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NANO COMPOSITES OF MAGNESIUM BY FRICTION STIR PROCESSING FOR BIOMEDICAL APPLICATION

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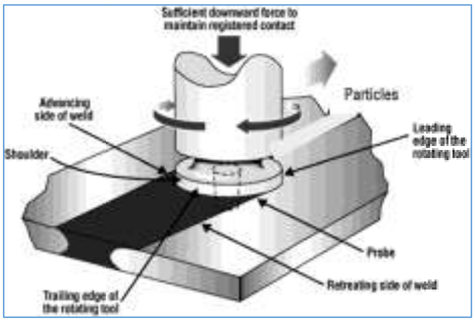
Abstract

Magnesium is the third most commonly used structural metal, following iron and aluminium. In this research work, an attempt has been made to investigate the mechanical properties of Magnesium based metal matrix composites manufactured through stir casting route. Friction Stir Processing (FSP) is a solid state processing technique that has unique capabilities like low amount of heat generation, extensive plastic flow of material, achieving very fine microstructure in the stirred region, healing of flaws and reducing porosity. The process parameters are identified for the FSP of both pure magnesium based hybrid composites and commercially available hybrid composites based on the refinements in the level of the grain sizes, improvement in mechanical properties such as tensile properties, hardness improvement and wear resistance characteristics of the Friction Stir Processed composites. Three different tool rotation speeds such as 220 rpm, 340 rpm and 560 rpm and three different linear velocities of the tool (10, 20 and 30 mm/min) were considered as the process parameters. Micro structural studies have been carried out to investigate the grain size variations and an appreciable refinement in the grain size from 84 to 7 μm was observed through Friction Stir Process. The produced composite was test for antimicrobial activity for biomedical purpose.

Keywords*: Magnesium, Friction Stir Processing (FSP), AZ91 hybrid, Nano composite*

Introduction

Nanotechnology has a crucial role in the development of advanced technologies including photonics, biotechnology, smart textiles and several other applications in the fields of science and technology. Nanotechnology is attracting universal awareness because it is offering enormous potential in a large range of scientific and technological areas. The unique and novel properties of nanomaterials have fascinated not only scientists and researchers but also trade due to their huge economical potential. Friction Stir Processing (FSP) is an adaptation of friction stir welding, and the following unique features of friction stir welding can be used to develop new processes based on the concept of friction stirring. FrictionStir Processing (FSP) uses the controlled-path metalworking characteristics of the process to perform metallurgical processing and microstructural modification of local areas on the surface of a part (Rajiv et al. 2007). In this process, a rotating tool with a pin and shoulder is inserted into a single piece of material for localized microstructural modification to achieve the specific property enhancement as shown in Figure 1.1.



(Source: Cinta et al. 2014)

Figure 1.1 Schematic representation of Friction Stir Processing

Friction Stir Processing (FSP), developed by (Mishra et al. 2003) a solid-state post-processing technique that can provide localized modification and control of microstructures in near-surface layers of metallic components is developed based on the principles of Friction Stir Welding (FSW) (Mishra et al. 2014). The Friction Stir Processing comprises of a rotating tool (non – consumable), which is pushed into the work piece and then traversedin the direction of interest.

Operating or processing parameters determine the amount of plastic deformation, heat generated and material flow around the non-consumable tool. The main process parameters of the FSP process are the following:

- Rotational speed,
- Travelling speed,
- Axial Force,
- Tilt angle,
- Tool geometry (pin length, diameter and shape of the pin, diameter and shape of the shoulder).
- Penetration depth of the tool,
- Cooling system,
- Clamping system.

