

**INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH
TECHNOLOGY****FABRICATION OF MG-FE COMPOSITE BY FRICTION STIR PROCESSING****Kavya H. M^{*1}, B. Yogesha² & Ranjit Bauri³**^{*1}Department of Mechanical Engineering, B.N.M. Institute of Technology Bangalore, Karnataka, India.²Department of Mechanical Engineering, Malnad College of Engineering, Hassan, Karnataka. India.³Department of Metallurgical and Materials Engineering, Indian Institute of Technology Madras, Chennai, Tamil Nadu, India**ABSTRACT**

In the present investigation, a commercially pure magnesium is chosen and carried out single pass FSP for grain refinement and also to process iron particulate reinforced magnesium matrix composite. Metal particle reinforced composites can provide beneficial effects on both strength and ductility. Due to the biodegradable nature of both Mg and Fe, a combination of the both the metals in one material as a composite can be explored as a new biodegradable material. The objectives of the present investigation are to develop a fine grained magnesium by FSP with homogeneous microstructure, to study and understand the grain refinement during FSP of Mg, and to develop iron particulate reinforced magnesium matrix composite without formation of deleterious intermetallic.

KEYWORDS: Friction stir, fabrication, composite, XRD, SEM.**INTRODUCTION**

One of the popular techniques to increase ductility and strength is via grain refinement of polycrystalline materials. Currently, there is growing demand to reduce the weight of components in the automobile and aerospace industries. Hence many groups are working towards development of high strength and ductility materials to cater to such demands. In this context, fine grained and ultrafine grained materials are being explored, given their improved mechanical properties. Crucially, materials can withstand plastic strains, mechanical damage if developed with a narrow grain size distribution and high fraction of high angle boundaries.

Magnesium alloys are potential candidate materials for applications as structural materials in automotive and aircraft industry, owing to their strength-to-weight ratio. With one quarter of the density of steel and only two thirds that of aluminum, and strength to weight ratio that is exceeding better than both, Magnesium and its alloys fall into the light weight category. Components in hand phone, CVD/DVD chassis, computer disk drives and camera casings are increasing being made up of Magnesium based materials. Another industry where the light weight is beneficial is the transport sector, where this reduction in weight leads to direct economic benefit.[Ambat, 1999].

Friction stir processing (FSP) is an emerging technique, wherein a thermo mechanical treatment involving severe plastic deformation at high homologous temperatures are used. This results in the formation of ultra- fine grains, a homogenous microstructure, removal of casting defects and the ability to make a wide range of composites (Mishra and Ma, 2005). More importantly, even after one pass, FSP has the capability to cause major grain refinement. The underlying mechanisms during such a grain refinement during FSP are not completely understood. In addition, FSP is an effective technique for processing metal matrix composites (MMCs). But such a processing of metal particulate reinforced MMCs by FSP has not been investigated well in the literature. Moreover, literature suggests that Friction stir processed MMCs offer much higher ductility when compared to their conventional counterparts.

A factor that limits the usage of Magnesium is its poor corrosion resistance. Magnesium, being a highly active metal, it is difficult to hamper the corrosion and any techniques to lower the dissolution rate will be a huge boost

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to the field. Additions of Rare Earths to the Magnesium matrix have been effective in improving the corrosion resistance. Coating is another alternate route to avoid corrosion. Despite this, the high corrosion rate of Mg has been used as an advantage in its application as a self- biodegradable material in temporary implants [Li, 2013]. Iron has also emerged as another biodegradable metal in recent times. Therefore, a combination of Mg and Fe in one material can be explored as a new biodegradable material. Grain refinement has shown to improve corrosion and surface adhesion property of Mg.

In the present investigation, fine grained magnesium and magnesium-iron composite were developed by friction stir processing. Given the limited understanding of the technique in literature, the microstructure evolution during FSP was investigated in detail. FSP is a simple and one step process for grain refinement. It is expected that incorporation of iron into magnesium does not affect the mechanism of grain refinement.

Corrosion resistance of processed Mg and Mg-Fe composite was evaluated using potentiodynamic polarization (PDP) tests in 3.5 wt% NaCl solution. The effect of grain refinement and presence of Fe particles on the corrosion property was evaluated.

