EXPERIMENTAL INVESTIGATION OF MAGNESIUM MATRIX COMPOSITE REINFORCED WITH TITANIUM NITRIDE NANO PARTICLES

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Abstract - In this work, powder metallurgy, compaction, sintering, and hot extrusion were used to create pure magnesium and magnesium matrix composite enhanced with titanium nitride nanoparticles. This study made use of magnesium powder and titanium nanoparticles. Mechanical and microstructural investigation of extruded specimens were performed using a universal testing machine, and scanning electron microscope with energy dispersive X-ray spectroscopy (SEM–EDS). The addition of TiN nanoparticles to the magnesium matrix increased mass density and ultimate tensile strength., and maximum UTS were investigated in Mg0.97 vol% TiN nanocomposite. Further SEM micrographs and EDS data corroborated the distribution and existence of TiN nanoparticles in the magnesium matrix.

Key Words: magnesium composite, hot extrusion, scanning electron microscope, hardness magnesium composite, hot extrusion, scanning electron microscope, hardness.

1. INTRODUCTION

Magnesium and its alloys are used as light weight structural materials for different applications in automobile, aerospace, telecommunication and electronic industries. Magnesium is potential material for light weight production and is highly prepared for strength to weight applications without compromising the overall strength. It is sixth most abundant element in the earth crust (~2.7% of weight). Further magnesium based material are recently attracted for bio medical application. Among the metals magnesium and pure iron have excellent bio compatibility without any local toxicity. Magnesium have lower young's modulus which is close to human bone and also eliminates secondary surgery in bio implant applications. Low modulus, limited ductility and poor corrosion resistant, wear resistant and poor surface property are some of the inherent properties of magnesium based material which restricts their extensive applications.

Magnesium Matrix Composites

Magnesium is the densest metal useful for critical structural and bio-medical applications. Magnesium based materials due to the low density offer high specific strength, high stiffness, high thermal conductivity and diffusivity, good castability, good machinability, good damping capacity and bio compatibility. The density of magnesium(1.74g/cc) is two third of aluminium, one third of titanium, one fifth of steel and iron

Selection of Reinforcement

Selection of reinforcement can be classified they are

Based on the type

1. Metallic reinforcement
2. Ceramic reinforcement

Based on size

1. Micron (1×10^-6m -100×10^-6m)
2. Submicron (0.1×10^-6m – 1×10^-6m)
3. Nano (1×10^-9m – 100×10^-9m)

Generally ceramic reinforcement can be grouped into oxides, carbides, nitrides and borides. The selection of reinforcement allows to make new magnesium-based composites by reinforcing with hard and inexpensive ceramics or metals for the purpose of tailoring the properties. Addition of inexpensive nanoparticles to enable improvements in strength and ductility of magnesium-based composites without compromising the gain in weight

PROCESSING OF MAGNESIUM MATRIX COMPOSITES

Processing of magnesium matrix composites can be classified into two types:

1.Primary Processing

a. Liquid state processing
b. Solid state processing

2.Secondary Processing

a. Hot extrusion

b. Hot forging

c. Hot rolling

d. Hot drawing

Liquid state processing-processed by addition of reinforcement into liquid metal.

Types of liquid state processing

- Conventional casting
- Liquid infiltration
- In-situ processes
- Spray deposition
- Disintegrated melt deposition (DMD)

Solid state processing-processing at a temperature below the solidus temperature of metal matrix. Example powder metallurgy

Mechanical and Physical Properties of MMCS

The mechanical properties of matrix metal and alloys have been enhancing the strength by decreasing reinforcement particle size of particulate reinforced metal matrix composites (Barekar et al., 2009). The aluminium reinforced metal matrix composite has been enhancing the strength 15-40%, stiffness 30-100% with superior wear resistance than the unreinforced aluminium and its alloys. But the reinforcement particles contribute a negative effect on matrix phase properties such as tensile strength, ductility and fracture toughness (Srivatsan et al., 2003). The mechanical and physical properties of metal matrix composites are tailored based on the interface region formation, which depends on the manufacturing methods. The region shared between the matrix and reinforcement phase is called an interface region in MMC. The interface region behavior influences the properties of composite, so it is also called the critical region. The soundness in properties of composites is associated with the interface region due to the interfacial chemical reaction, poor wettability of matrix with reinforcement particle, degradation of reinforcement, etc. (Rajan et al., 1998).

