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Study on Mechanical Behavior of Nanoparticulate Reinforced Magnesium Matrix Composite

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Abstract:- In this study, nano-sized titanium dioxide particles were reinforced with pure magnesium by powder metallurgy technique for five different weight fractions of reinforcement such as 4 wt%, 8 wt%, 12 wt%, 16 wt%, and 20 wt% respectively. Samples of these compositions are iso-statically cold compacted into cylindrical specimens of diameter and length each 15 mm respectively, followed by sintering and hot extrusion with an extrusion ratio of 1.56:1. SEM micrographs of blended composite powders before compaction confirmed the distribution of reinforcement has been uniform throughout the matrix with no presence of any agglomeration. Micro structural analysis of extruded specimens revealed the significant matrix grain growth. The reinforcement may interrupt the matrix grain growth on grain boundaries during extrusion. Targeting for biomedical applications like bone implants, orthopedic surgery etc., the mechanical behavior of the composite such as micro hardness, and compressive strength have been studied. It was observed from the study that the mechanical properties have been significantly improved by adding reinforcement compared to pure magnesium and better mechanical behavior have been noticed for composite with higher contribution of reinforcement.

Keywords:- *Magnesium Matrix Composite, Nanoparticulate, Titanium Dioxide, Biomedical, Bone Implants, Mechanical Behavior.*

I. INTRODUCTION

During the past few years, the researchers focused majorly on magnesium due to its attractive low density. Even though it has low density and good mechanical properties it suffers from certain barriers such as explosivity, poor ductility, poor wear, and corrosive nature which hindered it from being used in aerospace, automobile industries and much more fields in its pure form. Currently, researchers are working on adding reinforcements to magnesium metal matrix to improve its mechanical properties. Recent reviews has highlighted that there are about 60 types of components starting from instrument panels to engine components which has magnesium alloys such as cast alloys-AZ63, AZ81, AZ91 etc., wrought alloys-AZ31, AZ61, AZ80 etc., Elektron, Magnox, Magnuminium, Mag-Thor, Birmabright, Magnalium and ceramic reinforcements such as SiC, Al_2O_3, Si, Ti, Zr are added to magnesium are developed for use. The uses of magnesium alloy in automobile parts is predicted to increase at an average rate of 15% per year. Magnesium alloys are used in aircrafts, missiles, automobile wheels, housings, ladders, cell phones, power tools, textile machinery, seat frames etc. The next major field focusing on the usage of magnesium alloys is the biomedical field. Biomedical field has been using materials such as 316L SS, CoCrMo, Ti6Al4V in various implants division ranging from cardiovascular to otorhinology. These Implant alloys are fabricated using methods like investment casting, powder metallurgy, thermo-mechanical process(TMP), super plastic deformation (SPD), equal channel angular pressing (ECAP), accumulative roll-bonding (ARB), high-pressure torsion (HPT) etc. Magnesium can be used for bone implant fabrication since the modulus of the metal and bone is similar (40-45GPa) and hence secondary surgery for removal of the implant will not be necessary. But since pure magnesium is poor in certain mechanical properties, titanium dioxide is added to magnesium as reinforcement in powder metallurgy technique and the properties are studied.

The following are the studies on material currently used in biomedical applications. Jian Fang Li et al had worked on 316L SS by layering the alloy of Zr and ZrO_2 in plasma surface alloying apparatus. The cross-section microscopy, surface, phase structure was analyzed and the surface roughness, wear were also measured. It is found that ZrO_2 layers tremendously enhance wear resistance, improve adhesion and spreading of osteoblast cells [1]. M.M. Machado Lopez et al observed the property enhancement of 45s5 bioglass coating on Ti6Al4V alloy by a colloidal electrophoretic deposition process. The study included X-ray diffraction, chemical composition analysis, and coating evaluation. The result was that the corrosion resistance was increased and ensured good protection from ionic attack [3].

