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Microstructure and Corrosion Behaviour of Friction Stir Processed AZ91 Mg Alloy

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Abstract— ***Magnesium (Mg) and its alloys are gaining wide popularity as promising candidates for automobile, aerospace and structural applications. On the other hand, friction stir processing (FSP) is a solid state technique used to alter the microstructural characteristics of a metal. In the present study, AZ91 Mg alloy was processed by FSP at optimized process parameters. The microstructural changes were analyzed and found that the presence of secondary phase ($Mg_{17}Al_{12}$) was reduced. The network like structure of secondary phase that was appeared at the grain boundary before FSP has disappeared after the processing. Grain size was also found to be reduced from (66.5 ± 8.7 µm) to (4.1± 1.6 µm). The corrosion behavior was assessed by immersion test in 3.5%NaCl solution and found that the processed AZ91 has lower corrosion rated during the early hours of corrosion attack. However, after 72 h of immersion the corrosion resistance of the processed AZ91 was found to be reduced. Hence from the present study it can be understood that the presence of secondary phase has a great influence on the corrosion behavior of AZ91 mg alloy and that can be altered by grain refinement.***

Keywords— FSP, AZ91 Mg alloy, secondary phase, corrosion, grain refinement.

I. Introduction

Magnesium (Mg) and its alloys are attracting a considerable attention in the automobile and aerospace industry for light weight structural applications. Lower density compared with aluminium, high specific strength and excellent damping properties make magnesium as a promising choice among many other non-ferrous metals. [1]. However, the poor corrosion resistance of magnesium is a limitation which must be considered as a valid factor for designing the structures intended to use in corroding environments [2]. AZ series (Aluminum and zinc) Mg alloys are the widely known and commercially available materials in the families of Mg alloys. Mg alloy with 9% Al and 1% Zn (AZ91) is one of the compositions suitable for various applications. Usually, AZ91 Mg alloy is produced by die casting method and exhibits excellent damping properties. The presence of more secondary phase ($Mg_{17}Al_{12}$) at the grain boundaries significantly influences the mechanical and corrosion properties [3]. It was shown that the corrosion resistance was increased with the grain refinement [4-12] and also decreased in some studies [10-12, 15] of Mg alloys. Recently, friction stir processing (FSP), a solid state method developed from the friction stir welding (FSW) has shown tremendous effect on altering the mechanical and corrosion properties by modifying the microstructure of the metals. In FSP, a rotating non consumable rotating tool consisting of a shoulder and a pin is inserted and plunged through the work piece and the microstructure of metallic sheets or plates can be modified. The mechanism behind grain refinement during FSP can be seen elsewhere [16]. Improved corrosion resistance for FSPed Mg alloys was reported in the literature [5-8]. However, the information regarding the effect of FSP on the secondary phase in Mg alloys and further on electrochemical behavior is insufficient. Therefore, in the present study, AZ91 Mg alloy was processed by FSP and the effect of evolved microstructure on the corrosion behavior was investigated. The mechanism behind the corrosion behavior after FSP is discussed.

II. Material and Methodology

Die cast AZ91 Mg alloy (8.67%Al, 0.85%Zn, 0.002%Fe, 0.03%Mn and remaining being Mg by wt.%) ingot was obtained from Exclusive Magnesium, Hyderabad, India. Work pieces of size $100 \times 100 \times 4$ mm^3 were cut from the received ingot and friction stir processing (FSP) was carried out using an automated universal milling machine (Bharat Fritz Werner Ltd., India). FSP tool was made of H13 tool steel with a shoulder diameter of 20 mm. FSP tool has a tapered pin at the end of the shoulder with root diameter of 3mm and end diameter of 1 mm with a length of 3 mm. A few trial experiments were conducted and defect free processed zone was obtained at optimized process parameters. Then the FSP was carried out with a tool travel speed of 25 mm/min at a tool rotational speed of 1400 rpm. The penetration depth (3 mm) was given such a way that the tool shoulder touches the work piece surface. The processed AZ91 Mg alloy was coded as FSPed A931.

Samples of size 10×10×4 mm^3 were cut from the nugget zone of FSPed AZ91 and unprocessed AZ91 sheets. For metallographic observations, all the samples were polished with different graded emery sheets up to 2000 grade and followed by alumina and diamond paste (1-3 µm) polishing. The polished specimens were then cleaned using ethanol and dried in hot air. Etching agent comprised of 5 g of picric acid, 5 ml acetic acid, 5 ml distilled water and 100 ml ethanol was prepared and chemcial etching was done by immersing the polished samples for 20 sec. Then the etched samples were gently cleaned in de-ionized water, dried and then optical microscope (Leica, Germany) images were obtained at different regions.

