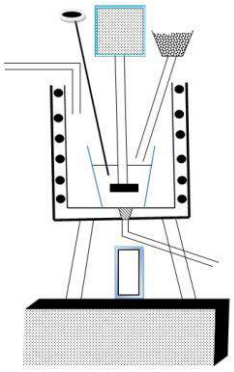
Chapter 2



EXPERIMENTAL DETAILS

2.1 Introduction

This chapter provides details of fabrication of composites and their characterization using different techniques.

2.2 Fabrication

Composites with single and multiple reinforcements were fabricated by the direct melt reaction (DMR) *insitu* technique. In this process inorganic salts were pre-heated in an electric oven (Fig. 2.1a) at desired temperature for few hours to remove moisture, on the other hand small pieces of Al alloy were charged into the graphite crucible kept in a vertical muffle furnace (Fig. 2.1b) for melting under argon atmosphere. Pre-calculated amount according to stoichiometric ratio of dehydrated inorganic salt/salts was/were added in the aluminium alloy melt with the help of graphite plunger to get the required vol.% of reinforcement particles. Temperature of the molten metal was maintained at specific temperature for completion of *insitu* reaction. The temperature of the melt was monitored by K-type thermocouple. During the *insitu* reaction, mechanical stirring was done with the help of graphite stirrer and the melt was degassed by tetra-chloro-ethane.

Finally, the melt was bottom poured into a preheated fire clay coated permanent mould having 45 mm inner diameter. Ingot of as cast composite (Fig. 2.2) was cut to prepare samples for different characterizations such as phase analysis, morphology, mechanical and tribological properties [Gautam and Mohan, 2015, 2016].

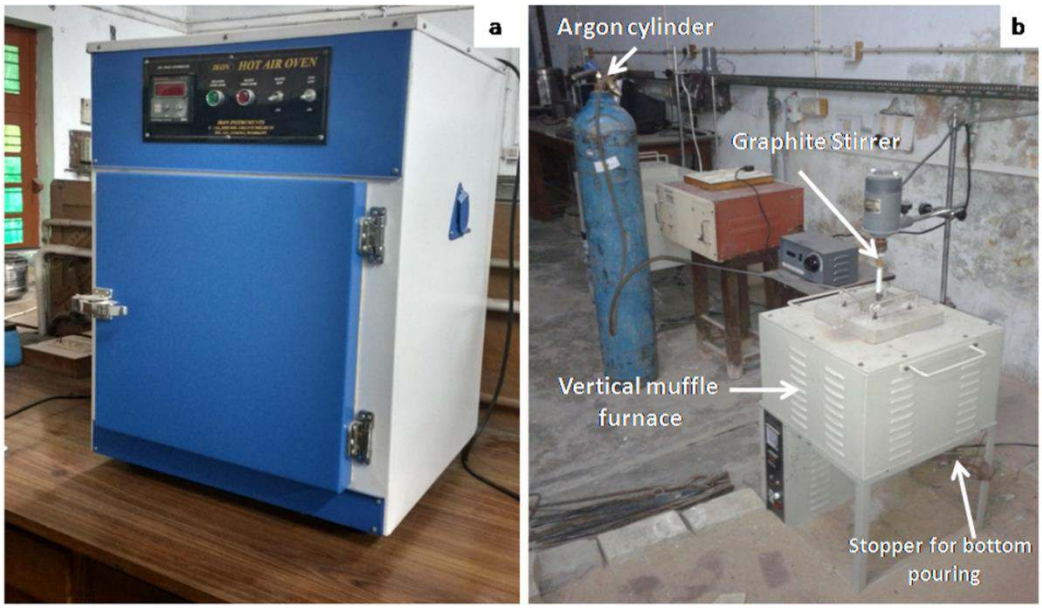


Figure 2.1 – (a) electric oven used to dehydrate inorganic salts and (b) stir-casting unit with bottom pouring arrangement.

Figure 2.2 – As cast composite ingot.

2.3 Phase Analysis by X-ray Diffractometer

The *insitu* formed reinforcement particles having different vol.% in as cast composites were identified by Rigaku Miniflex II X-ray diffractrometer using Cu Kα radiation (Fig. 2.3). The standard JCPDS files were used to compare the "d" spacing of *insitu* formed second phase particles.