Several studies in magnesium reinforced with various particulates are discussed as follows. Manoj Gupta et al analyzed the Al and Mg-based nanocomposites synthesized using powder metallurgy and extruded for characterization study. Density, microstructure characterization, X-ray diffraction analysis, tensile test, microhardness measurement was studied. It was observed that the hybrid reduced energy and time, hardness and work of fracture improved, and the processing time was considerably reduced [4]. Junko Umeda et al had studied on magnesium reinforced with titanium particulates by spark plasma sintering which was followed by hot extrusion. The optical microstructure, compression strength, tensile strength and

X-ray diffraction were studied. The composite material resulted in increased compression strength but there was no improvement in tensile strength [5]. Ganesh Kumar Meenashisundaram et al had worked on titanium (IV) oxide nanoparticulate reinforced magnesium composite material done by disintegrated melt deposition technique which was followed by hot extrusion. The materials characterization were studied such as microhardness, compression, tensile, thermal expansion and fracture behavior. The composites resulted in increased hardness and fracture strain was increasing with increase in the addition of titanium (IV) oxide [6]. Cheng-Jie Li et al has worked on Mg-3Zn-0.2Ca-0.5Y alloy using extrusion of billet at different temperature after heat treatment. The compression strength, microstructural study, and tensile strength have been studied. It resulted in increased grain size with increase in extrusion temperature [7]. AlirezaVahid et al had worked on magnesium reinforced with tantalum particles using sintering of compacted composite powder. Density, X-ray diffraction, microstructure study and thermal expansion behavior has been studied. The results of the above composites are increase in microhardness and compressive strength [9]. Dongfang Zhang et al has worked on hafnium coated magnesium alloy using sintering in tube furnace of hafnium coated magnesium. Crystal structure, electrochemical and corrosion test has been studied. The results are improved corrosion resistance with a change of microstructure and production of surface oxidation [10].

It was found from the literature that there is a wide scope for developing light weight composites targeting biomedical applications. The attempt is made with commitment to develop and characterize a light weight component which will be discussed in detail in the next section.

II. EXPERIMENTAL PROCEDURE

A. Materials

Magnesium powder supplied by S D Fine-Chem Limited with mean particle size 177µm and >99.995% purity is used as the base material. Titanium Dioxide particles of mean size <45 µm and purity >99.5% supplied by Merck Life Science Private Limited is chosen as reinforcement.

B. Procedure

➢ *Particle Size Conversion.*

Titanium Dioxide particle with average size of <45 µm was converted to an average particle size of 100 nm using planetary mill which included a jar with 16 metal balls (ball to powder ratio is 5:1) for size conversion process. This process took 17 hours at 250 rpm in ball mill.

➢ *Elemental Mixture of Mg-TiO2 Powders.*

Titanium Dioxide reinforcement was added to magnesium metal in five different weight fractions such as 4 wt%, 8 wt%, 12 wt%, 16 wt%, and 20 wt% respectively. This process of blending was carried out in the planetary mill at 200 rpm for 10 minutes for each composition.

