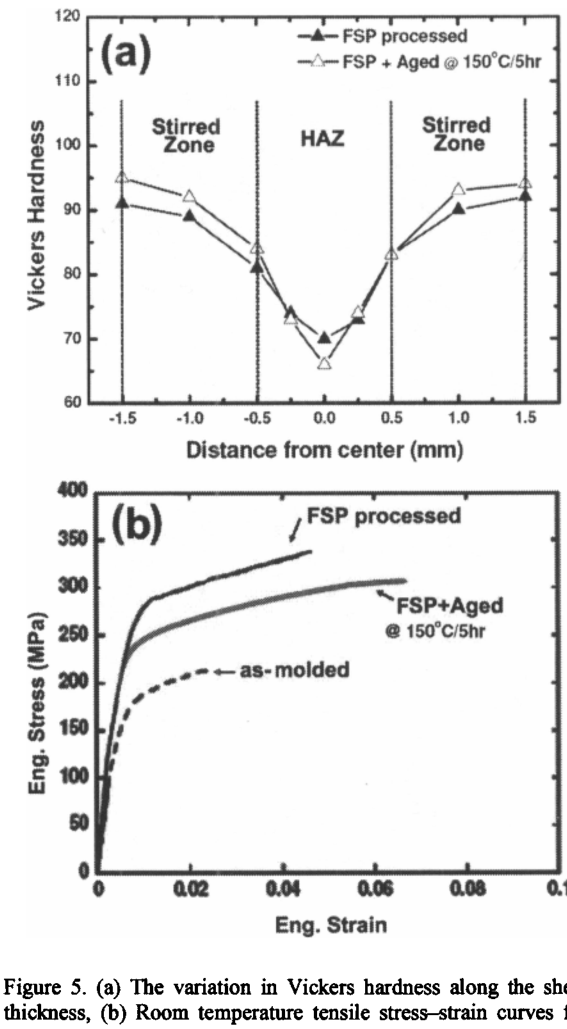

Figure 5. (a) The variation in Vickers hardness along the sheet thickness, (b) Room temperature tensile stress–strain curves for (a) as-molded, FSP processed and aged AZ91 Mg alloy.

Table I Mechanical Properties of thixomolded AZ91 Mg at different processing stages.

Condition	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation, %	Fracture Strain
Thixomolded	180	215	2.6	0.038
FSP	285	338	4.8	0.082
FSP + Aged	225	302	7.6	0.091