The samples of same size were immersed in 3.5% NaCl solution for 24, 48 and 72 h and the corrosion rate of the samples was calculated. Lab grade NaCl (Merc, India) was used to prepare 3.5% NaCl solution using de-ionized water. Each sample was immersed in 50 ml solution such a way that the ratio of the surface area of the sample to the volume of the solution was maintained as more than 1:10. The containers were kept at the room temperature. After different intervals of time, the samples were taken from the NaCl solution and gently rinsed in stable de-ionized water and dried. A boiling solution of chromic acid (180 g / one liter of de-ionized water) was prepared to remove the corrosion products. After the immersion test, the samples were immersed in the boiling solution of chromic acid and remained until the corrosion products were completely dissolved. The samples were then dried in open air and weight measurements were obtained. Corrosion rate was calculated according to ASTM standard NACE TM0169/ G31 – 12a as given below [17].

Corrosion rate (mm/year) = $k * \Delta W / (A * T * D)$ -- (1)

where, $k = 8.76x10^4$, T = time of exposure in hours, A = area of the specimen in cm^2, ΔW = weight loss in g , D = density in g/cm^3.

III. Results and Discussion

Fig 1 shows photographs of friction stir processed (FSPed) AZ91 sheets with defects and without defects obtained during the optimization of the process parameters. AZ91 Mg alloy is brittle and difficult to process material. The material plastic flow of AZ91 during FSP is also complex as it exhibits brittle nature. The tool rotational speed and traverse speed are the two important processing parameters which influence the heat generation during FSP. Insufficient material flow has produced defects as shown in Fig 1(a) which is due to insufficient heat generation during FSP at 1400 rpm tool rotational speed and 25mm/min tool travel speed. As the tool travel speed is decreased to 20 mm/min, the material flow was observed as defect free as shown in Fig 1 (b).

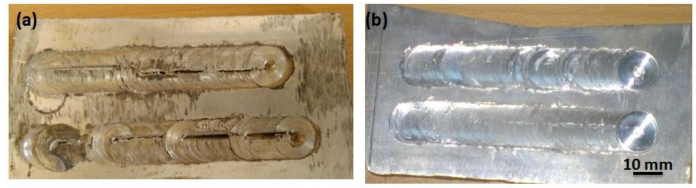

Figure 1 Photographs of FSPed AZ31 sheets: (a) 1400 rpm tool rotational speed and 25 mm/ min tool feed and (b) 1400 rpm tool rotational speed and 20 mm/ min tool feed.

Fig 2 shows the optical microscope images of the samples. It is clear from the observations that the FSP led to grain refinement. Before FSP, AZ91 has considerable amount of secondary phase (β-phase) as indicated with thick arrow in Fig 2 (a). The β-phase was appeared at the α-grain boundaries. Interestingly after FSP, the presence of β-phase was completely disappeared. The α-grains became rich in aluminium and turned into super saturated solid solution. The grain size in the nugget zone of FSPed AZ91 has found to be refined to (4.1± 1.6 µm) compared with the starting grain size (66.5 ± 8.7 µm). During FSP, the material flow is complex in nature that includes different regions such as preheat zone, extrusion zone, forging zone and post heat zones [18]. As the tool moves over the work piece, excessive material flow causes localized extrusion around the tool pin. As reported in the literature, dynamic recrystallization is the prime mechanism behind the grain refinement during FSP.

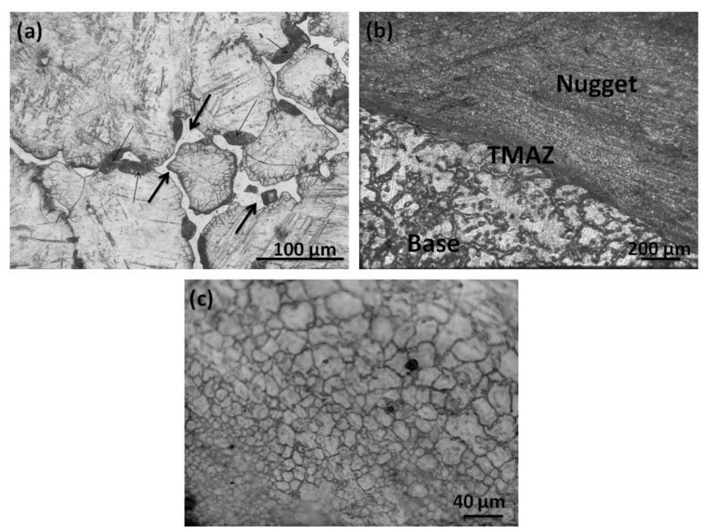

Figure 2 Optical microscope images of the samples: a) unprocessed AZ91, b) FSPed AZ91 and c) magnified image obtained at the nugget zone of FSPed AZ91