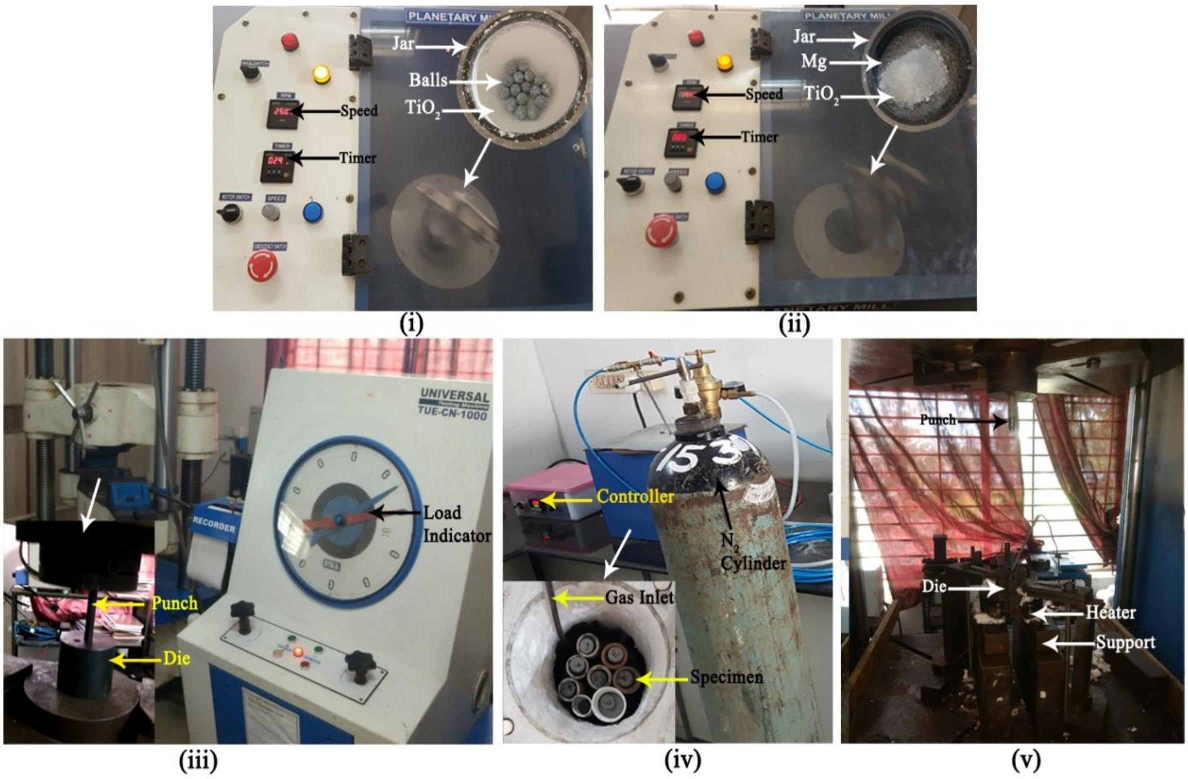

Fig 1:- Sequence of operations – i) Particle size conversion ii) Elemental mixture iii) Compaction iv) Sintering v) Hot extrusion.

➢ *Cold Compaction*

Compaction is a process of converting the powder form of the composite into a thick solid form to enable to conduct various tests on the specimen to tabulate its properties. The blended composite mixture was compacted at room temperature. The amount of powder weighed for each sample was 4.5 grams which produced a cylindrical billet of diameter and length of each 15 mm. This process was carried out in a Universal Testing Machine (100T) where the compaction die filled with weighed powders is fixed and a load of 50kN was applied using a punch. This load was held steady for 5 minutes to get applied completely to produce a solid specimen.

➢ *Sintering*

Sintering process was done in two stages. In stage 1, the compacted specimens were sintered at 500℃ for 4 hours in a muffle furnace at nitrogen atmosphere to prevent reaction of the specimen with air. A tray was prepared, and specimens were arranged in the tray in an individual crucible and placed inside the furnace. The gas was passed through a ceramic tube at 0.5 to 1 bar pressure into the chamber. Eight samples were heat treated at a time and took approximately 6 hours for one complete sintering process. The stage 2 sintering process is similar to stage 1, but the only difference is that the heating temperature and the time of sintering were reduced. Sintering was done at 400℃ for 1 hour at nitrogen atmosphere. The second stage sintering was done to improve growth of grain boundary. And it took 1 hour to reach 400℃ and the furnace was held at 400℃ for 1 hour.

➢ *Hot Extrusion*

The extrusion process is done to improve the matrix grain growth. An extrusion die had to be fabricated that supports the dimensions of the specimen. The extrusion temperature range for magnesium is 250℃ to450℃. The reduction percentage was 20% in diameter i.e. 15mm diameter was reduced to 12mm through extrusion. And hence the extrusion ratio is 1.56:1. The taper angle of the die is 14^o and length of taper is 10mm. The specimen was pre-heated at inert atmosphere to prevent reaction of specimen with air during heating. The die was pre-heated to 200℃ using band heater and the specimen was pre-heated to 350℃ in muffle furnace. The pre-heating of die was done to provide a smooth extrusion and to reduce the load acting on die. The extrusion process was done in 100T hydraulic press. A tapered punch of 15 mm diameter and 50 mm length along with a 10 mm length taper similar to die's internal taper was provided at end of the punch. This punch was fixed at the top die of hydraulic press and then pressure was applied with lubrication to reduce friction between punch and die during extrusion process. The dimension of extruded specimen obtained was 12 mm in diameter and 18 mm in length.

