Synthesis, Characterization And Tribological Investigation Of Magnesium Matrix Composites.


1Faculty Head, Department of Mechanical Engineering, Cape Institute of Technology, Kanyakumari, Tamilnadu, India.
2Assistant Professor, Department of Mechanical Engineering, University College of Engineering Nagercoll, Nagercoll, Tamilnadu, India.
3Associate Professor, Department of Mechanical Engineering, Pondicherry Engineering College, Pondicherry, India.

ABSTRACT
The increasing global demand for magnesium and rapidly growing applications in automotive markets drive the researchers to concentrate in the magnesium based composites. The Environmental and legislative influences will continue to promote the use of magnesium vie-à-vie steel and aluminium. The limitations of monolithic materials can be overcome by addition of reinforcing particles to matrix to get the desired properties of end products. Titanium and its alloys have attracted the industries because they offer high specific strengths and are widely used in the aerospace sectors. The objective of this experimental work is to elaborate the manufacturing processes of pure magnesium with varying percentage of Titanium oxide through the powder metallurgy route and investigating the improvements in mechanical properties. The Rockwell hardness tester and Universal testing machine were used to characterize the composite material. The tribological properties of the developed composite materials were investigated using pin-on-disc wear test apparatus under dry sliding conditions with varying the parameters of load, sliding distance and sliding speed. The mechanical properties are evaluated and the results proved that improvement of 13.8% in density, 17.8% in hardness value and 29% in ultimate tensile strength. The microstructural analysis by scanning electron microscopy (SEM) results revealed that there were uniform compositions throughout the structure of composites. It is concluded that the improvement in hardness and tensile strength is due to interfacial bonding between the matrix and reinforcement particles. The bonding strength was assured by the proper mixing of fine particles with the use of ball and jar mill, compaction pressure and sintering temperature. The wear resistance of the developed composites improved significantly than that of the magnesium matrix due to the upright effect offered by the reinforcements.

KEYWORDS: Magnesium, Titanium oxide, Tribology, SEM analysis, Mechanical properties, Powder metallurgy.

INTRODUCTION
Composite materials could be in the form of natural or synthetic which combines two or more chemically distinct materials in order to achieve improved properties over the individual materials. The density of magnesium is about one third of aluminium that found ideal candidate for light weight applications in automobiles and aerospace industries: [1]. Light-weighting vehicles can improve fuel efficiency and further reduce the dependence on fossil fuel resources used in the transportation industry. In order to improve the effective utilization of magnesium in practical applications the limitations like ductility, formability, corrosion resistance and wear resistance must be overcome by the addition of reinforcements:[2-4]. The strength to weight ratio of magnesium is 21% more than aluminium that will be the driving force for the increasing demand for magnesium composites :[5]. The mechanical properties of composites are controlled by the structure and
properties of the reinforcements: [6-7]. The small elements with near net shape could be manufactured with dimensional stability is the advantage of powder metallurgy process followed by hot extrusion: [8-9]. Reactive with atmospheric oxygen in direct alloying materials that led to use of powder metallurgy techniques which is more flexible than casting, forging or extrusion and achieved improvement in bonding strength, thermo-physical properties : [10]. The size of the matrix and reinforcement decides the uniform microstructure of the composite and relatively non uniform microstructure was obtained when the reinforcement particle size was smaller than the matrix particle size. The average particle size of matrix and reinforcements is 10 microns and the metal matrix samples are fabricated through powder metallurgy route and sintered temp is 550° C and the time in the range of 10-15 min.: [11-12]. The high energy ball mill like planetary ball mill is used to fabricate the nanosized composites to improve the hardness and density of the samples fabricated by powder metallurgy route followed by sintering and isostatic pressing:[13]. Titanium composites are used in high-temperature structures applications. The minimal porosity and hardness, yield strength, ultimate tensile strength were improved:[14-17]. Magnesium with 10%Ti and 1% Al composites were fabricated resulting enhancement of 74% in elastic modulus, 56% in yield strength, 45% in ultimate tensile strength, and 41% in failure strain respectively:[18]. The magnesium metal matrix composites with TiB2 are fabricated with powder metallurgy technique and hardness is increased up to 181%. The titanium alloys are used in the manufacturing of missiles, aircraft naval ships and spacecraft, with around two thirds of all titanium metal produced is used in aircraft engines and frames.: [19]. The hardness of the composites increase with the addition of Ti particles and when the content is 15% its highest value is obtained. The compression strength and the tensile properties depreciate when the Ti content is increased in the matrix composite:[20-23]. It is reported that well dispersed particles in magnesium matrix can improve the wear resistance:[24-26]. Instead of rare earth elements the titanium can be used to reduce the cost of the product:[27]. In this present investigation magnesium with TiO2 composites were fabricated in powder metallurgy route. Also the mechanical properties and tribological behaviour of composites were evaluated. Some researchers concentrated and developed the composites with the reinforcement of Titanium carbide and Titanium boride. From the literature review it is observed that no structured research was performed on this specific area upto present.

