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SYNTHESIS AND INVESTIGATIONS OF MAGNESIUM MATRIX WITH TITANIUM OXIDE COMPOSITES BY ARGON CONTROLLED STIR CASTING PROCESS

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ABSTRCT

The investigations of magnesium based matrix reinforced with the TiO_2 particles with various weight ratios of 2.5, 5, 7.5 and 10% are presented in this research paper. The purpose of this work is to elaborate the manufacturing processes of pure magnesium metal with the addition of Titanium oxide through the vacuum stir casting route with the use of Argon as shielding gas to prevent the oxidation. The prepared samples by stir casting are machined with EDM for conducting experiments. The parameters like density, hardness, tensile strength, ultimate tensile strength are evaluated by immersion test, Rockwell Tester and UTM respectively. The presence of particles in phase is examined by XRD analysis. The microstructural examinations of the obtained composite materials ensure that homogeneous distribution of the reinforcing particles in the magnesium matrix was revealed by SEM. The results proved that improvement of 24% in Hardness, 29.8 %in Ultimate tensile strength .

Keywords: Magnesium, Titanium oxide, XRD analysis, SEM analysis, Mechanical properties, Stir casting.

1. INTRODUCTION

Lightweight materials and its applications have always been an important concept in product design for automotive, aviation and where driving dynamics are of major considerations. The increasing challenges to achieve light weight of structural materials, magnesium alloys are potential candidates for various applications. Magnesium based metal-matrix composite castings have the potential to be used as replacements for many ferrous castings in power- train ,chassis including parts of engines, brake, suspension and steering . The corrosion resistance of modern high purity magnesium alloys is better than die cast aluminium materials. The distinct advantages of magnesium over aluminium alloys are lower latent heat fusion that makes longer die life, faster solidification, higher machinability and more precise tolerances due to higher fluidity. The final cost of magnesium components were less than that of aluminium and ferrous parts in large scale production [1].The interest in magnesium alloys for automotive applications is based on the combination of high strength properties, low density durability, design flexibility and reducing relative investment. For this reason magnesium alloys are very attractive as structural materials in all applications where weight savings are of great concern. In automotive applications the weight reduction is the great concern that improves the performance of a vehicle by reducing the rolling resistance and energy of acceleration, thereby reducing the fuel consumption and reduction of the greenhouse gas CO_2 can be achieved [2]. 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 if it 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 [1,3]. The density of magnesium is just about two thirds of aluminium, one fourth of zinc and one fifth of steel. The need for light weight materials with high performance in some demanding applications has led to all-embracing research and development efforts in processing magnesium matrix composites with cost–effective fabrication technologies [4,5]. Magnesium has the highest strength-to-weight ratio of any of the commonly used metals at present. The Advantages of magnesium include: good castability, high die casting rates, electromagnetic interference, shielding properties, part consolidation, dimensional accuracy and excellent machinability. These excellent properties promote its utilization in automobile-related products [6]. Magnesium composites can be attractive candidates for functional and structural materials. The defect free microstructures can be achieved by the use of stir casting process . The major merit of stir casting is its suitability for mass production applications with more economic manner [7]. A 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[8]. The volume fraction of 30% SiC particles added with Magnesium and successfully fabricated by stir casting process [9].In order to get the desirable properties of composites, the reinforcement particles should be stronger and stronger than matrix material [10].The preheating of reinforcement particles increase the surface energy thus increasing wettability between the matrix and particles and improving the strength [11]. The stirring temperature is significant in the process, because that causes clusters of particles, porosity and high oxidation may occur that will degrade the properties of composites [12].The effects of adding yttrium with magnesium are investigated and found that the improvement in grain size, mechanical properties and microstructure [13]. In some high-temperature industrial processes the presence of nitrogen or oxygen gases might cause defects within the material. Argon is sometimes used for extinguishing fires where valuable equipment may be damaged by water or foam [14]. 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 [15]. The technology for machining Magnesium matrix composites is not yet clearly developed yet. The Electrical discharge Machining is suitable for machining the composites without usage of tool and there is no difficulties in disposal of chips [16]. Many researchers reported that reinforcements strengthen the matrix by imparting better mechanical and tribological properties [17-18]. The tensile properties are enhanced by the addition of aluminum, SiC into magnesium by produced through stir casting process[19]. 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.

