CHAPTER 3

EXPERIMENTAL DETAILS

3.1 INTRODUCTION

The materials, experimental set-up and different characterizing equipment and the elaborate procedures used to carry out the experiments for fulfilling the objectives of the present study have been described in this chapter. The procedures adopted for characterization of microstructures, mechanical and tribological characteristics of stir cast as well as CS cast composites have also been described.

3.2 MATERIALS AND METHOD

The A356 aluminum alloy ingots used as base matrix materials in synthesis of hybrid composites was procured from the M/s. Khandelwal Aluminum Works Pvt. Limited, Varanasi. Mg turning (99.9% Mg), Potassium fluorotitanate (K_2TiF_6) (purity 97%) and potassium fluoroborate (KBF_4) (purity 98%) were procured from Alfa Aesar, while fluxes such as KCl (99.5%), $MgCl_2.6H_2O$ granule (98%) and degasser Hexa-Chloro-Ethane (C_2Cl_6) were procured from Molychem.

3.2.1 Preparation of A356-10%Mg_2Si composite

A356 alloy was charged into a crucible kept in an electric resistance furnace for melting. In-situ Mg_2Si was synthesised using Al-20%Mg master alloy. As a flux, KCl and $MgCl_2$ salts were utilised to prevent Al oxidation and Mg burning. To compensate for Mg loss, 10% additional Mg was supplied to the melt despite the use of flux [39].

When the master alloy was in the melt state, it was mechanically agitated for 5 minutes with a graphite stirrer to achieve homogeneity. Following that, 0.5 wt.% hexachloroethane (C_2Cl_6) powder was added to the melt to degas it[122]. Finally, slag was scooped out and melt was poured at 700°C into a mild steel mould (preheated at 300°C) in stir casting, while in CS casting melt was poured onto a cooling plate to produce A356-10%Mg_2Si composite.

3.2.2 Preparation of A356-10%Mg_2Si-xTiB_2 hybrid composite

The salt metal reaction route was used to synthesise insitu TiB_2 particles using Potassium fluoro-titanate (K_2TiF_6) (purity 97%, Alfa Aesar) and potassium fluoroborate (KBF_4) (purity 98%, Alfa Aesar) halide salts. The exothermic reactions (Eqs. 3.1 to 3.4) to synthesize TiB_2 particles [48] are as follows:

$$3K_2TiF_6 + 13Al \rightarrow 3TiAl_3 + 3KAlF_4 + K_3AlF_6 \quad (3.1)$$

$$2KBF_4 + 3Al \rightarrow AlB_2 + 2KAlF_4 \quad (3.2)$$

$$AlB_2 + TiAl_3 \rightarrow TiB_2 + 4Al \quad (3.3)$$

Direct reaction can be written as:

$$3K_2TiF_6 + 6KBF_4 + 10Al \rightarrow 3TiB_2 + 9KAlF_4 + K_3AlF_6 \quad (3.4)$$

The A356 alloy was melt in an electric furnace. Potassium fluorotitanate (K_2TiF_6) and potassium fluoroborate (KBF_4) halide salts were heated at 250°C for 2 hours, thoroughly mixed, and added to the molten metal at 850°C. To achieve homogeneous particle dispersion, the melt was stirred intermittently. Preheated Al-20%Mg alloy was added in the melt at 740°C for the synthesis of Mg_2Si phase to produce the A356-Mg_2Si-TiB_2 hybrid composite. 0.5wt% hexachloroethane powder was added in the melt to degas it. Then slag was removed and the melt was poured into a preheated (300°C for 3 hours) mild steel mould using stir casting process. Whereas, in case of cooling slope casting

process melt was poured in the mould through cooling slope plate as displayed in Fig. 3.1. The dimensions of the mould employed for casting are Φ 4 cm x 20 cm. The CS casting process variables such as slope length and slope angle were fixed at 50 cm and 45°, respectively[4, 123]. To reduce the melt temperature flowing over the cooling plate, water at ambient temperature has been circulated beneath the cooling plate. Four composites with varying TiB_2 amount 0, 1, 3 and 5 wt.% keeping constant Mg_2Si amount (10 wt.%) were fabricated as given in Table 3.1.

Table 3.1 Composites with varying TiB_2 content

S. No.	Composites	Mg_2Si	TiB_2
1	A356-10Mg_2Si	10 wt.%	0 wt.%
2	A356-10Mg_2Si-1TiB_2	10 wt.%	1 wt.%
3	A356-10Mg_2Si-3TiB_2	10 wt.%	3 wt.%
4	A356-10Mg_2Si-5TiB_2	10 wt.%	5 wt.%

