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MICROSTRUCTURAL STUDY, HARDNESS BEHAVIOR AND FRICTION STIR PROCESSING OF MAGNESIUM BASED METAL MATRIX COMPOSITES

Engineering

KEYWORDS: Stir casting process, Friction stir processing (FSP), Particle reinforced metal matrix composites

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ABSTRACT

The primary objective of the present study is to investigate the microstructure evolution and hardness behavior of Mg based metal matrix composites. These particulate metal matrix composites which incorporate magnesium and its alloy AZ91 as matrix phase and silicon carbide and titanium carbide as reinforced particulates were fabricated using the simple mechanical stir-casting process. The fabricated metal matrix composites were characterized by using optical and scanning electron microscopy in order to reveal their microstructural evolution and by Vickers micro hardness instrument to obtain hardness behavior. Furthermore, friction stir processing (FSP) was performed on composites for grain refinement. Microstructural studies revealed that grain refinement was observed in all fabricated composites in comparison to base metal. It was also observed that FSP tends to fragment the large reinforcement particles and produces near homogeneous distribution of fine particles in the composite. Increment in microhardness of fabricated metal matrix composites in comparison to their base metal was observed.

INTRODUCTION

In last decades, plenty of work has been carried out to use magnesium alloys in manufacturing of the engineering components such as gear box housings, and power train applications [1].However, some of the mechanical properties of these alloys, especially their high temperature mechanical behavior are not favorable to widen their application sector. Poor anti-high temperature creep performance, low corrosion resistance and lower elastic modulus are theissues related to magnesium and their alloys that limit their engineering applications [2, 3].

Particle Reinforced Metal matrix composite materials (cermets) are generally composed of two phases; one is termed the matrix which is a metal and continuous and surrounds the other phase, often called the dispersedphase. The properties of composites are a function of the properties of the constituent phases, their relative amounts, and the geometry of the dispersed phase. The light metal matrix composites include structural metallic matrix elements such as aluminum and magnesium alloys, and dispersed phase (orreinforcedparticulates) as strong ceramic materials such as carbides, oxides, and nitrides. These materials may be utilized at higher service temperatures than theirbase metal counterparts; furthermore, the reinforcement may improve specific stiffness, specific strength, abrasion resistance, creep resistance, thermal conductivity, and dimensional stability. Aluminum (Al) and magnesium (Mg) are most commonly used as base matrix materials in light-weight metal matrix composites as they offer high specific mechanical properties due to their low densities (2.7 and 1.7 g/cm3 forAl and Mg, respectively) when compared to iron. The development and application of high strength and light -weight Metal matrix composite materials could have significant impact as a replacement for heavier traditional metals or composites with resultant savings in fuel economy.[4].

In all magnesium alloys, AZ91 is easy to process, i.e., easy to cast into high strength products in low cost, obtained extensive research and development, as well as industrial application. Whereas the poor anti-high temperature (over 120°C) creep performance and the low corrosion resistance are limiting its further application. In traditional casting Mg-Al base alloy, -Mg17Al12 is major strengthening phase at room temperature, but the -Mg17Al12 phase's melting point is as low as 437°C. With temperature increasing to437°C, the atom diffusion accelerates, -Mg17Al12 phases are easily softened and coarsened. This causes the grain boundary to be weakened . So that -Mg17Al12 phase cannot pin the grain boundary thus the grain boundary sliding occurs. Currently in the most widely used AZ and AM series magnesium alloys, the microstructures are mainly composed of -Mg solid solution (the matrix) and a little amount of -Mg17Al12. Whereas in non-equilibrium solidification process, nearby each -Mg grain boundary aluminum supersaturated solid solution area exists, afterwards the -Mg17Al12phases formed in situ. During high temperature utility, -Mg17Al12 phases in discontinuous shape separate out of supersaturated solid solution, and become into lamellar precipitates owing to low thermal stability. This is one reason that conventional AZ and AM series alloys have poor creep performance. [5-9]

The price of calcium (Ca) element is low, the melting point low, and the density is quite close to the magnesium's; so at present it has become the most frequently using alkali soil element to enhance the magnesium alloy heat-resisting performance. Studies have shown that adding Ca element to the conventional magnesium alloy can not only enhance oxidation combustion temperature of magnesium alloy, but also refine as-cast structure so as to improve mechanical performance and high temperature creep of magnesium alloy. After Ca is added, the hardness of the alloy also increases significantly. [12, 13]

The primary objective of the present investigation is to investigate the microstructure evolution and hardness behavior of Mg based metal matrix composites fabricated using the simple mechanical stir casting process.