C. Material Characterization

➢ *SEM Analysis of Powder Blend*

This test is conducted to investigate the TiO_2 distribution in magnesium metal in the process of elemental mixture. The analysis was done on powders obtained after elemental mixture of Mg-TiO_2 in scanning electron microscope (SEM) in ASTM E986 – 04(2017) standards.

➢ *Microstructural Characterization*

This test is conducted to investigate the TiO_2 distributions and grain growth of Mg-TiO_2 nanocomposites. This microstructural characterization was done on polished surface of extruded composite samples in scanning electron microscope (SEM) in ASTM E986 – 04(2017) standards.

➢ *Density Measurements*

The density of extruded magnesium (4, 8, 12, 16 and 20) % TiO_2 was measured using Archimedes principle in ASTM D792 – 13 standards. Each sample was measured for 3 cycles to obtain accurate density value. Water was used as the liquid which has a density of exactly 1 g/cm^3.

➢ *Microhardness Test*

Using a microhardness tester-Wolpert Group with Vickers indenter, the Microhardness tests were conducted on flat and polished extruded specimens in ASTM E384 – 17 standards. Each sample was measured for 3 cycles to obtaine the hardness accurately.

➢ *Compression test*

The compressive properties of extruded Mg (4, 8, 12, 16 and 20wt%) TiO_2 samples was determined at room temperature in ASTM E9 – 09(2018) standards. Each sample was tested 3 times to ensure repeatable values.

III. RESULTS AND DISCUSSION

A. SEM image of powder

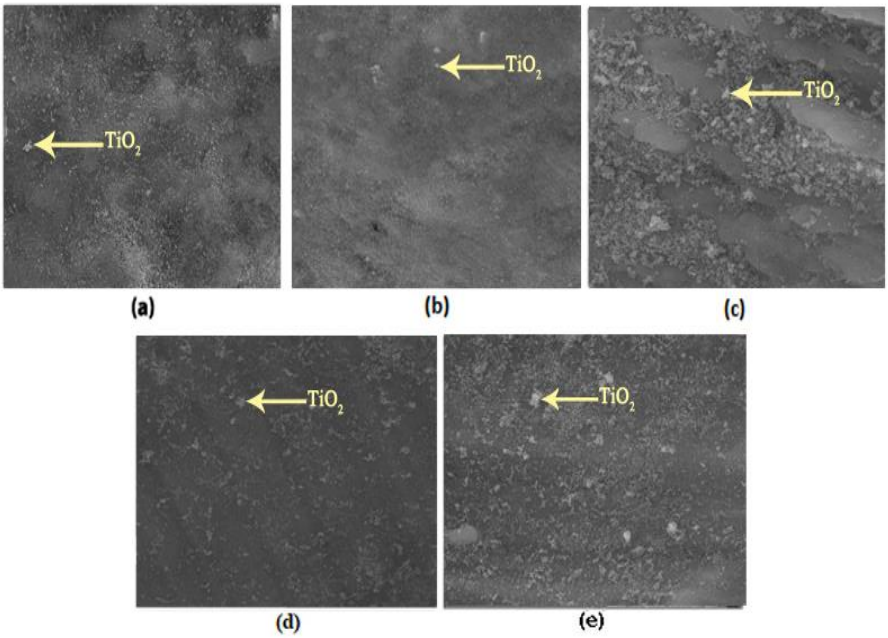

Fig 2:- SEM images that showing distribution of nano-TiO_2 particulates in Mg-TiO_2 powder mixture of different composition.

SEM analysis was conducted on the elemental mixture of Mg-TiO_2 to confirm the uniform distribution of TiO_2 reinforcement over Mg matrix in five different weight percentages. Only if there is uniform distribution, the properties of the resulting matrix would be improved along with a grain growth.