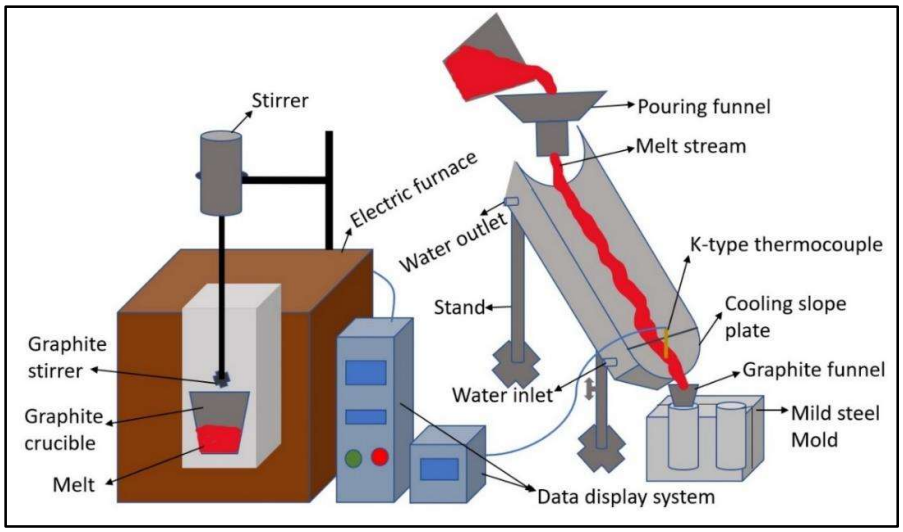

Fig. 3.1 Schematic diagram of CS casting set up.

3.3 X-RAY DIFFRACTOMETER FOR PHASE ANALYSIS

The phases incorporated into the hybrid composites were investigated using a Panalytical X-ray diffractometer with Co Kα radiations (wavelength 1.79 A^0) at 35 kV. The 2θ angle was varied in the range of 10º to 90º with a scanning rate of 5º/minute. To detect various x-ray peaks, 'd' values from XRD patterns were compared with the typical d-spacing of all possible values from the JCPDS database.

3.4 DENSITY AND POROSITY CALCULATIONS

The Archimedes principle was used to determine the density of the alloys and composites. Theoretical densities can be computed using the mixture rule for the weight percentage of the reinforcements following Eq. 3.5.

$$\rho th = \frac{1}{\frac{Wm}{\rho m}+\frac{Wf}{\rho f}} \tag{3.5}$$

Where ρ_{th} is theoretical density of composite, W_m and W_f are wt. fraction of matrix and reinforcement, ρ_m and ρ_f are densities of matrix and reinforcement

The actual density and theoretical density of alloys and composites were used to calculate the porosity percentages using Eq.3.6 [122].

$$\% \text{ Porosity} = \frac{\rho th - \rho ac}{\rho th} \times 100 \tag{3.6}$$

Where ρ_{th}: theoretical density, ρ_{ac}: actual density

3.5 MICROSTRUCTURAL CHARACTERIZATION

The specimen was cut from the middle section of the casting as shown in Fig. 3.2 and polished with different grades of emery paper for metallographic inspection. The microstructural characteristics were examined employing an optical microscope (OM) and scanning electron microscopy (SEM).

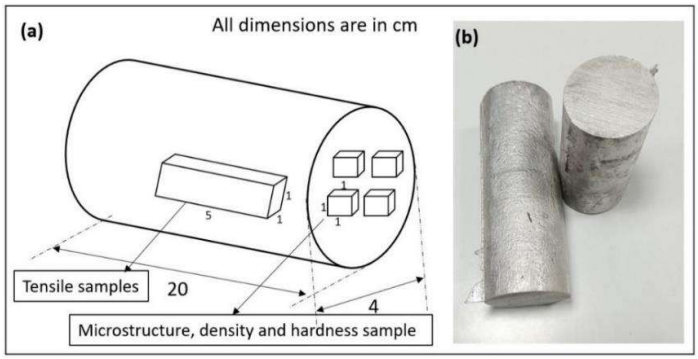

Fig. 3.2 (a) Location of test sample from casting (b) Image of the castings

3.5.1 Optical Microscope (OM)

A Leica Metallux-3 microscope attached with an image analyser was employed for microstructural investigations for optical microscopy. Samples were prepared for microstructural examination applying conventional metallographic practices. To prepare the sample, different grade of emery papers ranging from 400 to 2500 grit were utilized, followed by cloth polishing using brasso paste. The polished samples were etched by Keller's reagent (2.5%HNO_3, 1.5%HCl, 1%HF and 95%H_2O by volume) for about 10-15 seconds.

3.5.2 Scanning Electron Microscope (SEM)

The polished and etched specimens were further observed under scanning electron microscopes (SEM) of either ZEISS -EVO 18 FEI or NOVA Nano SEM 450. The elemental analysis and distribution of various elements in the hybrid composites was analysed by EDX analysis attached to the scanning electron microscope. The particle size distribution in composites was analysed using the SEM image.

3.5.3 Measurement of Particle size and shape factor

ImageJ software was used to calculate particle size using the following equation (Eq. 3.7) [73].

$$\text{Particle Size} = 2\sqrt{(Ao/\pi)} \quad (3.7)$$

Where A_o represents the area of the particles.

