CHAPTER 3 EXPERIMENTAL PROCEDURES

The present work involves the fabrication of pure Mg and Mg/MgO composites through powder metallurgy process. The fabricated samples were subjected to different characterisation tools like XRD, SEM, hardness and compression tests, etc. to study different physical, mechanical and wear properties. This chapter describes the details pertaining to procurement and characterisation of feedstock powders, synthesis of pure Mg and composites samples, different characterisation tools and techniques and procedures and parameters for carrying out the tests.

The pure Mg and Mg/MgO composites were fabricated by powder metallurgy route by two different sintering methods. In the first part of the work, the fabrication was done using rapid microwave sintering method. Pure Mg and Mg/5wt. % MgO composite were fabricated by varying sintering temperature of 450 °C, 500 °C and 550 °C. The effect of varying sintering temperature on different physical and mechanical properties of pure Mg and Mg/5wt. % MgO composite were studied.

In the second part of the work, pure Mg and Mg/MgO MMCs were fabricated by a cost effective sintering technique, the details of which are described in the section 3.2.2. Mg/MgO metal matrix composites were fabricated with varying MgO content of 0, 5, 10, and 15 wt. % at a fixed sintering temperature of 500 °C. The fabricated samples were characterised by XRD, SEM, hardness and compression tests and the effect of varying MgO weight fraction on different properties were studied. The same samples were also tested on pin on disc wear tests and dry sliding wear behaviour was studied at varying load and sliding velocity. Post wear characterisation of worn samples was done by SEM and optical profilometer. The worn track micrographs and

elemental maps were studied for wear mechanism. The topographical characteristics were studied by optical profilometer graphs.

3.1 Fabrication of pure Mg and Mg/5wt. % MgO MMCs by rapid microwave sintering

3.1.1 Materials procurement

Elemental magnesium powder of more than 99% purity (supplied by Alfa Aesar, US) having APS ≅ 40 μm was used as matrix material, and magnesium oxide powder (supplied by Alfa Aesar, US) of APS ≅ 12 μm was used as reinforcing phase. Figure 3.1 shows the morphology of staring powders and their corresponding XRD diffractograms.