Fig 3 shows weight loss measurements for the samples after 1, 2 and 3 days. The weight loss for AZ91 was higher compared with FSPed AZ91 during the initial hours and interestingly found to be reduced after 72 h of immersion. The decreased corrosion rate after FSP can be related with the presence of secondary phase (β-phase) and the reduced grain size. In Mg alloys, grain boundary behaves as cathode and grain interior acts as anode during the electrochemical events when exposed to any corroding environment. Presence of secondary (β) phase influences the electrochemical events as the (β) phase acts as cathode and therefore the corrosion mechanisms are severely affected. The role of β phase is prominent in AZ91 Mg alloy. Due to the presence of active cathode and anodic sites within the AZ91 Mg alloy, galvanic couple is established and a quick localized corrosion is initiated. [7,8]. Bobby Kannan et al., [5] reported a slight increase in the corrosion resistance for FSPed AZ31 Mg alloy and suggested that the β phase ($Mg_{17}Al_{12}$) dissolution into the α-grains decrease the galvanic corrosion and enhanced the corrosion resistance. Similarly, in the present study after FSP, the fraction of β phase ($Mg_{17}Al_{12}$) was reduced to a great extent as observed from the microstructural studies (Fig 2). Therefore, the super saturated α-grains in FSPed AZ91 have shown higher corrosion resistance compared with unprocessed AZ91. Surprisingly, The corrosion rate of the FSPed AZ91 (3 ± 1.9 mm/year) was found to be increased compared with AZ91 (1.8 ± 0.3 mm/year) as the immersion time was increased to 72 h. This interesting behavior can be related with the effect of the corrosion products which are formed on the surface of Mg alloys when immersed in chloride containing solutions.

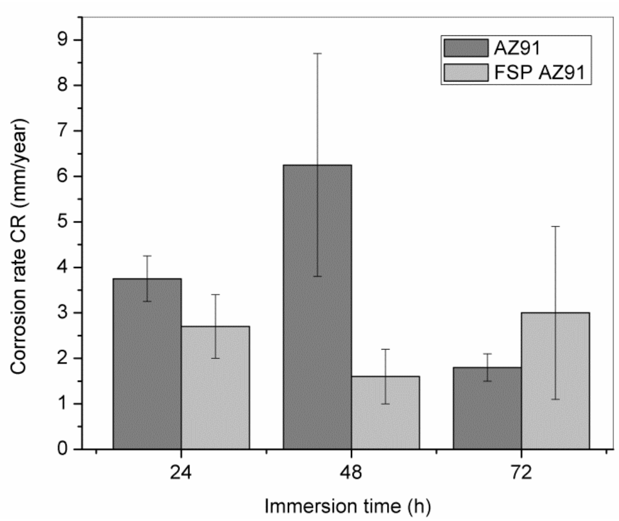

Figure 3 corrosion rates of the samples calculated from the immersion tests.

Magnesium hydroxide and magnesium oxide are the two important corrosion products usually formed on mg alloys. These phases are semi-protecting ceramic phases which do not withstand for longer periods in the presence of chloride ions. Therefore, the protection offered by these layers on the surface is insufficient and further corrosion is advanced if the material is under the attack of the chloride ions. However, in the present study, FSPAZ91 has lower amounts of the corrosion products compared with AZ91 during early hours of immersion and which has offered lower protection as the immersion time increased to 72 h. Hence from the present study, it can be understood that FSP can be used to produce fine grained super saturated AZ91 alloy which increase the corrosion resistance particularly during the early stages of the corrosion attack.

IV. Conclusion

In the present study, AZ91 Mg alloy was successfully processed by friction stir processing and a grain refinement from 66.5 ± 8.7 µm to 4.1± 1.6 µm was achieved. From the microstructural observations, it was found that the β phase ($Mg_{17}Al_{12}$) phase was completely dissolved and super saturated AZ91 Mg alloy was produced. Corrosion behavior assessed by immersion tests carried out in 3.5%NaCl solution has clearly shown the influence of secondary phase on corrosion behavior of FSPed AZ91. The increased corrosion resistance for FSPed AZ91 can be attributed to the smaller grain size and the negligible presence of the secondary phase. Hence, it can be concluded that corrosion resistance of AZ91 can be improved by FSP by developing super saturated fine grains

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