Table 1: Chemical Composition of Mg Alloy AZ91Showing the Weight Percentage

Element	Aluminium	Zinc	Magnesium
AZ91	9%	1%	Balance

EXPERIMENTAL DETAILS

In the present investigation, commercially available magnesium and its alloy designated as AZ91 (The chemical composition of AZ91 is listed in Table 1.)were used as matrix phase and silicon carbide and titanium carbide ceramic particles with average particle size of 40 μm were used as reinforcement particles, to fabricate particle reinforced metal matrix composites (mentioned in table 2) by using stir casting process . Stir casting process not only helps in the transfer of the particles to the melt but also retains them in a state of suspension and it also allows a homogenous distribution of the reinforcing material in the metal matrix.[10]

Table 2: Composition of fabricated particle reinforced metal matrix composites

Nomenclature Used For Fabricated Metal Matrix Composites	Matrix Phase	Dispersion Phase	Percentage of Dispersion Phase In Composite By Weight	Percentage of Calcium In Composite By Weight	Percentage of Matrix Phase In Composite By Weight
Mg-12%TiC	Mg	Titanium carbide	12	5	Balance
AZ91-12%SiC	AZ91	Silicon carbide	12	5	Balance
AZ91-12%TiC	Az91	titanium carbide	12	5	Balance

The process of casting to fabricate above mentioned metal matrix composites was obtained by using commercially built stir casting instrument. The melting and stirring of metal were carried out in controlled environment which is mixture of inert argon gas and sulfur hexafluoride in order to avoid the oxidation of magnesium or its alloy AZ91. This special bottom pouring set-up for casting offers several advantages in comparison to conventional top pouring technique; namely the removal of the slag inclusion that otherwise goes directly in to cast mould, and also the reduction in pouring time. The processing procedure is explained briefly in the following. At first, as-cast magnesium and its alloy AZ91ingots were cut in to several pieces by machining process. AZ91 alloy machined pieces were heated to temperature of 750ºC in the steel crucible, as AZ91 alloy reach completely to molten state, preheated silicon carbide(SiC) particles (at temperature of 400ºC in order to overcome the humidity and enhance wet ability) and calcium granules (average size 1mm)were added, and homogenously mixed with mechanical stirrer at the desired set of operating conditions i.e. stirrer geometry of three-blade axial type, stirrer speed of 400 rpm, stirrer position of 0.3 times the height of molten metal, and holding time of 20 minutes. Finally, mixed slurries melt were then bottom-poured down into a plain carbon steel split-mold that was preheated at temperature of 400 ºC, to fabricate a cylindrical casting having length 300mm and diameter 32mm.The above mentioned procedure was repeated to fabricate cylindrical castings of the composites Mg-12%TiC and AZ91-12%TiC. All the castings were then left to air-cool under room temperature conditions.

A rectangular specimen with dimensions of 55mm ×26mm×5mm was prepared for FSP. Friction stir processing was carried out on a computer numerical control vertical milling machine (5 horsepower) with the help of FSP tool. The tool material used for FSP was high-speed steel. The FSP tool was a commonly used cylindrical tool with 12-mm shoulder diameter, 4-mm pin diameter, and 2.7-mm pin length. The parameters used, consist of tool rotational speed of 1500 r.p.m., linear speed of 56 mm/min, and target depth of 4mm. The FSP specimen was cut along transverse (so as to reveal cross section) and longitudinal (so as to reveal surface) directions and then it was hot mounted. Note that FSP is performed only in the case of fabricated Mg-12%TiC composite.