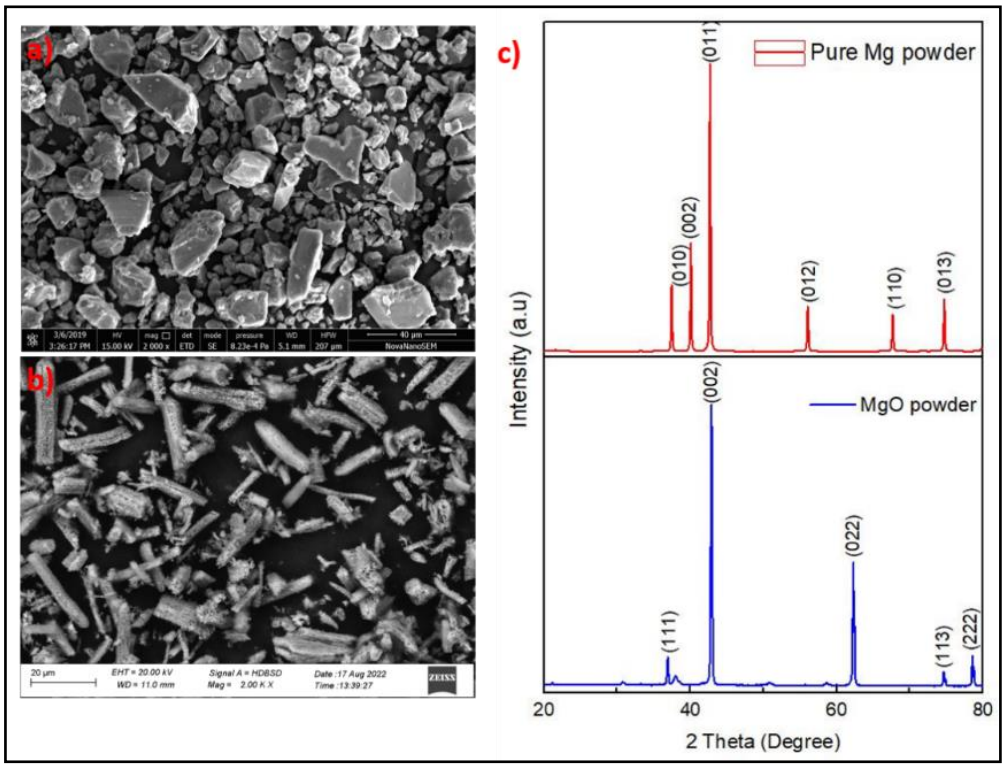

Figure 3.1 SEM image of starting powders for microwave sintered samples, a) Pure Mg, b) MgO and c) XRD diffractograms of pure Mg and MgO powders

3.1.2 Fabrication of pure Mg and Mg/MgO composites by rapid microwave sintering

The fabrication of pure Mg and Mg/5wt. % MgO composites consists of mixing of powders, compaction of mixed powder and sintering of compacts in microwave furnace. The schematic of the process is shown in Figure 3.2.

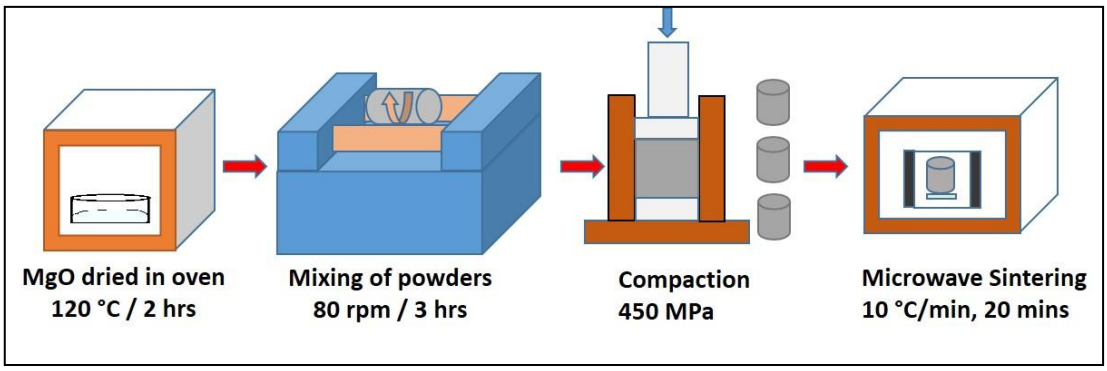

Figure 3.2 Schematic of fabrication of Mg/MgO composites by rapid microwave sintering

The details of each steps are described in the following sub – sections:

3.1.2.1 Mixing of powders

Prior to mixing, MgO powder was first heated at 120 °C for two hours in an oven to remove the moisture on MgO powders as it is hygroscopic in nature. Pure Mg and MgO powders were then weighed and mixed in a steel jar on a roller mixer at 80 rpm for 3 h. No balls were used during the mixing of powders. About 1% stearic acid was added to reduce the agglomeration of powders particles to each other and to the wall of the container. Ball milling of powders was not preferred as powders may get oxidized due to increase in temperature inside the container.

3.1.2.2 Compaction of mixed powders

The compaction of mixed powders was done with a manual hand held press in a hardened D2 steel cylindrical die of 12 mm diameter. An optimum pressure of 450 MPa and holding time of 45s was selected for compaction of all the samples. Graphite powder was used as a lubricant to reduce the wall friction of die. A cylindrical billet (compact) of size 12 mm diameter and 10 mm height was obtained after compaction.

3.1.2.3 Sintering of compacts

Sintering of compacts was done in a microwave furnace (Make—Nano Tec, Chennai, India 4.4 kW, 2.45 GHz). The green compacts of both pure magnesium and Mg/5wt. % MgO were sintered at 450 °C, 500 °C, and 550 °C for 25 min with a heating rate of 10 °C/min under the protective atmosphere of argon. The compact was placed at centre on an alumina disc surrounded by porous SiC blocks for hybrid two directional microwave sintering.

