CHAPTER 3:

MATERIALS AND EXPERIMENTAL METHOD

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This chapter describes the details of the experimental techniques and equipment used in the present investigation in respect of the heat treatment of steels to develop dual phase and fully martensitic structure in the plain carbon steels and their microstructural characterization by optical, scanning and transmission electron microscopy. The chapter also contains the details of hardness and tensile tests along with the machines used for the same. A specifies of the characterization of erodent material and a detailed procedure for carrying out the erosion and the corrosion tests along with the testing parameters are also included in the chapter. The presentation of the techniques/instruments used for the examination of the eroded and corroded surface & sub-surface of steels also forms a part of the present chapter.

3.1 Selection of Steel

The medium carbon hypo eutectoid steel rods of commercial grade of 25 × 25 mm^2 square cross section have been used to develop normalized, dual phase and fully martensitic steels. The steel has been procured from All India Metal India Corporation, Mumbai.

3.2 Determination of Chemical Composition of Steel

The chemical analysis of the plain carbon steels used in the current study has been carried out through the spectrophotometer and the results of the analysis are presented in Chapter4.

3.3 Experimental Set-up for Heat Treatment

An experimental set-up shown schematically in Fig. 3.1 has been used to develop dual phase and fully martensitic structure by inter critical annealing heat-treatment, Essentially, it consists of an electric resistance muffle furnace; a device for suspending steel samples in the furnace tube for heat treatment and a quenching device. A vertical electric resistance muffle furnace is locally fabricated using a sintered alumina tube, with both ends open, of internal diameter 6.5 cm and length 67 cm by winding non-inductive Kanthal wire of 18 SWG gauge and resistance of ~ 30 Ω as to yield in the center of the furnace a uniform temperature zone (UTZ) of approximately 20 cm length. A circular sheet of asbestos, acted as a cover for the top end of alumina tube and had two openings-one for passing a chromel-alumel thermocouple encased in protective refractory sheath to measure temperature in the UTZ and the second for suspending square shaped steel sample in the UTZ for heat treatment. Immediately below the furnace, is placed a quenching bath-a cylindrical steel tank containing water as the quenching medium. The lower end of the tube is submerged in the water for the instantaneous quenching of the specimen. Quenching is carried out by cutting the wire from which the sample is suspended allowing thus the heated specimen to fall directly into the quenching bath placed just below the furnace tube.

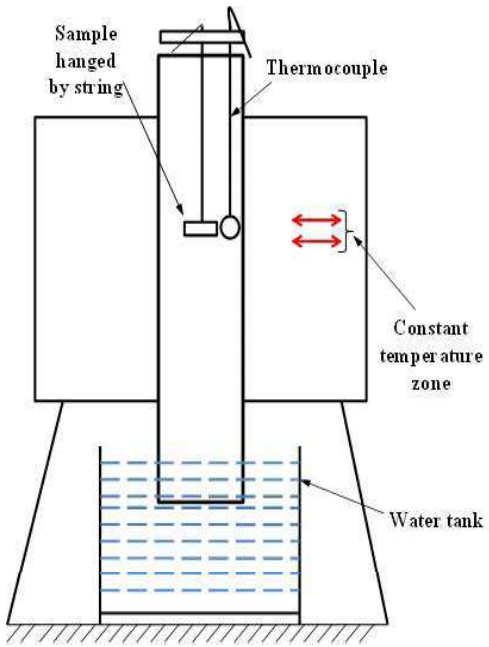

Figure 3.1 Schematic of vertical tube furnace for intercritical annealing time heat treatment

The furnace (Fig. 3.1) is supplied with single phase 220 V ac power through an automatic solid state ac Servo-Voltage Stabilizer (NELCO make, 7.5 kVA, 175-266 V range, maximum current 30 amp.), a variable resistance (ESCORP make, Variac-240V, 28 A maximum load) and an automatic on-off type relay-operated temperature controller with indicator (APLAB make-Applied Electronics, Thane, India, range up to 1200 $^{\circ}$C), all mounted on a control panel with suitable indicator lights and electrical fuses and switches to enable precise temperature control up to ± 5°C in the UTZ of the furnace. For temperature measurement, a Leads-Northup type 8694 potentiometer has been used.