In order to reveal microstructures, samples of length 5mm and diameter 3mm were cut from all the fabricated cylindrical castings. Samples having magnesium as matrix phase were hot mounted and samples having AZ91 as matrix phase were cold mounted to avoid the influence of their age-hardening behavior. The surfaces of above mounted samples were prepared by standard metallographic techniques in order to avoid the influence of roughness effect on hardness response of the composites. Then Vickers micro indentation hardness Test was performed on all mounted samples, at a 300-g load for 22 seconds and 30 observations of microhardness were taken for each specimen. All the mounted samples were etched with a solution of 3 mL hydrogen peroxide, 6mL nitric acid and 15 mL ethanol for 30 seconds. The microstructure features of fabricated composites were examined using Scanning Electron microscopy instrument.

MICROSTRUCTURE

The SEM micrographs of the fabricated composites namely AZ91-12%TiC, AZ91-12%SiC,Mg-12%TiCand pure AZ91and pure magnesium are shown in Figures 1,2,3,4, and 5 respectively. Optical micrographs of FSP specimen are shown in Figures 6 and 7. The grain size (average grain diameter as determined from a random cross section) of fabricated composites, pure magnesium and pure AZ91 and size of reinforced particles of fabricated composites, measured by software Image J are being tabulated in Table 3.It is observed from the Table 3 that the grain size of fabricated composites is quite less than the grain size of base metals magnesium and AZ91.Hence it can be concluded that grain refinement has taken place on fabrication of particle reinforced metal matrix composites.

The grain refinement effect has been attributed to heterogeneous nucleation of the primary magnesium andAZ91phase on reinforcement particles and the restricted grain growth of magnesium and AZ91crystals caused by the presence of rigid reinforcement particles. In the literature, it is also mentioned that in heterogeneous nucleation mechanism the smaller the reinforcement particles, the finer are the grains of the obtained composite matrix .This is because more nucleation sites are provided during solidification process. [10]

Table 3 : Grain and reinforced particle size of fabricated composites.

Sr. No.	Sample Investigated	Grain size(µm)	Reinforced Particle Size(µm)
1	Mg(as cast)	500	-
2	AZ91(as cast)	91.11	-
3	Mg-12%TiC	44.10	16.84
4	AZ91-12%SiC	24.42	21.94
5	AZ91-12%TiC	20.03	11.68

