CHAPTER 3

EXPERIMENTAL PROCEDURE

This chapter describes the experimental methods and techniques used in the present investigation in respect of synthesis and characterization of atmospheric plasma sprayed coatings. The procedure for evaluating the friction and wear behavior of deposited coatings 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 for the synthesis of coatings. The powders of Nickel (Ni), Molybdenum di-sulphide (MoS_2), Silver (Ag) and Aluminum (Al) have been procured from Loba Chemie, Mumbai, India whereas hBN powder has been purchased from SRL Pvt. Ltd., India. Inconel 718 with a composition of Ni (50-55%), Cr (17-21%), Niobium (4.75-5.50%), Molybdenum (2.80-3.30%) and Ti (0.65-1.15%) has been used for as the substrate for deposition of coating and it has been procured from All India metal corporation, Mumbai.

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. The chemical composition of Nickel powder, as provided by the supplier is given in Tables 3.1.

Table 3.1 Chemical composition of nickel (Ni) powder.

Element	Fe	S	C	Ni
Wt. %	0.01	0.001	0.08	balance

3.2 SYNTHESIS OF COATINGS

3.2.1 MIXING OF POWDERS

Table 3.2 gives the designation of coatings and composition (wt. %) of the powders used in the present study. For getting a homogenized mixture all powders have been mixed through ball milling (PM 200, RETSCH, Germany) for 8 hours at a speed of 200 rpm. The zirconia balls have been utilized during ball milling with a ball to powder weight ratio of 10:1.

Table 3.2 Coating designation and composition (wt. %) of powders.

Coating designation	**Ni (wt. %)**	**Al (wt. %)**	**Ag (wt. %)**	**MoS_2 (wt. %)**	**hBN (wt. %)**
Ni-Al-Ag-MoS_2 **(NAMB0)**	69.4	10.6	10	10	0
Ni-Al–Ag–MoS_2-hBN (5 wt. %) **(NAMB5)**	65.025	9.975	10	10	5
Ni-Al–Ag–MoS_2-hBN (10 wt. %) **(NAMB10)**	60.69	9.38	10	10	10

3.2.2 PREPARATION OF SUBSTRATE

The square (30 mm × 30 mm, 6 mm thickness) specimens of Inconel 718, which has been chosen as substrate materials in the present study, have been grit blasted at a pressure of 3 kg/cm^2 using alumina grits (grit size of 60). Stand-off-distance has been kept between 120-150 mm for blasting. The particles of alumina have been directly fed into an air blast from a pressurized container into a high pressure air stream. The venturi type nozzle has been used to impart kinetic energy to the grit particles for impinging the surface of the substrate material. The surface of the substrate specimens has been cleaned with acetone in an ultrasonic cleaning unit after grit blasting. The specimens have been exposed to plasma spray deposition immediately after completing the cleaning process to avoid formation of any oxide layer on their surface.

3.2.3 PLASMA SPRAY COATING DEPOSITION

The atmospheric plasma spray deposition on Inconel square samples has been done by 3 MBM Plasma Gun, Sulzer Metco equipment at Anod Plasma Spray Limited, Kanpur, India. Plasma torch input power level has been varied from 11kW to 21 kW, by varying the gas flow rate, voltage and arc current. The powder has been injected at the outlet of the nozzle and directed towards the plasma. The Ar and H_2 gases have been used as primary and secondary gases during the plasma spray deposition. The powder has been kept in a hopper and is allowed to come out of the nozzle through hoses after supplying the reactive gases. The powder flow has been kept constant at a rate of 45 g/min. A four stage closed-looped centrifugal pump (water cooling) has been used under a regulated pressure of 10 kg/cm^2 supply for providing the cooling in the whole system. To deposit the coatings the powders have been sprayed through the nozzle at an angle of 90°. The

microstructure and the properties of the coating are highly reliant on the spraying parameters. The parameters used for the coating deposition are listed in Table 3.3.