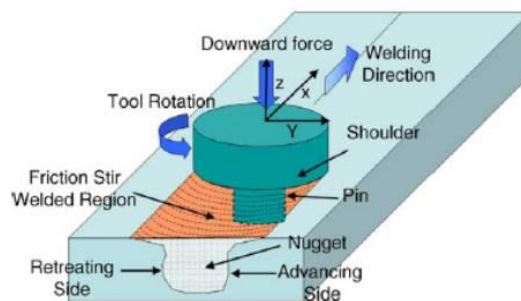


Fig.1: Schematic of Friction Stir Processing [Sattari, 2012]

EXPERIMENTAL DETAILS

Mg Melting

For melting Mg, a bottom pouring type stir Mg/Al casting furnace was used. The furnace was then pre-heated to 720°C with the stirring speed set at 300-400 rpm. The rectangular shaped mold that is to be used for the casting experiment was heated to 500°C in the oven for 2 hours. During the pouring process, the melt temperature was maintained 720°C. Argon gas was continuously passed while the melt was being poured to avoid oxidation and porosity.

Friction stir processing of Mg-Fe composite

To fabricate the Mg-Fe composite, a groove of width, depth and length of 1mm, 2mm and 8mm respectively was made on the cast Mg plate and filled with Iron powder (200 Mesh) of 99% pure (Alfa Acer). A threaded tool of shoulder diameter, pin diameter and pin length of 15mm, 5mm and 3.5mm respectively was used to incorporate the iron particles into magnesium. A tool, made of Inconel 718, was used for FSP at a rotation speed of 1200 rpm and a traverse speed of 60mm/min with a downward force of 8 KN. The Fig.1 shows Friction Stir Processing Setup.



Fig. 1: Friction Stir Processing Setup used in this study

Etching

In order to reveal the grain boundaries for grain size measurements, etching of base sample had been carried out using a mixture of 1ml nitric acid, 3ml acetic acid, 12ml ethanol and 4ml water etchant. The sample was immersed for 20 seconds in this solution. After immersing, the sample was washed and immersed in ethanol till grains became visible under the Optical Microscope.

Characterization

X- Ray Diffraction

X-ray diffraction (XRD) experiments were conducted using the X'Pert Pro (PANalytical). X-ray Diffractometer in Bragg-Brentano geometry equipped with X'Celerator detector with Cu-K α radiation in the 30°- 90° 2 θ range with a step size of 0.15 and a time per step of 10s.

Scanning Electron Microscope

Samples were metallographically polished and examined in FEI Quanta 200 Scanning Electron Microscope equipped with BSE and EDX detectors and the Field Emission Gun attached Quanta 400 equipment for high resolution imaging.

RESULT & DISCUSSION

Optimization and Characterization of rolled Mg plate

Initially FSP was carried out on a rolled Mg plate to optimize the parameters for pure Mg. Then these parameters were used a benchmark to optimize the parameters during FSP of Mg plates cast in the lab. All further studies were carried out on the cast Mg plates. A narrow window of processing parameters has been identified for defect free microstructure. The rotational and traverse speeds seem to play a crucial role in obtaining sound FSP microstructure.

Optical Microscopy

The optical micrographs have revealed that FSP has resulted in the formation of new strain free recrystallized grains which can improve mechanical and corrosion properties as observed in Fig. 2.

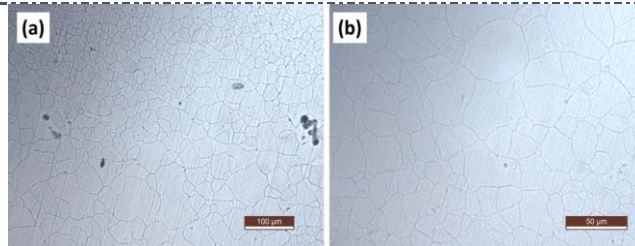


Fig. 2: (a) Optical Micrograph showing the base (rolled plate) region with the fine grained FSP region and (b) magnified view of the fine grained region.

XRD analysis

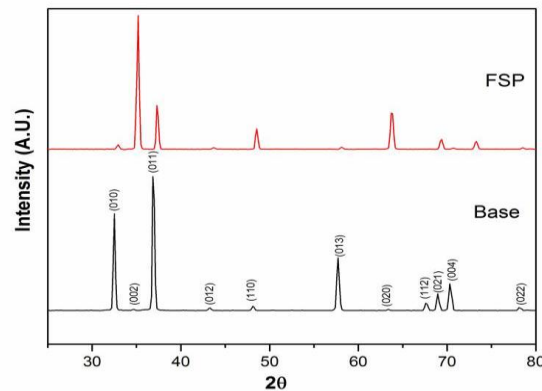


Fig. 3: Comparative XRD patterns of the Base and FSP'ed Mg

In the base sample the most intense peak is the (011) peak at 36.93°. In the case of FSP'ed sample the most intense peaks changes to the (002) peak at 35.16° as shown in Fig.3. The change of the most intense peak could be a result of FSP induced texture. No other secondary phases were found to form.

