CHAPTER 3

CHAPTER 3

EXPERIMENTAL PROCEDURE

This chapter describes the experimental methods and techniques used in the present investigation to synthesize and characterize Ti-TiB-based composites. The procedure for evaluating the friction and wear behavior of these composites and the techniques adopted for the characterization of worn surfaces are also included in the chapter.

3.1 MATERIALS USED

The following section highlights the procurement and chemical composition (as given by the supplier) of the different material powders used in the present study to synthesize composites. The metal powders used for making the sample were Ti powder (99.4% pure; average particle size of 100 μm; Alfa Aesar, USA), TiB_2 powder (99.5% pure; average particle size of 325 μm; Alfa Aesar, USA), Fe (99.7% pure; average particle size of 6 μm; Thermo Fisher Scientific, USA). All the powders have been examined under high resolution scanning electron microscope (NOVA NANOSEM 450, FEI, USA) to acquire a knowledge of their shape and size distribution.

Table 3.1 Chemical composition of titanium (Ti) powder and titanium diboride TiB_2.

Element	Fe	O	C	Other's(N,H)			Ti
Wt. %<	< 0.030	< 0.068	< 0.05	< 0.031			99.9
Element	**Al**	**Cr**	**Cd**	**Fe**	**Pb**	**Zr**	**TiB_2**
Wt. %	< 0.01	< 0.01	< 0.0003	< 0.03	<0.0003	<0.05	99.5
Element	**Cr**	**Mn**	**Nb**	**Ni**	-	-	**Fe**
Wt. %	< 0.002	< 0.003	< 0.002	< 0.006	0	0	99.998

Table 3.2 Composite designation and composition (at. %) of B and Fe.

Composite designation	B (at. %)	Fe (at. %)
TiBFe1010	10	10
TiBFe1020	10	20
TiBFe1030	10	30
TiBFe2010	20	10
TiBFe3010	30	10

3.2 FABRICATION OF COMPOSITES

3.2.1 MIXING OF POWDERS

Table 3.2 gives the designation of composites and composition (at. %) of the present study's powders. The powder was mixed mechanically for 24 h in a jar mill for homogenous mixture of the composite. Five different compositions were mixed and kept separately in a poly bag zip sealed before placing them in the graphite die mould for Spark plasma sintering.

Table 3.3 gives the designation of composites and composition (wt. %) of the present study's powders. The powder was mixed mechanically in a mixing bowl for a homogenous mixture of the composite. Five different compositions were mixed and kept separately in a poly bag zip sealed before placing them in the copper mold.

Table 3.3 Composite designation and composition (wt. %) of powders.

Composite designation	**Ti (wt. %)**	**TiB_2 (wt. %)**
Ti-TiB (0 vol. %) **(Ti)**	100	0
Ti-TiB (50 vol. %) **(TiB50)**	70.3	29.7
Ti-TiB (60 vol. %) **(TiB60)**	64.4	35.6
Ti-TiB (70 vol. %) **(TiB70)**	58.5	41.5
Ti-TiB (80 vol. %) **(TiB80)**	52.5	47.5
Ti-TiB (85 vol. %) **(TiB85)**	49.5	50.5

3.2.2 SPARK PLASMA SINTERING

The Ti powder, TiB_2 powder, Fe powder were used in at.% for synthesizing the composites. The powders were mechanically mixed for 24 h in a jar mill, and mixed powders were placed in a graphite die. The spark plasma sintering system (Model 10-4, Thermal Technologies) of 10-ton capacity was used for preparing the composites. The die was fixed in SPS chamber where uniaxial pressure of 10 MPa was applied, the temperature was maintained at 1100°C, the heating rate was 50 °C/min, the holding time was 30 min to allow for the reaction to take place and the cooling rate was 22.5 °C/min. Joule heating was provided by the current supplied to achieve sintering conditions. The sintering is restricted through the holding time and heating rate to inhibit the formation of undesired phases, even in the highly reactive system. Five different composites having 10, 20, 30 at.% of Fe and 20, 30 at.% of B were synthesized.