Figure 3.1 displays the typical arrangement of the plasma spray set up and its various units. The equipment consists of the following units:

- Plasma spraying equipment
- Control console
- Powder feeder
- Power supply
- Stand-off-distance of torch
- Torch cooling system (water)
- Carrier gas supply
- Hoses, cables, gas cylinders and accessories.

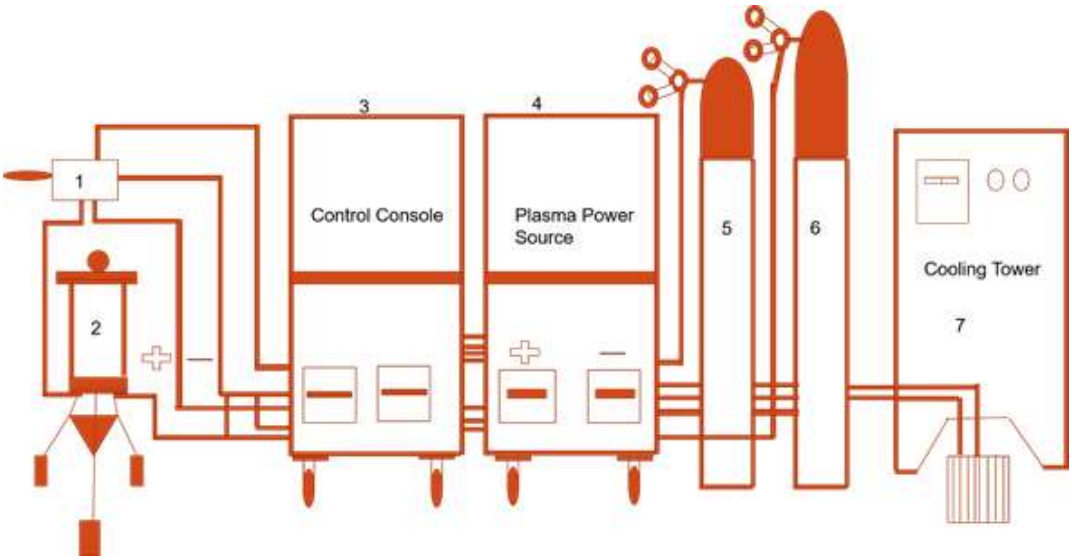

Fig. 3.1 General arrangement of plasma spraying equipment 1. Plasma Torch 2. Powder feeder 3. Control Console 4. Plasma Power Source 5. Ar gas cylinder 6. H_2 gas cylinder 7. Cooling Tower.

Table 3.3 Plasma spray deposition parameters.

Items	Value
Argon flow rate, L/min	40
H_2 flow rate, L/min	6
Injector angle,°	90
Powder feed rate, g/min	45
Spray distance, mm	100
Current, A	550
Voltage, V	55

3.3 CHARACTERIZATION OF COATINGS

3.3.1 X-RAY DIFFRACTION ANALYSIS OF COATINGS

X-ray diffraction technique has been used to identify the different phases (elemental phase/ intermetallic phase/ crystalline phase) present in the coating, 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 20° to 90°. 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 COATING THICKNESS MEASUREMENT

To measure the thickness of coating on the substrate, specimen cross section has been polished and examined under the field emission scanning electron microscope FESEM **(**Nova Nano SEM 450, FEI, USA**).** Five readings have been taken on each specimen and the average value is reported as the coating thickness.

3.3.3 HARDNESS MEASUREMENT

The coated samples have been transversally mounted and polished. The microhardness of the as-sprayed coatings has been measured on polished surfaces using an MH-5-VM micro hardness tester **(**Hengyi Precision instrument Co. Ltd; China**)** at a load of 200 g and dwell period of 10 s. At least ten indentations have been taken and the average value has been reported.

3.3.4 POROSITY MEASUREMENT

Area percentage porosity of the composite coatings 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 on the basis of the measurement of the area covered by the pores and the total area of the coating. At least five images have been analyzed for these

measurements to get a proper estimation of porosity and the average value of porosity has been reported.