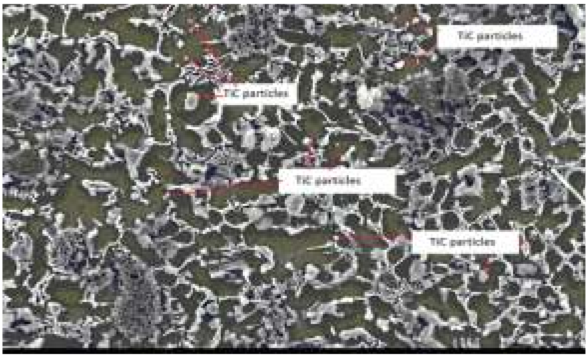

Fig.1: SEM image of AZ91%TiC composite showing TiC particles

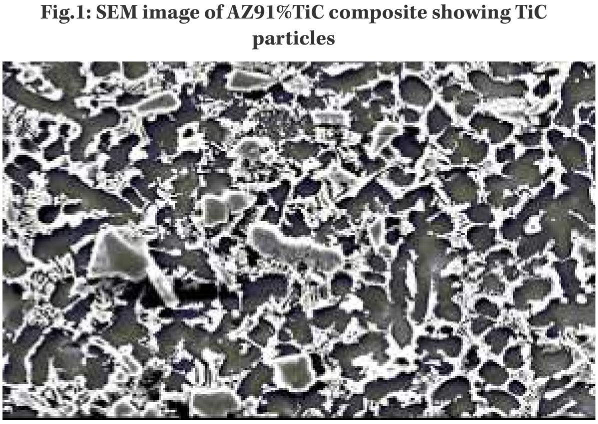

Fig.2: SEM image of AZ91%SiC composite

Fig.3: SEM image of Mg12%TiC

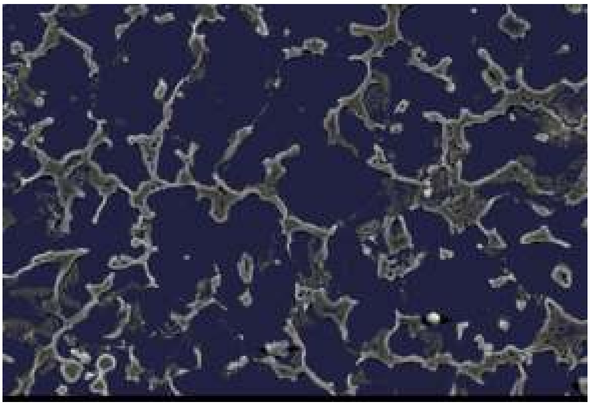

Fig.4: SEM image of pureAZ91

Table 4: Microhardness of prepared specimens.

S. No.	Specimen	Micro Hardness(HV)		
		Matrix Rich Region	Particle and Matrix Interface	Mean Microhardness (HV)
1	Mg	42.43	-	42.43
2	AZ91	89.83	-	89.83
3	Mg-12%TiC	58.11	92.17	75.14
4	AZ91-12%SiC	108.33	152.6	130.46
5	AZ91-12%TiC	110.10	154.4	132.25
6	FSP Specimen	76.10 (Nugget Zone)	-	76.10

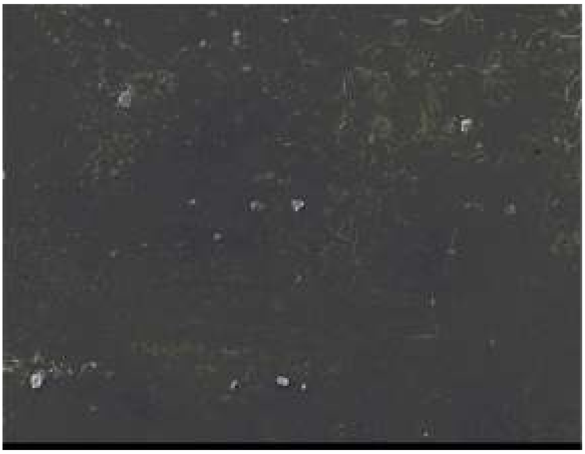

Fig.5: SEM image of pure magnesium

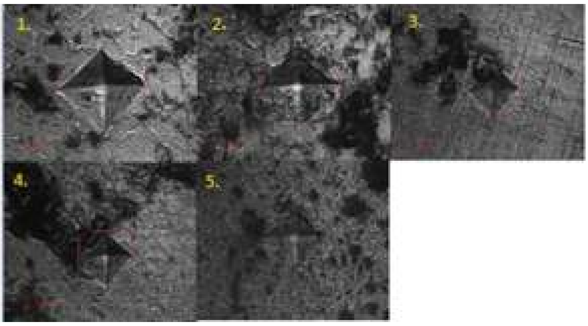

Fig.6: Evolution of indentation depression

Figure 6 depicts the evolution of indentation depression in,Mg-12%TiC,Mg-12%SiC, AZ91-12% TiC,AZ91-12%SiC And Mg-12%TiC (FSP sample).

It is observed that in AZ91-12%SiC, reinforced particles of SiC are more clustered than reinforced particles of TiC in AZ91-12%TiC.In AZ91-12%SiC, all SiC reinforced particles are segregated at the grain boundary but in AZ91-12%TiC although most of TiC reinforced particles are segregated at the grain boundary but some of TiC particles are trapped in the grains of composite. Some of the trapped TiC reinforced particles are clearly marked in Figure 1.This unusual behavior is not clearly understood. Nugget zone, basemetal and transition zone of FSP specimen are clearly shown in Figure 6,it can be observed in Figure 7 that large particles of TiC that were present in Mg-12%TiC composite were fragmented into smaller particles during friction stir processing.

