

Production and Evaluation of TiO₂ Reinforced with Magnesium Matrix Composites

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Abstract: Experimental investigations of TiO₂ particles reinforced with magnesium are presented in this research paper. The objective is to elaborate the manufacturing processes of pure magnesium adding with particles weight ratios of 2.5 to 10% by vacuum stir casting process with Argon as shielding gas to prevent the oxidation. Electrical discharging machining is used to resize the specimens for conducting experiments. The parameters like density, hardness, tensile strength, ultimate tensile strength are evaluated by immersion test, Rockwell Tester and Universal testing machine respectively. The presence of particles in phase is examined by X-ray powder diffraction (XRD) and scanning electron microscope (SEM) analysis. The homogeneous distribution of the reinforcing particles in the magnesium matrix was revealed by SEM. The results confirm that improvement of 23.7% in hardness and 29.8 % in ultimate tensile strength.

Key words: Magnesium • Titanium oxide • Stir casting • XRD analysis • SEM analysis • Mechanical properties

INTRODUCTION

The growing challenges to achieve light weight of structural materials, magnesium alloys are potential candidates for various applications. Magnesium based metal-matrix composite castings have the prospective to be used as replacements for many ferrous castings in power- train chassis including parts of engines, brake, suspension and steering . Cole and Sherman [1] insisted that corrosion resistance of modern high purity magnesium alloys was better than die cast aluminium materials. The discrete advantages of magnesium over aluminium alloys are lower latent heat fusion that makes longer die life, faster solidification, higher machinability, more precise tolerances due to higher fluidity and minimum cost in large scale production. Blawert *et al.* [2] summarized that the interest in magnesium alloys for automotive applications are based on the combination of high strength properties, low density, durability and design flexibility. For these reasons the magnesium alloys have been used as structural materials in all applications where weight savings are of great concern. Due to the weight reduction in automotive applications, the performance of a vehicle was improved by reducing the

rolling resistance and energy of acceleration, thereby reducing the fuel consumption and reduction of the greenhouse gas CO₂. Cole and Ibrahim *et al.* [1, 3] emphasized that the ability to produce the functional component at an affordable price is the real challenge for light weight materials. The new materials are considered to be part of vehicle designs since that satisfies the expected performance in fuel economy and drivability. The composites combine metallic properties like ductility and toughness with ceramic properties like high strength and high modulus leading to greater strength in shear and compression and higher service temperature capabilities. The reinforcement materials could include oxides, nitrides and carbides. NADCA and Ye *et al.* [4, 5] reported that density of magnesium is just about two thirds of aluminium, one fourth of zinc and one fifth of steel. The embracing research and development efforts in processing magnesium matrix composites with cost-effective fabrication technologies are required for future. Jayamathy *et al.* [6] described that the highest strength-to-weight ratio of the commonly used metals at present is magnesium. The Advantages of magnesium include good castability, high die casting rates, electromagnetic interference, shielding properties, part

consolidation, dimensional accuracy and excellent machinability. Venetti [7] claimed that magnesium composites were the attractive candidates for functional and structural materials. The defect free microstructures can be achieved by the use of stir casting process. The stir casting is used for mass production applications with an economic manner. Gui *et al.* [8] estimated that Vacuum stir casting method is used to fabricate the reinforcement of SiC particles upto 15% Vol. with magnesium and prevented the entrap of gases onto the melt and oxidation of magnesium during synthesis. Saravanan and Surappa [9] proved that the volume fraction of SiC particles upto 30% added with magnesium and successfully fabricated by stir casting process. Kumar *et al.* [10] reported that, the reinforcement particles should be stronger than matrix material in order to get the desirable properties of composites. Aravindan *et al.* [11] concluded that the preheating of reinforcement particles increased the surface energy thus in turn the wettability between the matrix and particles and strength were improved. Poddar *et al.* [12] anticipated that the stirring temperature was significant in the process and the properties of composites were affected by clusters of particles, porosity and high oxidation. Li *et al.* [13] mentioned that grain size, mechanical properties and microstructure are improved by the addition of rare earth metal yttrium particles with magnesium. Joseph *et al.* [14] wrote that Argon was used for extinguishing fires where valuable equipment may be damaged by water or foam. European Patent EP0268841 [15] informed that the porosity of melted metal matrix can be reduced by the use of Dichlorodifluoromethane with argon to create the protective atmosphere. The blanketing atmosphere protects the melt from oxidation, burning and evaporation of materials, improves alloy cleanliness and can be used in any furnace, transfer or casting operations. Ponappa *et al.* [16] intimated that the technology for machining Magnesium matrix composites is not yet clearly developed yet. The Electrical discharge Machining was suitable for machining the composites without usage of tool and there was no difficulties found in disposal of chips. Viswanath *et al.* and Liu *et al.* [17, 18] claimed that reinforcements strengthen the matrix by imparting better mechanical and tribological properties by many researchers. Samal *et al.* [19] proved that the tensile properties are enhanced by the addition of aluminum, SiC into magnesium by produced through stir casting process. Nuckols *et al.* [20] presented that the hardness, Elastic modulus are increased with addition SiC particles into Mg alloy but the ultimate tensile strength and