MATERIALS AND METHODS

2.1 Physical properties for Matrix:
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.

<table>
<thead>
<tr>
<th>Material</th>
<th>Magnesium</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase</td>
<td>Solid</td>
</tr>
<tr>
<td>Melting point</td>
<td>650 °C [923 K]</td>
</tr>
<tr>
<td>Boiling Point</td>
<td>1091 °C [1363 K]</td>
</tr>
<tr>
<td>Density</td>
<td>1.738 g/cm³</td>
</tr>
<tr>
<td>Heat of fusion</td>
<td>8.48 kJ/mol</td>
</tr>
<tr>
<td>Heat of vaporization</td>
<td>128 kJ/mol</td>
</tr>
</tbody>
</table>

Table 2: Properties of reinforced material.

<table>
<thead>
<tr>
<th>Material</th>
<th>Titanium</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase</td>
<td>Solid</td>
</tr>
<tr>
<td>Melting point</td>
<td>1668 °C [1941 K]</td>
</tr>
<tr>
<td>Boiling Point</td>
<td>3287 °C [5560 K]</td>
</tr>
<tr>
<td>Density</td>
<td>4.506 g/cm³</td>
</tr>
<tr>
<td>Heat of fusion</td>
<td>14.15 kJ/mol</td>
</tr>
<tr>
<td>Heat of vaporization</td>
<td>425 kJ/mol</td>
</tr>
</tbody>
</table>

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 (TiO₂). The corrosion resistance can be improved by forming a thin layer of titanium dioxide (TiO₂) 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 (TiO₂) is a whitening pigment used in paints, foods, medicines and cosmetics.

2.3. Experimental Methodology:
Powder Metallurgy methodology is used to fabricate the samples in which materials or components are made from metal powders. This process has an influence to get near expected shape and avoid or greatly reduce the need to use metal removal processes, thereby considerably reducing yield losses in manufacture and often ensuing in lower costs.

2.3.1 Synthesis:
Even though various techniques are available for the fabrication of metal composites, Powder metallurgy makes materials properties relatively easy to control by mixing materials with different properties in various proportions. The processes involve collection of matrix and reinforcement particles in powder form and then they are blended by using vibratory ball mill in order to get fine grain sizes. Then the mixture is placed in the dies setup and compacted by cold compaction process. The samples are entered into the sintering process to improve the bonding between the matrix and particulates. The extruded billets are then machined to the desired sizes for the conduction of tests like density, hardness and tensile tests. The purchased matrix material used in this research work is pure magnesium powder of 100 microns. The Reinforcement material titanium oxide of particle size 100 microns. It was reported that particle size of matrix and reinforcement particles have a substantial effect on the hardness, microstructure and mechanical properties of the composite. In order to get uniform microstructure the selection of both particle sizes are very important. Hence in the present work both matrix and TiO₂ of equal size particle size of 100 microns are chosen. Fig 1 and Fig 2 represent the appearance of matrix and reinforcement materials in powder form.

The powders are milled to the requirement size of 10 microns separately. The weighed powder for the five proportions is mixed (0%, 2.5%, 5%, 7.5% and 10% of mass) in a high energy ball mill from VBCC Ltd Chennai. Since the particle size of matrix and the reinforcements are 10 microns which is adequate for compaction ball milling was focused only on mixing of 60 min with a speed of 45 rpm for all the reinforcement composition. Fig. 3 shows the arrangement of ball and jar mill used for blending the composites. Powder compaction is the process of compacting metal powder(s) in a die through the application of high pressures or load. The compaction of the powders has been carried out in a standard die – setup to prepare the specimens. The arrangement of die set up is as shown in Fig 4. Then the compactions were carried out in a hydraulic press attached with the universal testing machine with compacting pressure of 650 Mpa is given to carry out the uni-axial pressing of specimens. Zinc stearate is acted as lubricant to improve the powder processing properties of formulations. The purpose of adding lubricant is to decrease the friction at the interface between billet surface and the die wall during ejection so that the wear on punches and dies are get reduced. Typically the tools are held in the vertical orientation with the punch tool forming the bottom of the cavity. The powder is then compacted into a shape and then ejected from the die cavity. The die was manufactured by using the material ASTM D2 high carbon steel, hardened, tempered and carbide inserted. Punch and die were prepared to close tolerances.
Table 3: Percentage of particles in specimens