2. PROPERTIES OF MATERIAL USED.

2.1 Physical properties of Magnesium

Magnesium is the lightest metal among the metals available for structural applications. It is a silvery-white alkaline earth metal and one third lighter than Aluminium. The Magnesium powder or form of ribbon is heated to certain temperature that ignites or burns with an intense of white light and releases large amount of heat. It also burns in pure nitrogen and pure carbon dioxide. Magnesium reacts with cold water very slowly. It forms a thin protective coating of magnesium carbonate when it is get in touch with moist air. The fire produced by magnesium is not extinguished by water, since water reacts with hot magnesium and releases hydrogen which can cause the fire to burn more ferociously. The effective way to stop the burning fire is by using chemical extinguisher and covering with sand. The properties of the material are listed in Table 1. Magnesium may be prepared from electrolysis of fused magnesium chloride, most repeatedly obtained from sea water. It can be used as reducing agent in the preparation of Uranium and other metals that are purified from their forms of salts.

2.2 Physical properties for Titanium

Titanium is present in most igneous rocks and their sediments and it is always found bonded with another element that does not occur in natural pure form. Pure titanium is a transition metal with a lustrous silver-white color and is resistant to corrosion including sea water and chlorine. Titanium has the highest strength to weight ratio of any metal. Even though titanium is used in many products, nearly 95% of the purified metal is used to make titanium dioxide (TiO_2).The corrosion resistance can be improved by forming a thin layer of titanium dioxide (TiO_2) on its surface that is extremely difficult to penetrate for these materials. It is non-magnetic, biocompatible and is not good as to conduct the electricity and heat. The properties of Titanium are listed in Table 2. Many elements like Aluminium, Vanadium and nickel are alloyed with titanium to produce light weight alloys. Its resistance to cavitations and erosion makes it, is an essential structural metal for aerospace and automotive applications. Titanium dioxide (TiO_2) is a whitening pigment used in paints, foods, medicines and cosmetics.

Table 1 Properties of matrix material.

Material	Magnesium
Phase	Solid
Melting point	650 °C [923 K]
Boiling Point	1091 °C[1363 K]
Density	1.738 g/cm^3
Heat of fusion	8.48 kJ/mol
Heat of vaporization	128 kJ/mol

Table 2 Properties of reinforced material.

Material	Titanium
Phase	Solid
Melting point	1668 °C [1941 K]
Boiling Point	3287 °C [3560 K]
Density	4.506 g/cm^3
Heat of fusion	14.15 kJ/mol
Heat of vaporization	425 kJ/mol

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3. EXPERIMENTAL METHODLOGY.

3.1 Sample Preparation

The vacuum stir casting method in stages is used to fabricate five samples. Table 3 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 raw materials used in experiments are represented in Figures 2 &3.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 3 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

Figure 1 Experimental set up.

Figure 2 Magnesium raw material.

Figure 3 Titanium oxide in weighing machine.

Under an applied stress, slip of dislocations and initiation of micro crack can occur in these areas relatively easily, eventually resulting in failure of the material. In the areas of significant segregation or agglomeration of normally highly brittle hard particles, weak bonds are formed in the material which can lead to the reduced mechanical properties of composites. The stirrer is used in stir casting process to avoid segregation of reinforcing particles which is caused by the surfacing or settling of the reinforcement particles during the melting and casting processes. 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 control panel set up to indicate the various temperatures to control the process is as shown in Figure 4.

Figure 4 control Panel setup.

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 5. The first three samples represent the composites after machining and next two represent before machining stage.

Figure 5 Prepared Samples.

4 MICROSTRUCTRE.

4.1 XRD Analysis.

The XRD patterns of the specimens prepared by stir casting process are as shown in Figures 6& 7. 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 Figures 6& 7, 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 TiO_2 is formed completely and a large quantity of molten magnesium fullyinfiltrates through the aperture gap of the particulate.

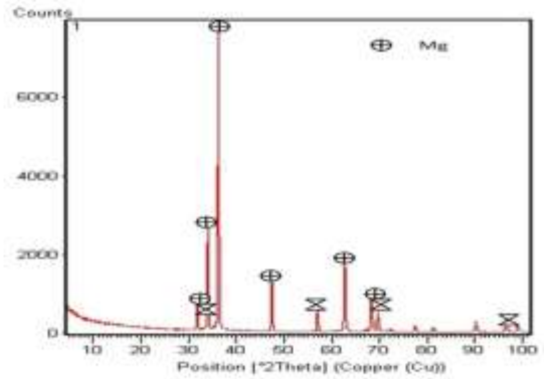

Figure 6 XRD pattern for sample 4

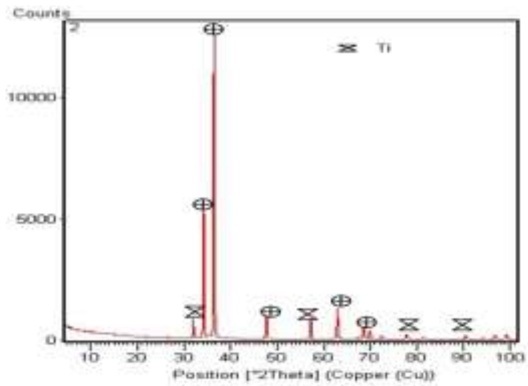

Figure 7 XRD pattern for sample 2

4.2 SEM Analysis.

In order to characterize the microstructure of Mg-TiO_2 composites, Scanning Electron Microscope is used. The interface of Mg and TiO_2 is examined through the SEM. The grain size with its boundaryof pure

