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## MAGNESIUM BASED COMPOSITE VIA FRICTION STIR PROCESSING

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#### Abstract

Friction stir processing (FSP) was used to incorporate 6 vol.% of micron sized  $B_4C$  (6µm) reinforcements in WE43 alloy to form a surface composite. Multiple passes were utilized in order to achieve homogenization. Better distribution of the reinforcements, grain size refinement and an increase in hardness was observed after every FSP pass. The composite was 4-6 mm thick and showed higher values of hardness and modulus than that of the base material. Post FSP aging was performed at 210 °C for 48 h. The tensile properties were evaluated at room temperature for samples in as-FSP and after FSP+aging. The yield strength remained unaffected by the reinforcement addition.

#### Introduction

Composites have increasingly gained technological importance because they provide the opportunity to fabricate materials by a combination of a large variety of reinforcements and matrices. These result in achievement of hybrid properties in a single material which are otherwise not achievable in a conventional material. Although, the origin of metal matrix composites (MMCs) dates long back in history, it was not until the 1920s that dispersion strengthened alloys attracted scientific attention as the first composite materials [1]. Next came interest in continuously reinforced fiber composites in the 1960s, which displayed positive results but were discontinued owing to the high costs associated with the reinforcements and production [2]. This was followed by the era of discontinuously reinforced composites in which the costs of the reinforcements became much more competitive. It also permitted higher volume fractions of the reinforcements than that of a dispersion strengthened systems resulting in greater tailorability. As a result, discontinuously reinforced MMCs have recently attracted considerable attention in the automotive and aviation industries due to their remarkable properties and marked development in various processing techniques for MMCs [3, 4]. Today, composites have led to substantial advancements in various spheres of mechanical properties like strength, stiffness, wear resistance, creep resistance, fatigue resistance, etc. This has been possible due to marked improvements in various processing techniques. Various liquid phase or solid state processes exist for the fabrication of discontinuously reinforced composites. Though liquid phase processes have been quite successful in fabricating composites, there are still a few shortcomings associated with them [5, 6]. The liquid phase processes involve melting of the materials which can lead to formation of detrimental phases, segregation of second phase particulates, presence of a large number of defects, or difficulties associated with the wetting of the second phase particulates. These shortcomings may be overcome to a considerable extent by the use of solid state processes to manufacture composites [4, 7].

Friction stir processing (FSP), a solid state process developed by Mishra et.al [8] based on the principles of friction stir welding (FSW) is getting increased attention. It involves the traverse of a high strength rotating tool to locally heat the work piece and produce intense plastic deformation. The interplay between temperature and strain leads to a fine grained recrystallized grain structure. FSP is also stated as a 'green' process and consumes much less time, energy and money as compared to other thermomechanical processes [9]. Mishra et al. [10] have used FSP to fabricate a surface composite fabrication in aluminum alloys with various reinforcements, like Al<sub>3</sub>Ti, Al<sub>2</sub>Cu, Al<sub>2</sub>O<sub>3</sub> etc. via FSP [11,12,13]. Although aluminum forms an important part of lightweight materials, increasing demand for weight reduction has expanded the interest of the automotive and aircraft industries to magnesium alloys as well. Efforts have been made to fabricate various Mg based MMCs [14,15,16] with various ceramic reinforcements through processes like disintegrated melt deposition, powder metallurgy, and casting.

Fabrication of various composites can have different objectives. The goal for the addition of reinforcement to magnesium is to increase the stiffness so that light weighting of the structure can be done effectively. Significant focus has recently been given to the precipitation strengthened Mg-RE alloys, especially the Mg-Nd-Y system, owing to their enhanced mechanical properties along with improved castability, creep resistance and corrosion resistance. However, there are no reports of composites with WE43 as a matrix. B<sub>4</sub>C on the other hand is the lightest ceramic reinforcement with the highest elastic modulus. Hence, an attempt was made in this study to merge the structurally efficient properties of WE43 alloys with high stiffness of  $B_4C$  through FSP.