3.3 Heat Treatment Variables and Procedure

After homogenizing the specimens has taken for intercritical annealing heat treatment to obtain a DP steels with varying martensite volume fractions. This involved a specific

route, as depicted in Fig. 3.2. The time temperature transformation diagram depicts the two curves out of which one shows the path for the formation of ferrite + martensite phase in the steel while the other depicts how the fully martensitic steel forms (Fig. 3.2 (a)). The two phase region till where the specimens for preparing dual phase structure have been held and subsequently quenching starts has been shown in the Fig. 3.2 (b).

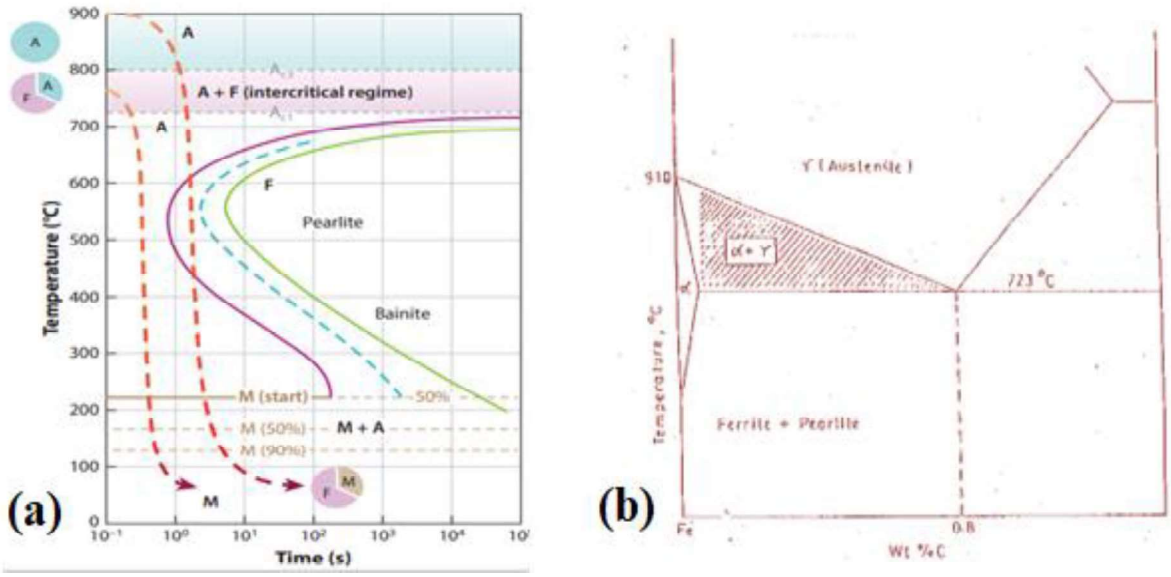

Figure 3.2 (a) Schematic indicating TTT diagram indicating heat treatment methods used to obtain DP steels and (b) Schematic shows the two phase region in the Iron rich portion of iron - iron carbide phase diagram

3.3.1 Normalizing

Square samples (25 mm × 25 mm) of medium carbon steel have been used in the present study is depicted in Fig. 3.3. The thickness has been reduced to 5 mm by the grinder. Sample thickness has been reduced to 5 mm to attain the sufficient hardenability in the quenched samples. All specimens are then normalized in a muffle furnace.

Normalizing has been carried out at a temperature of 860ºC for 20 minutes followed by air cooling. This normalizing treatment is done in batches comprising of four (4) samples per batch.