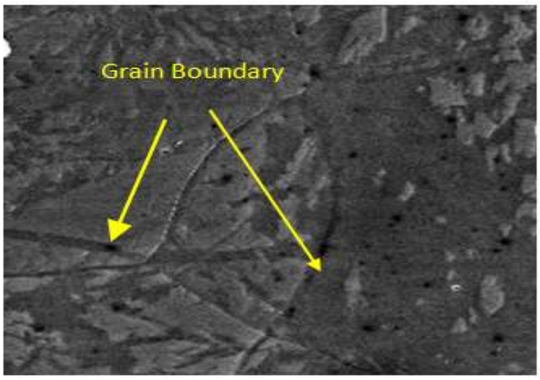

Figure 8 SEM Image for Pure Magnesium Sample.

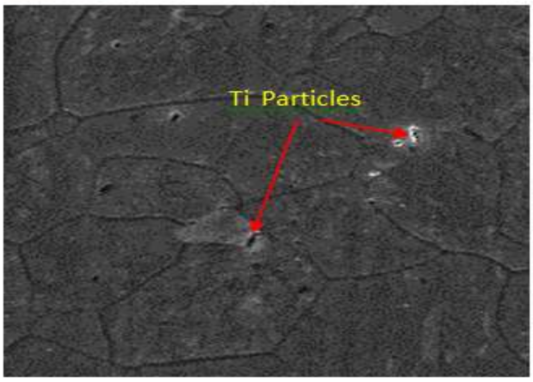

Figure 9 SEM Image for Mg+5% TiO_2 Sample.

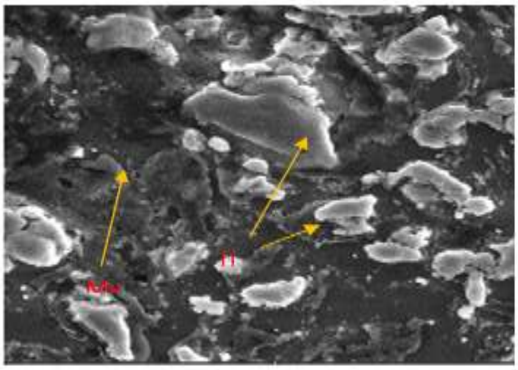

Figure 10 SEM Image for Ti particles in Mg Matrix.

magnesium specimen is presented in Figure 8. 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 TiO_2 particles which is shown in Figure 9. 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 TiO_2 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 TiO_2 in variation in sizes that is not soluble in magnesium matrix is presented in Figure 10. In the magnification of 1000 x shows that very little micro pores are present on the surface.

5. TESTING AND EVALUATION

5.1 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 samples is as shown in Figure 11. 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.

% of porosity = 1 - (measured density /theoretical density) *100

5.2 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 diameter for specimens produced by the stir casting process. Then the average values of each specimens are taken into account and listed in the table.

5.3 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 prepared cylindrical tensile specimens are as shown in Figure 12. 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.

Figure 11 Sample in density Test.

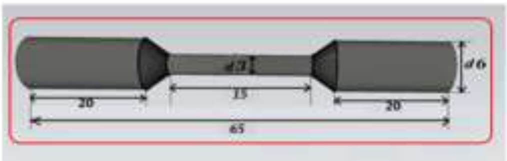

Figure 12 The ASTM standard tensile test specimen.

Figure 13 Tensile test samples

6. RESULTS AND DISCUSSIONS.

6.1 Density and Porosity

The actual density measurements of each samples and their comparison with theoretical values are shown in Table 4. 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.

6.2 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. Mean hardness values of the pure magnesium and of the fabricated composite materials reinforced with the TiO_2 ceramic particles with the weight ratios of 2.5, 5, 7.5 and 10% are shown in Table 5. The values of investigated composite materials are characterized by the higher hardness compared to the non-reinforced material. Hardness of composite materials increases with increasing content of the reinforcing material in the metal matrix

6.3 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

Table 4 Calculation of porosities.

Specimen	Theoretical density g/cm³	Measured density g/cm³	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 5 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

values of Ultimate tensile strength and yield strength for the fabricated samples by vacuum stir casting are listed in the Table 5. 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.

7. CONCLSIONS.

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 TiO_2 in the Magnesium matrix.
- Due to the presence of TiO_2, 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 TiO_2 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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