3.4 MICROSTRUCTURAL EXAMINATION

For metallographic examination, coating samples have been manually polished following the standard metallographic procedures described below. Coated samples have been first of all mounted on the cross sections. The coated 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. Thereafter, the coated samples have been polished on a sylvet-cloth in 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 coatings have been performed using 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 coating compositions are photographed. The specimens have been also subjected to energy dispersive spectroscopy (EDS) to acquire a 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 composite coatings, namely, Ni-Al-Ag-MoS_2 (designated as NAMB0), Ni-Al-Ag-MoS_2-5 wt. % hBN (NAMB5) and Ni-Al-

Ag-MoS_2-10 wt. % hBN (NAMB10) have been carried out on a 'rotary ball on disk configuration' using Pin-on-Disk tribometer under different loads, speeds and temperatures. Figure 3.2 presents a schematic diagram of ball-on-disc set up for friction and wear tests. The coated specimens have been rotated continuously during the experiment against a counterface of alumina ball (φ 6 mm) which is held stationary. Before conducting the tribological tests, the coated samples have been ground with the help of emery paper to obtain a uniform roughness of around 0.3 μm and then cleaned with ultrasonic cleaner (LMUC-2A, LABMAN Scientific Instruments Pvt. Ltd., India) in presence of acetone solution for a time period of 5 min to get rid of loose particles from coated samples which might have stuck from emery paper to coated specimen during polishing. Initially, the friction tests have been conducted at the normal loads of 5, 10, 15, 20 N and at a sliding velocity of 0.5 m/s corresponding to a distance of 500 m at room temperature (RT) only using a friction and wear test rig (TR-20LE-CHM600, DUCOM, Bengaluru, India). All the wear tests have been performed under ambient conditions at a relative humidity (RH) of 40-55% which has been recorded by Ambient Condition Recorder Testo 623 (Testo SE & Co. KGaA, Germany). Further, the high temperature friction and wear characteristics of composite coatings NAMB0, NAMB5 and NAMB10 have been also examined by conducting the tests at different temperatures i.e., 200, 400, 600 and 800 °C at a constant load of 5 N and a fixed sliding velocity of 0.3 m/s for a total sliding distance of 480 m using a High Temperature Computerized Friction and Wear Machine (TR-20E DHM-850 PHM-300 CE DUCOM, Bengaluru, India). In order to explore the effect of speed and temperature on the tribological behavior of coatings, namely, NAMB0, NAMB5 and NAMB10, the friction tests have been also carried out at four different sliding speeds of 0.3, 0.5, 0.7 and 0.9 m/s and temperatures RT, 200, 400,

600 and 800 °C at a constant load of 5 N for a total sliding distance of 480 m. A Table 3.4 illustrates the details of experimental conditions and the machine used for friction and wear tests. The high temperature tribometer had the arrangement of heating the disk from bottom through induction heater. Initially, the coated specimen and the alumina ball have been fixed in the jaw plate and make parallel through lever arm, without being in contact with each other. The chamber is then covered and the required temperature has been set in the panel attached with the tribometer. The disc is heated with induction heater while being simultaneously rotated for obtaining uniform temperature throughout the coated sample. After reaching the desired temperature, the sample has been stopped and temperature has been allowed to stabilize. Before starting the test, the required load has been put on the arm and the contact has been made between the alumina ball and coating. The temperature of the disc has been maintained within ± 5°C during each elevated temperature test. The coefficient of friction during each run has been recorded through data acquisition system in a computer that had an interface with tribometer. The data from the starting to end of the test has been used to estimate the average coefficient of friction. Each tribological test has been performed at least three times for getting consistency in the results and the average value has been reported in present work. Optical profilometer has been used for wear volume loss calculation of the wear track. The wear track has been scanned by the profilometer at five different locations and the images have been obtained. After getting the images of the wear track by profilometer, wear profile has been measured across the wear track by using Gwyddion 2.30 software and then the area under the profile has been calculated by using origin pro software. The average value of the calculated area has been multiplied by πD to get the wear volume loss,

where D is the diameter of wear track. The wear rate, *W*, is calculated by using Eqn. (3.4) given below.

