Consolidation of AZ31 magnesium chips using equal channel angular pressing

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Abstract. Equal channel angular pressing (ECAP) as a severe plastic deformation (SPD) technique was employed for consolidation of AZ31 magnesium chips. The chips were consolidated at a temperature of 300 °C following ECAP route A up to two passes. The optical and scanning electron micrographs reveal that consolidation have taken place and the quality of consolidation improves with number of ECAP passes and hence the total imposed strain. The Vickers hardness of the processed samples increased from 41 of the as cast used to generate the chips to 47.7 ad 58.5 of consolidated samples after one and two passes respectively. The consolidation of chips was accompanied by the breakage and dispersion of the oxide layer developed on the surface of the chips during and after machining.

Introduction

During the last two decades, magnesium (Mg) and its alloys have attracted great interests as structural materials for many engineering applications because of their high strength-to-weight ratio. However, frequent observations on the relatively low strength and ductility of these materials presents great concerns and have limits their applications. The consolidation or solid state recycling of chips is considered as a possible option to improve the mechanical properties of materials especially the lightweight materials like magnesium alloys. In the recent years many studies have been conducted to consolidate lightweight materials like magnesium, aluminum and titanium alloys. Mabuchi et al., [1] solid-state recycled (SSR) AZ91 Mg alloy using hot extrusion and were able to obtain a good combination of mechanical properties of the consolidated material. The solid-state recycling of Mg alloys using hot extrusion followed after the success of this study [2–8].

Wan et al [9] and Peng et al [10] suggested the consolidation mechanism of Mg alloys chips during solid-state recycling. Mg alloy chips are usually covered with a layer of oxide that may prevent the consolidation process. The oxide layers are broken down due to the large shear forces and high pressure at elevated temperature during the deformation process [11,12]. This break of the oxide layer helps meet the first requirements for the perfect bonding of chips, where the broken oxides particles are moved to the boundary between the recrystallized grains [89,101]. Therefore, direct contact between fresh metal surfaces, and interface bonding of chips can be accomplished. Cracks and pores are also expected to be distributed around the interface between chips [3,6]. However, the pores or voids can be reduced due to the plastic flow of the matrix under severe deformation and hence enhancing the compactness of the recycled Mg alloys [14]. Moreover, the physical consolidation may be fulfilled when the interfaces of the chips get dissolved because of the metallurgical bonding by atom diffusion under severe plastic deformation at elevated temperature [13]. The interfaces and crystal defects such as dislocation and grain boundaries caused by shear deformation act as diffusion paths of atoms [10,14]. Eventually, a fully dense material is obtained with uniform dissipation of oxide particles in the Mg alloy matrix.

The AZ31 alloy, as the most commonly used Mg alloys, has been solid-state recycled using hot extrusion [8], hot rolling [3], spark plasma sintering [15], and friction stir extrusion [16]. ECAP-based SSR method, to the best of the authors' knowledge has not been used to recycle Mg alloy chips before the current study. Wu et al. [8] found that double extrusion of AZ31 chips is necessary for the breakage and dispersion of the oxide particles into the structure and hence improving the consolidation of the recycled material. Similarly, Chino et al. [17] reported that high extrusion ratio of 1600:1 produced more uniform dispersion of oxide contaminant than lower extrusion ratio of 45:1. One of the advantages of ECAP over other SPD techniques is the possibility of continuous deformation without affecting the shape of the workpiece and hence accumulating imposed strain with more number of passes. This technique of solid-state recycling metals has been used before for solid-state recycle several titanium and aluminum alloys. In this study, ECAP is investigated as a means of consolidating the Mg AZ31 alloy chips into the bulk material.

Experimental Procedures

The raw material used in this investigation was cast AZ31 Mg alloy. The required AZ31 chips were generated using milling machine. Prior to production of the chips, all surfaces of AZ31 ingot have been machined to remove oxide layers. The milling process was carried out with a cutting tool speed of 900 rpm, a feed rate 75 (mm/min) and a depth of cut of 1 mm. No coolant or lubricant has been used to avoid any contamination with the chips. The produced chips have a curly shape with various sizes as shown in Figure 1.

