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Synthesis and Investigation of Magnesium Matrix Composite with Titanium Oxide by Powder Metallurgy

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Abstract: The investigations of magnesium based matrix material 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 with the addition of Titanium oxide through the powder metallurgy route. The powder blending was done with the energetic ball and jar mill. The compaction pressure of 650Mpa is given to improve cold compaction. The Sintering temperature of 550 °C and holding time of 30 min was used to sinter the compacts to get better results. The microstructural analysis by scanning electron microscopy (SEM) for the starting powders and fabricated samples are carried out for the investigation. 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.
Keywords: Magnesium, Titanium oxide, SEM analysis, Mechanical properties, Powder metallurgy.

I. 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. Usually matrix is a ductile or tough material and reinforcing materials are strong with various properties and in the form of particles, platelets, whiskers, short fibers, and continuously aligned fibers are combined together to get desired properties, otherwise that was not available in any single conventional material. The incorporation of reinforcement increases stiffness, specific strength, design flexibility, corrosion resistance, fracture resistance, durability and reducing relative investment. The advanced composites are used in aerospace, sporting goods, military and commercial aircraft, etc. magnesium based composites used in space and satellite structures, Antenna structures, Helicopter transmission structures with the reinforcing particles of Graphite, boron, alumina respectively. The composites can be used in thermal management and support systems for electronic packages like integrated circuit materials. Magnesium is useful in manufacturing of electronic devices such as cameras, laptops and mobile phones[1-3]. It is well known that the elastic properties of metal matrix composites are strongly influenced by micro structural parameters of the reinforcement such as shape, size, orientation, distribution and volume fraction [4]. The mechanical properties of composites are controlled by the structure and properties of the reinforcements [5]. There are several metal matrix processing technologies available in modern world era. The powder metallurgy process followed by hot extrusion and casting methods is playing vital roles among them. The major advantage of powder metallurgy over other methods is that small elements with near net shape could be manufactured with dimensional stability, whereas manufacturing of locally reinforced elements from composite materials with excellent surface quality shall be obtained by using pressure infiltration method [6-8]. There were certain constraints on manufacturing that imposing unwelcome chemical and thermal properties and some substances 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 [9-10]. By using powder metallurgy route the aluminium, titanium and vanadium particles reinforced with magnesium matrix composites are prepared and results showed that certain improvements in mechanical properties like yield strength, Ultimate tensile strength and elastic modulus than MB15 alloy [11]. 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. [12-13]. The high energy ball

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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, and best hardness was obtained by more than 70 hours of milling with a speed of 350 rpm and particle size of steel is reduced to less than 100 nm.[14].Titanium with SiC composites are used in high-temperature structures applications. The nanosized hybrid particles of SiC and Al_2O_3 are reinforced with magnesium material by using powder metallurgy route involving microwave sintering followed by hot extrusion and results obtained with minimal porosity and fairly well distribution and found that improvements in hardness, yield strength and Ultimate Tensile Strength [15].The reinforcement of varying B_4C particles with Magnesium metal matrix composites are fabricated with minimal micro porosity by powder metallurgy technique and results revealed that improvements in hardness and wear resistance [16]. The magnesium metal matrix composites with TiB_2 are fabricated with powder metallurgy technique and increase in hardness from 41% 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 [17].The usage of composite materials gains prominence when it is reinforced with hard ceramic particles which can be used to control the tribological properties. This can be obtained with various composition of the reinforcing particle in the matrix. 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 [18-19].The magnesium alloys can be tailored to bio-medical applications with adjustable corrosion rates specific mechanical properties and biodegradable cardiovascular magnesium stents for the advanced clinical applications [20-23].

II. PROPERTIES OF MATERIAL USED

A. 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.

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 1 Properties of matrix material.

B. 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.

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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

Table 2 Properties of reinforced material.

III. EXPERIMENTAL METHODLOGY

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.

A. 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.

1) *Powders:* 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 TiO_2 of equal size particle size of 100 microns are chosen. Fig 1 represents the appearance of matrix material in powder form.

Fig 1 Magnesium powder.