II. LITERATURE SURVEY

S.C. Cifuentes et al (2017) [1] studied the thermal behaviour and mechanical behaviour of PLLA/Mg composites synthesised by hot extrusion. optical microscope, XRD, FTIR and DSC was used to characterise the extruded samples. From the results microstructure, thermal stability, heat flow, absorbance peaks, deformation behaviour under compression and young's modulus were observed.

Chen Q et al (2016) [2] synthesised the Mg-Nd-Zn (ZM6) alloy matrix reinforced with SiC particles using semisolid thermal transformation route (SSTT) and recrystallization and partial melting route (RAT). Microstructural and thermal properties of extruded samples was examined by Optical microscope, SEM-EDS, XRD and DSC. From the results micrographs, partial remelting of composite elemental composition, solidification and liquid fraction temperature were observed.

Ganesh kumar and manoj gupta et al (2015) [3] synthesised the Mg (0.58,0.97 and 1.98) vol % of TiC nanocomposite using DMD processing technique followed by hot extrusion at 350°C with an extrusion ratio of 20.25 :1 using 150 ton hydraulic press.mg turnings >99.9% pure and TiC particles of >98% pure and approximately 30-50 nm size was used in this study. The physical, optical, microstructural and mechanical properties were examined by pycnometer, TMA (Thermo mechanical analysis), Optical microscope, FESEM, XRD and microhardness tester and servo hydraulic testing machine. from the results it was concluded that, and compared to pure Mg the experimental results exhibited the fine grain size, increase in microhardness, increase in 0.2% TYS, UTS and fracture strain of Mg matrix with an addition of TiC particles further Mg 0.97 TiC composite exhibited the maximum of 0.2% CYS and minimum TCA compared to that of other composite samples

Kumar et al. (2013) stated that the pure magnesium was enhanced by adding Si as an alloying element and proved that the wear resistance of Mg–Si alloys increased with higher amount of Si addition and reduced when higher normal load was applied. The addition of Si content in Mg decreased the specific wear rate.

Srinivasan et al. (2012) studied and compared the AZ31B magnesium alloy and the nano-composite of magnesium alloy reinforced with alumina and calcium, which shows lower wear rates due to higher hardness improvement in nano-composite.

Sun & Ahlatci (2011) mentioned that the Al-12Si-xMg/Mg_2Si composite was studied and enhanced the mechanical properties and wear behavior. The addition of different amount of Mg and Mg_2Si reinforcement in the Al increases the bulk hardness, compressive strength and wears resistance.

Shanthi et al. (2010) investigated the dry sliding wear behavior of AZ31B/Al_2O_3 and AZ31B/Al_2O_3 (1-3) wt % Calcium nano-composites at a range of 1-10 m/s sliding velocity under 10 N constant loads and reported that the composite reinforced with alumina and calcium particulate, dominate the higher wear resistance than composite reinforced with alumina particle alone in either lower or higher sliding speeds. They also observed that abrasion and adhesion wear are the dominant wear mechanisms to transition for thermal softening when it is sliding at higher speeds.

Mondal & Kumar (2009) conducted a wear test under dry sliding condition with a constant sliding velocity and distance of 0.837 m/s and 2.5 km, respectively by varying the applied load from 10-40 N. They reported that AE42 magnesium alloy reinforced with SiC particles and saffil short fiber hybrid composite has a lower wear rate than magnesium alloy and saffil reinforced composite samples. They also concluded that the presence of SiC ceramic particles in the magnesium alloy matrix improves the wear resistance of hybrid composite material.

Objectives

- To develop the pure Mg & Mg-TiN nanocomposite using powder metallurgy followed by hot extrusion.
- To study the Microstructural properties of pure Mg & Mg-TiN nanocomposite.
- To study the Thermal properties of pure Mg & Mg-TiN nanocomposite.
- To study the Physical properties of pure Mg & Mg-TiN nano composite.