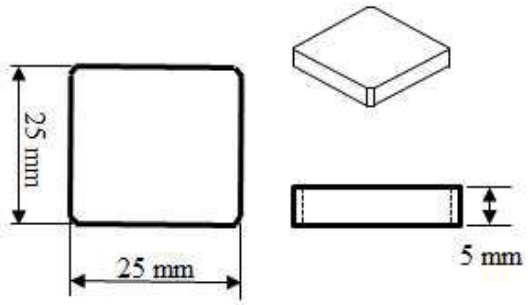

Figure 3.3 Geometry of Erosion test specimen

3.4.2 Intercritical Annealing

In the present investigation one parameter viz., intercritical annealing time has been varied at a fixed intercritical annealing temperature for obtaining different martensite volume fraction (MVF) in the DP steels. A close examination of available published literature on production of DP steels (Davies, 1978; Koo *et al*, 1980; and Nath, 1989) reveals that, in general, the intercritical annealing time used has been either 10 minutes or 15 minutes at any one intercritical annealing temperature to obtain a fixed martensite volume fraction (MVF) in DP steels. These are the time duration for a plain carbon steel having carbon from 0.42 wt. pct. However, the objective of present study is to have the DP steels with varying microstructure having different volume fraction of martensite, to analyze the effect of the martensite volume fraction on the erosive wear behavior of DP steels.

For obtaining dual phase structure, the square shaped specimen is suspended by a wire in the UTZ of the vertical tube furnace and the top end of the alumina tube is

covered with the asbestos sheet in order to prevent heat loss. The temperature of the furnace drops during charging of the sample inside the furnace. Therefore, the start of the intercritical time is considered from the moment the furnace temperature regains the original desired Intercritical Annealing (ICA) temperature. After the lapse of specified soaking time for each heat treatment, the wire is cut, and the specimen is made to fall directly into the quenching water bath placed just below the furnace tube. The schematic shown in Fig. 3.4 represents the intercritical annealing routes followed for the development of DP steel.

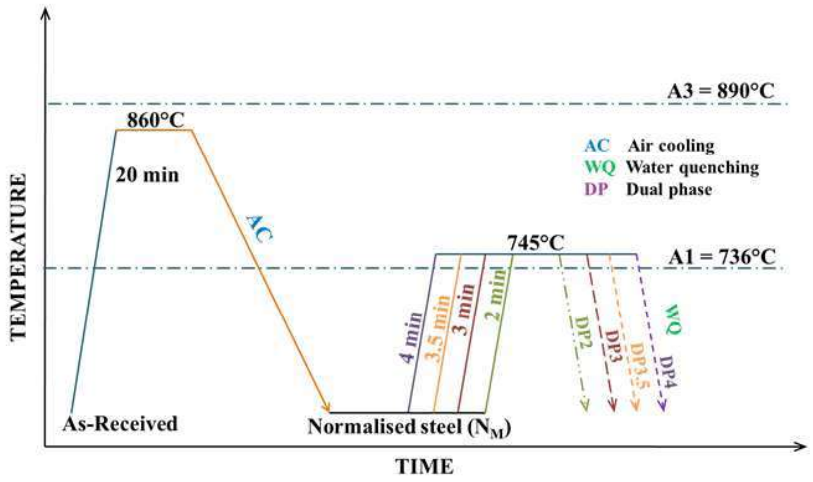

Figure 3.4 Schematic representation of intercritical annealing cycle

The samples of the medium carbon steel have been intercritical annealed to develop the desired dual phase structure of martensite and ferrite. The intercritical annealing has been conducted at a temperature of 740°C for different holding times viz., 2.0, 3.0, 3.5, 4.0, 5.0, and 7.0 minutes followed by water quenching to obtain different volume fraction of martensite in the dual phase structures. The heat treatment methods used for the development of normalized, DP and fully martensitic steels is depicted in the layout as shown in Fig. 3.5