Figure 1 *AZ31 Mg alloy chips generated by milling machine.*

Prior to ECAP process, chips were pre-compacted using a locally fabricated steel mold. The compaction process was carried out at room temperature using 100 MPa pressure. Compacted billets with an approximate diameter of 14.5 mm and a length of 80 mm were produced. The compacted AZ31 billets were then inserted into aluminum cans for the ECAP process. The extrusion process was carried out using locally designed and fabricated ECAP die with a 90° angle and a 24.5 mm by 24.5 mm channels' cross-section.

Optical and electron microscopy were carried out on the consolidated specimens extracted from the fully consolidated region of the processed workpiece. All measurements and investigations were carried out on the flow plane normal to the flow direction FD. Samples were cut from 1-A and 2-A specimens were prepared following the standard metallographic procedures. For optical microscopy (OM), the samples were etched using Acetic Glycol etchant solution which is composed of 20 mL acetic acid, 1ml HNO_3, 20 ml H_2O and 60 ml of ethylene glycol. The samples were immersed in the etching solution for 25-30 seconds. VHX 1000E Keyence digital microscope was used for optical microscopy (OM) investigation. The same metallographic procedures were followed to prepare samples for scanning electron microscopy (SEM) investigation. Back-scattered electron (BSE) and secondary electron (SE) micrographs of the samples were taken to

check the quality of the consolidation after 1 and 2 passes of route A. Elemental mapping of both samples was done using energy-dispersive X-ray spectroscopy (EDS) attached to the SEM.
Vickers hardness (HV) with a diamond indenter was carried out on the flow plane of the recycled AZ31 samples. The sample face was subjected to a load of 10 kgf by indenter having a shape of right pyramid with a square base and an angle of 136 degrees between opposite faces. Several hardness measurements were taken at different regions per sample to evaluate the homogeneity of the recycled materials.

Experimental Results and Discussion

The optical micrographs (OM) of the consolidated AZ31 alloy samples taken from flow plane after one pass (1-A) and two passes (2-A) are shown in Figure 2.a and 2.b, respectively. It is well known that Mg and its alloy are easily oxidized during milling even at room temperature and hence a thick oxide layer will be formed on the chips surface. The thick dark boundaries seen in the micrographs of the consolidated AZ31 alloy samples are oxide layers [18–21]. It is expected that the consolidated chips after experiencing simple shear deformation during severe plastic deformation ECAP process will be distorted and rearranged from random directions to specific inclination angle. This angle changes with the number of passes and processing route used. In the current study the chip boundaries in the 1-A sample are oriented along the principal strain direction with an inclination angle of about 24-27° from the extrusion direction (ED). In previous chips consolidation studies using ECAP, inclination angles of 21°[18]and 22.5° [20] from the ED were measured.

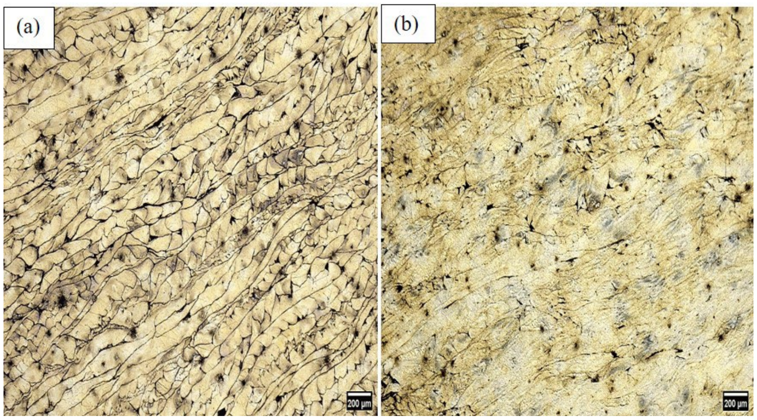

Figure 2 *Optical micrographs of consolidated AZ31 samples using ECAP at 300 C after (a) 1-A (one pass) and (b) 2-A (two passes). Extrusion direction (ED) is to the right.*