<table>
<thead>
<tr>
<th>Sample</th>
<th>proportion</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Pure magnesium</td>
</tr>
<tr>
<td>2</td>
<td>Mg+ 2.5 % TiO$_2$</td>
</tr>
<tr>
<td>3</td>
<td>Mg+ 5 % TiO$_2$</td>
</tr>
<tr>
<td>4</td>
<td>Mg+ 7.5 % TiO$_2$</td>
</tr>
<tr>
<td>5</td>
<td>Mg+ 10 % TiO$_2$</td>
</tr>
</tbody>
</table>

This process step involves heating of the mixed materials, usually in a protective atmosphere, to a temperature that is below the melting point of the major constituent. Under the use of heat the bonding takes place between the porous aggregate particles and once cooled the powder has bonded to form a solid piece. Sintering was carried out for prepared billet samples upto 550°C with the holding time of 20 min in Muffle sintering furnace at a variation in rate of heating up to 10°C per minute. After the sintering process the specimens were allowed to cool up to 250°C inside the furnace to avoid atmospheric contamination and sudden cooling and finally to room temperature. Some prepared samples are shown in Fig. 5.

2.4. Microstructure:

The Microstructure examination of cross-sections from the prepared specimens revealed that there were equiaxed grains with uniformly distribution of Ti O$_2$ within the magnesium matrix are shown in Fig 6 (a-f). The sizes of the pure magnesium powder and of the reinforcing TiO$_2$ powder particles were determined based on observations of SEM. It was found out that particles of powders used for fabrication of composite materials were irregular in shape and their size did not exceed 100 μm for the magnesium powder r and 25 μm for the TiO$_2$ powder. The Metallographic examinations of the composite materials after the fabrication of samples revealed the uniform distribution of the TiO$_2$ reinforcing particles in the aluminum matrix. The structure obtained from the observation ensures the perfect bonding between the matrix and particles of composites. In the magnification of 1000 x shows that very little micro pores are present on the surface.
Fig. 6: SEM images a) magnesium powder particles b.) TiO$_2$ powder particles c.) Mg + 2.5% TiO$_2$ sample d.) Mg + 5% TiO$_2$ sample e.) Mg + 7.5% TiO$_2$ sample f.) Mg + 10% TiO$_2$ sample

2.5. Testing:

The density of prepared specimens was estimated with Archimedean principle, by determining the specimen mass and volume before and after the immersing the specimen in air and in distilled water. The hardness tests of the fabricated composite materials were made on Rockwell hardness tester. The samples were machined up to the required size to conduct the experiments. The macro hardness of polished cross-sections was determined on the Rockwell 15 T superficial scale using a 1/16 in. diameter steel ball indenter with a 15 kg major load, in accordance with the ASTM E18-92 standard. Three indentations were made on each of the transverse section of samples. The hardness values are estimated for both pure alloy and fabricated composite materials reinforced with the TiO$_2$ phase particles. Finally the average hardness of each samples were calculated and plotted as a curve. Static tensile tests of the fabricated composite materials were made on the ZWICK 100 type testing machine at room temperature. The cylindrical tensile specimens of 5 mm diameter and 18 mm gauge length according to ASTM E8M-96 standard. The samples were machined from the extruded bars. The Yield stresses (YS), ultimate tensile strength (UTS) and Young module (E) were determined employing at least
two specimens for each material. Dry sliding wear tests were performed in accordance with the ASTM: G99-05 test standards using pin-on-disc equipment. The counter disc material was made of EN31 steel. The tribological performance of the composites were studied as a function of reinforcement content (wt. %), sliding speed (m/s), applied load (N) and sliding distance (m).

