CHAPTER-3

EXPERIMENTAL PROCEDURE

3.1. Introduction

The materials, experimental set-up and different characterizing equipments 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 techniques adopted for characterization of fabricated FGMs such as microstructures, mechanical properties and tribological characteristics in as-cast as well as solution treated and ageing conditions have been described.

3.2. Procurement of materials

The A356 aluminum alloy ingots used as base matrix materials in synthesis of FG composites was procured from the M/s. Khandelwal Aluminum Works Pvt. Limited, Varanasi. The Mg-turning (99.9% Mg), Silicon metal (98.5%), fluxes like KCl (99.5%) and $MgCl_2.6H_2O$ granule (98%) and degasser Hexa-Chloro-Ethane (C_2Cl_6) used were of Molychem, India make.

3.3. Melting and casting

The A356 ingots and Al-20%Mg master alloy were melted at 750 °C in an electric resistance heating furnace in a clay-graphite crucible of 5 kg capacity. The melting was done under a flux cover of $MgCl_2$ and KCl mixture (1:1) to avoid any dross formation. After melt down the slag is skimmed off and melt was degassed with hexachloroethane (C_2Cl_6). The molten composite was then transferred to a preheated stir casting melting

furnace attached to vertical centrifugal casting machine (Fig.3.1) and was stirred for 10 min with a graphite stirrer for homogeneous mixing.

The melt was then poured through the bottom opening to a rotating cast iron mold at a constant rotational speed of 1200 rpm. The mold was preheated to 250 °C before pouring. The tubes of dimensions 100 mm in the outer diameter, 18 mm in thickness and 150 mm in length were produced in the centrifugal casting as shown in (Fig.3.2). The flow-chart for the synthesis process has been illustrated in (Fig.3.3). The chemical composition of the base alloy and the synthesized composites (Table 1) were analyzed with an Optical Emission Spectrometer (Foundry Master) as shown in (Fig.3.4).

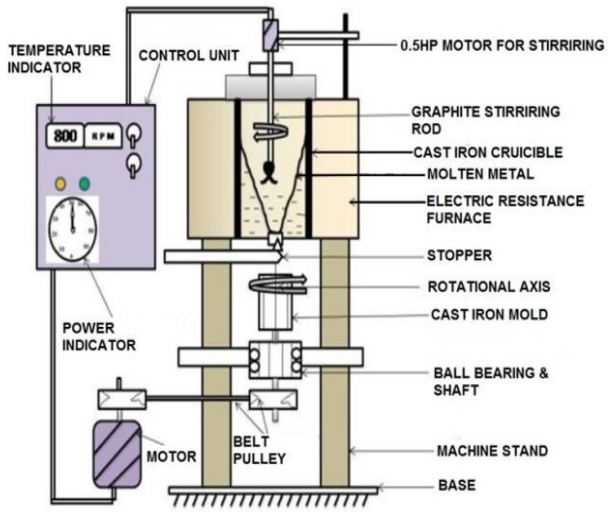

Fig.3.1 Set-up for stir casting attached with centrifugal casting machine.

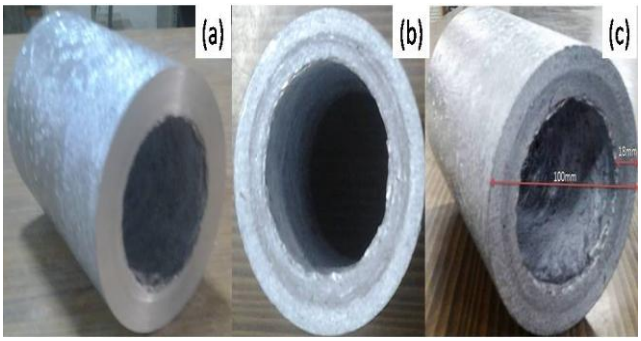

Fig.3.2 Centrifugally cast FGM tubes.