III. EXPERIMENTAL WORK

Materials

Magnesium powder is used as matrix material and Titanium nitride was selected as ceramic reinforcement in the Magnesium matrix. The specification and properties of magnesium and titanium nitride powders are shown in table

Table-1: Specification and properties of Mg-TiN powder

S.No	Description	Magnesium (Mg)	Titanium Nitride(TiN)
1	Size	60-300 µm	~20 nm
2	Purity	>98.5	>99.5
3	Density	1.74 g/cc	5.22 g/cc
4	Crystal type	HCP	Cubic
5	Melting point	651 °C	2930 °C
6	Boiling point	1107 °C	0
7	Youngs modulus	40-45 Gpa	390 Gpa
8	Coefficient of Thermal expansion	27 µ/K	9.35 µ/K
9	Supplier	Merck, Germany	US Research and Nano materials Inc., USA

Fabrication

The synthesis of pure Mg and Mg matrix with different proportions of (1.5, 2.5 and 5 wt.%) nano sized TiN ceramic reinforcement was done by powder metallurgy (solid state processing) incorporated with microwave sintering followed by hot extrusion. Finally, the 8mm extruded samples are cut down to required length by using diamond saw for further characterisation studies. The sequence of operation in fabrication process and extruded samples are shown in fig 1.1 (I) and (II).

Fig-1.(I) Extruded samples (a) Pure Mg, (b)Mg-1.5 TiN, (c) Mg-2.5 TiN and (d) Mg-5 TiN

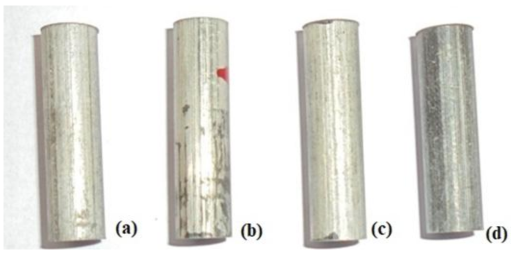
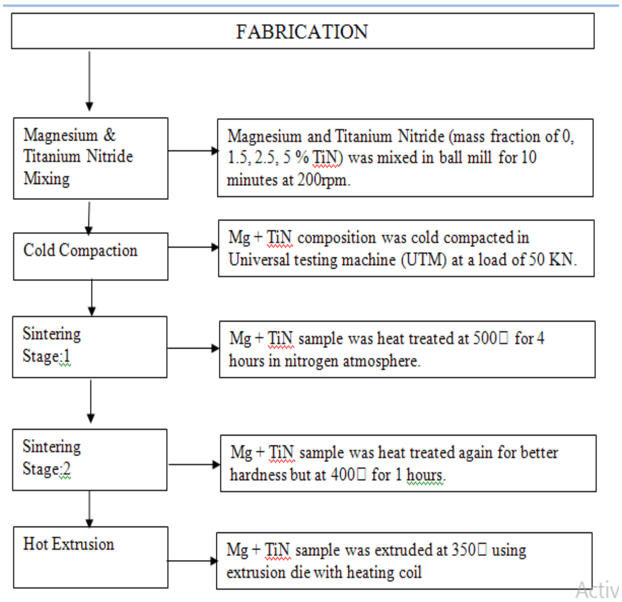