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

Where, *V*, is the volume loss in mm^3, *S*, the total sliding distance (m) and *L,* the normal load in N.

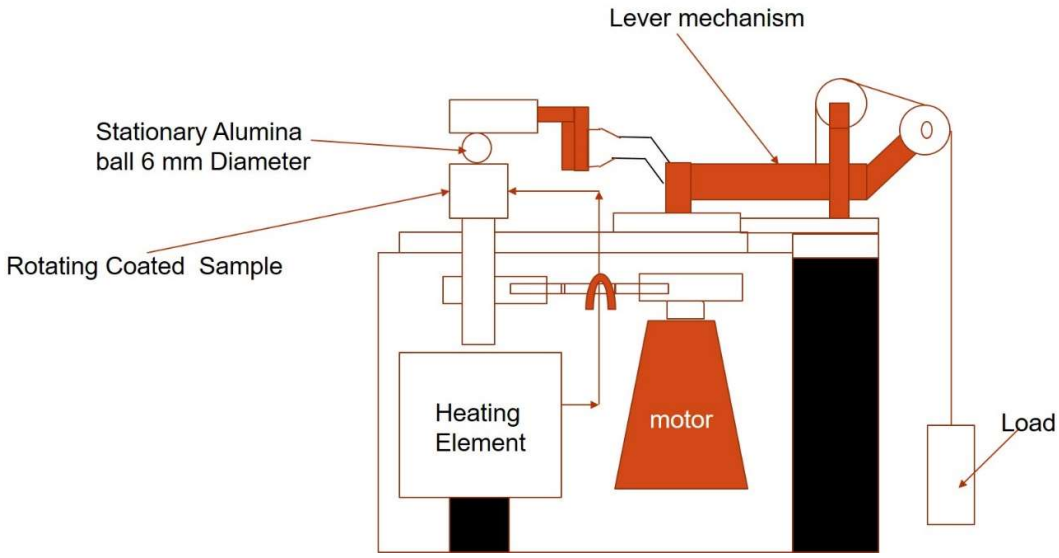

Fig. 3.2 Schematic diagram of the ball on disk testing rig.

3.6 EXAMINATION OF WORN SURFACES

The worn surfaces of coated discs as well as the counterface balls of alumina have been examined using scanning electron microscope, X-ray diffractometer and Raman spectrometer to explore the possibility of formation of 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 coatings NAMB0, NAMB5 and NAMB10 worn under each test condition have been examined under 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.

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

Variable/s	Load (N)	Tribometer used	Test temperature (°C)	Sliding speed (m/s)	Sliding distance(m)
Load	5 10 15 20	TR-20LE-CHM 600 DUCOM, Bengaluru, India	(RT)	0.5	500
Temperature	5	TR-20E DHM-850 PHM-300 CE DUCOM, Bengaluru, India	Room temperature (RT), 200, 400, 600 and 800	0.3	480
Speed and Temperature	5	TR-20E DHM-850 PHM-300 CE DUCOM, Bengaluru, India	RT, 200, 400, 600 and 800	0.3 0.5 0.7 0.9	480

3.6.2 X-RAY DIFFRACTION ANALYSIS AND RAMAN SPECTROSCOPY

The worn surfaces of coated 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) having Cu $K\alpha_1$ radiation (λ=0.1541nm), 40kV

operating voltage, 2θ scanning rate of 0.02 °/s, over an angular range from 20° to 90° 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 detection of X ray. To overcome this limitation, Raman spectroscopy, which is a more powerful technique has been utilized by analyzing the worn surfaces of composite coatings through Raman spectrometer (Raman-HR-TEC, Stellar Net Inc, USA) with excitation wavelength of 785 nm. Both the X-ray and the Raman spectra are presented and discussed in Chapter 4.