The presence of these particles in the composites was further confirmed by the XRD analysis of extracted particles. The particles were extracted from the composite by making its solution in the 10% HCl which took several days. The matrix of the composite dissolved in 10% HCl. The solution was filtered by ash less filter paper and extracted particles were heated in electric oven at 100°C for 24 hours to eliminate the moisture content.

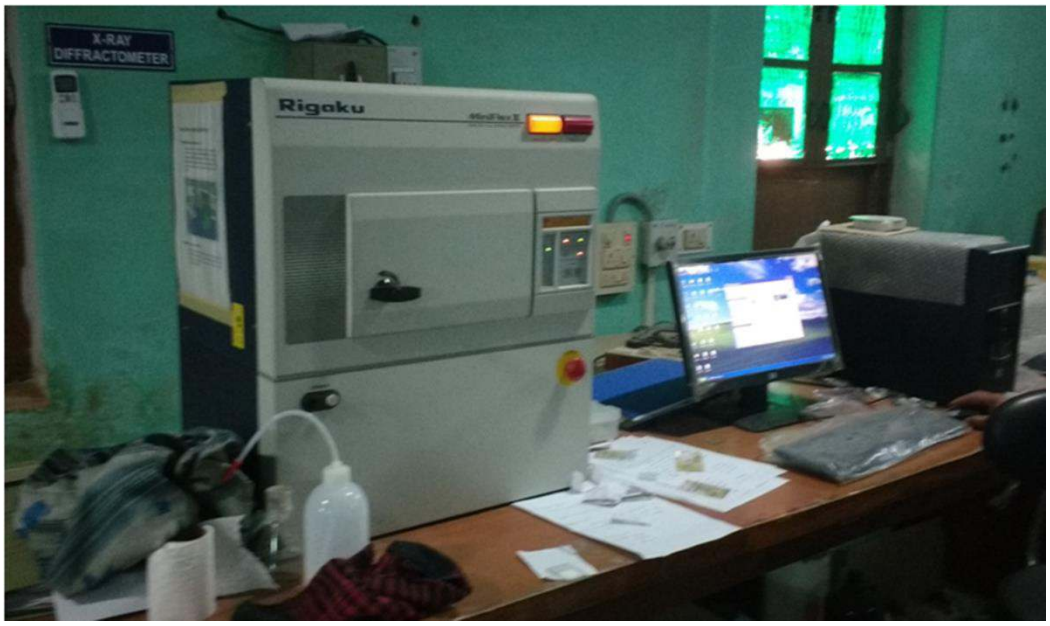


Figure 2.3 – XRD machine for XRD analysis of composites.

2.4 Compositional Analysis

To find out the actual vol.% of reinforcement particles in all composites, optical emission spectrometer (Fig. 2.4a) study was carried out. Scratch free samples were prepared to eliminate the problem of electric spark during testing and these were cleaned with acetone before test. The chemical composition of all composites was taken at four regions (Fig. 2.4b) and their average was taken to calculate the vol.% of reinforced particles [Gautam and Mohan, 2015; Gautam et al., 2016c].

Figure 2.4 - (a) Optical emission spectrometer for chemical composition analysis of composites, and (b) composite sample after test.

2.5 Thermal and Physical Properties

2.5.1 Differential Thermal Analysis (DTA)

Differential thermal analysis (DTA) of composite sample was carried out on DTA/TGA apparatus (NETZSCH STA 449F3 STA449F3A-0843-M) as shown in Fig. 2.5. The study was conducted to find the reaction temperature between base alloy and inorganic salts. In this testing, small quantity of composite sample was taken and was heated in the furnace up to 1100°C in an Al_2O_3 crucible under nitrogen atmosphere. The heating rate was maintained at 10°C/min. Endothermic and exothermic peaks were analyzed to find out the corresponding temperatures [Gautam et al., 2016c].

2.5.2 Coefficient of Linear Thermal Expansion

The theoretical value of coefficient of linear thermal expansion for all compositions was evaluated using Turner's model [Agrawal et al., 2014] as given below –

$$\alpha_c = \frac{V_r K_{Br} \alpha_r + V_m K_{Bm} \alpha_m}{V_r K_{Br} + V_m K_{Bm}} \quad (2.1)$$

Figure 2.5 – DTA/TGA machine for DTA analysis of composite.