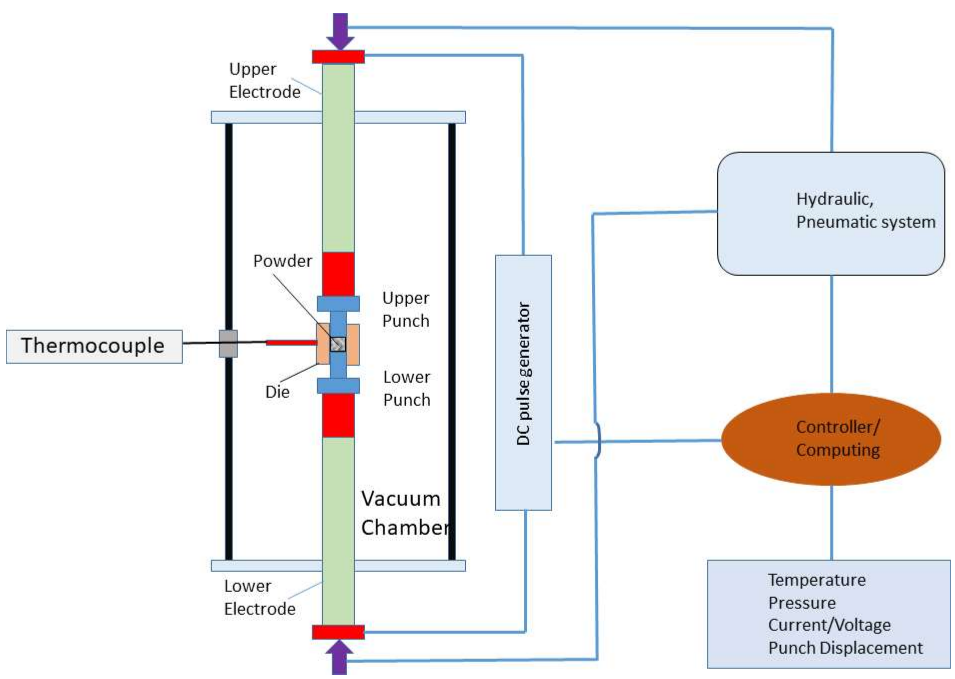

Figure 3 1 Schematic diagram of a typical spark plasma sintering machine.

3.2.3 VACUUM ARC MELTING

Powder were taken out from the sealed polybag and were placed inside the vacuum arc furnace, where half of spherical copper mould of 30 cm diameter was engraved. Tungsten electrode was also connected to a hollow copper cylinder pipe and placed just above the copper's engraved mould. The arc produced from the tungsten electrode was pointed to the mould containing the sample. After completing the cleaning process to avoid any oxide layer formation on their surface, the specimens were exposed to arc immediately after melting the gutter material. To obtain homogeneous sample remelting it 5-7 times by flipping each time, holding the arc of 1-3 minutes over the surface at 15-25V and 150-350 A. The mold and specimen surface has been cleaned with acetone to avoid any impurity insertion in the specimen every time after melting completes.

Microstructures play a very important role in deciding the end properties of an alloy or a composite. The optimized microstructure could be obtained by a careful choice of processing technique and selection of materials. However, it is important to understand the microstructural aspects to understand the behavior of the component in different environmental and working conditions. MMCs can be tailored with myriad of variations to suit for a particular application and hence they belong to a unique class of materials.

Figure 3.1 displays the typical arrangement of the vacuum arc melting furnace set up and its various units. The equipment consists of the following units:

- Tungsten electrode
- Control console
- Powder feeder

- Power supply
- Handle for arc movement
- Torch cooling system (water)
- Carrier gas supply
- Hoses, cables, gas cylinders and accessories.