Figure 3.3 a) Microwave sintering furnace, b) heating space for sample and c) roller mixer

Table 3.1 Microwave Sintered Samples' composition

S. No	Sintering Temperature (°C)	Sample Composition
1	450	Pure Mg
2	500	Pure Mg
3	550	Pure Mg
4	450	Pure Mg + 5 wt. % MgO
5	500	Pure Mg + 5 wt. % MgO
6	550	Pure Mg + 5 wt. % MgO

3.1.3 Characterisation of pure Mg and Mg/MgO composites

This section describes the procedure of carrying out each tests and details of each characterisation tools and techniques used for rapid microwave sintered samples.

3.1.3.1 Sample preparation for different tests

Samples for each test were cut to the required dimension using Wire Cut Electric Discharge Machine (W-EDM). Then they were cleaned and polishing was done in two steps. Coarse polishing was done using SiC emery paper of grit size 400 to 2000. Then samples were cleaned using acetone and fine polishing was done using diamond paste on velvet cloth on disc polishing machine.

3.1.3.2 Density and porosity of Mg/MgO composites

The theoretical density (ρ_t) of samples were calculated using rule of mixture method.

$$\rho_t = \sum_{i=0}^{n} v_i \times \rho_i = (v_m \times \rho_m) + (v_r \times \rho_r) \quad (3.1)$$

Where, ρ_t = theoretical density of composite, v_m and ρ_m = vol. fraction and density of matrix, v_r and ρ_r = vol. fraction and density of reinforcement.

The sintered (measured) density of samples was calculated using Archimedes' Principle on precision balance having 0.0001g accuracy with the help of density measuring kit. Ethanol ($\rho = 0.789 g/cm^3$) was used as liquid medium and three samples of each composition were measured to get an average value. The weight of sample in air and in liquid medium was taken and then density was calculated by following formula:

$$\rho_s = \frac{w_a \rho_e - w_e \rho_a}{w_a - w_e} \quad (3.2)$$

Where, ρ_s is sintered density of sample, w_a and w_e are weight of sample in air and ethanol respectively, ρ_a and ρ_e are density of air and ethanol.

The relative density was calculated by comparing the measured (sintered) density with the theoretical density. Structural porosity was estimated using the following formula:

$$\% \, Porosity = \left(1 - \frac{\rho_s}{\rho_t}\right) \times 100 \quad (3.3)$$

3.1.3.3 Phase analysis and identification

The X-ray diffraction of polished sintered specimens was done using X-ray diffractometer (Make - Rigaku Corp. Model: Rigaku Smart Lab 9 kW Powder type) using Cu K_α ($\lambda = 1.540$ Å). The pattern was recorded in the 2θ range from 20° to 80° with step size of 0.02°. The phase analysis of samples was done using XPert Highscore Plus software. The average volume fraction of MgO phase formed in sintered samples was calculated using relative intensity method [81]

$$Wp = \frac{P}{\Sigma Pi} \times 100 \quad (3.4)$$

Wp = volume fraction of phase p, P = relative intensity of phase p and ΣP_i = sum of relative intensities of all observed phases

3.1.3.4 Microstructural characterisation of composites

To carry out the microstructural investigation, the samples were polished as described in section 3.1.3.1. They were then etched in acetic picral solution for microstructural characterisation using optical microscope and Scanning Electron Microscope (SEM) (Make - CARL ZEISS EVO 50) equipped with Energy Dispersive Spectroscopy (EDS). EDS analysis was performed on specific features within a sample. The X-ray mapping technique was performed to analyse the distribution of elements within the polished samples.

3.1.3.5 Hardness test of composites

Hardness testing of samples was conducted on Vickers' Microhardness tester (Micro Mech Technologies, Pune, India) according to ASTM standard E384 – 08. The samples were first coarse polished and then fine polished to mirror finish using diamond paste and finally etched using acetic - picral solution. Five indentations of 50g load and 15s dwell time were made at different places using 136° Vickers' diamond pyramidal indenter. The lengths of diagonals of pyramid were calculated by focal lens of microscope and the average hardness value with standard deviation were calculated.