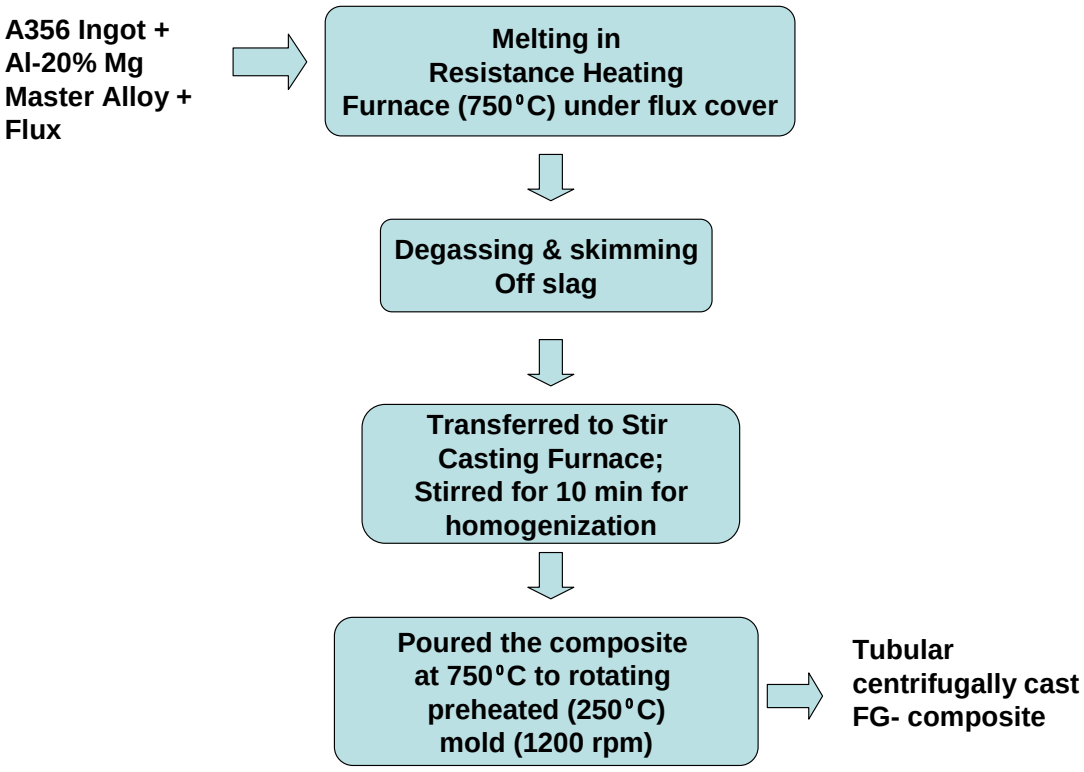

Fig 3.3. Flow-chart for synthesis of FG-composites.

Fig.3.4 Optical Emission Spectrometer (Foundry Master)

3.4. Solution heat treatment and ageing (T6) of Al-Mg_2Si in-situ FG-composites

The base A356 alloy and the FG composites were solution treated temperature(Ts) at 495^0 C ± 5°C for 7 h in a electrical muffle furnace and subsequently quenched in hot water at 90°C. The solution treated composites were subjected to artificial ageing temperature (Ta) at 160^0 C for different ageing times up to maximum of 10 hours in steps of 1hour.The heat treatment processes sequences are given as shown **Fig.3.5**

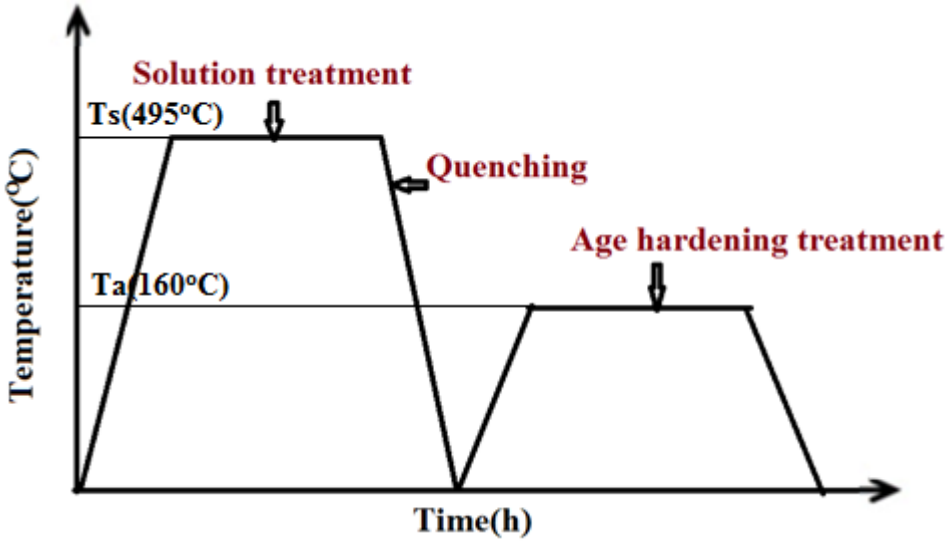

Fig.3.5. Solution treatment and ageing cycles (T6).