Fig-1. (II) Sequence of operation in Fabrication Process



Sample preparation

Fig-2. Wire cut EDM machine

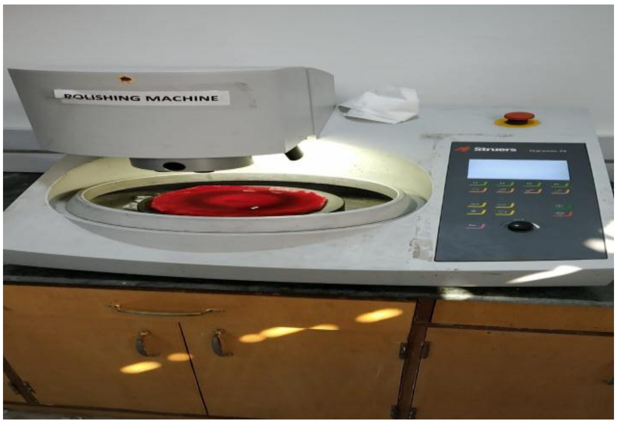


Fig-3 Polishing machine

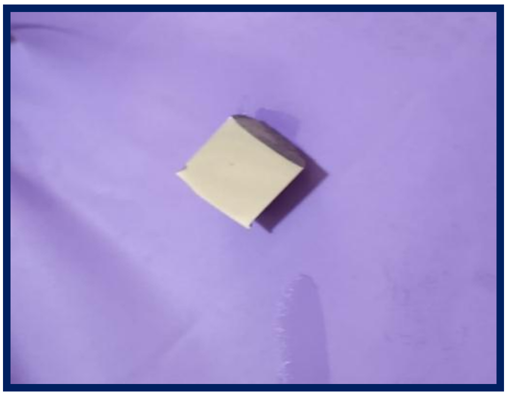


Fig-4 Prepared sample

PHYSICAL PROPERTY MEASUREMENT

The true density Mg alongside hierarchical Mg-tin nanocomposite measured by using Helium gas pycnometer . Each sample were run for six cycles to measure the true density more precisely. Pure helium gas was purged with a pressure of 21psig (2.4611 bar) for all six cycles. using rule of mixture principle, the theoretical density of Mg-TiN nanocomposite sample was calculated and compared to that of true density (experimental density) values, The theoretical density of pure Mg and Mg-TiN nanocomposite samples were calculated by using the following equations.

$$\rho_{the} = v_r \rho_r + (1 - v_r)\rho_m \qquad \text{....... (Equation.1)}$$

Where,

ρ_{the} is the theoretical density in g/cm^3

ρ_{exp} is the experimental density in g/cm^3

ρ_r is the density of reinforcement (TiN – 5.22) in g/cm^3

ρ_m is the density of matrix material (Mg - 1.74) in g/cm^3

v_r is the volume fraction of reinforcement in cm^3

IV. RESULTS & DISCUSSION

FIELD EMISSION SCANNING ELECTRON MICROSCOPE (FE-SEM)

Sample for microstructure study was prepared using standard procedure for sample preparation, they were etched using acetic-picral agent. Microstructure examination was carried out using a KARL ZEISS Ultra55 field emission scanning electron microscope (FE-SEM). The micro-structure of etched sample along with EDS spectrum presented in fig. 5 and fig.6 which shows that titanium nitride particles distributed in the matrix.

Element	Weight%	Atomic%
Mg	97.41	96.31
Ti	2.39	3.59
N	0.20	0.10
Totals	**100.00**	

Fig-5 FE-SEM of Mg+2.5TiN

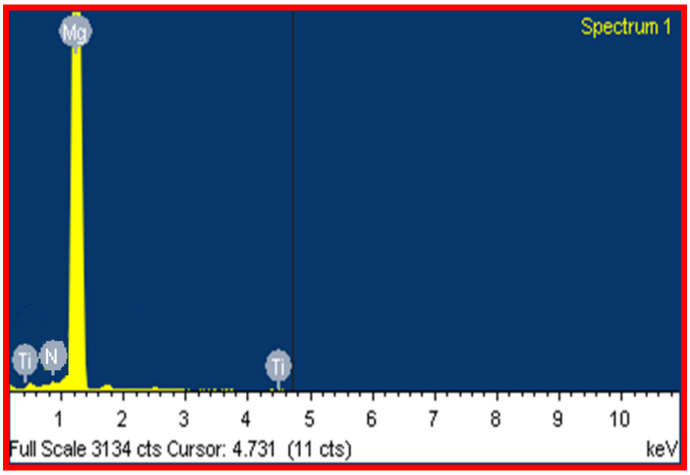
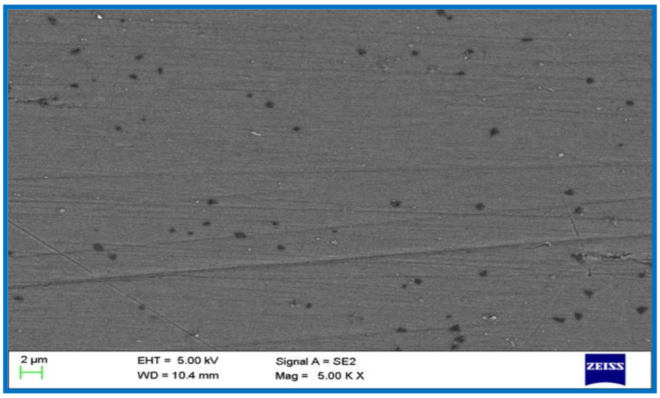