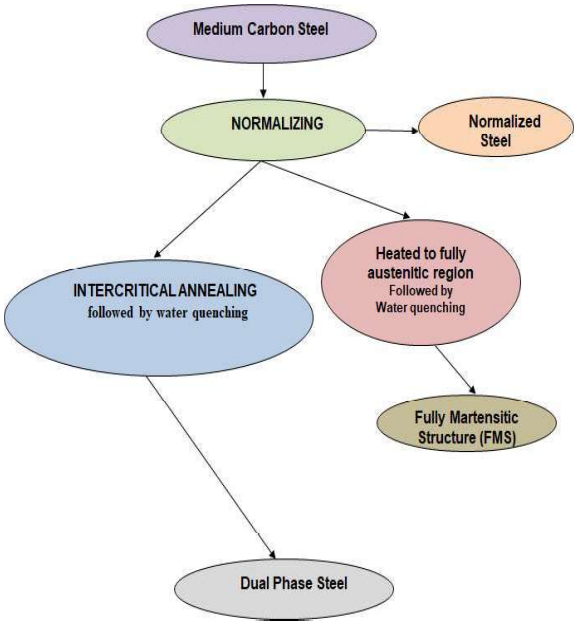

Figure 3.5 Layout for development of DP steel

For developing fully martensitic structure in the steel the sample is heated at 920°C for 20 minutes followed by water quenching.

3.5 Metallographic Studies

The microstructure of as-received samples and shot-peened has been characterized using optical microscopy, scanning electron microscopy, transmission electron microscopy and atomic force microscopy. XRD has been utilized to analyze the constituent phases in steel, and to assess the phase stability after corrosion

3.5.1 Optical Microstructural Examination

For metallographic examination, samples are first prepared using hot mounting in bakelite employing METCO (Model: MPH007), Chennai, specimen mounting press. These mounted samples are next manually polished following the standard

metallographic procedures described below. The surface of the specimen that is to be examined is first made plane by means of a specially designed motor-driven emery belt. The sharp edges of the specimen are then beveled to avoid the tearing of the emery paper in the subsequent polishing. The specimens were then polished manually using the SiC metallographic emery papers (400, 600, 800 and 1000 grit). During polishing on each emery paper the direction of grinding was such as to introduce scratches at right angles to those introduced by the preceding paper. The final polishing is carried out on a sylvet-cloth using 0.1 μm size alumina powder suspension on a METCO (Model: PMV009), Chennai, make cloth polishing machine. After polishing, all the samples are etched with 2 pct. nital (2 pct. HNO_3 + 98 pct. Methanol), washed, dried and finally examined under Leica DFC 295 optical microscope made in Germany. The microstructural features of all the samples are photographed and the optical micrographs are presented and discussed in Chapter 4.

3.5.2 Scanning Electron Microscopy

The polished specimens were rinsed in water, cleaned with acetone, etched with 2% Nital; and were examined under scanning electron microscope (Jeol JSM 840 A, Japan). Quantitative measurements of microstructural features have been carried out to determine the volume fraction and the size of the islands of the phases present in the microstructure. The volume fraction of martensite phase in the DP2, DP3, DP3.5, and DP4 steels was measured using image analyser software, Image J 32-bit Java 1.6.0_20 version.

3.5.3 Transmission Electron Microscopy

The phase characterization of the normalized and DP steel specimens, as well as the dislocations formed during phase transformation, have been carried out using TEM

(TECNAI 20 G2, USA) at 200 kV. Thin slices have been cut from the steel specimens using diamond cutter and then the specimens were polished manually using the SiC metallographic emery papers (800 and 1000 grit) to reduce the thickness of these slices to ~ 50 μm. Discs of 3 mm diameter were punched out from the 50 μm thinned slice and after that the TEM foils were prepared by electrolytic thinning from the side opposite to the treated surface, in the electrolyte containing 90% methanol and 10% perchloric acid, using a twin jet polisher (TenuPol-5). After electrolytic polishing the specimens acquire transparency to further evaluate the microstructure under transmission electron microscope.

3.5.4 Atomic Force Microscopy (AFM)

The surface morphology has been assessed using atomic force microscopy (Model: NTEGRA PRIMA, NT-MDT, Netherlands) to analyze the corroded surface and worn surface features. In the AFM analysis, the 10 × 10 μm surface area was scanned for extracting the solid profile. The arithmetical mean height and root mean square height value of the ground surface after corrosion has been calculated. The peak-to-valley variation has been observed on the corroded surface profile of all the steels. The average surface roughness values have been calculated for both corroded and eroded samples. A comparative study has been conducted for tested and untested steel specimens.