B. SEM micrograph

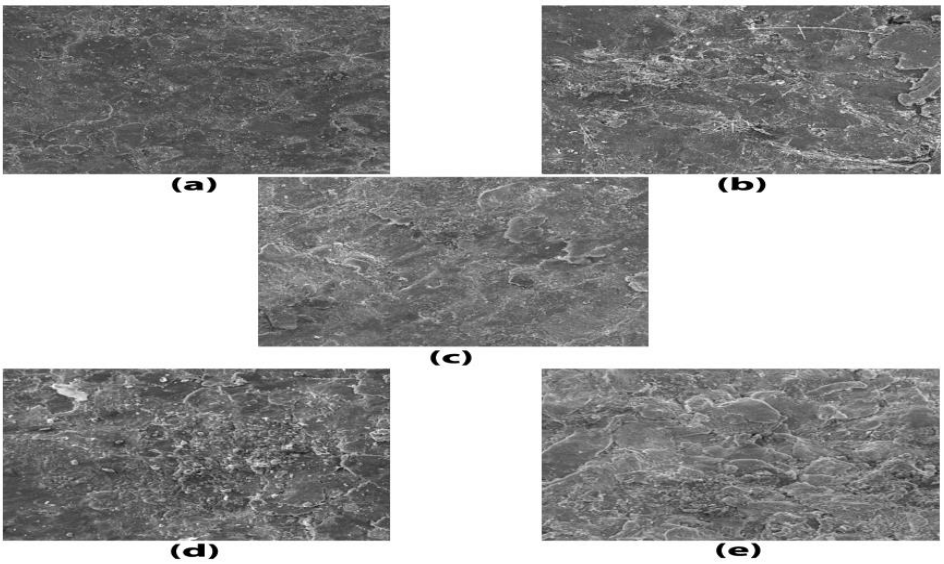

Fig 3:- SEM Micrograph showing distribution of nano-TiO_2 particulates in Mg-TiO_2 composites.

SEM analysis was conducted on the extruded specimen to obtain the micrographic images of each composition of nanocomposites. As shown in Fig.3 the structure and grain growth of the nanocomposites continuously improved with increase in the addition of TiO_2 reinforcement. The grain size of pure magnesium is large and has a lot of micro pores. But the addition of nano TiO_2 refined the grain structure. Mg-TiO_2 composite exhibits a smooth surface with less presence micro pores on its surface. It can be seen that the reinforcement is uniformly distributed in the matrix which is due to the efficient strategy adopted while fabrication.

C. Density and Porosity

S. No	Composition	Density		
		Theoretical ρ (g/cm^3)	Experimental ρ (g/cm^3)	Porosity (%)
1	Mg-4%TiO_2	1.839	1.837	0.1087
2	Mg-8%TiO_2	1.938	1.937	0.1012
3	Mg-12%TiO_2	2.039	2.037	0.1339
4	Mg-16%TiO_2	2.138	2.136	0.1183
5	Mg-20%TiO_2	2.239	2.236	0.1589

Table 1:- Results of density and porosity measurements.

The presence of minimal porosity in the composite material is due to better bonding between Mg-TiO_2. The results of density and porosity measurements conducted on the extruded specimen are shown in above Table. The porosity of pure magnesium is higher when compared to reinforced magnesium [15], the porosity level is low on an addition of 8 wt% of TiO2 with Mg. The density of composites increased with the increase in weight percentage of TiO2 particulates. The density and porosity values revealed minimal oxidation of magnesium and no presence of micro pores.

D. Microhardness

The addition of nano-sized TiO_2 particulates to pure Mg leads to enhancement in hardness. The improvement in hardness of nanocomposites is due to the strengthening effects that have risen from the refined grains. The synergetic effect of TiO2 particulates in magnesium matrix revealed improved hardness values. The value of micro hardness continuously increased with increase in addition TiO2 reinforcement. An intending load of 0.1 kg for a dwell time of 15 s was apllied.

S. No	Composition	Average Hardness in HV
1	Mg-4%TiO_2	37
2	Mg-8%TiO_2	40
3	Mg-12%TiO_2	43.67
4	Mg-16%TiO_2	44.67
5	Mg-20%TiO_2	50

Table 2:- Result of hardness.