2) *Powder Blending:* The powders are milled to the requirement size of 10 microns separately. The weighed powder for the five proportions are 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. 2 shows the arrangement of ball and jar mill used for blending the composites.

Sample	proportion
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

Table 3 Percentage of particles in specimens

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Fig. 2 Ball and Jar mill.

3) *Cold Compaction:* 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 3.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.

Fig 3 Die setup .

4) *Sintering:* 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^{o} C with the holding time of 20 min in Muffle sintering furnace at a variation in rate of heating up to 10^{o}C per minute. After the sintering process the specimens were allowed to cool up to 250^{o}C inside the furnace to avoid atmospheric contamination and sudden cooling and finally to room temperature. some prepared samples are shown in Fig. 4.

Fig.4. Samples.

IV. MICROSTRUCTURE

The Microstructure examination of cross-sections from the prepared specimens revealed that there were equiaxed grains with uniformly distribution of Ti O2 within the magnesium matrix.The sizes of the pure magnesium powder and of the reinforcing TiO_2

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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 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.

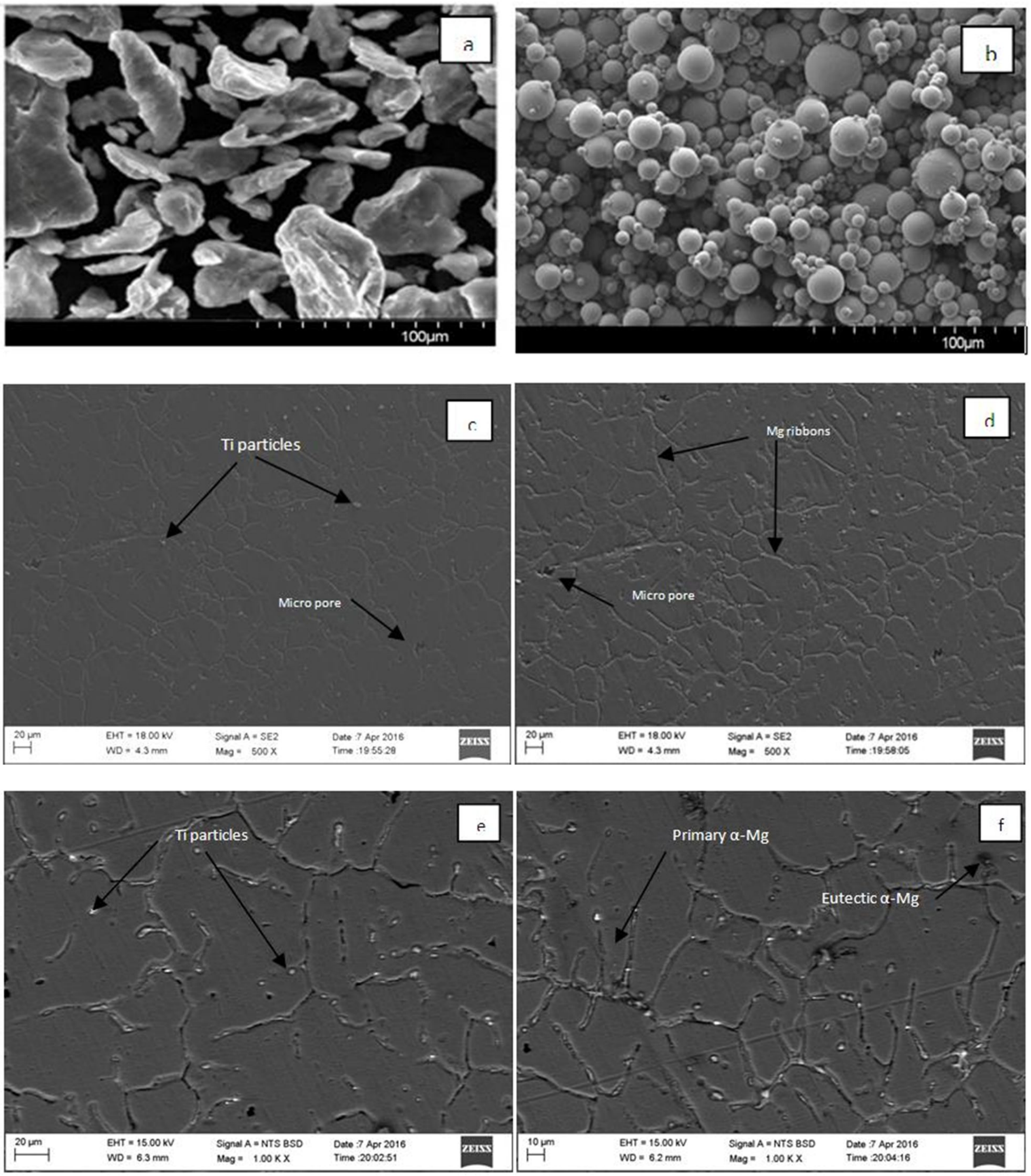

Figure 5 SEM images a) magnesium powder particles b.) TiO_2 powder particles c.) Mg + 2.5% TiO_2 sample d.) Mg + 5% TiO_2 sample e.) Mg + 7.5% TiO_2 sample f.) Mg + 10% TiO_2 sample

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V. 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 macrohardness of polished cross-sections was determined on the Rockwell 15 T superficial scale using a 1/16 in. diameter steel ball indentor 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.

VI. RESULTS AND DISCUSSIONS

A. *Density Measurement*

The density measurements and their comparison with theoretical values are shown in Figure 6. The differences between the real and theoretical densities indicate the presence of porosity.