3.5.5 X-Ray Diffraction (XRD)

The Rigaku Miniflex X-ray diffractometer, Japan has been employed to study the XRD patterns of normalized and DP steel samples to characterize the phases of both types of samples. To compare the effects of intercritical annealing on the phases present samples have been subjected to Cu-Kα radiation with a Ni filter at a wavelength of 1.5402 over a 2 theta range of 20° to 90° at the scan rate of 2 °/min operating at 40 kV

and 15 mA. XRD has been utilized to examine the byproducts of corroded samples after corrosion at room temperature.

3.6 Measurement of Mechanical Properties

3.6.1 Hardness Measurement

The hardness of the normalized steel, DP steels and fully martensitic steel has been measured by Vickers hardness tester (LECO LV-700 AT, USA) at a load of 3 kg and a dwell period of 20 s. The indentation mark is measured from a projection of the indentation on a screen with the help of an optical lens provided along with the equipment. The time of application of load should be such as to ensure that the plastic flow of the metal in the area under indentation has ceased. The Vickers hardness number has been estimated from the values of the mean indentation diagonal and from the load applied, at least five indentations for hardness measurement are made at different locations and the average of these readings is reported as the hardness of the steel.

Vickers micro hardness tests have been carried out on the samples of normalized, DP steel and fully martensitic steels. The samples have been polished using the emery paper up to 4/0 grade. The hardness has been measured at a load of 10 g for 30 seconds by using a square base pyramid shape diamond indentor with an angle of 136°. The diagonals of the square indentation are measured under optical microscope at a magnification of 500 X provided with the micro hardness tester (MMV-SA, MICRO-MACH TECHNOLOGIES, India) and the average of the two diagonals is used to find out the corresponding micro hardness. At least five readings are taken on the martensite islands and the ferrite phase respectively. Average of these readings for each sample is reported as the micro hardness of the martensite and ferrite in DP steels.

3.6.2 Tensile Testing

The tensile tests have been carried out at ambient temperature for the normalized steel, DP steels and fully martensitic steel developed out of the medium carbon steel. The yield stress in the case of DP steels has been calculated by adopting the 0.2 pct. offset proof stress method. The engineering stress *vs*. engineering strain curves have been plotted from the load extension curves. Dumb-bell shaped tensile test specimens shown in Fig. 3.6 were machined according to ASTM (E8/E8M-15a) standard test method with gauge length and gauge diameter of 15 mm and 4 mm, respectively, from the treated specimens. The tensile tests have been performed on a 100 kN screw-driven Instron^{TMKJ} Universal Testing Machine (Model: 5982) at the cross-head displacement rate of 0.1 mm.min^{-1}. It has an arrangement for the computerized printing of the load *vs*. extension curve. The diameter and the gauge length of each specimen are measured prior to and after the tensile test. The ultimate tensile strength of the specimens has been estimated by dividing the maximum tensile force by the initial cross-sectional area of the specimens in units of MN/m^2 or MPa. After fracture of the specimen, the increase in gauge length is measured and the engineering fracture strain has been estimated as a change in gauge length per unit initial gauge length of 15 mm.

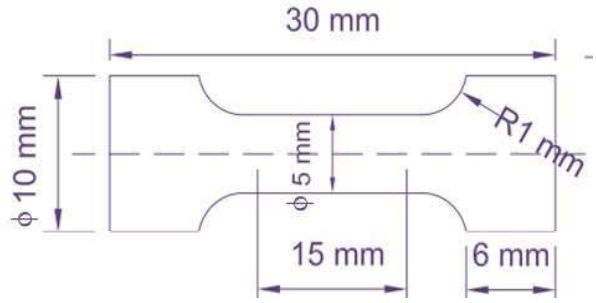

Figure 3.6 Schematic diagram of dumbbell shaped tensile specimen

3.7 Fractographic Studies

The fractographic studies have been carried out on the broken tensile test specimens of normalized steels, DP steels and fully martensitic steels. To have

knowledge of the operating mode of fracture, all these samples have been examined under scanning electron microscope (Jeol JSM 840 A, Japan) and the salient features have been photographed.