Fig-6 Composition of Mg+2.5TiN

EDS analysis exposed the quantitative composition of pure Mg, Mg -1.5%TiN, Mg-2.5%, TiN, Mg-5%TiN. Further oxides(O) presence was observed in the Pure magnesium. The distribution of major chemical elements, including Mg, Ti, N and O within the microstructure, was traced by EDS to detect differences in the chemistry of micro regions between the matrix and phase. It is clearly seen that the center of the Mg grain contains small traces of Ti, N and O. The compounds are almost the stoichiometric proportion of the phase, which contains the elements of Mg, Ti and N.



COMPRESSION TEST

Table-2 Compression Test(N/mm²)Values

S.NO	SAMPLE NO.	COMPRESSION TEST(N/mm²)VALUES
1	1.5Mg	310
2	2.5Mg	350
3	5Mg	353
4	PURE Mg	50

The goal of a compression test is to determine the behavior or response of a material while it experiences a compressive load by measuring fundamental variables, such as, strain, stress, and deformation. Here,four samples taken in the names of 1.5Mg, 2.5Mg, 5Mg, PURE Mg for this samples had been tested and based upon the stress-strain relationship graph values are noted.5Mg has been found the best compression value compare with the other samples

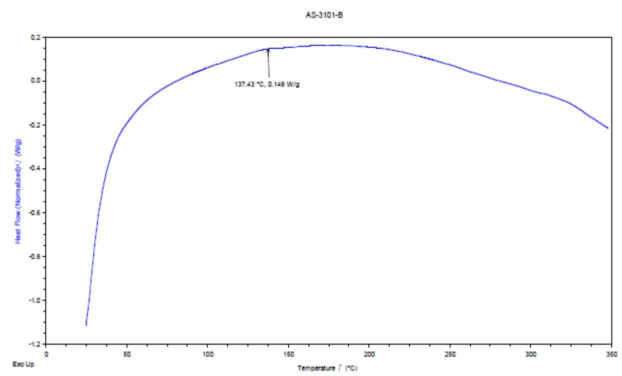
TENSILE TEST

Table-3 Tensile Test(N/mm²)Values

S.NO	SAMPLE NO.	TENSILE TEST(N/mm²) VALUES
1	1.5Mg	108
2	2.5Mg	85
3	5Mg	30
4	PURE Mg	60

A compression test is conducted in a manner similar to the tensile test, except that the force is compressive and the specimen contracts along the direction of the stress. Here,four samples taken in the names of 1.5Mg, 2.5Mg, 5Mg, PURE Mg for this samples had been tested and based upon the stress-strain relationship graph values are noted.5Mg has been found the best compression value compare with the other samples

DIFFERENTIAL SCANNING CALORIMETRY (DSC)



Graph-1 DSC graph shows the glass transition temperature of Mg+5TiN

DSC graph shows the glass transition temperature, T_g at 137.43⁰C and weight loss of (0.148w/g) was observed in Mg+5TiN.

V. CONCLUSIONS

Pure Mg & Mg-TiN nanocomposite was successfully fabricated by powder metallurgy followed by hot extrusion and SEM results exposed the micrograph, distribution of TiN nanoparticles in Mg matrix. In SEM results Mg+2.5TiN has found the best values for Weight% as well as Atomic% compared with other samples. In Compression test, among the four samples '5Mg' will be the best compression value(N/mm^2). In Tenslie test, among the four samples '1.5Mg' will be the best tensile value(N/mm^2). DSC graph shows thebest glass transition temperature, T_g at 137.43^{0}C and weight loss of (0.148w/g) was observed in Mg+5TiN.

EDS results confirmed that TiN presence in Mg matrix and also quantitative compositional of Mg-TiN nanocomposites was recorded and DSC results reveals that composite samples exhibits the weight loss values of 0.57, 0.49 and 0.148 w/g respectively.

VI. REFERENCES

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