3.1.3.6 Compression tests of composites

MgO has high compressive strength which when added in magnesium matrix is expected to improve the compressive strength of composites. Compression test of pure Mg and Mg/MgO composites were performed according to ASTM standard E9-89a. Specimen were first cut in cylindrical shape using W - EDM having diameter of 10 mm and l/d ~ 1. Then the flat end of samples were fine polished. Tests were carried

out on INSTRON 3367 (30 kN) at room temperature with a cross-head speed of 0.5 mm/min (equivalent to a strain rate of 0.0003 s^{-1}).

3.2 Fabrication of Mg/xMgO (x = 0, 5, 10, 15 wt. % MgO) MMCs by a novel sintering technique

The second part of work involves the fabrication of Mg/MgO composites having varying weight fraction of 0, 5, 10, and 15 % MgO as reinforcement through powder metallurgy route employing a novel sintering method. This section describes the details pertaining to procurement and characterisation of feedstock powders, synthesis of pure Mg and composites samples, procedures of carrying out tests and different characterisation tools and techniques. A schematic of the experimental procedure established for the whole study is shown in Figure 3.4. Each of the main steps will be discussed in the following sub - sections.

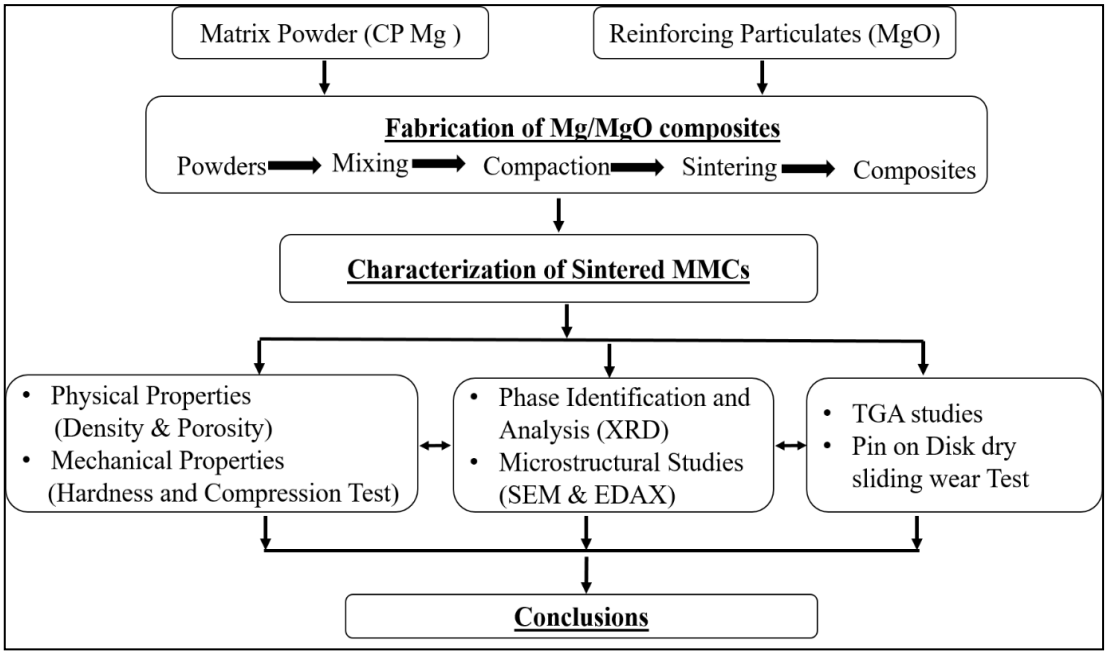

Figure 3.4 A flowchart depicting overview of total experimental procedure for Mg/MgO composites fabricated by the novel sintering method

3.2.1 Materials procurement

Commercially pure Magnesium powder was purchased from CDH, India having more than 96 % purity. MgO powder was procured from Alfa Aesar, US having more than 99% purity. Figure 3.5 shows the morphology and XRD plots of Mg and MgO powders and Figure 3.6 shows the size distribution of both the powders.