Figure 3 shows scanning electron micrographs (SEM micrographs) of 1-A and 2-A samples. These micrographs were taken from as polished samples without etching. The chips boundaries are visible as dark lines in 1-A sample as shown in Figure 3.a. These chips boundaries are clearly seen at high magnification SEM micrograph of 1-A (Figure 3.c). In contrast, 2-A sample shows less and thinner chip boundaries (see Figure 3.b. and 3.d) .This suggests that a higher number of passes may induce more breakdown and dispersion of chip boundaries, and hence achieve better consolidation between the chips. Similar chips boundaries appearance of SSR Mg alloy have also been reported in earlier studies utilizing different consolidation techniques like hot extrusion [6,22–24] and cyclic extrusion compression(CEC) [10,25].

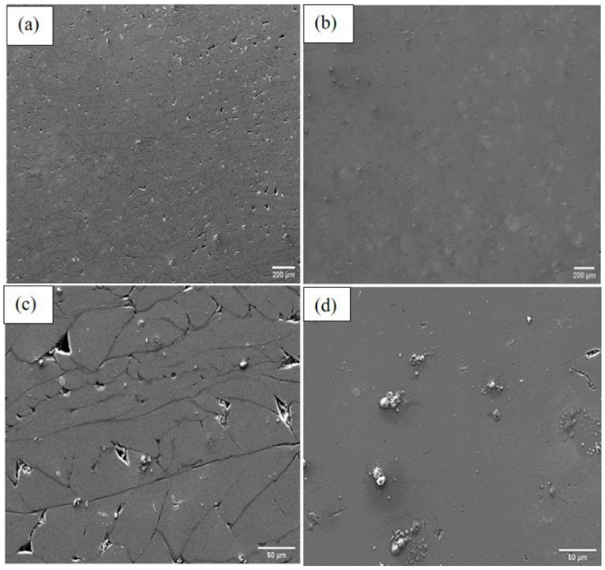

Figure 3 *SEM micrographs of 1-A and 2-A samples: (a) low magnification 1-A, (b) low magnification image 2-A, (c) high magnification 1-A and (d) high magnification 2-A.*

Figure 4 shows the elemental mapping of oxygen measured using EDS mounted in SEM. The mapping reveals more clearly the difference in oxide distribution between 1-A and 2-A samples. The oxide is concentrated at the chips surface and formed boundaries between the chips. After one pass shear deformation (1-A) of ECAP as shown in figure 4.a the oxide is still concentrated at the chips surfaces and formed boundaries between them. Oxide layers work as a barrier to fulfil the requirement of consolidation between the chips. In contrast to 1-A sample, it is clearly observed that most of the oxide layers between the chips are no longer visible in 2-A sample. The accumulation of shear deformation after two ECAP passes is capable of breaking up oxide layers into particles distributed in the structure of the recycled 2-A sample as shown in Figure 4..b. The destruction of oxide layers allows stronger bonding between chips.