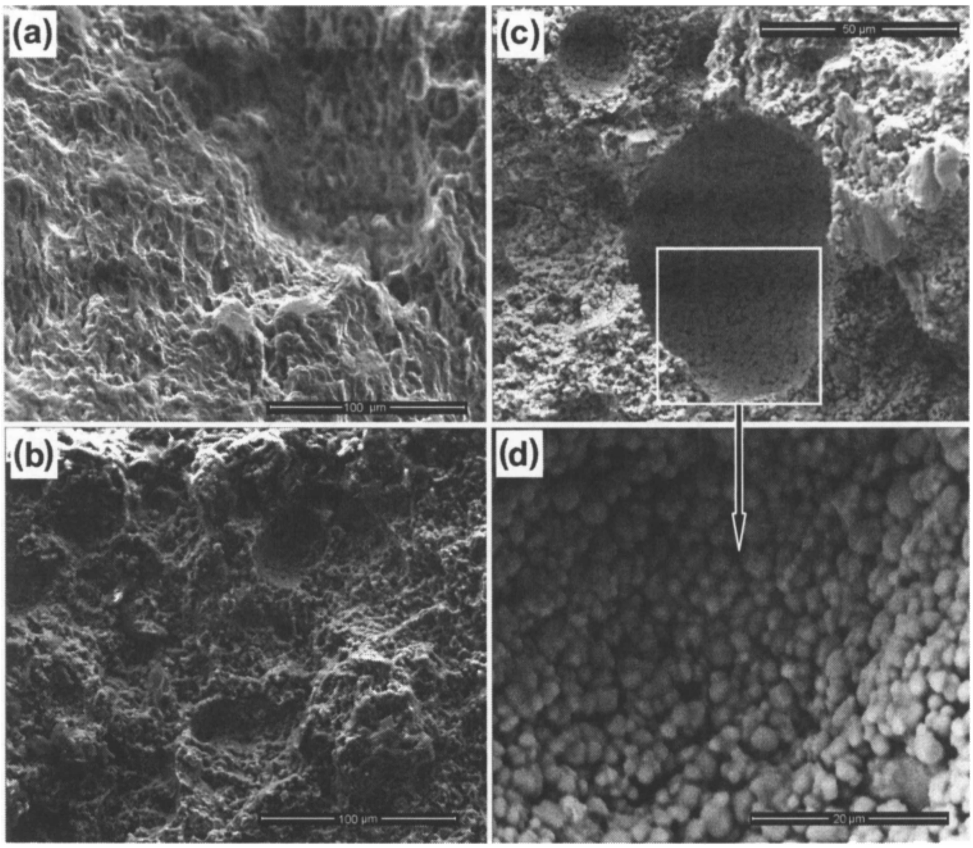

Figure 6. (a) SEM images show the fracture surface after tensile test for (a) as-molded condition (b) FSP processed and aged condition. The SEM images in (c) and (d) show the pull-out of a primary solid grain on the fracture surface observed in (b) at higher magnifications. This region of the sample corresponds to the un-processed HAZ present in the middle of the AZ91 plate.

It is well known that strain incompatibility between the (bcc) β-intermetallic phase and the (hcp) α-Mg phase leads to brittle interfaces in the as-molded condition [24]. This causes low ductility in the as-molded condition. The FSP processed material with UFG in stirred zones and bimodal microstructure in HAZ exhibits high strength. Improvement in ductility here is determined by reduction in the bcc/hcp brittle interfaces. This happens by complementing effects resulting from morphological changes and dissolution of eutectic films at α-Mg grain boundaries and the incorporation of eutectic rich material into the stirred zone. However, since the FSP material contains residual stresses and possibly some cold work, the over-all ductility is still low (< 5%). Further improvement in ductility is noticed after low temperature (150 °C) aging treatment. Aging/annealing causes recovery (relaxation of residual stresses by dislocation rearrangement), possibly some degree of recrystallization (and precipitation of fine nano-sized particles in the stirred zones [15]. It is nevertheless interesting that the 5 hrs aging treatment at a temperature much lower than recrystallization temperature of Mg (~200 °C) causes evolution of equiaxed microstructure in stirred zone (not shown here). These processes, in addition to Hall-Petch strengthening are most likely responsible for the improved mechanical behavior [25-27]. Figure 6 shows the fracture surfaces of tensile tested samples for as-molded and FSP processed and aged condition. The SEM images in (c) and (d) show several small recrystallized grains located in and around a crater possibly left by the pull-out of a primary solid particle on the fracture surface of the aged material. This region of the tensile tested sample corresponds to the heat affected core zone present in the middle of the AZ91 plate. Some precipitates are also visible in the

SEM images of the fracture surfaces. Ductile dimples were evident on the aged material (Figure 6(b)) but the fracture surface for the as-molded condition (Figure 6(a)) shows sharp and brittle characteristics.

Conclusions

Severe plastic deformation is imparted to a thixomolded AZ91 Mg plate by friction stir process. This creates a unique, layered microstructure with three distinct microstructural regions: (i) a stirred zone with homogeneous, ultrafine (~1 μm) microstructure; (ii) a transition zone with bimodal microstructure and partially dissolved, fragmented β network; and (iii) a HAZ, which largely remains unchanged but shows partial dissolution of α_p-Mg globules along with morphological changes in β network. After FSP processing, significant improvement in strength is observed but with some improvement in ductility. The super-saturated α-Mg phase stirred region by FSP eliminates the need for a high temperature solutioninzing treatment. Also, the aging treatment can be carried out at a much lower temperature of 150 °C without causing grain growth. The FSP processed and aged material exhibits superior mechanical properties with a good combination of strength and ductility as compared to the as-molded condition.

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