Magnesium Oxide Nanoparticles

MgO is an important inorganic material with a wide band gap. MgO used in many applications like catalysis, toxic waste remediation, paint, superconducting products, antibacterial activities. Among the metal oxide nanomaterials, MgO is particularly interesting as a low cost, environmentally friendly material. The toxicity of MgO has found to be lower than other metal oxide nanoparticles; including ZnO. Small size MgO nanoparticles have an efficient antibacterial activity towards E.coli and S.aureus. MgO nanoparticles penetrate the bacterial cell wall and cell membrane. So the quantity of killed bacteria was strongly dependent on particle size. Generally, the specific surface area of MgO nanoparticles increases as the size of the nanoparticles decreases. The increase in surface area determines the potential number of reactive groups on the particle surface, which are expected to demonstrate high antibacterial activity. Therefore the findings are similar to the antibacterial activity of Ag nanoparticles against gram positive and gram negative bacteria. The exact antibacterial mechanism of MgO nanoparticles is still unknown. A number of mechanisms, such as the formation of reactive oxygen species (ROS), the interaction of nanoparticles with bacteria, subsequently damaging the bacterial cell, and an alkaline effect have been proposed. It has been reported that the increase of the surface area of MgO particles leads to an increase of the O_2^- concentration in solution and thus results in a more effective destruction of the cell wall of the bacteria.

Experimental Process

Magnesium based hybrid composite materials were fabricated through Stir route. The composite castings prepared were further processed through Friction Stir Processing in order to further enhance the surface properties of the hybrid composites.

Composite Preparation

Selection of the Reinforcement Particles

The objective of enhancing the mechanical and metallurgical properties of the Magnesium based alloys, appropriate reinforcement particles is selected based on the earlier research work carried out by many researchers around the globe. One of the main criteria based on which the selection of the reinforcement particles are made normally is its mechanical properties such as strength and hardness. Based on literature, it was decided to use Al_2O_3 and SiC as reinforcement for preparing magnesium hybrid composite, with a particle size of 20□ m, which is commercially available.

Silicon Carbide (SiC)

Silicon Carbide is the only chemical compound of carbon and silicon. It was originally produced by a high temperature electro-chemical reaction of sand and carbon. Silicon carbide is an excellent abrasive and has been produced and made into grinding wheels and other abrasive products for over one hundred years. Today the material has been developed into a high quality technical grade ceramic with very good mechanical properties. It is used in abrasives, refractories, ceramics, and numerous high-performance applications. The material can also be made an electrical conductor and has applications in resistance heating, flame igniters and electronic components. Structural and wear applications are constantly developing. The key silicon carbide properties are: low density, high strength, low thermal expansion, high thermal conductivity, high hardness, high Elastic Modulus, excellent thermal shock resistance and superior chemical inertness.

Alumina (Al_2O_3)

Alumina is one of the most cost effective and widely used materials in the family of engineering ceramics. The raw materials from which this high performance technical grade ceramic is made are readily available and reasonably priced, resulting in good value for the cost in fabricated alumina shapes. With an excellent combination of properties and an attractive price, it is no surprise that fine grain technical grade alumina has a very wide range of applications. The key properties of the Alumina are: Hard, Wear resistance, excellent dielectric properties, resists strong acid and alkali at elevated temperatures, good thermal conductivity, high strength and stiffness. The Physical and Mechanical properties of the reinforcements ($Al_2O_3$and SiC_p) are given in Table 1.1.

Table 1.1 Physical and Mechanical Properties of Reinforcements

Property	Unit	Al_2O_3	SiC
Density	(g/cc)	3.69	3.10
Young's modulus of elasticity	GPa	300	410
Flexural Strength	MPa	330	550
Compressive Strength	MPa	2100	3900
Hardness	Kg/mm^2	1175	2800
Thermal Conductivity	W/m.K	18	120

Stir Casting of Magnesium Based Based and Az91 Hybrid Composites

Commercially available elemental Magnesium (99.9% purity) were procured from Sigma Alrich company, Bangalore, whose physical and chemical properties are recorded in Table 3.2 for further processing. Magnesium ingot pieces of weight 5kgs are mixed with 5wt% of SiC (20 μm) and 5wt %Al_2O_3(20 μ m) in a steel crucible kept in a furnace, under the cover of a flux consisting of NaCl and MgO. A charge containing a mixture of 5kg Magnesium ingot and 250 gms of Al_2O_3 with flux is taken in a mild steel crucible and heated up to 400° C in a furnace. 250gms of SiC particles of size 20 μm are preheated up to 300° C and were added to the charge. The furnace temperature was raised to 500°C and the melt homogenized for one hour. The temperature was increased further up to730°C and then reduced to 700°C. A Stainless Steel impeller was used to stir the composite melt. The stirring was done at the rate of 120 rpm for 20 minutes.