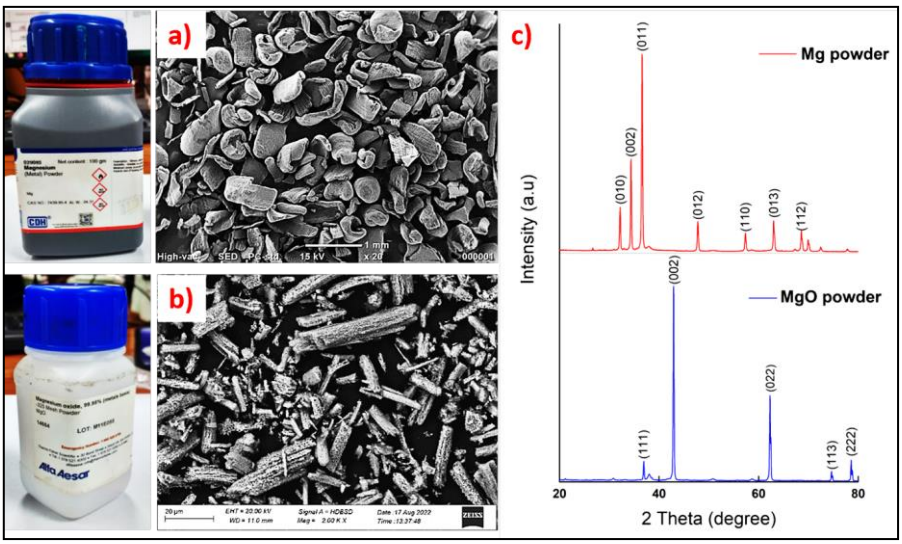

Figure 3.5 Morphology of starting powders a) Pure Mg, b) MgO and c) XRD plot of pure Mg and MgO powders

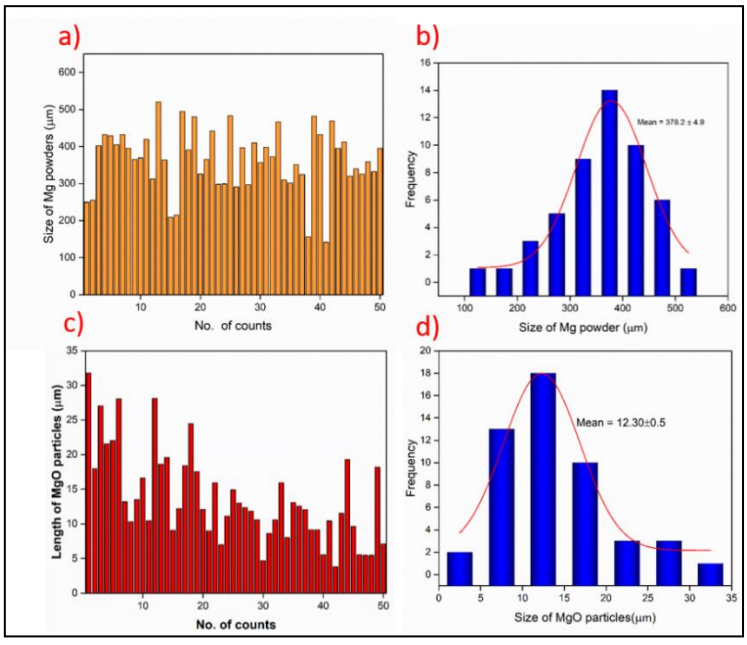

Figure 3.6 Bar chart for size distribution of a) Pure Mg powder, c) MgO powder and frequency distribution of b)Pure Mg , d) MgO powders

3.2.2 Fabrication of pure Mg and Mg/MgO composites

Figure 3.7 shows the schematic of fabrication procedure of Mg/MgO composites. The mixing was done by mechanical stirring in wet medium and then drying. Uniaxial compaction of mixed powders was done with the help of manual press. Sintering of compacts was done by low cost approach in a muffle furnace.

