CHAPTER-3

MATERIALS AND EXPERIMENTAL PROCEDURES

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This chapter focuses on the selection of matrix materials as well as reinforcements in metal matrix composites. The base alloy composition and reinforcement's morphology along with EDAX have been presented. The different characterization techniques and testing methods which are used for magnesium alloy and its composites have also been discussed.

3.1. The selection of matrix materials

Based on available literature review and objective of the current research the properties of different matrix materials and reinforcement has been reviewed. The commercial magnesium alloy AZ91 has been used as matrix materials in the current research. AZ91D is the extensively used magnesium alloy due to following outstanding properties: good fluidity, castability, corrosion resistance, mechanical properties, low specific heat per unit volume and reduced tendency to adhere to dies [135]. The compositions of the base alloy were obtained through optical emission spectroscopy at IIT Kanpur. The main alloying elements of the base alloy are given in Table 3.1

Table 3. 1: The composition (% wt.)of commercial alloy AZ91

1	**2**	**3**	**4**	**5**	**6**
Mg	Al	Zn	Mn	Si	Other minor elements
89.467	**8.87**	**1.017**	**0.18**	**0.086**	**rest**

3.2. The reinforcement

The two different types of reinforcement are used for the synthesis of magnesium alloy AZ91 based metal matrix composites. The SiC particulates (average size of 40 microns) are used as the first type of reinforcement. The TiC particulates (average size of 20 microns) are used as a second type of reinforcement. The small variations (3 %, 6 %, 9 %, and 12 %) of both the reinforcement are added in the magnesium alloy AZ91. The basis of selection of reinforcement is the bonding and wettability between the reinforcement and matrix materials. The different characterization techniques are used to determine the properties of monolithic AZ91 and its composites reinforced with above-discussed reinforcements. The morphology of the SiC particulates reinforcement with the help of a scanning electron microscope is given in Figure 3.1, and Figure 3.2 shows the EDAX of SiC particulates. Figure 3.1 shows the size of the reinforced particle, and it is clear from the figure that the reinforced particles have different size and shape. Figure 3.3 shows the scanning electron microscope image of TiC particulates and Figure 3.4 shows the EDAX of TiC particulates. The elemental compositions of the reinforcements were almost the same as per our requirement.