B. *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 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

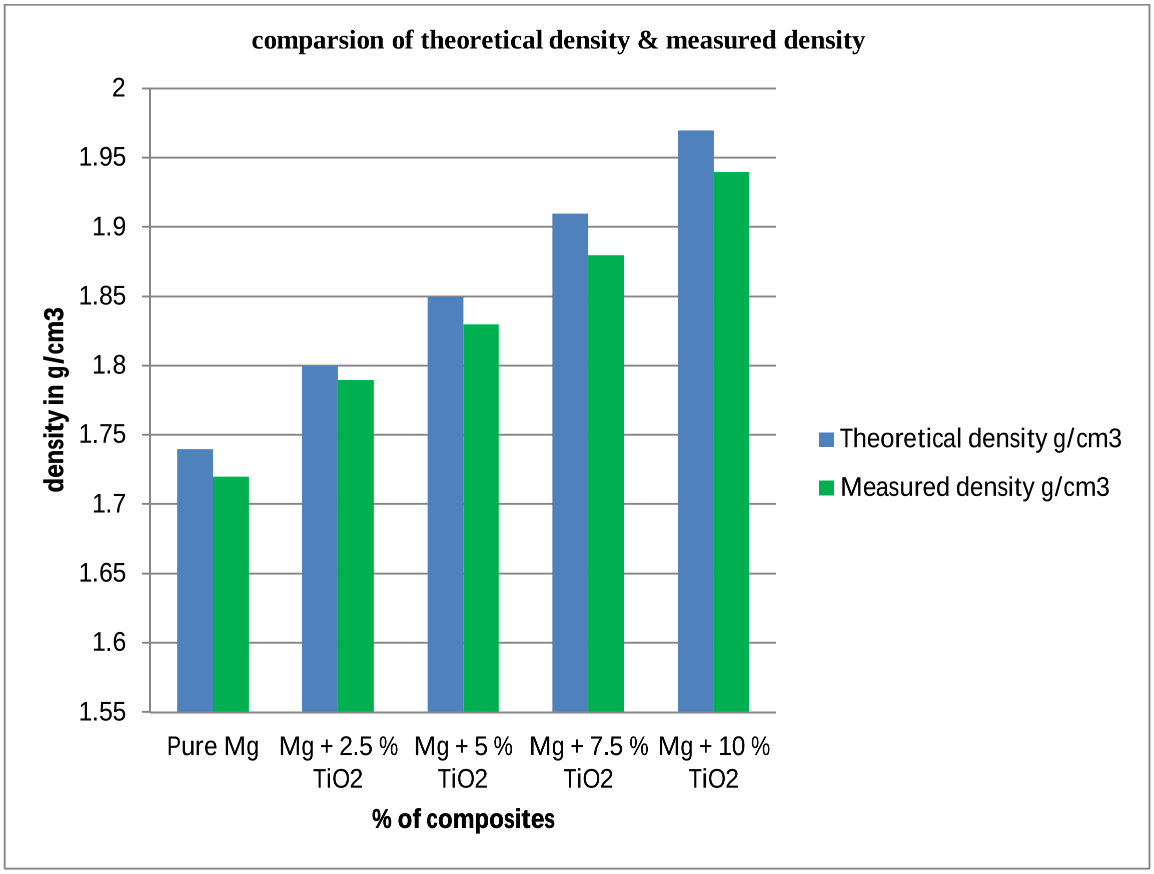

Figure 6. Graphical representation of density values.