Where, α is the coefficient of thermal expansion, V is volume fraction, K_B is the bulk modulus, c denotes composite, r and m denote reinforcement and matrix phase respectively. Value of coefficient of thermal expansion for ZrB_2, Al_3Zr and base alloy are $6.88 \times 10^{-6} K^{-1}$ [Tian et al., 2014], $12.60 \times 10^{-6} K^{-1}$ [Belov et al., 2002] and $23.80 \times 10^{-6} K^{-1}$ [Handbook ASM Vol. 2, 1990] respectively, while bulk modulus are 240 GPa [Okamoto t al., 2003], 100.05 GPa [Clouet et al., 2002] and 70.70 GPa [Handbook ASM Vol. 2, 1990] respectively.

2.5.3 Density and Porosity

The theoretical density of the composites with different vol.% of reinforcement particles was calculated by the rule of mixtures (ROM) technique which is given below [Varin, 2002] –

$$\rho_C = \rho_R VF_R + \rho_M VF_M \quad (2.2)$$

Where ρ_C, ρ_R and ρ_M denote density of composite, reinforcement phase, and matrix phase respectively, and VF_R and VF_M are volume fraction of reinforcement and matrix phase respectively.

For experimental density measurements composite samples were taken from the middle section of the ingot. The experimental density was evaluated by Archimedes principle as given in equation 2.3 [Prasad et al., 2014] –

$$Density = \left(\frac{Weight\ in\ air}{Weight\ in\ air - Weight\ in\ water}\right) \quad (2.3)$$

Where, density is in g/cm^3 and weight of samples in air and water is in g.

The porosity was evaluated with the help of theoretical and experimental densities for all composite samples as given below [Prasad et al., 2014] –

$$Porosity = \left(\frac{Theoretical\ density - Experimental\ density}{Theoretical\ density}\right) \times 100 \quad (2.4)$$

Where, porosity is in volume percent while theoretical and experimental densities are in g/cm^3.

2.6 Microstructural Characterization

2.6.1 Optical Microscope (OM)

The Leitz Metallux-3 optical microscope (Fig. 2.6) was used to study the morphology of Al-rich grain in composites with different vol.% of reinforcement particles. The composite samples were prepared following standard polishing procedure. After initial grinding the samples were prepared using standard emery paper grades and finally cloth polishing was done to get mirror finish. Keller's reagent consists of 5 ml nitric acid, 3

ml hydrochloric acid, and 2 ml hydrofluoric acid in 190 ml water was used as etchant. Then the samples of all compositions were studied under optical microscope.

Figure 2.6 – Optical microscope.

2.6.2 Scanning Electron Microscope (SEM)

FESEM Quanta 200FEG scanning-electron microscope (SEM) and ZEISS (Model-EVO 18) SEM (Fig. 2.7) were used to study the morphology and distribution of second phase reinforcement particles in the composites. Second phase particles were also confirmed by elemental analysis through EDS attached to FESEM Quanta 200FEG SEM.

2.6.3 Transmission Electron Microscope (TEM)

The morphology of second phase reinforcement particles & their distribution in the composites, crystal structure of different phases and presence of dislocations in the composites were studied by transmission electron microscope, model TECNAI G^2 20

(Fig. 2.8) at 200 kV. The specimens were thinned up to 50 µm by different grades emery papers and discs of 3 mm diameter were prepared by punching these specimens. Punched disc specimens were electro-polished by twin-jet polishing facility using a solution of 20% HNO_3 and 80% CH_3OH at -35°C and 20V.

Figure 2.7 – Scanning-electron microscope.