ductility are reduced [20]. The elements of various ceramic particles and other rare earth materials have been added to magnesium alloy for improving their mechanical properties, thermal stability, strengthening mechanisms for the recent years. But there were no systematic investigations and research about the addition of Titanium oxide or Titanium dioxide upto present. In this experimentation work the effects of addition of Titanium particles on mechanical properties are examined.

Experimental Methodology

Sample Preparation: The vacuum stir casting method in stages is used to fabricate five samples. Table 1 represents the proportion of particles with matrix material. The experimental set up to produce the samples is presented in Figure 1.

In the stir casting process the reinforcing phases usually in powder form are distributed into the molten magnesium by means of mechanical stirring. The effect of high strength can be achieved by homogenous distribution of secondary particles in the matrix by stirring process.

Otherwise uneven distribution can lead to premature failures in both reinforcement free and reinforcement rich areas. The main concerned with this process is segregation of reinforcement particles that is caused by surfacing or settling of the reinforcing particles during the melting and casting processes. Since the magnesium alloy is highly sensitive to oxidation, there is a possibility of entrapment of gases and other inclusions in the stir casting process. This will further increase the viscosity of the molten metal and produce imperfections within the material. Thus the stirring process needs to be more astutely controlled for magnesium alloys than aluminum alloys in order to prevent the entrapment of unwanted gases and other inclusions. Since magnesium is a flammable material and easily gets oxidized in the presence of oxygen a shielding gas is required to control the atmosphere inside the furnace. The protection of this environment from the oxygen is prevented by the use of Argon. Argon is used for thermal insulation in energy efficient windows that the element undergoes almost no chemical reactions. The outer atomic shell makes argon stable and resistant to bonding with other elements. It is mostly used as an inert shielding gas in welding and other high-temperature industrial processes where ordinarily unreactive substances become reactive. At room temperature, Argon is chemically inert under most conditions and low thermal conductivity thus forms no confirmed stable compounds [21].

Table 1: Percentage of particles in specimens

Sample No	Proportions
1	Pure magnesium
2	Mg+ 2.5 % TiO ₂
3	Mg+ 5 % TiO ₂
4	Mg+ 7.5 % TiO ₂
5	Mg+ 10 % TiO ₂



Fig. 1: Experimental set up



Fig. 2: Prepared Samples

The final distribution of the particles in the solid depends on material properties and process parameters such as the wetting condition of the particles with the melt, strength of mixing, relative density and solidification rate. The distribution of the particles in the molten matrix depends on the geometry of the mechanical stirrer, stir- ring parameters, placing of the mechanical stirrer in the melt, melting temperature and the characteristics of the particles added. In our research work the matrix material is heated to above liquidus temperature and the allowed to cool in between the stage of semi solid. The particles are preheated and then mixed with matrix material. Then the combinations of composites are heated again to above melting temperature of matrix material. The melting and casting is performed in stages to avoid the gas layer around the surface. Normally Particles have a thin layer of gas absorbed on their surface, which impedes wetting

between the particles and matrix metals. In comparison with conventional stirring, the mixing of the particles in the semi-solid state can more effectively break the gas layer because the high melt viscosity produces a more abrasive action on the particle surface. The produced final samples are shown in Figure 2. The first three samples represent the composites after machining and next two represent before machining stage.

XRD Analysis: The XRD patterns of the specimens prepared by stir casting process are as shown in Figures 3a&b. The samples are polished with mirror like surfaces with an automatic polisher. The phase analysis was carried out with a speed of 3 degree/ minute with a range of 0-100 degrees. As the intensities agree with the theoretical values, the increase in the peak areas gives the information about the kinetics of the reaction process.