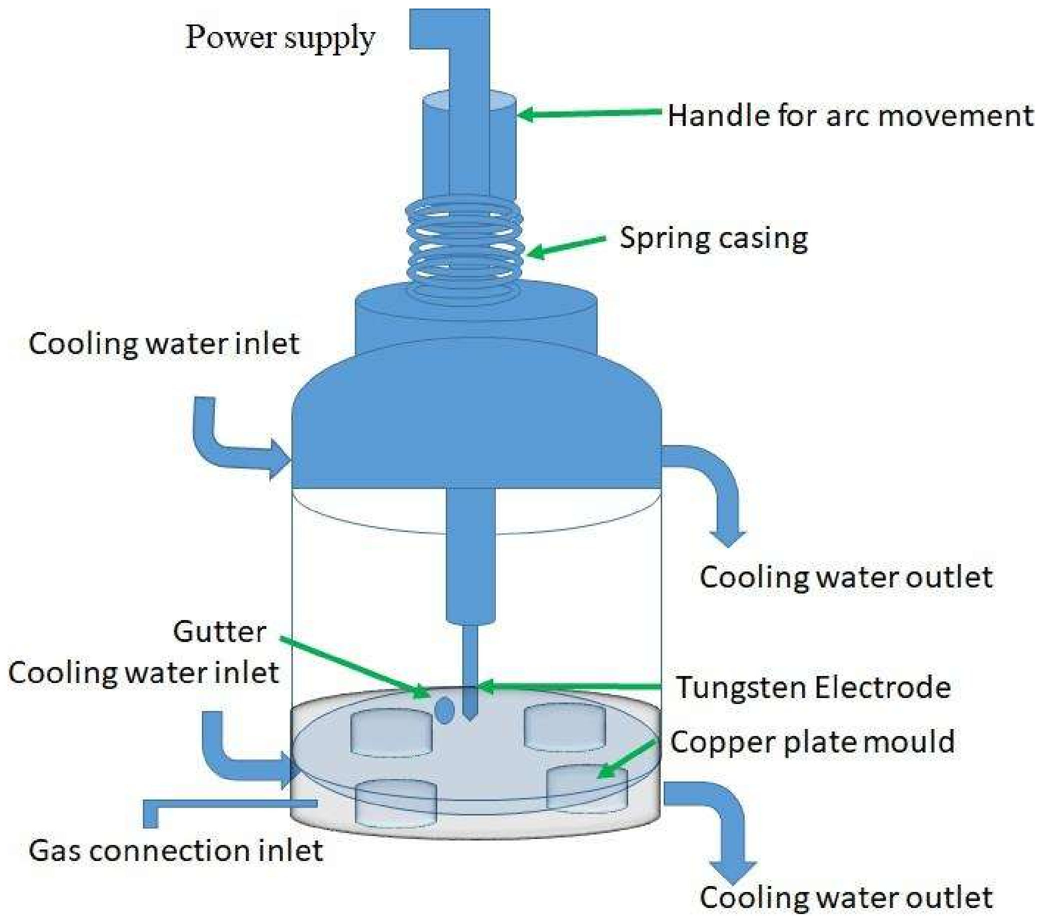

Figure 3 2 General arrangement of Arc melting equipment 1. Arc Torch 2. Powder melting copper hearth 3. Gutter 4. Control handle 5. Arc Power Source 6. Argon gas inlet 7. Water cooling system.

Table 3.4 Arc produced for synthesis parameters.

Items	**Value**
Argon flow rate, L/min	40
Electrode holding angle,°	90
Arc distance, mm	15-30
Current, A	550
Voltage, V	55

3.3 CHARACTERIZATION OF COMPOSITES

3.3.1 X-RAY DIFFRACTION ANALYSIS OF COMPOSITES

X-ray diffraction technique has been used to identify the different phases (elemental phase/ intermetallic phase/ crystalline phase) present in the composites, by using X-ray Diffractometer (XRD) (Smart Lab, Rigaku, Germany) having Cu $K\alpha_1$ radiation (λ=0.1541nm), 40 kV operating voltage, 2θ scanning rate of 0.02 °/s, over an angular range from 30° to 80°. For all the intensity peaks, the interplanner spacing, *d*, has been calculated using Bragg's law given by Eq. (3.1), corresponding to the values of 2θ, which has finally been used for identification of various phases with the help of X-ray diffraction data cards (JCPDS).

$$2d\ sin\theta = n\lambda \tag{3.1}$$

Where 'θ' is the incident angle, 'λ' is the wavelength of the x-ray, and 'n' is an integer representing the order of the diffraction.

3.3.2 HARDNESS MEASUREMENT

The composites samples have been transversally mounted and polished. The microhardness of the as-melted composites has been measured on polished surfaces using a Microhardness tester (model no: HV-50A, Vertex Engineering, and Association, India) at a load of 1kg and dwell period of 10 s. At least ten indentations have been taken and the average value has been reported.

3.3.3 POROSITY MEASUREMENT

Area percentage porosity of the composite has been measured by image J analyzer. In this technique, cross section has been polished and placed under the field emission scanning electron microscope FESEM **(**Nova Nano SEM 450, FEI, USA**)**. The image is analyzed in the computer having image J analyzer software. The porosity has been estimated based on the area covered by the pores and the total area of the composite. At least five images have been analyzed for these measurements to estimate porosity properly, and the average value of porosity has been reported.