#### **Experimental Procedure**

#### Processing

A commercial hot rolled F temper WE43 (30 cm x 5 cm x 1.6 cm) plate and commercial purity  $B_4C$  powder (6 µm) were used in this study. FSP was carried out with a stepped spiral conical tool with a featureless shoulder and a pin length of 6.5 mm, which was made of H13 tool steel. A set of holes with a depth of about 6 mm were drilled into the plate in the pattern shown in Fig.1 (a) and the  $B_4C$  powder was then filled into these holes. The FSP was carried out by employing one capping pass and four overlapping passes. The capping pass was performed with a tool with no pin at a tool rotation speed of 600 rpm, traverse speed of 101.6 mm/min and a tilt angle of 2.5°. The capping pass was performed to close the holes and thus reduce the loss of reinforcement. There was a complete overlap between all the overlapping passes. All the overlapping passes were performed with the same process parameters – a tool rotation rate of 1100 rpm, a traverse speed of 25.4 mm/min and with the tool tilt angle of 2.5°, but each subsequent pass was in opposite direction to ensure higher mixing of particulates. The plate was allowed to cool to room temperature after every pass.



Fig. 1. (a) Schematic showing the FSP process, plate dimensions, pattern of the drilled holes, location of samples for characterization and (b) the tool used for processing.

## Microscopy and Mini-tensile testing

The FSP plate was cut in the transverse direction, ground and polished for optical microscopy and scanning electron microscopy. Mini tensile samples with gage dimensions (2 mm x 1 mm x 1 mm) were cut out from the plate with tensile axis parallel to transverse direction for room temperature testing. Aging at 210°C for 48 hours was carried out after FSP. The tensile tests were performed both before and after aging to evaluate the change in strength and ductility with aging. The initial strain rate in these tensile tests was  $10^{-3}$  s<sup>-1</sup>. The tensile results were averaged over 2-3 samples.

#### Microhardness and Modulus Measurements

The microhardness and modulus measurements were done on samples from the transverse face of the plate. There samples were ground and polished. The hardness measurements were recorded at an interval of 5 mm across the transverse face of the weld as shown in Fig. 2(a). Composite stiffness values were obtained by instrumented Vickers hardness equipment, by

running the equipment in 'load-unload' mode with a test force of 500 mN and a hold time of 10 seconds.

### **Results and Discussions**

## Microstructure

The macrostructure of the nugget after four passes is shown in Fig. 2(a) which depicts a basin shaped nugget. It is evident that the nugget is free of porosity and processing defects. The average grain size in the nugget after the first pass was  $3.37\pm0.48$  µm. This is much finer than  $29\pm2.80$  µm of the base material as shown in Fig. 2(b). Hence, significant grain refinement has taken place due to dynamic recrystallization in the nugget during FSP. Fig. 2(c) shows the grain size to be  $2.71\pm0.69$  µm after four passes. Hence, a further reduction in grain size is evident after four passes as compared to that after a single pass.

Figs. 2(c) and 2(d) depict the change in distribution of  $B_4C$  with overlapping passes. It is seen that the distribution of the reinforcements is banded after the first pass (Fig. 2(c) which becomes more uniform after four passes (Fig. 2(d)). This suggests that the tool geometry and the processing parameters were effective in distributing the reinforcements in the matrix resulting in a basically even microstructure. The SEM image in Fig. 2(e) showing a uniform microstructure also further confirms that FSP is an effective way of distributing second phase reinforcements in the matrix to modify the microstructure locally.

## Microhardness

Fig. 3 shows the microhardness results after every pass. The dotted lines mark the ends of the nugget separating the base material from the nugget. The various curves represent the hardness evolution after every pass. The nugget shows a significant increase in hardness of about 19% in the nugget as compared to the base material. This is attributed to the presence of the hard  $B_4C$  ceramic reinforcements in the nugget. Apart from this, the grain size refinement brought about by dynamic recrystallization also contributes to the hardness improvement.