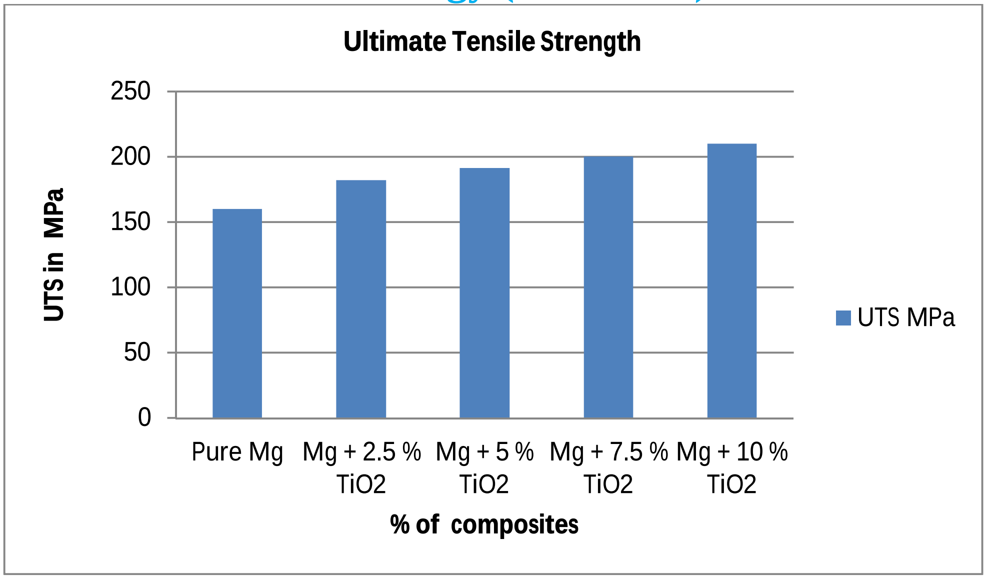

Figure 7. Graphical representation of UTS values.

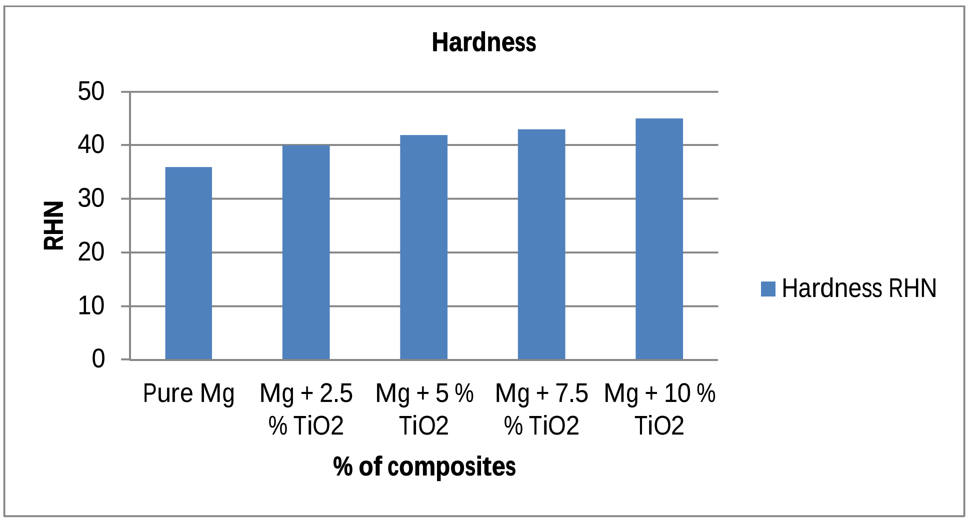

Figure 8 . Graphical representation of Hardness values.