3.4 MICROSTRUCTURAL EXAMINATION

For metallographic examination, composite samples have been manually polished following the standard metallographic procedures described below. Composite samples have been first of all mounted on the cross sections. The specimens have been manually polished using the SiC metallographic emery papers (400, 600, 800, and 1000 grit). The direction of grinding on the emery paper is such as to introduce scratches at right angles to those introduced by the preceding paper. After that, the samples have been polished on a sylvet-cloth in the presence of alumina water on a polishing machine

(Chennai Metco Pvt. Ltd., India). This has been followed by polishing using diamond paste and aerosol on a sylvet-cloth to attain a mirror-polished surface. After polishing, the specimens have been flushed with acetone and dried. Morphological studies of the composite have been performed using a high resolution-scanning electron microscope (HR-SEM) Nova Nano SEM 450, FEI, USA, equipped with energy dispersive spectroscopy (EDS) EDAX Inc. Typical microstructural features of all the compositions are photographed. The specimens have also been subjected to energy dispersive spectroscopy (EDS) to acquire knowledge of the compositional details. SEM micrographs and EDS patterns are presented and discussed in Chapter-4.

3.5 DRY SLIDING FRICTION AND WEAR TESTING

Dry sliding wear tests for the composites, designated as TiB50, TiB60 TiB70, TiB80, and TiB85 have been carried out on a 'reciprocating ball on flat configuration' using a Ball-on-Flat tribometer under different loads and frequencies. Figure 3.2 presents a schematic diagram of the ball-on-flat setup for friction and wear tests. The fabricated specimens have been fixed on the continuously reciprocating table during the experiment against a counterface of bearing steel ball (φ 6 mm, 0.1-0.21 R_a(μm) of surface finish), which is held stationary. Before conducting the tribological tests, the composite samples have been ground with emery paper's help to obtain a uniform roughness of around 0.3 μm and then cleaned with acetone solution to eliminate loose particles from a specimen that might have stuck from emery paper to the specimen during polishing. Initially, the friction tests have been conducted at the normal loads of 10, 15, 20, and 25 N and at a frequency of 5 Hz at room temperature (RT) only using friction and wear test on Multi-Functional

Tribometer (Rtec instrument, USA) for the TiBFe composites to know the potential of Fe and B addition in the Specimens. Whereas for TiB composites, the friction tests have been conducted at the normal loads of 10, 15, 20, and 25 N, stroke length of 2 mm and at a frequency of 4 Hz at room temperature (RT) only using friction and wear test on Multi-Functional Tribometer (Rtec instrument, USA) for the TiBFe composites. All the wear tests have been performed under ambient conditions at a relative humidity (RH) of 40-55%, recorded by Ambient Condition Recorder Testo 623 (Testo SE & Co. KGaA, Germany). Further, the friction and wear characteristics of composite TiB50, TiB60 TiB70. TiB80 and TiB85 have also been examined by conducting the tests at different frequencies and a stroke length of 5 mm. To explore the effect of frequency and load on composites' tribological behavior, namely, TiB50, TiB60, TiB70, TiB80, and TiB85.

The friction tests have also been carried out at three different frequencies of 7, 10, and 15 Hz at RT and loads 10, 15, 20, and 25 N. A Table 3.4 illustrates the details of experimental conditions and the machine used for friction and wear tests. Initially, the composite specimen has been fixed in the jaw plate and ball in the ball holder spring collection placed just above the specimen touching the load sensor, without being in contact with each other. Then the desired load and time are entered in the system's software and made the system run with a dwell time of 1 minutes so that the load gets stabilized. The contact has been made between the bearing steel ball and composite forming a point contact with the specimen surface, and after reaching the desired load, the load has been allowed to stabilize. The test was carried out at room temperature within ± 2°C during each test due to frictional heating. During each run, the friction coefficient was recorded through a data acquisition system in a computer with an interface with a tribometer. The data from the starting to end of the test has been used to estimate friction's average coefficient. Each tribological test has been performed at least three times to get consistency in the results, and the average value has been reported in the present work. The weight of specimens was taken after every test to measure the loss of material to calculate the wear track wear volume loss. The weight was measured on Keroy (GE 202, Japan) for every repetition, and the average weight loss of three test values has been reported. The wear rate, W, is calculated by using Eqn. (3.2) given below.

$$W = \frac{V}{S} \tag{3.2}$$

Here, V is the volume loss in mm^3, S, and the total sliding distance (m).