VICKERS MICROINDENTATION HARDNESS TEST

The mean microhardness values obtained from 30 observations carried out on each of the fabricated composite, pure magnesium, pure AZ91andFSP specimen are tabulated in the Table 4. Microhardness of AZ91-12%SiC and AZ91-12%TiC is more than the base metal of the composites i.e. AZ91, because it is observed that grain refinement has taken place on fabrication of composites AZ91-12%SiC and AZ91-12%TiC .Strength of material increases as grain size decreases in accordance with Hall-Petch equation given below:

$$\sigma_o = \sigma_i + kD^{-1/2}(1) \qquad \text{(Equation – 1)}$$

where σ_o is the yield stress,σ_i is the friction stress representingthe overall resistance of the crystal lattice todislocation movement, k is the locking parameter that measures the relative hardening contribution of the grain boundaries, and D is the grain diameter. The equation states that the hardness value is inversely proportional to the grain size. Thus, in accordance with Eq.1, as the grain diameter/grain size decreases, the hardness of the material increases.[11]

Since grain size of AZ91-12%TiC is less than grain size of AZ91-12%SiC so, according to Hall–Petchequation, microhardness of AZ91-12%TiC is more than AZ91-12%SiC. Microhardness of AZ91-12%TiC in matrix rich region is more than that of AZ91-12%SiC which means Load-Bearing Effect is more prominent in AZ91-12%TiC than in AZ91-12%SiC.Microhardness of matrix rich region of FSP specimen is more than microhardness of matrix rich region of Mg-12%TiC because large particles of TiC that were present in Mg-12%TiC were broken into smaller particles during friction stir processing and grain size of Mg-12%TiC was also reduced after FSP hence increasing hardness of compositein accordance with Hall–Petch equation.

CONCLUSIONS

Grain refinement has taken place on fabrication of particle reinforced metal matrix composites due to heterogeneous nucleation of the primary phase on reinforcement particles.

Microhardness of all the fabricated particle reinforced metal matrix composites is more than their base metals in accordance with Hall–Petch equation.

Friction stir processing tends to fragment the large reinforcement particles and produces near homogeneous distribution of fine particles in the composite.

Friction stir processing leads to increase in microhardness of fabricated particle reinforced metal matrix composite.

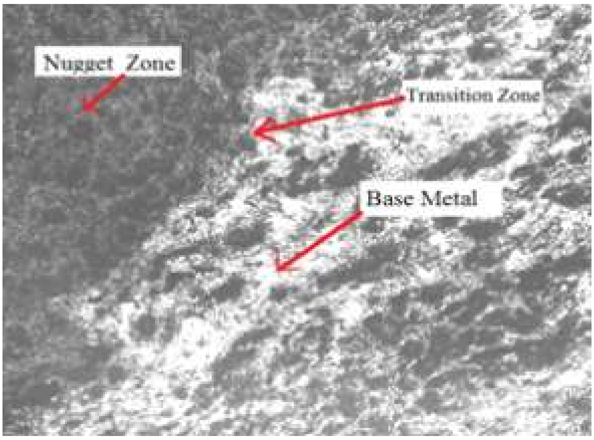

Fig.6: Optical micrograph of FSP specimen(Mg12%TiC) showing Nugget Zone Transition Zone and Base Metal

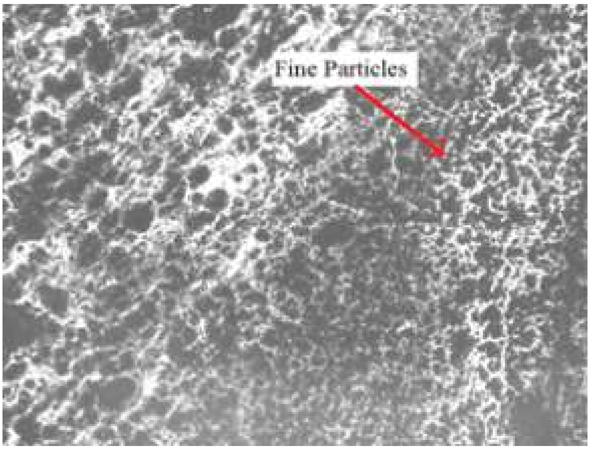

Fig.7: Optical micrograph of FSP specimen

(Mg12%TiC) showing fine Particles of TiC after FSP of Mg12%TiC .

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