It means that the composite was formed with in the systems. From the XRD pattern shown in Figures 3 a &b, The main diffraction peaks corresponding to the phases of Mg and Ti were detected. It is expected that the powder particle size can affect the process. it is observed that smaller particles of the elemental powder are more beneficial in the reaction between Mg and Ti. It is evident that TiO₂ is formed completely and a large quantity of molten magnesium fully infiltrates through the aperture gap of the particulate.

SEM Analysis: In order to characterize the microstructure of Mg-TiO₂ composites, Scanning Electron Microscope is used. The interface of Mg and TiO₂ is examined through the SEM. The grain size with its boundary of pure magnesium specimen is presented in Figure 4(a).

From the microscopic point of view the bonding within the magnesium matrix is ensured with less porosity. The image reveals the presence of small amount of TiO₂ particles which is shown in Figure 4(b). It is noted that free interference of the components are strongly connected and precipitate obtained The interface looks in a fine comportment shows the good bonding between the ceramic and matrix. The Metallographic examinations of the composite materials after the fabrication of samples revealed the uniform distribution of the TiO₂ reinforcing particles in the magnesium matrix. The structure obtained from the observation ensures the perfect bonding between the matrix and particles of composites. The particles of TiO₂ in variation in sizes that is not soluble in magnesium matrix is presented in Figure 4(c). In the magnification of 1000 x shows that very little micro pores are present on the surface.

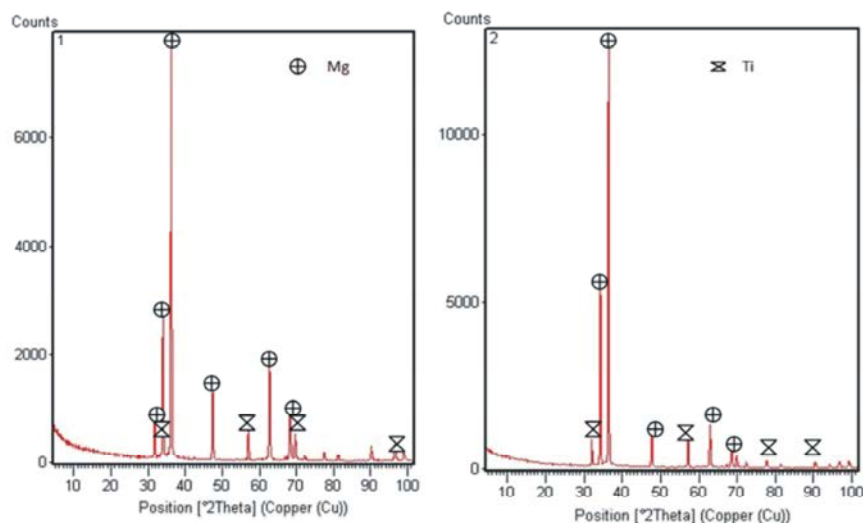


Fig. 3: (a) XRD Pattern for sample 4, (b) Sample 2

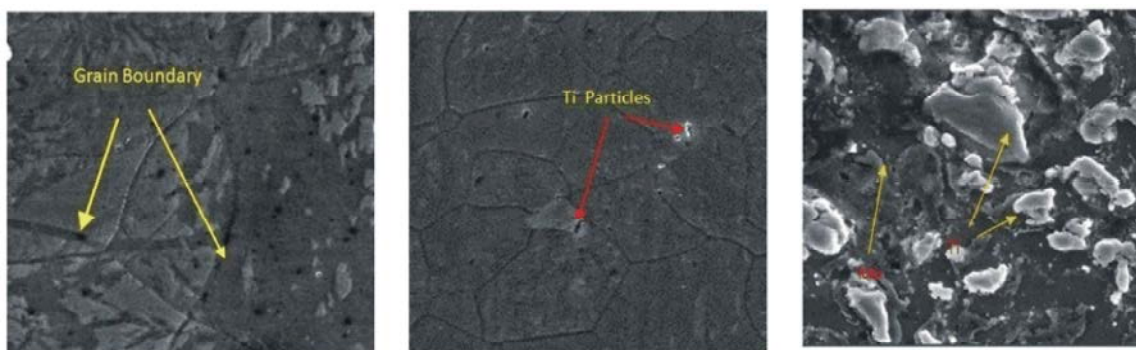


Fig. 4: SEM images a.) Pure Magnesium, b.) Mg + 5% TiO₂, c.) Ti particles in Mg matrix

Porosity Test: The Density of extruded specimens was estimated with Archimedean principle, by determining the specimen mass and volume and basing on the apparent loss of weight after immersing the specimen in water. The Archimedes principle practically allows the buoyancy of an object partially or fully immersed in a liquid to be calculated. The percentage of porosity is calculated by using the following equation.

$$\% \text{ of porosity} = 1 - (\text{measured density} / \text{theoretical density}) \times 100$$

Hardness Test: Hardness tests of the fabricated composite materials were made by using Rock well harness tester. Five indentations were made on the transverse section gdiometer for specimens produced by the stir casting process. Then the average values of each specimens are taken into account and listed in the Table 3.