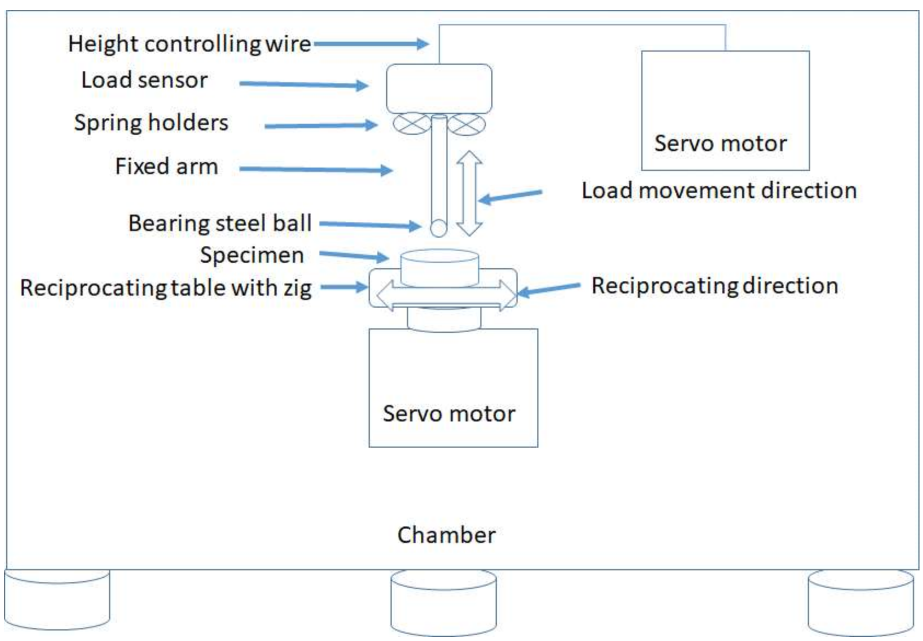

Figure 3 3 Schematic diagram of the reciprocating ball on flat testing.

3.6 EXAMINATION OF WORN SURFACES

The worn surfaces of composites and the counterface balls of bearing steel have been examined using scanning electron microscope, X-ray diffractometer and SEM-

EDS to explore the possibility of forming new phases or transfer of material during sliding.

3.6.1 HIGH-RESOLUTION SCANNING ELECTRON MICROSCOPY (HR-SEM)

In order to explore the prevailing mechanisms of wear, the sliding surfaces of the composite TiBFe1010, TiBFe1020, TiBFe1030, TiBFe2010, TiBFe3010, and also for Ti, TiB50, TiB60 TiB70, TiB80 and TiB85 under each test condition have been examined under the high resolution-scanning electron microscope (HR-SEM) (Nova Nano SEM 450, FEI, USA) equipped with energy dispersive spectroscopy and the salient features in each have been photographed which are presented in Chapter 4 and Chapter 5.

Table 3.4 Experimental conditions and equipment used for friction and wear testing.

Variable/s	Load (N)	Tribometer used	Test temperature (°C)	Sliding Frequency (Hz)	Stroke length (mm)	Sliding time(minutes)
Load	10 15 20 25	Rtec Multifunctional tribometer, USA	27°C(RT)	5	2 mm	20 20 20 20
Load	10 15 20 25	Rtec Multifunctional tribometer, USA	27°C (RT)	4 7 10 15	5mm 5mm 5mm 5mm	20 20 20 20

3.6.2 X-RAY DIFFRACTION ANALYSIS

The worn surfaces of composite specimens have been subjected to X-Ray diffraction analysis to examine the formation of new phases that might have resulted from the tribo-chemical reactions at the interface due to sliding. The X-ray Diffractometer (XRD) (Smart Lab, Rigaku, Germany) has Cu $K\alpha_1$ radiation (λ=0.1541nm), 40kV operating voltage, 2θ scanning rate of 0.02 °/s, over an angular range from 30° to 80° with two-dimensional D/MAX RAPID II-CMF detector of micro area diffraction unit has been used to reveal the presence of various compounds at the worn surface. However, a particular phase cannot be detected. Its amount is beyond the limit of the detection of X-ray. The X-ray and the SEM are presented and discussed in Chapter 4 and Chapter 5.