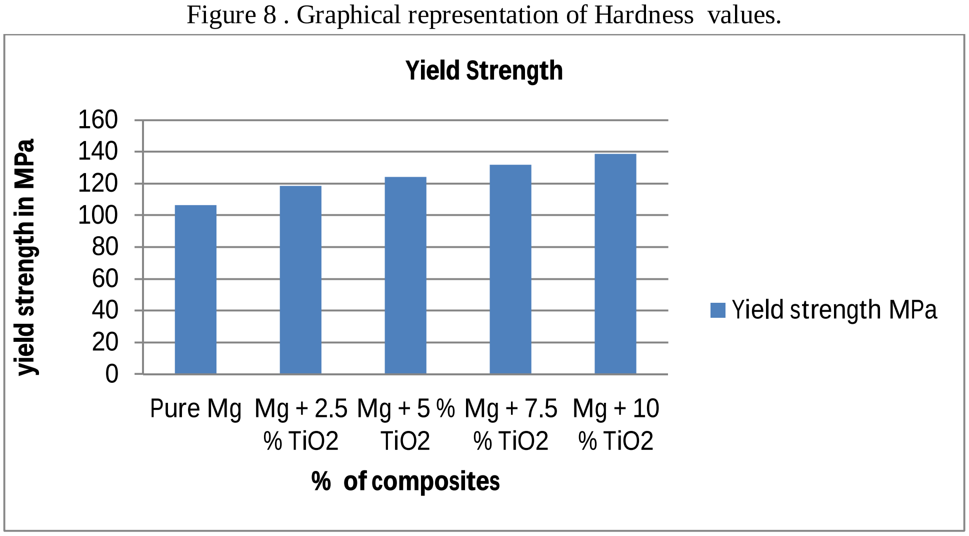

Figure 9. Graphical representation of yield strength values.

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The measured values of mechanical parameters are represented in the Figures 7,9 and 10 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. There was little bit reduction elastic modulus.

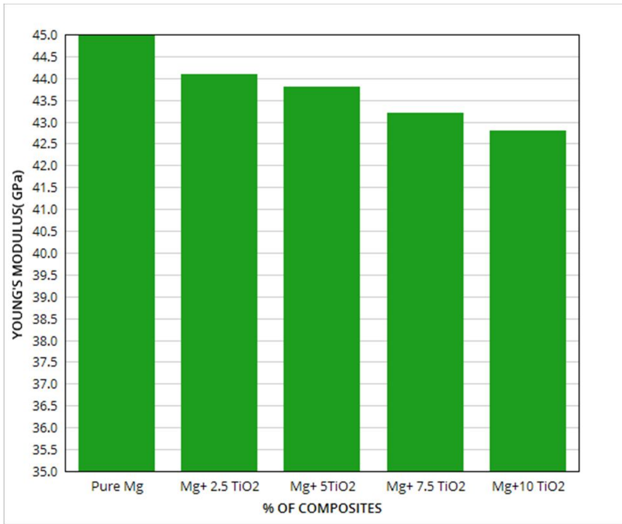

Figure 10. Graphical representation of young's modulus values.

VII. CONCLSIONS

From the experimental investigations of the composites ,it is concluded that as follows..

A. The Powder metallurgy method is suitable for producing magnesium based composites with an economic manner.

B. 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.

C. The uniform distribution of particles into the matrix is ensured by the investigations' of SEM.

D. The values of density for the prepared composite materials are near to the theoretical one but existing differences indicate presence of porosity..