After stirring, the composite melt was then rapidly heated to about 730° C and poured into a mould for preparing rectangular plates of 200 mm × 80mm × 12mm size as illustrated.

Table 1.2 Properties of Pure Magnesium

Chemical composition (Mass) %								
Mg ≥	**Impurities ≤**							
	Fe	**Si**	**Ni**	**Cu**	**Al**	**Mn**	**Cl**	**Others**
Mg 99.9	0.01	0.03	0.001	0.002	0.02	0.05	0.005	0.01
Physical Properties								
Density						1.738 g/cc		
Young's modulus of elasticity						44.8 GPa		
Tensile strength						135-285 MPa		
Impact Strength						28 J		

Table 1.3 Chemical compositions of AZ91 magnesium alloy

Chemical composition (Mass) %							
Impurities ≤							
Fe	**Si**	**Zr**	**Cu**	**Al**	**Mn**	**Zn**	**Remaining**
0.02	0.02	<0.002	0.005	8.84	0.018	0.88	Mg
Physical Properties							
Density					1.81g/cc		
Young's modulus of elasticity					44.8 GPa		
Tensile strength					135-285 MPa		
Impact Strength					2.7 J		

Microhardness

The micro hardness measurements were performed under Vickers hardness tester of Wilson Wolpert – Germany make with a load of 10 N for a dwell period of 10s to assess the effect of Friction stir process on the Magnesium metal matrix composites. The Vernier calliper least count of the Vickers Hardness is 0.01mm. The measurements for the hardness of the FS processed composites are made at different zones for various operating parametersand are tabulated for further investigations. The test specimens were cleaned to ensure flatness and mounted using specimen mounting press.

Method of Bactericidal Test

The antibacterial activity was tested against six important bacteria, *Staphylococcus aureus* (MTCC737), *Escherichia coli* (MTCC 1679), *Klebsiella pneumoniae* (MTCC 3384), *Pseudomonas aeruginosa* (MTCC 2295), *Bacillus cereus* (MTCC 1272), and *Bacillus subtilis* (MTCC 619). We have adopted the diffusion method for the antibacterial investigations. We also have performed the quantitative analysis. For this purpose we have followed the commonly used AATCC test method 100-2004.

Method of Fungal Test

Antifungal study was done by agar diffusion method, Investigation were done against *Aspergillus niger* (MTCC 478) and *Candida albicans* (MTCC3018). Control fabrics did not show any activity. The inhibitory spectrum was measured in mm scale.

Results and Discussion

FSP is a solid state novel technique to produce surface composite. It is a method of changing the properties of a metal through intense, localized plastic deformation. Previous literature studies have shown microstructural refinement, densification and homogeneity of the work material after FSP.The Friction Stir Processing of pure & AZ91 magnesium based composites is discussed in the following section. The mechanical properties such as hardness, tensile properties and wear resistance of the Mg based and AZ91 based hybrid composite materials, before and after FSP were studied with the help of microhardness tester, tensile testing machine and pin on disc test set-up.

Friction Stir Processing of Pure Magnesium Based Hybrid Composites

The mechanical properties of Friction Stir Processed pure Magnesium based hybrid composites are studied by conducting Micro Hardness studies and tensile test. FSP of pure Magnesium based hybrid composite is carried out with three different tool speeds namely 220, 360 and 540 rpm and three different tool linearvelocities such as 10, 20 and 30 mm/min. The hardness value of various specimens, tensile strength and percentage elongations of the respective specimens obtained for various composite materials and processes through tensile tests were recorded and their average values are compared in the following section.