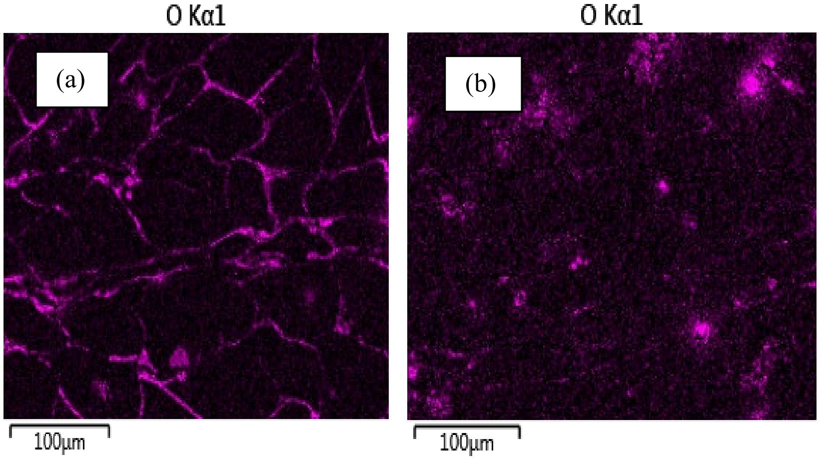

Figure 4. *EDS elemental mapping of oxygen in (a) 1-A (b) 2-A samples.*

In order to evaluate the mechanical properties of the recycled materials, hardness measurements were carried out on the flow plane of the recycled samples. The Vickers hardness (HV) of recycled material after one pass 1-A, two passes 2-A and as cast AZ31 were measured. Table 1 lists the HV values of 1-A, 2-A and as cast AZ31 samples taken from different locations. For easy comparison, Figure 5 shows the average HV value of recycled material 1-A, 2-A and as cast AZ31 samples. It can be clearly noticed that the hardness of the SSR materials is higher than the as cast material used to generate the chips. The average hardness values increased from 41± 1HV for as cast to 47.7±1.15 HV (~15% increase) after the first ECAP pass and further increased to 58.5±1.4 HV after the second pass about (~23% increase).

***Table 1** Vickers hardness values of as-cast and ECAP SSR AZ31 samples after one and two passes.*

Location	**Cast AZ31 (0 pass) [HV]**	**1-A (One pass) [HV]**	**2-A (Two passes) [HV]**
1	41.5	44	61.1
2	40.4	49.5	61.3
3	38.5	49.8	56.4
4	42.8	50.3	60.4
5	43.2	47.3	55.4
6	39.7	45.7	56.7
Average	**41.0**	**47.7**	**58.5**

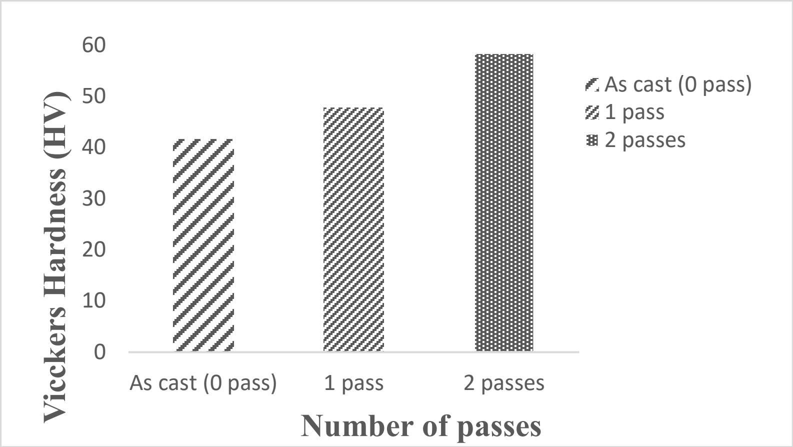

***Figure 5** The average values of Vickers hardness of as cast and ECAP SSR AZ31 samples.*

Conclusions

- Machining chips of AZ31 have been successfully consolidated at moderate a temperature below melting temperature using equal channel angular pressing (ECAP).
- The consolidation quality is significantly improved with higher number of passes, wherein the accumulation of shear deformation leads to easier destruction of chip boundaries (oxide layers), dissolve the interfaces between chips and hence better solid bonding occurred.
- Oxide contaminants were dispersed more uniformly in the recycled AZ31 material after multiple passes ECAP.
- The hardness of recycled AZ31 alloy is higher than that of the starting as cast AZ31 and it is further increased with increasing number of passes as a result of the greatly improved physical contact of chips and more uniform dispersion of the fine oxide particles.

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