RESULTS AND DISCUSSIONS

3.1 Density Measurement:
The density measurements and their comparison with theoretical values are shown in Figure 7. The differences between the real and theoretical densities indicate the presence of porosity. The results proved that addition of reinforcements into the matrix increase the overall density of the composites:[21].

3.2 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 TiO₂ ceramic particles with the weight ratios of 2.5, 5, 7.5 and 10 are shown in Figure 8. The values of investigated composite materials are characterized by an higher hardness compared to the non-reinforced material. The Hardness of composite materials increases with increasing content of the reinforcing material in the metal matrix.[21,27,28].

3.3 Mechanical properties:
The measured values of mechanical parameters are represented in the Figures 9,10 and 11 respectively. From the results it is found that there will be significant improvements in mechanical properties of the composites due to reinforcement of titanium oxide particles. The mechanical properties are evaluated and the results proved that improvement in 13.8%, 17.8% and 29% for the density, hardness values and universal tensile strength. The results proved that the interfacing between the matrix and particles decide the strength of the composite:[21,27,29]. There was little bit reduction elastic modulus.

![Comparsion of Theoretical density & Measured density](image)

**Fig. 7:** Graphical representation of density values.
Fig. 9: Graphical representation of UTS values.

Fig. 8: Graphical representation of Hardness values.
3.4 Wear behaviour

The sliding speed is kept as constant with the variation of loads from 5N to 30N the wear losses were measured and represented in Fig 12. The Variation in load helps us to understand the behavior of candidate material during metal to metal contact even under higher loads. It is noticed that while the percentage of reinforcement increases conversely the wear loss of specimen decreases. An increase in wear loss of Mg–TiO$_2$ composites at high loads which may be due to the brittle nature of the reinforcement particles: [30].

The presence of ceramic reinforcements raises the wear resistance of matrix materials at a lower range of applied loads. The variation in distance could easily be acknowledged for the want of continuous sliding over the hard surface for a longer period and by the way increase temperature at the interface. The increase in temperature may cause to soften the specimens which results in increased wear loss. Reduction in wear loss of the specimens with significant increase in Ti particles quantity at higher sliding distances may be due to the wear

![Graphical representation of yield strength values.](image1)

![Graphical representation of young’s modulus values.](image2)
resistance offered by the interfacing effect of magnesium and reinforcements:[31,32]. The wear losses with varying sliding distances were presented in Fig 13. The wear losses for the varying sliding speed from 0 to 3 m/s are presented in the Fig 14. From the interpretation it is observed that the wear loss decreases for increase in sliding speed invariably to the quantum of reinforcements. When the sliding speed increases, thinner and less adherent lubricant layer peels off from the surface of the composite and in turn forms an oxide layer which prevents the test specimen from further loss. The increase in sliding speed decrease the contact time between pin and disc resulting for a down trend in wear loss:[33].

![Load vs Wear loss](image)

**Fig. 12:** Wear loss for varying loads.

![Sliding distance vs Wear loss](image)

**Fig. 13:** Wear loss for varying distance
Fig. 14: Wear loss for varying distance.

Conclusions:
From the experimental investigations of the composites, it is concluded that as follows:
- The Powder metallurgy method is suitable for producing magnesium based composites with an economic manner and near net shape can be manufactured.
- 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 can be minimized by improving the wettability.
- The improvement of mechanical properties of composites is due to perfect interfacing between matrix and 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.
- The percentage of reinforcement increases in composites conversely the wear loss of specimen decreases.
- The increase in sliding distances causes the increase in temperature that further soften the specimens which results in increased wear loss.
- An increase in wear loss of Mg–TiO$_2$ composites at high loads which may be due to the brittle nature of the reinforcement particles.
- The wear loss decreases for increase in sliding speed invariably to the quantum of reinforcements.

Future work:
In high temperature applications the magnesium based composites have some limitations. The limitations can be overcome by addition of suitable materials. Hybrid composites can be fabricated to resolve the problems for high temperature applications. The future studies can be made to determine suitable materials for the coatings to improve the wear resistance and corrosion resistance of the composites.

REFERENCES
33. Tee Zhen Wei, Siti Rahmah Bt Shamsuri, Chang Siang yee, Mohd Warikh Abd Rashid, Qumrul Aksan, 2013. Effect of sliding velocity on wear behaviour of magnesium composite reinforced with SiC and MWCNT, Procedia Engineering, 68: 703-709.