Table I lists the hardness values and scatter in the hardness values with increasing number of passes. An improvement in hardness is observed up to the third pass. The scatter in hardness readings within the nugget are considered to be secondary and could be related to minor microstructural variations. This scatter is also seen to reduce after every pass up to the third pass.

	1 pass	2 pass	3 pass	4 pass
Microhardness	86 - 99	88 - 98	93 - 95	88 - 93
range (HV <sub>0.2</sub> )				

Table I. A summary of the range of hardness values after every pass.



Fig. 2(a) Macrostructure of the nugget after FSP – the microhardness results were taken across the black dotted line, Optical micrographs of (b) the base material, (c) nugget after one pass, and (d) nugget after four passes. (e) A backscattered SEM micrograph showing the  $B_4C$  particles (darker).



Fig 3. Variation in the microhardness across the processed region after every pass.

#### Tensile Properties

Table II shows the tensile properties for the composites tested at room temperature. The yield strength of the composite was found to be  $189\pm17$  MPa which improved to  $281\pm58$  MPa after aging. An improvement in ultimate tensile strength was also observed changing from  $193\pm18$  MPa to  $281\pm51$  MPa after aging. There was however no improvement in yield strength of the composite (189 MPa) as compared to that of the base material (185 MPa). The elongation to failure decreased very significantly in the composite specimens.

Material	YS	(MPa)	UTS (MPa)		Elongation to failure (%)	
	Before	After	Before	After	Before aging	After
	aging	aging	aging	aging		aging
WE43	185± 8.66	270±15 [17]	293±8	348±6 [18]	17.7±1.02	16±3
WE43+6 vol% B <sub>4</sub> C	189±17	281±58	193±18	281±51	1.2±0.5	0

Table II. A summary of the tensile properties at room temperature.

#### Modulus Measurements

Table III compares the elastic modulus of the alloy with that of the reinforced composite. The modulus of WE43 obtained in this study matched with the values reported in another study [18] and is listed in Table 3. A marked improvement of the modulus in the composite (about 41%) was observed as compared to the base material. The  $B_4C$  phase has an elastic modulus which is

an order of magnitude higher than that of the matrix alloy and this accounts for the increased stiffness of the composite. The rule of mixture calculation suggest the WE43-6 vol.% B<sub>4</sub>C composite modulus should be ~ 51 GPa (assuming, WE43 ~ 25 GPa, B<sub>4</sub>C ~ 460 GPa). The current results show the improvement in modulus is consistent with the rule of mixture approach and indicate an effective load transfer. The force vs indentation depth curves for various readings are shown in Fig. 4, and these were used for modulus measurement of WE43 with and without reinforcement.

Material	WE43	WE43+6 vol.% B <sub>4</sub> C
Elastic modulus (GPa)	26.54±0.23	43.47±2.87
	25.0 – (F temper) [18]	
	25.8 – (T5 temper) [18]	

Table III. A summary of the modulus values for both the base material and the composite.



Depth (µm)

Fig. 4. A graph showing force vs indentation depth for various modulus readings of WE43+6 vol.% of  $B_4C$  composite.

## Conclusions

Friction stir processing fabricated the composite successfully with a uniform dispersion of the reinforcements in the matrix which resulted in uniform and consistent properties. Following conclusions can be drawn based on foregoing discussion.

- I. Grain refinement was observed as compared to base material. A decrease in grain size was also observed with overlapping passes.
- II. An appreciable increase in hardness of about 19% as compared to the base material.
- III. An improvement in modulus of about 41% was observed in the composites as compared to the base material by addition of 6 vol% of reinforcements.
- IV. Though an improvement in ultimate tensile strength was observed for the composites, the yield strength remained unaffected.

- V. Considerable reduction in ductility was observed for the composites.
- VI. The composites show a similar aging response as that of the base material.

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