Micro Hardness

The micro-hardness values of the tested composite specimens before and after the FSP as well as the heat treatment process at seven different locationsin the nugget zone were measured. The average values of the individual specimen readings obtained through Vickers micro hardness testing facility for three different process conditions are given in Table 4.1. It is inferred from Table 4.1, that the hardness value for pure magnesium based hybrid composite before FSP is 59.3 Hv. This value is increases to a maximum of 69.7 Hv when the composite is Friction Stir Processed with the following process parameters i.e., high tool speed (540 rpm) and low tool linear velocity (10 mm/min). This is due to decrease in size of the grain owing to mechanical recrystallization.

This in turn reflects the Hall – Petch relationship which states that as the grain size decreases, the hardness increases.

(Hall-Petch relation: $H = H_o + K_H d^{-1/2}$,

where, the hardness of materials, H, is dependent on grain diameter, d, Ho and KH are constants).

Further it is noticed that as the linear velocity of the tool increases, the contact time between the composite surface and the tool material decreases, resulting in reduced the Friction Stir Processing time. As the FSP time decreases, the grain growth get affected which positively influence the hardness value of the composite. From the data on microhardness shown in Table ,it is observed that there is only amarginal increase in hardness, especially for the sample processed at lower tool rotating speed and larger linear speed(FSP7),this is due to relatively insufficient processing time for the grains to undergo recrystallization.

Another interesting observation is, the rotational speed of the tool playing a vital role in achieving enhanced hardness value because of a higher temperature developed on the surface of the composite. This temperature will result in grain growth refinement and hence increase the micro hardness values near on the surface(NZ). The known average grain sizes were also measured and recorded in Table.1.4

Table 1.4 Micro hardness values - Friction Stir Processed Mg-Hybrid Composite

Sl. No.	Sample Code	Rotating Speed (rpm)	Linear Speed (mm/min)	Average Grain Size (□m)	Average Hardness value (Hv)
1	As cast	-	-	82	59.3
2	FSP 1	220	10	22	64.7
3	FSP 2	360	10	15	67.6
4	FSP 3	540	10	07	69.7
5	FSP 4	220	20	14	65.2
6	FSP 5	360	20	09	68.5
7	FSP 6	540	20	09	68.5
8	FSP 7	220	30	30	62.7
9	FSP 8	360	30	15	67.5
10	FSP 9	540	30	15	67.3

Antibacterial Studies

Antibacterial Activity of Coated and Uncoated Fabrics

The antibacterial efficiency of uncoated and Mg nano composites coated fabrics against *S, aureus*, *E.coli*, *K.Pneumoniae*, *P.aeruginosa*, *B.cereus* and *B.subtilis* is shown in Figure. The results of all the coated fabrics and uncoated fabrics are based on the zone of inhibition formed on an agar medium of petri dishes.