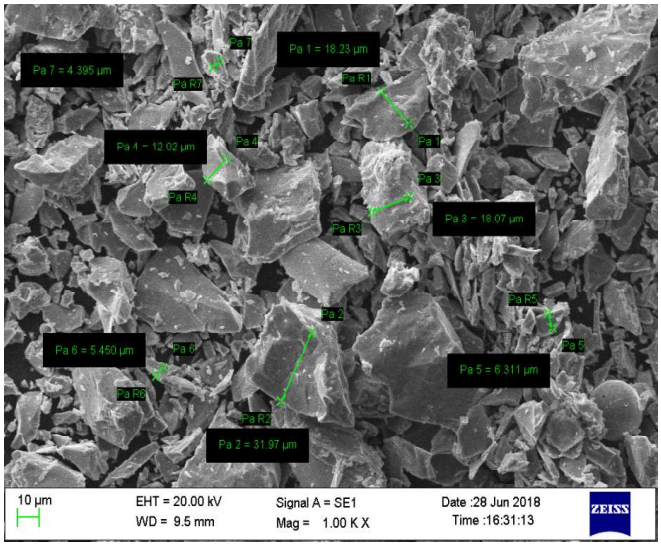

Figure 3. 1: Morphology of SiC particulates

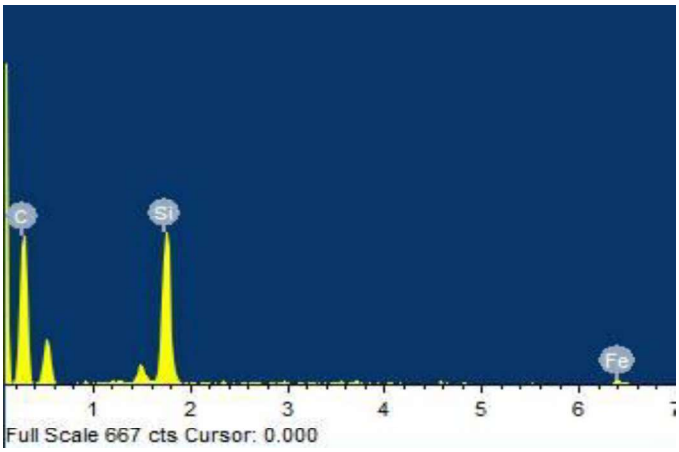

Figure 3. 2 : EDAX of SiC particulates

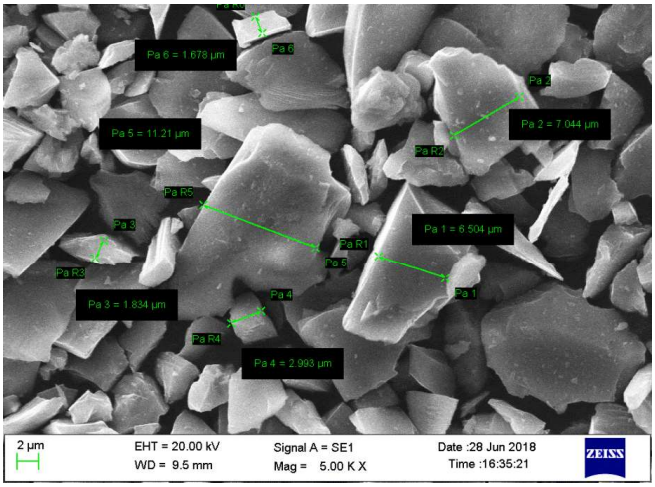

Figure 3. 3: Morphology of TiC particulates

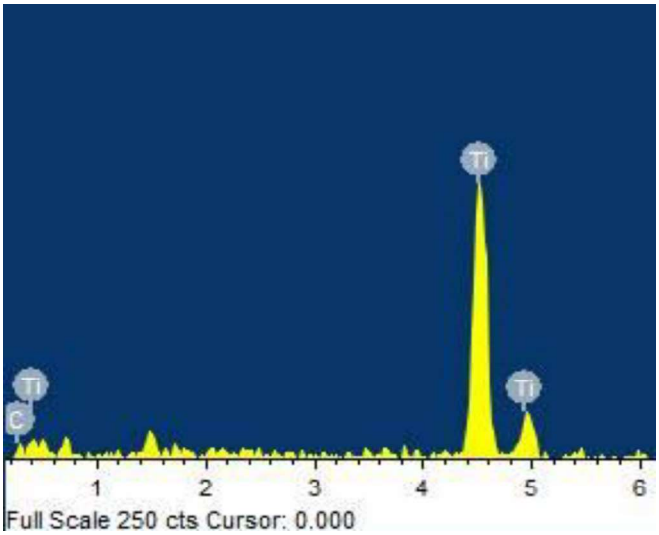

Figure 3. 4: EDAX of TiC particulates

3.3. Design and development stir casting set-up

The design and fabrication of stir casting experimental set-up for casting have been discussed in Chapter 4. Two different stir casting facilities have been developed for casting magnesium alloy and its composites. The detail processing capability of the stir casting set-up and way of incorporating reinforcements in the matrix materials are also discussed in Chapter 4.

3.4. Density and porosity measurement

Density (ρ) measurements of magnesium alloy (AZ91) and its composites were performed on the polished sample. The density measurement was carried out in accordance with Archimedes principle [170]. Distilled water was selected as the immersion fluid. Three samples were arbitrarily selected and carefully weighed both in the air and when fully immersed in distilled water [171]. An electronic balance with an accuracy of 0.0001 g is used for measuring the weights. The theoretical densities of each of the composites have been calculated using the rule of mixtures equation as shown below:

$$\rho_{th} = V_r \rho_r + (1 - V_r)\rho_m \qquad (3.1)$$

Where ρ_{th} is the theoretical density (g/cm^3), V_r is the volume fraction of reinforcement, ρ_r and ρ_m is the density (g/cm^3) of the reinforcement and the matrix respectively.

Experimental density is calculated using the following equation:

$$\rho_e = \frac{W_a \rho_w}{W_a - W_w} \qquad (3.2)$$

Where ρ_e is the experimental density (g/cm^3), W_a is the weight of the specimen in air, ρ_w is the density of the water and W_w is the weight of the specimen in water.

The porosity of the composite material is calculated using the following equation

$$porosity = \frac{\rho_{th} - \rho e}{\rho_{th}} \times 100 \qquad (3.3)$$

For theoretical computing value of the density of the composites, the density value of 1.81 g/ cm^3 for matrix material and 3.216 cm^3 for SiC particle and 2.66 g/cm^3 for TiC particulates were used.

3.5. Microstructural characterization

Microstructure characterization studies are performed on the sample, which is metallographically polished [67]. The primary objective of these studies was to investigate the composite in term of reinforcement distribution, interfacial integrity between the matrix and reinforcement [172] and morphological characteristics of grains in comparison to AZ91 alloy. The die-cast composites with the different percent of SiC particles are sectioned into small pieces for mechanically grinding and polishing. The mirror polished sample etched with a suitable etchant for 5 seconds to determine the related microstructure. The etchant is prepared by mixing ethyl glycol, distilled water, and nitric acid in the ratio of 75:24:1. The grinding paper of 200,400, 800, 1000, 1200, 2000 and 2500 grit size of SiC abrasive is used for grinding/polishing. The metal matrix composites (MMC) specimen finally polished on velvet cloths with diamond paste (Mono-crystalline) of 1-micron size. The obtained microstructure samples are examined by a Leica optical microscope (O.M.) equipped with a Leica application suite. Further ZEISS scanning electron microscope equipped with E.D.S. is used to investigate the samples surface topography with particle distribution and elemental compositions.