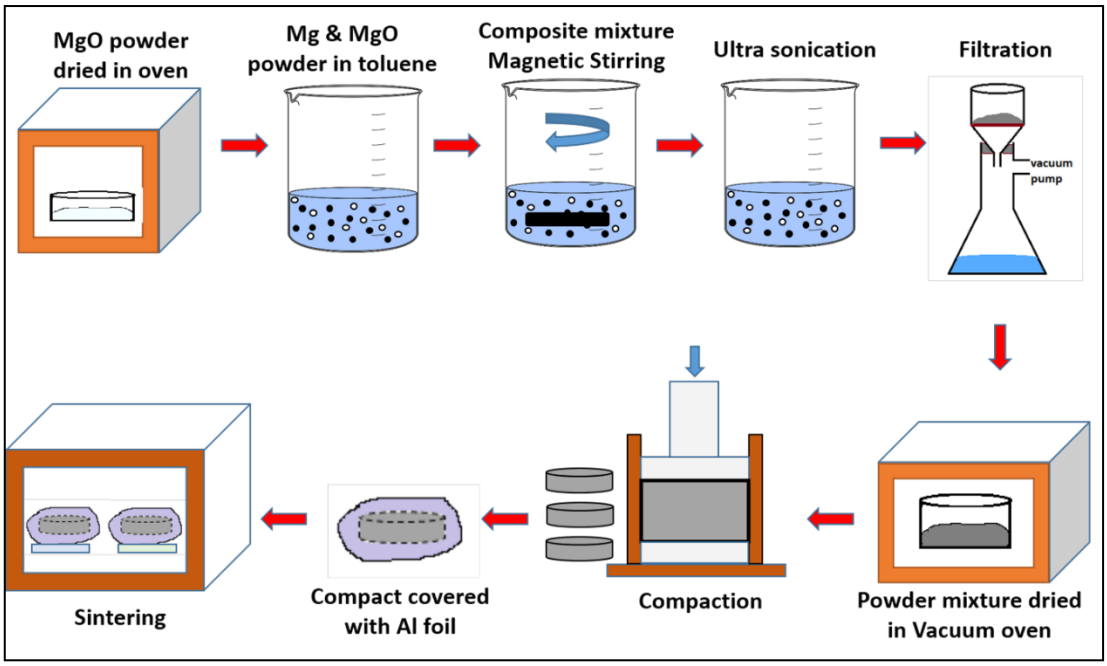

Figure 3.7 Schematic of cost effective sintering procedure for composites

The details of each steps are covered in the following sub sections:

3.2.2.1 Mixing of powders

Before mixing the powders, MgO powder was taken in a petri dish and heated to 350 °C for 2 hours to ensure that all the moisture adsorbed on the powder surface were removed. This was done because MgO is hydroscopic in nature. Then pure Mg powder and MgO powder were weighed on precision balance according to the required composition. The weighed powders were poured in a beaker containing toluene. Toluene was selected as the wet medium because both powders are neither soluble nor they form suspension. The wet medium containing both powders were mechanically

agitated at 150 rpm for 2 hours using magnetic stirrer. Then it was ultra-sonicated for 20 minutes so that powder particles get separated from each other. The powders were then filtered and toluene was separated. The wet powders were then dried in a vacuum oven at 70 °C for nearly 3 h. After taking out of oven, powders were weighed for compaction.

3.2.2.2 Thermo - gravimetric analysis (TGA) of mixed powders

TGA analysis were performed on mixed powders (Mg/xMgO, x = 0, 5, 10, 15 wt. %) on TGA analyser (Make – M/s Shimadzu Pvt. Ltd. Model – TGA - 50). The analysis was done from room temperature to 500 °C with heating rate of 10 °C/min on a platinum pan in nitrogen environment to determine any weight change with respect to temperature.