3.5 Microstructural characterization

For microstructural observations, samples were prepared following standard metallographic procedures. The polished surface was etched with Keller's reagent for about 5-10 seconds. For microstructural observations under optical microscope a Lieca Metallux-3 microscope equipped with an image analyzer as shown in Fig.3.6 was used. The volume percent of primary Mg_2Si, eutectic phase and intermetallic phase were quantitatively determined by the image analyzer. The polished and etched samples were further observed under scanning electron microscopes (SEM) of either FEI, Quanta 200F or ZEISS -EVO

18 as shown in Fig.3.7 . The elemental distribution of various elements in different phases in the FG composites was analyzed by EDX analysis attached to the scanning electron microscope. The as-cast and heat treated samples were examined by Transmission Electron Microscopy (TEM) Tecnai G^2-20 microscope as shown in Fig.3.8). Thin foils were prepared from sectioned bulk samples with the help of slow speed precision cutter. Further thinning down to a thickness of approximately 50 μm was made by mechanical thinning on fine emery paper. Discs of 3 mm diameter were punched form the mechanically thinned slice. Finally the discs were subjected to electrolytic thinning by twin jet polisher (TenuPol-5) using an electrolyte of 20% nitric acid and methanol at -35^0C at 20.5V.

Fig.3.6. Metallurgical microscope (Lieca)

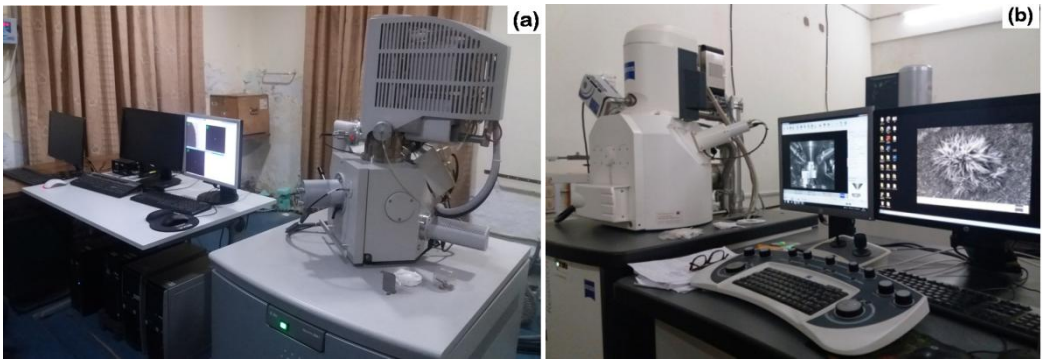

Fig.3.7. (a) SEM FEI, Quanta 200F and (b) ZEISS Model-EVO 18

Fig.3.8. Transmission electron microscope (Tecnai G^2-20)

3.6. Phase detection by X-Ray Diffraction (XRD) analysis

The phases present in the as-cast and heat treated composites were examined with a Rigaku Miniflex 600 DTex Ultra X-ray diffractometer (Fig.3.9.) using Mo Kα as well as Cu Kα radiations at an accelerating voltage of 35 kV. The 2θ angle was varied in the range of 20° to 80° with a scanning rate of 5°/minute. 'd' values obtained from XRD profile were compared with the characteristic d-spacing of all possible values from JCPDS cards to identify different x-ray peaks. A NaOH (20%) and water solution was used as etchant to extract primary Mg_2Si phase from Al–Mg_2Si in-situ composite. In this process, a small section was cut from the inner zone of cast Al-Mg_2Si FG composites and emerged in enchant for 20h. After that, Mg_2Si phases were separated from Al-Mg_2Si cast composites due to dissolution of the Al matrix. The residue of Mg_2Si phase were treated with an ultrasonic cleaner to clean repeatedly and alternatively washed out with water and alcohol. The XRD analysis of extracted Mg_2Si particles was carried out for observation of peak intensities and conforming the Mg_2Si phases as shown in result and discussion chapter.

Fig.3.9. X-Ray diffractometer (Rigaku Miniflex 600 DTex)

3.7. Determination of secondary dendrite arm spacing (SDAS) and solidification time

The SDAS is calculated following the relationship, SDAS= L/nM, where L is the length of the line, M is the magnification, and n is the number of dendrite cells. The solidification times (t_E) have been calculated knowing the secondary dendrite arm spacing from the equation:

$$t_E = \{SDAS/A\}^3$$

where, 'A' represents a material-dependent constant, for the type Al-Si7-Mg aluminum alloy, this is taken as 11.7 for Al.