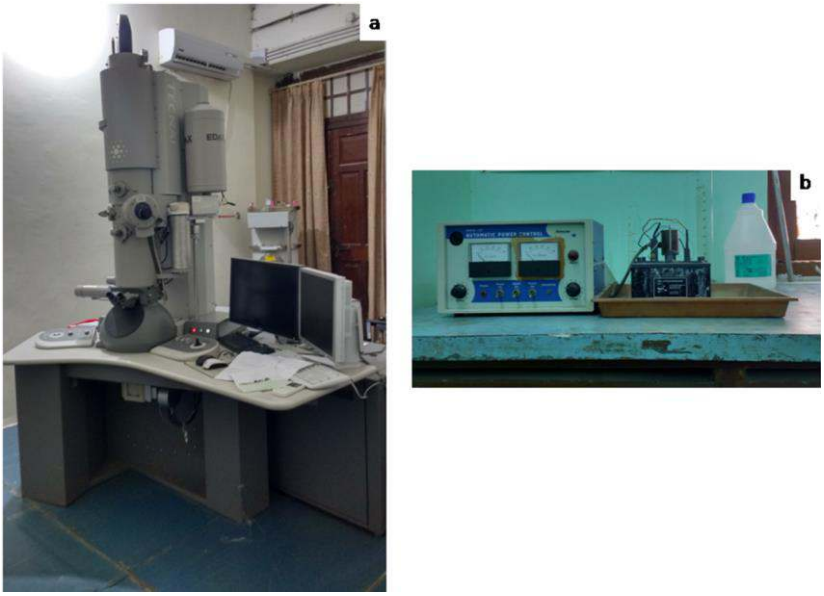


Figure 2.8 – (a) Transmission-electron microscope and (b) twin-jet electro-polishing machine.

2.7 Mechanical Properties

2.7.1 Hardness Test

To see the effect of vol.% of reinforced particles on hardness, bulk hardness tests were performed on Aktiebolaget Alpha Brinell hardness testing machine (Fig. 2.9a). 10 mm steel ball was used as an indenter under 500 kgf load and 30 s dwell time was taken. The testing specimens were prepared following standard procedure and the average of five readings was taken and reported.

To check the hardness of reinforcement particles and their effect on the hardness of matrix in the composite, Vickers micro-hardness testing was performed by Shimadzu micro hardness tester (Fig. 2.9b) with diamond indenter under a load of 0.01kg with 5 s dwell time.

Figure 2.9 – (a) Brinell hardness tester and (b) Vickers micro-hardness tester.

2.7.2 Tensile Test

To evaluate the effect of vol.% of reinforced particles on the tensile properties, the tensile tests were carried out on 100 KN screw-driven Instron™ Universal Testing Machine Model 4206 (Fig. 2.10a) at room temperature at a strain rate of 1.07×10^{-3} s^{-1}. Standard cylinder specimens with 15 mm gage length and 4.5 mm diameter of as per BS specification no. 12-1950 were used for tensile testing. The schematic representation of the tensile specimen is shown in Fig. 2.10b. During testing, the tensile data was recorded to plot the engineering stress and strain curve. The plots between engineering stress and engineering strain give the value of ultimate tensile strength (UTS), yield strength (YS) and percentage elongation. Average of three tests has been reported for UTS, YS and % elongation. The fractured surfaces were studied under FESEM Quanta 200FEG SEM.