3.2.2.3 Compaction of mixed powders

The compaction process was same as described in section 3.1.2.2 but a different cylindrical die of 20 mm diameter was used. A cylindrical billet (compact) of size 20mm diameter and 8 mm height was obtained after compaction.

3.2.2.4 Sintering of compacts

Sintering of compacts was done by a novel sintering approach. The compacts were covered by aluminium foil very carefully such that foil don't get torn. 4 – 5 layers of wrapping was done carefully to ensure that air doesn't get trapped inside and sample is completely covered. Sintering of all samples was done at 500 °C with a heating rate of 5 °C/min and held for 2 hours followed by furnace cooling to room temperature. The sintered samples were taken out and aluminium foils were removed to get the sample as shown in Figure 3.8. Table 3.2 shows the sample composition and notation which will be used throughout the work.

Figure 3.8 Sintered samples a) just after removing aluminium foil and b) after coarse polishing

Table 3.2 Composition and notations of samples sintered using cost effective sintering

S. No	Reinforcement (MgO)		Sample Composition	Sample notation
	Wt. %	Vol. (%)		
1	0	0	Pure Mg + 0 wt.% MgO	Mg/0MgO
2	5	2.49	Pure Mg + 5 wt. % MgO	Mg/5MgO
3	10	5.11	Pure Mg + 10 wt. % MgO	Mg/10MgO
4	15	7.89	Pure Mg + 15 wt. % MgO	Mg/15MgO

3.2.3 Characterisation of Mg/MgO composites

This section describes the procedure of carrying out each tests and details of characterisation tools and techniques used for the same.

3.2.3.1 Sample preparation for different tests

Sample preparation was done as described in section 3.1.3.1.

3.2.3.2 Density and porosity measurement of Mg/MgO composites

The density and porosity were measured as described in section 3.1.3.2.

3.2.3.3 Phase analysis and identification

Phase Analysis and Identification was done as described in section 3.1.3.3. The volume fraction of MgO phase was also calculated with same method as described in section 3.1.3.3.

Crystallite size and lattice strain of the samples were calculated using equation (3.5) & (3.6) respectively.

$$\boldsymbol{Dc = \frac{k\lambda}{\beta\ cos\theta}} \tag{3.5}$$

$$\boldsymbol{\varepsilon = \frac{\beta}{4\ tan\theta}} \tag{3.6}$$

Where, Dc – Crystallite size (nm), k – shape factor, λ - X – ray wavelength (nm), β - Full width half maxima (FWHM), θ - Bragg's angle (degree), ε - lattice strain

3.2.3.4 Microstructural characterisation of composites

The microstructural characterisation was done using Scanning Electron Microscope (Make - CARL ZEISS EVO 50) equipped with Energy Dispersive Spectroscopy (EDS). The samples were first polished as described in section 3.2.3.1 and then they were etched in acetic picral solution. The SEM was done in both secondary electron and back scattered mode at 20kV. Secondary electron imaging (SEI) was used on samples to determine the distribution of MgO, porosity, cracks, etc. Backscattered electron imaging (BSE) provides contrast between chemically different phases in composites due to differences in effective atomic number. EDS point and line scanning analysis was performed on specific features within the sample. The X-ray elemental mapping technique was performed to analyse the distribution of elements within the selected areas.

3.2.3.5 Hardness test of composites

Hardness test of samples were done as described in section 3.1.3.5.

3.2.3.6 Compression tests of composites

Compression tests of pure Mg and Mg/MgO composites were performed according to ASTM standard E9-89a. Specimens were first cut in cylindrical shape using W - EDM having diameter of 6 mm and l/d ~ 1 as shown in Figure 3.9 Then the flat end of samples were fine polished. Tests were carried out on INSTRON 3367 having 30kN load capacity at room temperature with a cross-head speed of 0.5 mm/min (equivalent to a strain rate of 0.0003 s^{-1}).