D. Compression Test

The compressive strength of extruded Mg (4-20 wt%) TiO_2 samples were tested at ambient temperature. The test specimens of diameter 12 mm of five different compositions were used. The specimen was subjected to compression test at a ram speed of 3 mm per min. The test was carried out on five different compositions of Mg (4-20 wt%) of TiO_2 nanocomposites.

S. No	Composition	Compressive strength in MPa
1	Mg-4%TiO_2	292
2	Mg-8%TiO_2	307
3	Mg-12%TiO_2	312
4	Mg-16%TiO_2	321
5	Mg-20%TiO_2	330

Table 3:- Result of compressive strength.

The variation in microhardness and compressive strength of the composite samples complement each other. At higher weight fraction of reinforcement, in spite of original variation in it, there is a drastic increase in compressive strength. This may be due to the restricted grain growth at a higher content of reinforcement.

Density and hardness increase with respect to the quantity of reinforcement added to the matrix. This is attributed to the fact such as the non-uniform crystalline structure and abrasive nature of reinforcement.

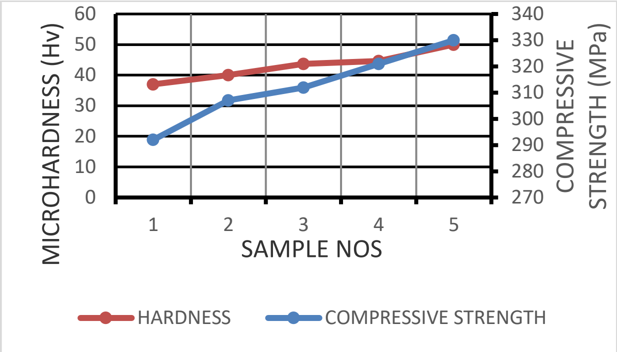

Fig 4:- Graph correlating Microhardness and Compressive Strength.

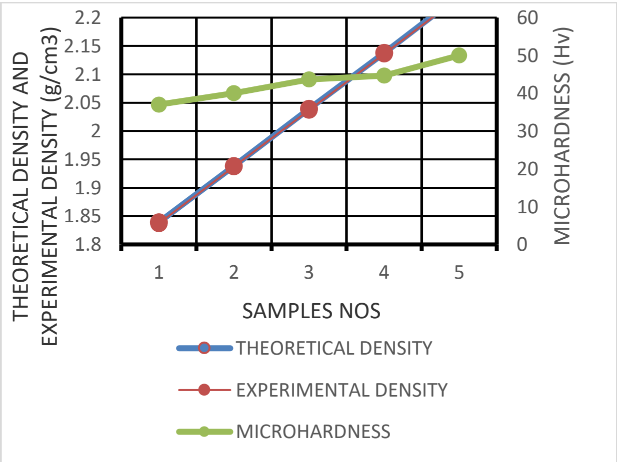

Fig 5:- Graph correlating Theoretical density, Experimental Density and Microhardness.

IV. CONCLUSION

An Mg-TiO2 nanocomposite was synthesized through powder metallurgy technique followed by sintering and hot extrusion. The technology route follows as conversion of macro titanium particles to nano particles, preparing the elemental mixture, compacting the elemental mixtures into cylindrical specimens, sintering the specimens, extruding the specimens, and characterizing mechanical properties. Based on the micro structural and mechanical characterization various conclusions can be drawn.

- This is an efficient method which prevents oxidation of Mg and less porosity is achieved.
- SEM image confirms the uniform distribution of TiO_2 over Mg matrix and thus proves homogeneity of the composite.
- Density is observed to be increasing as the weight percentage of reinforcement increases and porosity is minimum due to the fine bonding of Mg and TiO_2 in the mixture and hence proves the minimum oxidation of Mg.
- Hardness has been considerably improved due to the synergetic effect of TiO_2 particles as the weight percentage of TiO_2 increases.

The compressive strength as well as the hardness increases proportionally with weight percentage of Titanium dioxide reinforcement due to its ceramic nature and thus enhances the mechanical property of the composite.

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