3.6. X-Ray diffraction studies

The X-ray diffraction analysis is carried out on the polished samples [173] of the monolithic magnesium alloy AZ91 and its composites using MiniFlex 300/600 Regaku table top XRD diffractometer to determine the possible available phases. The samples are exposed to Cu K-alpha radiation (λ= 1.5406 Å) at a scanning speed of 10°/min and a step width of 0.020 °. The scan range was 10.0000° - 90.0000° with continuous scan mode. The Bragg's angle, the value of the interplanar spacing (d) and intensity height (counts) is subsequently matched with the standard Mg, $Mg_{17}Al_{12}$ SiC, TiC, and other related phrases.

3.7. Mechanical characterization

3.7.1. Microhardness

The hardness test for the entire composite has been performed to estimate the distribution homogeneity of reinforcement particles as well as the variation of hardness with different percentage of reinforcement. The Vickers microhardness measurements are conducted across the section of polished composites by applying a load of 9.80 N with a dwell time of 10 seconds. LECO's LV Series micro-Vickers hardness machine is used for testing hardness. A pyramidal diamond indenter with a facing angle of 136^0 is used for indentation.

3.7.2. Tensile test

The tensile test of particulate reinforced metal matrix composites is performed to estimate the mechanical properties of vacuum-assisted stir die-cast composites. The tensile samples are prepared from composites reinforced with different percentage of SiC/TiC particles as well as commercial magnesium alloy AZ91. The gauge length and

gauge diameter of the prepared tensile specimen was 20 mm and 8 mm respectively, according to the ASTM E8/E8M- 16a standard [174]. The tensile tests are carried out at room temperature on the Instron-4208 universal testing machine under the initial strain rate of 0.005 s.

3.7.3. Compressive test

Uniaxial compressive tests at room temperature are performed on cylindrical monolithic AZ91 alloy and its composite sample according to ASTM standard E9 [175]. The sample length and diameter of the compressive specimen was 12 mm and 8 mm respectively to make the aspect ratio (l/d) of 1.5. Aimil makes semi-automatic universal testing machine is used to performed compression tests.

3.8. Factograph of tensile and compressive tests

Factograph studies are performed on the tensile and compressive fractured specimens of monolithic magnesium alloy and its composites to investigate various possible fracture mechanisms operating insight the sample during tensile and compressive loading. These studies are performed using a ZEISS Field Emission Scanning Electron Microscope (FE-SEM) at different magnifications.

3.9. Wear behavior studies

Pin on disk friction and wear testing machine is used to evaluate the tribological characteristics of the magnesium alloy and its composites under dry conditions at room temperature. The wear sample is made as per ASTM standard G 99 [176]. The disc of the pin on the disc is made of EN31 steel having surface roughness 0.1. The friction and wear weight loss report is generated by varying the three parameters namely velocity, sliding distance, and normal load. The dimensions of prepared wear test

samples were 8 mm in diameter and 30 mm in length. The end surfaces of the wear test samples are polished adequately with different grades of abrasive paper (i.e., 200, 800, 1000, and 2500). All wear test samples are weighed at the start and end of each test. The wear weight loss is measured by Ohaus adventurer weighing machine with an accuracy of 0.0001 g. The worn surfaces of the wear tests sample are also characterized by scanning electron microscopes (SEM) examinations.

3.9.1. Test parameters

The following test parameter is generally used to evaluated wear behaviors of the pin materials [176]**.**

1. **Load**: The normal force values used in Newton at the wearing contact.
2. **Speed:** The relative sliding speed between the pin and disk surfaces is selected in meters per second.
3. **Distance:** The sliding distance was measured in meters.
4. **Atmosphere:** The atmosphere (laboratory air, relative humidity, etc.) around the wearing contact was assumed as constant during the period in which the test was performed.

Out of these parameters, the only variation in load, speed, and distance are taken for the current study.

3.9.2. Wear test procedure

The following test procedures are used to evaluate wear behaviors of the materials [176]**.**

1. Clean the specimens with acetone and dry it before measuring its dimension and weight. The least count for dimensions measurement is 2.5 μm, and the least count of weighing machine is 0.1 mg.

2. Insert the specimens in its holder and adjust the specimen perpendicular to the disk surface, at the time of contact, to maintain the necessary contact condition.
3. Add the proper weight to the lever, so that the desired normal force can be developed for pressing the pin against the disk.
4. Start the machine by holding the pin specimen out of contact with the disk and adjust the speed to the desired number of revolution.
5. Start the test with the specimen in contact with the disk under different load. When the desired number of revolution is achieved, the machine will automatically stop.
6. Remove the specimens and clean off any lose wear debris. Examine the features on or near the wear scar such as a protrusion, micro-cracking, discoloration, or spotting.
7. Re-measure the specimen dimension and weight.
8. To get sufficient data for statistically significant results, the tests were repeated with additional specimens.

3.9.3. Calculation and reporting

The procedure for evaluating and reporting different wear parameters are given as [176]

1. The wear measurement should be reported as the weight loss or volume loss in cubic millimeters for the pin.
2. The following equation can be used to calculate wear volume loss, assuming that there is no significant disk wear.

$$Pin\ volume\ loss\ (mm^3) = \frac{Weight\ loss(g)}{Density\ (g/cm^3)} \times 1000 \qquad (3.4)$$

3. The coefficient of friction is also be reported in dry sliding wear test.