3.8 Erosive Wear Testing

Erosive wear tests for the normalized steels, DP steels and fully martensitic steels have been conducted using an air jet erosion test rig model TR-20E, supplied by M/S DUCOM, Bangalore (India) shown schematically in Fig 3.7 (a). A complete schematic of the set-up of the machine is shown in Fig. 3.7. Erosive wear tests have been conducted using Al_2O_3 erodent particle. The square shaped (25 × 25 mm^2) heat treated specimens is held stationary against the impact of a 50 µm diameter erodent made of Al_2O_3. The specimen holders used for holding the samples against the impacting erodent particles to test under various impact angles. The specimen holders used during the test has been shown in Fig. 3.7 (b).

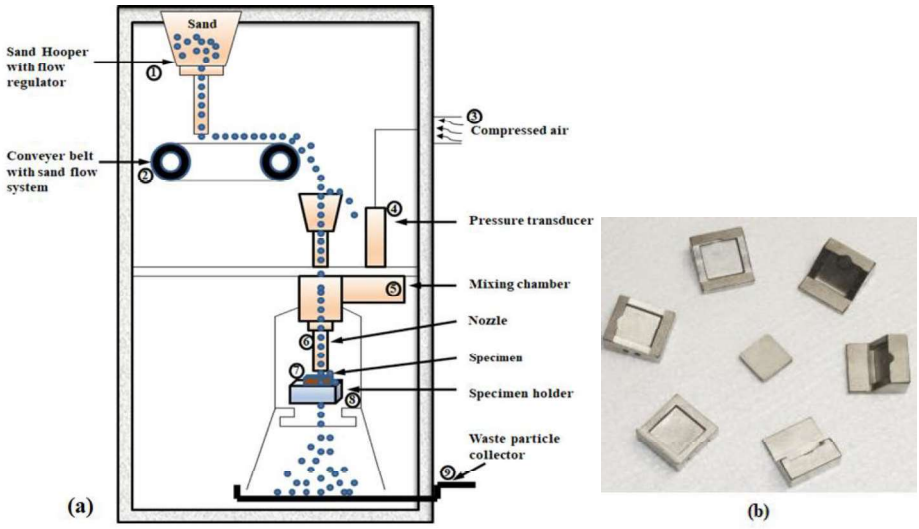

Figure 3.7 (a) Set-up of air jet erosion test rig and (b) Specimen holder for impact at different angles

The specimens have been polished up to 4/0-grade emery paper and the erodent were sieved and dried by heating in an oven at 100 ºC. The erodent at high pressure is impacted against the specimen through a nozzle of 1.5 mm diameter. The erosive wear tests for medium carbon normalized steels, DP steels and fully martensitic steels have been conducted under different impact velocities of 30 m/s, 60 m/s, 90 m/s and 120 m/s. Furthermore, four different impact angles of 15°, 45°, 75°, and 90° are used for conducting the air jet erosive wear tests in the present study. The mass flow rate of erodent particles is kept constant i.e. ~ 5.4 g/min. The distance between the specimen and the tip of nozzle is kept constant at 10 mm. Each erosive wear test has been carried out for 1 hour of duration. Specimen weight losses have been measured at different intervals of time (10 minutes) to determine weight loss. Weight loss data has been converted to volume loss data using a steel density of 7760 kg/m^3. The specimen is removed from the holder after each run, brushed lightly to remove loose wear debris, cleaned with acetone, weighed and fixed again in exactly the same position in the holder so that the orientation of the wear surface remained unchanged. The weight has been taken in a semi-micro balance to within an accuracy of 1 x 10^{-7} kg. Each test at a given impingement angle and impact velocity has been repeated three times and the average data for volume loss after each interval of time has been used for the analysis of wear rate.