3.8. Determination of density and porosity% with increasing wt.% of Mg

The experimental densities (ρ_{exp}) of the composites were measured by Archimedes principle. The theoretical density values were computed applying the rule of mixtures using measured volume percentage of Mg_2Si particles.

$$\rho_{th} = v_f * \rho_{f+} v_m * \rho_m \text{.....i}$$

where 'v_f' Volume Fraction of particles and 'v_m' Volume Fraction of Matrix, 'ρ_f' is density of particles, 'ρ_m' is density of matrix. The % porosities in the composites were calculated from the theoretical and experimental densities of formulae as expressed follows.

$$\%\text{Porosity}=\left(\frac{\rho_{th}-\rho_{exp}}{\rho_{th}}\right)*100 \quad \text{.....ii}$$

where ρ_{th} is theoretical density and ρ_{exp} is an experimental density of functionally graded composites.

3.9. Measurement of hardness

The hardness of the base alloy and the FG composites in the as-cast and ageing condition were measured by a Vickers Hardness tester (Leco make) as shown in Fig.3.10, using a load of 5 kg with dual time of 10 seconds. In as-cast FG composites the hardness vs. distance profiles in the radial diameter direction with 1 mm distance interval have been plotted. For a particular distance, at least six measurements were taken on the same arc radius and average values have been reported. In case of solution treated and aged samples average of three readings at different locations has been reported.

3.10. Measurement of tensile properties

The tensile tests were performed as per ASTM- E8 at temperatures of 25 °C (room temperature), 150 °C and 300 °C using an Instron-5848 tensile testing machine as shown in Fig.3.11 with a crosshead speed of 0.15 mm/min (corresponding to engineering strain rate of 1 $x10^{-3}s^{-1}$). Three samples were tested at a particular temperature for each zone of the FG- composites. Before testing, each specimen was homogenized for 20 min at the test temperature in the heating chamber of the testing machine. The schematic view of

dimension of tensile test sample is shown in Fig.3.12 having 15.5mm of gauge length and 4.5mm of gauge diameter.

Fig.3.10. Vickers Hardness tester (Leco LM248 AT)

Fig.3.11. Instron-5848 tensile testing machine

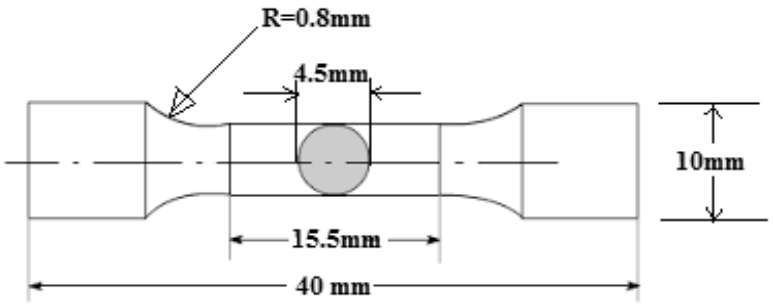

Fig.3.12. Schematic view of tensile test sample dimensions.

3.11. Evaluation of tribological characteristics

3.11.1. Dry sliding wear at room temperature

Dry sliding wear behavior were evaluated using a Pin-on-Disc type tribometer (DUCOM, TL-20) as shown in Fig.2.13 with data acquisition system of FG composites and matrix alloy against hardened high-carbon chromium steel counterface (HRC-64) shown in Fig.2.14 of surface roughness (Ra) 0.511μm. Cylindrical samples (30 mm length and 8 mm diameter) were used for wear and friction under dry sliding conditions at room temperature. The wear tests were carried out with varying normal loads (10, 20,30 and 40N) and sliding distances (1200, 1800, 2400 and 3000 m) at fixed sliding velocity of 1.15 m/s. The samples were cleaned with acetone in ultrasonic bath and weight losses were measured in a digital balance (least count of 0.1 mg). Wear rate was calculated from the weight loss measurements for each sliding conditions. The frictional force and applied loads was used for determination of coefficient of friction. The reported values are based on the average of three readings. The roughness profile of the worn surfaces of the inner reinforced zones of the FG composites at various sliding distances with applied normal load 40N were measured by a contact stylush profilometer (Mitutoyo, Surftest, SJ-410). The examination of worn surface topography and surface elemental analysis were

performed under SEM attached with EDX facility. The subsurface characteristics of the worn surfaces were observed by optical microscopy in the transverse sections.