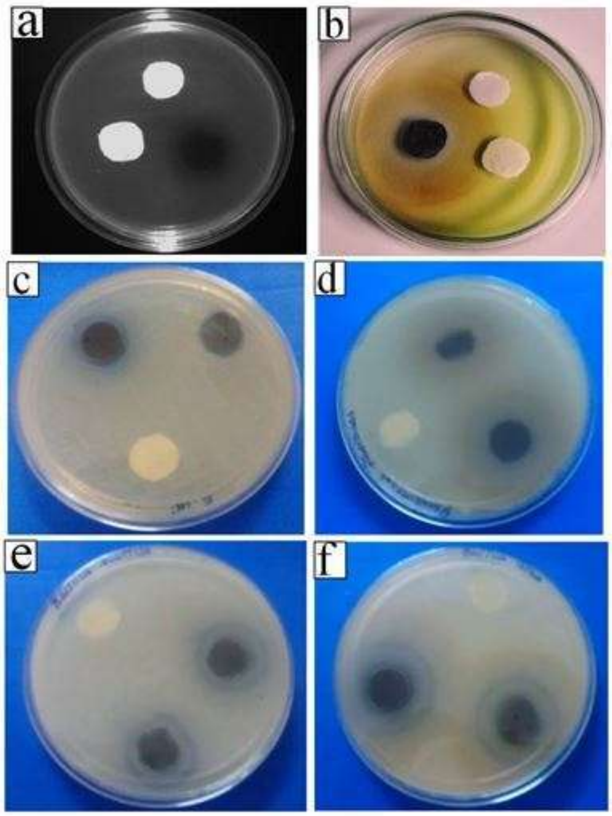


Fig. 4. 12 Antibacterial activities of uncoated and Mg nanocomposites coated fabrics against *S, aureus, E.coli, K.Pneumoniae, P.aeruginosa, B.cereus and B.subtilis*

The antibacterial activity of nanocomposites coated fabrics against S, aureus, E.coli, K.Pneumoniae, P.aeruginosa, B.cereus and B.subtilis. It is clear that the uncoated fabric shows no clear zone of inhibition for all of the pathogens because of the high hydrophilicity of cellulose, but nanocomposites coated fabrics showed a greater level of zone of inhibition against all pathogens compared to uncoated fabrics with a zone of inhibition of 14mm against *S.aureus* and *K.Pneumoniae*, where as 13mm zone of inhibition against E.coli and P.aeruginosa, and 12mm against *B.cereus* and *B.subtilis*. We have also done the experiments on the affect of washing on the antibacterial activity of the nano composites. The zones of inhibition before washing and after washing the fabric a number of times are shown in Figure. As can be seen in the experimental results shown in the figure the washing durability of the coated fabrics decreased the rate of antibacterial activity due to the nanoparticles removal from the fabric surface. Generally, nanoparticles release the copper and zinc ions and interact with microbes, which easily oxidise and die immediately. The surfaces of the nanoparticles coated fabric interact with bacterial cell walls, which results in the reduction of bacterial growth.

Antifungal Studies

Antifungal activity of uncoated and nano composites coated fabrics were considered against *C. albicans* and *A. niger* by agar diffusion method. Only few studies were carried out on the antifungal activity of nanoparticles. Antifungal results are displayed from figure

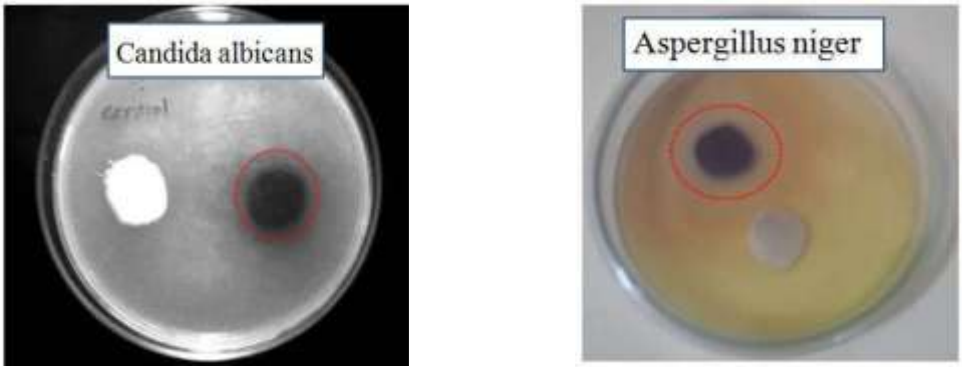


Fig. 4.13 Antifungal activity of uncoated and nanocomposites against *C. albicans* and *A. niger*

Summary and Conclusion

In this research work, magnesium based hybrid composite material is prepared through stir casting technique and further processed through Friction Stir Processing. Magnesium based hybrid composite and is found to be equated fine grains of size 7 μm with distinct grain boundaries for the process parameters of tool rotation of 560 rpm with tool linear velocity of 10 mm/min. The average grain size of 84 μm has been observed in the as cast magnesium composite. Microhardness studies were also conducted. Antimicrobial studies were conducted for the prepared nano composite. The application of nanocomposites could significantly enhanced the properties of starch based materials, which would be strongly required for biomedical

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