3.6 MECHANICAL PROPERTIES

3.6.1 Hardness Test

Vickers Hardness tester (Leco LV 700 AT) was used to measure the hardness of the matrix alloy and hybrid composites with varying TiB_2 percentage in the stir cast and CS cast conditions. ASTM E384 was followed in the preparation and testing of the samples. At least six measurements were taken at different locations using 5 kg load with 10 seconds dwell time and average values have been reported in the results.

3.6.2 Tensile Test

Tensile tests were performed on a screw-driven Instron™ Universal Testing Machine (UTM) at atmospheric temperature with a strain rate of 0.01 s^{-1}. Tensile testing was performed in accordance with the ASTM E8 standards, with specimens measuring 4.5 mm in diameter and 15.5 mm in gauge length. The graphs of engineering stress against engineering strain were used to calculate yield strength (YS), ultimate tensile strength (UTS), and percentage elongation. Average of three tests were taken to find out the actual value of YS, UTS and % elongation. The fractured surfaces were studied using NOVA EVO 18 SEM.

3.7 TRIBOLOGICAL TEST AND WORN SURFACES ANALYSIS

3.7.1 Wear and friction

Wear testing was performed on a standard pin-on-disc (POD) tribometer (Ducom TR-20 M26) under dry sliding conditions in accordance with the ASTM G99 standard. For the test, cylindrical pin samples with dimensions of 6 mm diameter and 30 mm length have been used. An EN31 steel disc with a hardness of 60 HRC was utilised as a counter surface for pin samples. The pin samples were polished before the test with different grades of emery paper and cloth polishing. Wear tests were performed at different applied loads (10 N to 40 N), sliding distances (1000 m to 4000 m), and sliding speeds (0.75 m/s to 3 m/s). The weight loss of the sample was measured using an electronic weight scale (METTLER TOLEDO) with a 0.1 mg accuracy.

The wear and friction data are expressed as wear volume, wear rate, wear coefficient, and coefficient of friction.

Wear volume has been defined by the following equation (Eq. 3.8) [ASTM standards G99, 2010]:

$$\text{Wear volume} = \frac{\Delta w}{\rho} \times 1000 \tag{3.8}$$

Where wear volume in mm^3, Δw is weight loss in g, ρ is density in g/cm^3

The wear rate was calculated using weight loss given in Eq. 3.9, and the coefficient of friction (COF) was determined using friction force. The friction coefficient is defined as the frictional force divided by the normal force.

$$W = \frac{\Delta w}{\rho \times d} \tag{3.9}$$

Where W- wear rate (mm^3/m), Δw- weight loss (g), ρ- density (g/cm^3) and d- sliding distance (m).

The wear coefficient is a more effective parameter for elaborating material wear properties, and it is determined by the following Eq. 3.10 [112]:

$$\text{Wear coefficient (K)} = \frac{\Delta w * H}{\rho * L * d} \quad (3.10)$$

where, Δw-weight loss, H-hardness (HV), ρ-density, L-applied load and d-sliding distance

SEM with EDS was utilized to analyse worn surfaces and wear debris in order to better understand the wear mechanisms involved during sliding wear. A three-dimensional (peaks and valleys) roughness profile was obtained using atomic force microscopy (AFM) to further analyse the worn surfaces.

3.7.2 Surface Topography

Wear surface topography was analysed using FESEM (Nova Nano SEM 450) connected with EDS and atomic force microscopy (AFM) using INTEGRA Prima microscopy to better understand the wear mechanism. These instruments have been utilised to assess the surface unevenness and roughness value (Ra in μm) of alloys and composites.

3.8 STATISTICAL MODELLING USING RESPONSE SURFACE METHODOLOGY

Response surface methodology (RSM) is a statistical method for optimizing process parameters. It is widely used where several independent variables influence the output response [33]. The input variables like load, sliding distance, sliding velocity and wt.% of TiB_2 reinforcement have been used in present study. RSM is used to establish a mathematical relation between input and output variables. Influence of input variables on wear rate and COF were studied using central composite design of experiments on Design Expert-13 software. Four factors with three level of variables were used in present

investigation as presented in Table 3. The optimal wear parameters for composites were validated using RSM based on central composite design (CCD). Load (A), sliding speed (B), sliding velocity (C), and varying TiB_2 wt.% (D) were used as independent variables, with wear rate and COF serving as response variables. The optimization of input variables to minimize wear rate and COF was obtained by desirability function method from the developed model. In order to validate the predicted value of wear rate and COF, set of confirmation test were performed using the optimised input parameters.

Table 3.2 Sliding wear parameters with their levels used in RSM

Sl. No.	**Parameters**	**Level**		
		-1 (Low)	**0 (Medium)**	**+1 (High)**
1	A: Load (N)	20	30	40
2	B: Sliding distance (m)	1000	2000	3000
3	C: Sliding velocity (m/s)	1.5	2.25	3
4	D: Wt.% of TiB_2 (%)	1	3	5