Optimization and characterization of cast Mg plate

The same optimized parameters of rolled Mg have been used for the FSP of cast Mg plate.

Optical Microscopy

For the remaining part of the study, cast Mg plate was used. Fig. 4 shows the optical micrograph of such cast magnesium. The average grain size was estimated to be $534 \pm 238 \mu\text{m}$.

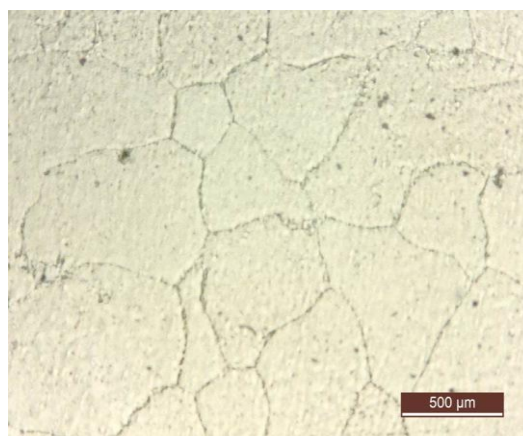


Fig. 4 Optical Micrograph showing the cast Mg

SEM study

SEM studies of the polished surface indicated that there is no sign of any intermetallic formation in the Mg matrix of the Mg-Fe composite. Iron was found to be present in elemental form, clearly indicating that iron had not dissolved in the matrix. The temperature of the stir zone during FSP depends on the ratio of tool rotation speed to the traverse speed.

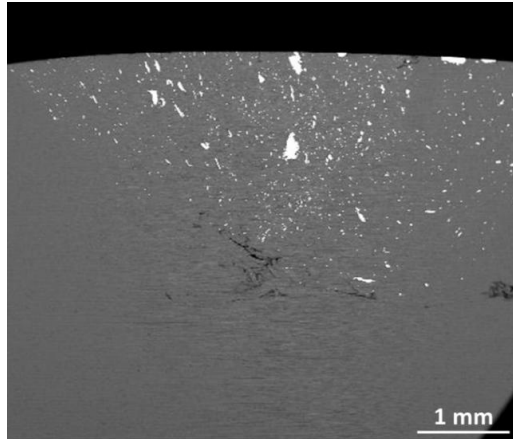


Fig. 5: SEM image of the nugget formed after FSP of the Mg-Fe composite

As no intermetallic was found in the matrix of the composite, it is believed that the ratio of tool rotation speed to traverse speed was not high enough to raise the temperature of the stir zone to the level required to initiate magnesium iron reaction. Formation of any intermetallic will act as source of fracture initiation and failure since intermetallic have lower strain to failure and considerably reduces the toughness of the composite. However, it is generally believed that a very thin reaction layer at the particle-matrix interface can improve the interfacial bonding between Mg and Fe. However, it should be noted that such a low amount cannot be detected by XRD.

Nugget formation was clearly observed from the SEM micrograph (Fig. 5). FSP resulted in incorporation of the Fe particles into the Mg matrix with uniform distribution of Fe particles and good interfacial bonding. The SEM micrograph in Fig. 5 shows the distribution of Fe particles on the surface of stir zone.

CONCLUSION

From the detailed investigations of the grain refinement of Mg, processing of Mg-Fe composite, and microstructure development. the following conclusions can be drawn.

Grain refinement of Mg

FSP was found to be an effective technique for grain refinement. A single pass of FSP resulted in significant grain refinement in Mg. Starting with $534 \pm 238 \mu\text{m}$ grain size, a single pass of FSP resulted in dynamically recrystallized, equiaxed fine grains with average grain size of $9 \mu\text{m}$ throughout the stir zone. FSP gave a narrow grain size distribution with a high fraction (>80%) of high angle grain boundaries, which is higher than that achieved by other thermo-mechanical processes. Presence of grains with different orientations indicates possible development of texture which was also indicated by the XRD patterns.

Processing of Mg-Fe composite

FSP resulted in incorporation of Fe into Mg matrix to form composite and significant grain refinement of the Mg matrix. Fe particles were uniformly distributed with good bonding with the matrix. There was no sign of harmful Fe-Mg intermetallics in the matrix. The matrix is characterized by fine grains with narrow grain size distribution and high fraction of high angle boundaries. Low angled grain boundaries are also formed by dislocation rearrangement into low energy configuration due to dynamic recovery.

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