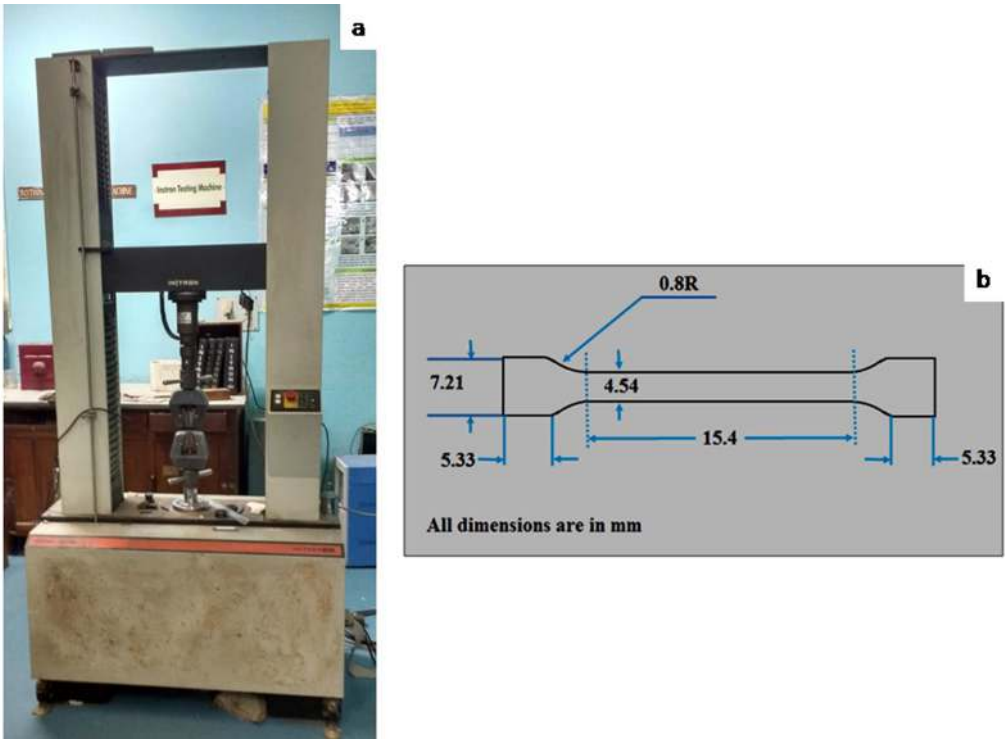


Figure 2.10 – (a) Tensile testing machine, and (b schematic representation of tensile specimen.

Tensile properties were also studied at elevated temperature up to 250°C at an interval of 50° with same test conditions and fractured surface were observed under ZEISS (Model-EVO 18) SEM.

2.8 Tribological Properties

2.8.1 Wear and Friction

The composites with different vol.% of reinforcement particles were subjected to dry sliding wear and friction on a pin-on-disc configuration of a multi-function tribometer from Rtec Instruments, USA (Fig. 2.11). As per ASTM standards, cylindrical specimens of 8 mm diameter and 30 mm length were used. Tests were conducted against a hardened steel disc. Before conducting the wear test, the surface of cylindrical test specimens and disc were polished and cleaned with acetone. An analytical balance with the precision of 0.1 mg was used to weigh the samples before and after the test. The wear rate was evaluated from the weight-loss measurements, and volume loss per unit sliding distance was calculated [Handbook ASM Vol. 18, 1992]. Volume loss has been defined by following equation according to ASTM standard [ASTM standards G99, 2010]:

$$\text{volume loss} = \frac{\text{mass loss}}{\text{density}} \times 1000 \tag{2.5}$$

Where, volume loss is in mm^3, mass loss is in gram, and density is in g/cm^3.

The coefficient of friction was calculated with the help of recorded frictional force data during experiment.

Wear and friction tests were carried out for different distance slid from 600 to 3000 m, load range of 10 to 40 N, and sliding velocity in a range of 1 to 4 m/s for all compositions. The initial (before test) surface roughness of the cylindrical pin sample and steel disc was measured as ≤ 0.39 μm by profilometer and the conformal contact was maintained throughout the study [Gautam and Mohan, 2016, Gautam et al., 2016a, 2016c].

Figure 2.11 – Pin on disc configuration of a multi-function tribometer.

Wear and friction tests were also conducted in air on a pin-on-disc configuration of a multi-function tribometer from Rtec Instruments, USA, with a high temperature module furnace as shown in Fig. 2.12a. The diameter of pin sample was 9 mm and the length was 12 mm as shown in Fig. 2.12b. The counterpart EN31 steel disc of 50 mm diameter and 6 mm thickness was used (Fig. 2.12c). The tests were carried out at different applied loads (10 to 40 N) and different temperatures i.e. room temperature, 50°C, 100°C, 150°C, 200°C, 250°C. All tests were conducted at constant sliding velocity (0.5 m/s) for 1000 m sliding distance.