Fig.3.13. Pin-on-Disc type tribometer (DUCOM, TL-20, Bangalore, India)

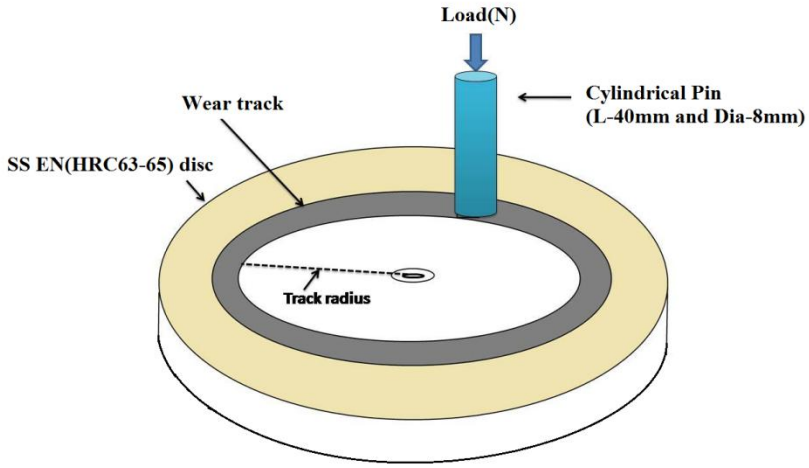

Fig.3.14. Schematic representation of Pin-on Disc sliding wear test

3.11.2. Linear reciprocating wear at elevated temperature

The condition of scuffing wear takes place in between the piston and the cylinder bore wall was evaluated by Linear Reciprocating Tribometer (Ducom Make) which complies with **ASTM G133** standard was used to perform the wear test at 200°C temperature (Fig.3.15). In this test upper specimen is in the form of ball and lower

specimen is in the form of flat square block (40x40x5mm), it is mounted on a stage which can be heated to desired temperature. Linear relative contact of ball with flat block takes place for the wear tests till the specific time periods. The test samples are held in holders for testing conformal contacts of ball and blocks. The constant test load 200N was applied by dead weights on the top of ball holder. This option facilitates a predefined stepwise or ramp loading profile over the test. Frictional force was measured with piezoelectric sensor and plotted against time. The linear reciprocating wear test parameters are shown in Table.3.1 to conform the test conditions.

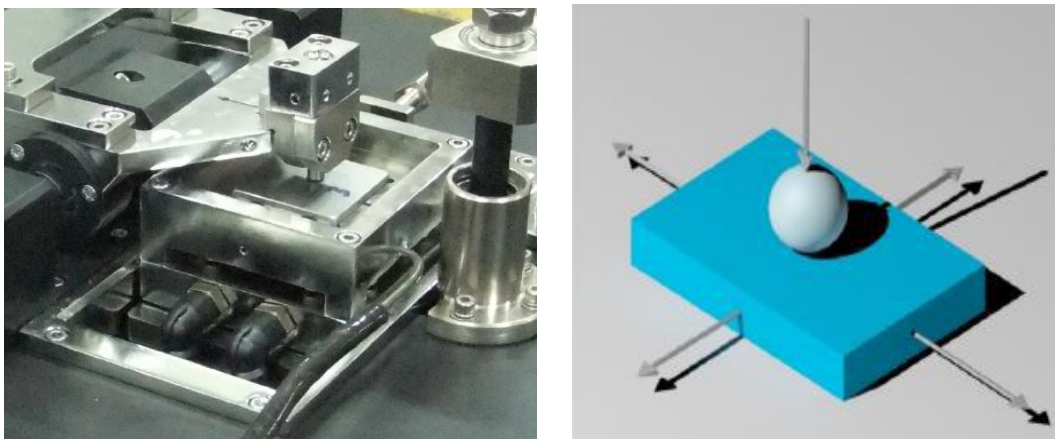

Fig.3.15. Ducom Linear reciprocating tribometer and ball on test block specimen.

Table.3.1 Linear reciprocating wear test parameters

S.N.	**Test parameters**	
1	Load in N	200
2	Frequency in Hz	25(1500RPM)
3	Duration in seconds	2400 (40 min)
4	Temperature in °C	200
5	Stroke in mm	5
6	Top specimen	Ø10 mm, hardened steel ball

Precautions were taken to drive the ball specimen in a smooth manner as per the ASTM standard suggesting implementing a drive mechanism which would allow for the ball specimen to follow a reciprocating motion without the need for the drive motor to stop and reverse direction

between each stroke. Additionally, the apparatus was fitted with a mechanism to count the number of cycles, such that the test may be automatically terminated after a specified number of cycles. After the wear test the worn surfaces, ball surface and subsurface characteristics were examined under scanning electron microscope and the elemental distributions with the attached EDX facility.