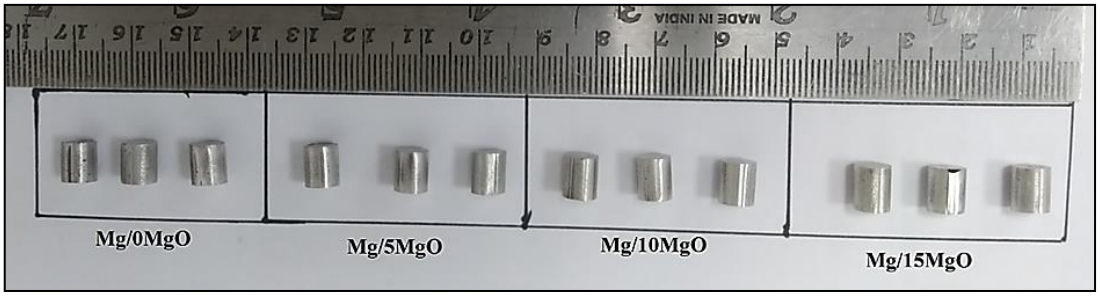

Figure 3.9 Test samples of pure Mg and composites for compression testing

3.2.3.7 Pin on disc dry sliding wear test of composites

Friction and wear tests of pure Mg and Mg/MgO composites were conducted using pin on disc rotary tribometer according to ASTM G99 - 17. The tribometer was supplied by M/S DUCOM, Bangalore, India. The torque moment of the sample was calibrated in terms of frictional force as indicated on the machine, using a fixed distance of lever arm of the apparatus. The tangential force was continuously monitored during the tests and data was acquired using data acquisition software installed in the tribometer. The coefficient of friction was determined using friction force and normal loads; only pre calibrated dead loads were used.

The dry sliding pin on disc wear tests were conducted at ambient conditions having relative humidity in the range of 30 to 35 %. The test specimens were cut in shape of cylindrical pin of diameter 6 mm using W- EDM machine. The pin was polished up to 4/0 – grade emery paper and then subsequent polishing was done on cloth wheel using diamond paste. EN31 grade stainless steel polished disc was used as counter face for wear test having hardness of 62 HRC. The wear tests were conducted at 5, 10, and 15 N load at each sliding speed of 0.6, 0.9, and 1.2 m/s for a fixed sliding distance of 1000 m as shown in Table 3.3.

Table 3.3 Pin on Disc testing parameters for Pure Mg and Mg/MgO composites

Configuration	**Pin on Disc**
Standards	ASTM G99-17
Type	Dry Sliding
Test Specimen	Pin (length = 7mm, Diameter = 6mm)
Disc	EN31 steel, Hardness = 60±2 HRC, ϕ165 mm
Temperature	30 °C
Load (N)	5, 10, 15
Sliding Velocity (m/s)	0.6, 0.9, 1.2
Total Sliding Distance (m)	1000

Each wear test was conducted thrice with fresh set of tribo - pair and average values are reported here. Prior to each test, both disc and pin were cleaned with acetone and dried before conducting the tests. Then the initial weight of the sample was taken and testing was done. After the test, each sample was cleaned with acetone to remove the wear debris. The cleaned sample was weighed on precision balance to get final weight of specimen after wear. The sample was carefully stored for other characterisations. Wear rate was calculated by volume loss per unit sliding distance.

Volume loss was obtained by weight loss upon density. It can also be written in mathematical expression as follows:

$$\boldsymbol{Volume\ Loss(mm^3) = \frac{Weight\ Loss\ (g)}{Density\ (g/cc)} \times 1000} \quad (3.7)$$

$$\boldsymbol{Wear\ Rate\ (mm^3/m) = \frac{Volume\ Loss(mm^3)}{Sliding\ Distance(m)}} \quad (3.8)$$

3.2.3.8 Characterisation of worn surface

The worn surface was characterised further using scanning electron microscope (Make - CARL ZEISS EVO 50 Tungsten SEM) to study the wear mechanism at varying load and sliding velocity. EDS elemental distribution maps of worn surface were obtained to ascertain the distribution of elements on the worn surface.

The worn surface was also characterised using optical profilometer to get three dimensional profile of worn surface. The surface roughness was calculated from the profilometer data using Gwyddion software.