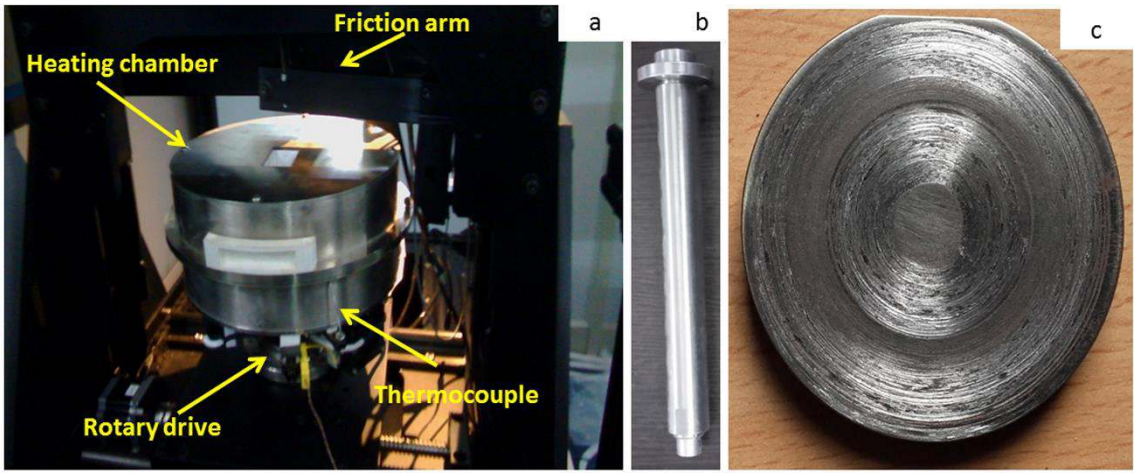


Figure 2.12 – (a) High temperature wear testing machine, (b) sample holder with sample, and (c) EN31 disc.

2.8.2 Surface Topography

The worn surfaces (wear tracks) of the test specimens with different test conditions at room and elevated temperatures were studied under FESEM Quanta 200FEG scanning electron microscope (SEM) and ZEISS (Model-EVO 18) SEM to understand the mode of damage and wear mechanism. The 3D-profilometer (Fig. 2.13) from Rtec Instruments was also used to study the topography of worn surfaces. Profilometer provides the colour contour with peaks and valleys of the worn surface. Surface roughness values and 3D pattern of worn surface were also observed by 3D-profilometer to understand wear phenomenon [Gautam and Mohan, 2016; Gautam et al., 2016a, 2016c].

The wear debris was also analyzed by energy-dispersive X-ray spectrometer (EDS) of Oxford instrument (Model-51-ADD0048) attached with ZEISS (Model-EVO 18) scanning-electron microscope (SEM) to confirm the presence of elements under different operating conditions [Gautam and Mohan, 2016; Gautam et al., 2016a].

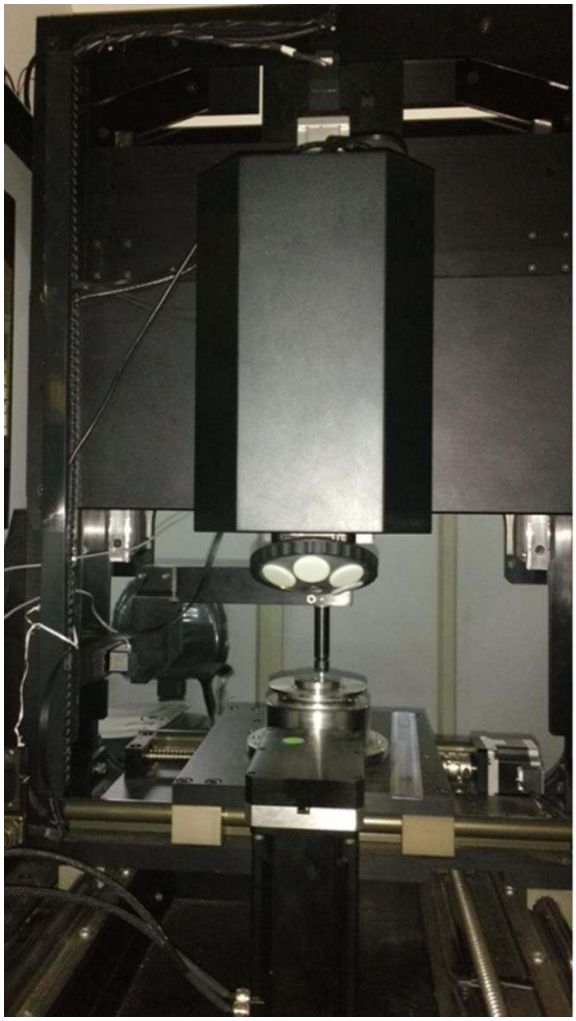


Figure 2.13 – 3D-Profilometer.