E. The improvement of mechanical properties of composites is due to perfect interfacing between matrix and particles.

F. The Hardness, Yield strength and Ultimate Tensile Strength of composites were increased to significant level due to addition of reinforcement particles.

G. 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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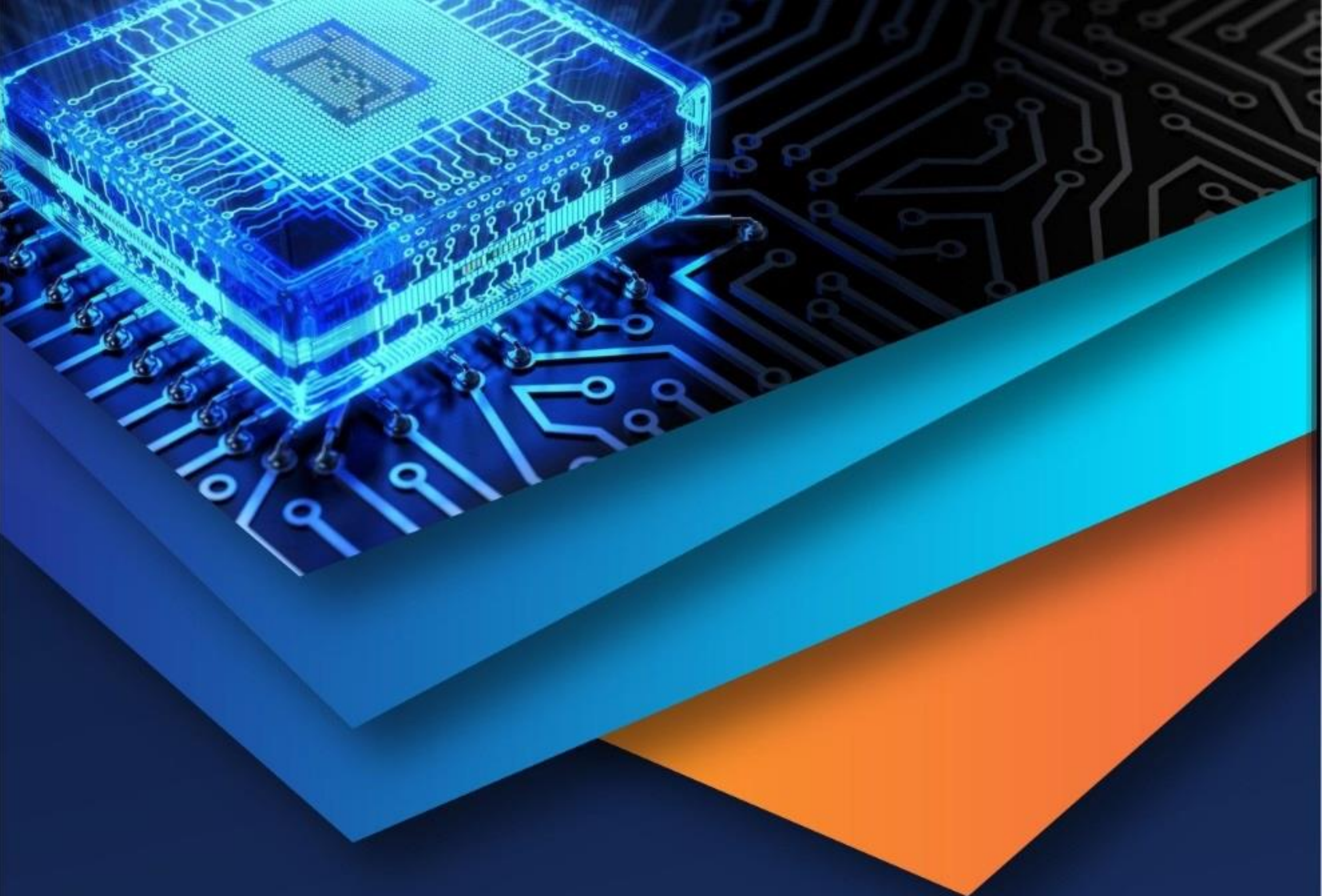

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