Tensile Test: Static compression and tensile tests of the fabricated composite materials were made with the ASTM Standard B557-06 is followed at room temperature. The examined test pieces in the tensile have a overall length of 65 mm and gauge length 15 mm. The Yield stresses (YS) and ultimate tensile strength (UTS) were determined employing at least two specimens for each combination by using standard Universal Testing Machine.

RESULTS

Density and Porosity: The actual density measurements of each samples and their comparison with theoretical values are shown in Table 2.

Hardness: Mean hardness values of the pure magnesium and of the fabricated composite materials reinforced with the TiO₂ ceramic particles with the weight ratios of 2.5, 5, 7.5 and 10% are shown in Table 3. The hardness is increased to 24% more than unreinforced material.

Table 2 Calculation of porosities.

Specimen	Theoretical density g/cm ³	Measured density g/cm ³	Ratio of actual density to the theoretical density in %.	% of Porosity
Pure Mg	1.74	1.73	99.43	0.57
Mg + 2.5 % TiO ₂	1.80	1.79	99.44	0.56
Mg + 5 % TiO ₂	1.85	1.83	98.81	1.19
Mg + 7.5 % TiO ₂	1.91	1.88	98.43	1.57
Mg + 10 % TiO ₂	1.97	1.94	98.48	1.52

Table 3 Mechanical properties and its values.

Material	Hardness RHN	UTS MPa	Yield strength MPa
Pure Mg	38	163.8	108.4
Mg + 2.5 % TiO ₂	41	184.5	121.3
Mg + 5 % TiO ₂	43	193.1	127.6
Mg + 7.5 % TiO ₂	44	204.5	134.8
Mg + 10 % TiO ₂	47	212.6	140.9

Tensile Strength: The measured tensile parameters are presented in Table 3. The UTS is increased from 163.8 to 212.6(Mpa). The yield strength is increased from 108 to 141(Mpa).

DISCUSSIONS

Porosity: The differences between the real and theoretical densities indicate the presence of porosity. From the calculations the samples 1& 2 got very low porosity values and sample 4 got little bit higher value but the range is acceptable one.

Hardness Values: Hardness tests of the fabricated composite materials revealed its diversification depending on the weight ratios of the reinforcing particles in the Magnesium matrix. The values of investigated composite materials are characterized by the higher hardness compared to the non-reinforced material.

Mechanical Properties: The reduced stiffness and strength of the magnesium alloys set a limit on its applications in the field of automobile and aerospace industries. Magnesium alloy composites can overcome such difficulties with improvement in mechanical properties were proved by addition of hard particles in matrix alloys. The values of Ultimate tensile strength and yield strength for the fabricated samples by vacuum stir casting are listed in the Table 3. It reveals that there were significant improvements in UTS and YS due to grain refinement of particles and matrix. It ensured that there was perfect interfacial bonding between the matrix and reinforcements.

CONCLUSIONS

From the experimental investigations it is concluded that as follows.

- The Vacuum stir casting method is one of the cost effective methods with protected argon atmosphere and easy process to disperse the TiO₂ in the Magnesium matrix.
- Due to the presence of TiO₂, the morphology of the Mg phase is changed to discontinuous and fine. There are no imperfections in the interfacial bonding between the matrix and particles.
- The uniform distribution of particles into the matrix is ensured by the investigations' of SEM.
- The values of density for the prepared composite materials are near to the theoretical one but existing differences indicate presence of porosity.
- The porosity test revealed that stir casting method in stages with argon atmosphere is suitable for preparing Mg-Ti composites with very less porosity.
- The improvement of mechanical properties of composites is attributed to the grain refinement of matrix as well as particles.
- The Hardness, Yield strength and Ultimate Tensile Strength of composites were increased to significant level due to addition of reinforcement particles.
- The addition of the TiO₂ particles of the reinforcing material to the magnesium matrix increased the expected hardness of the composite materials and got the value of 17.8% more than the unreinforced material.

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