3.8.1 Impact velocity calibration

The double disc method is employed to measure the impact velocity of erodent particles. This method involves two metal discs that are mounted on a common shaft. The erodent particle stream is directed towards the upper disc, which is equipped with a thin radial

slit. When the particles exit the nozzle, they collide with the upper disc, with some of them passing through the slit and eventually creating an erosion mark on the lower disc. Two erosion exposures are conducted: one with a stationary disc and the other with a rotating disc at a known revolution per minute (rpm). These exposures generate erosion marks as shown in Fig. 3.8.

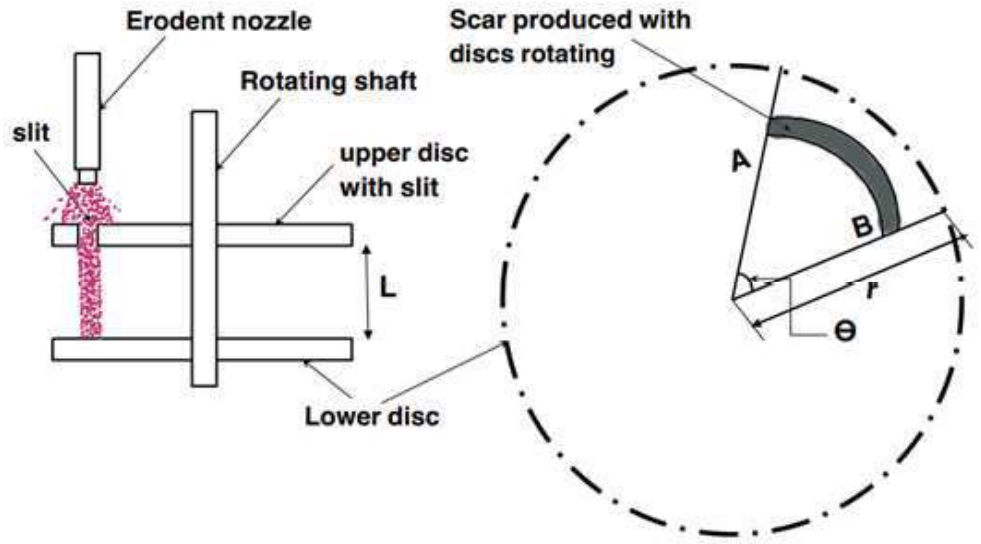

Figure 3.8 Representation of Double disc arrangement set up

By measuring the angular displacement between these marks, we can determine the flight time of the particles as they traverse the space between the discs. The time of flight of the particle pulse admitted through the slit is L/V and during this time the discs will rotate by $\left(\frac{L}{V}\right)v$ revolutions. The average velocity (V) of particles has been calculated using relation, $V = \frac{(2\,\pi rvL)}{s}$ where L is separation of the two discs (mm), r is radius of the disc (mm), v is rotational speed of disc (s^{-1}) and S is linear separation of the two marks at a radius r from the disc center.

3.8.2 Erosion test procedure

Erosion tests were performed under ambient conditions using an air jet erosion test rig (TR-471-1200, DUCOM Instruments Pvt. Ltd., Bangalore, India) which is shown in Fig. 3.7 (a). The erosion test rig consists of a compressor, drying unit, a conveyor belt-type particle feeder which helps to control the flow of erodent particle, and an air particle mixing and accelerating chamber. The compressed air is then mixed with the selected range of the alumina particles which is fed constantly by a conveyor belt feeder into the mixing chamber and then the mixture is passed through a convergent alumina nozzle of 1.5 mm internal diameter. The operating parameters for erosion test have been listed in Table 3.1. The uncertainty in the impact velocity has been calibrated against the air pressure is shown in Fig. 3.9. However, the weight loss of the specimen was measured after every 10 minutes to determine the steady state erosion rate (SSER) defined as the weight loss of the target material per unit weight of the erodent particle impinging (i.e. testing time × particle feed rate) on the target surface. The specimens were cleaned in an ultrasonic bath using acetone and weighed using semi-micro analytical balance (GR 202, A&D Company Ltd., Japan) having an accuracy of 10^{-7} kg before and after the test.

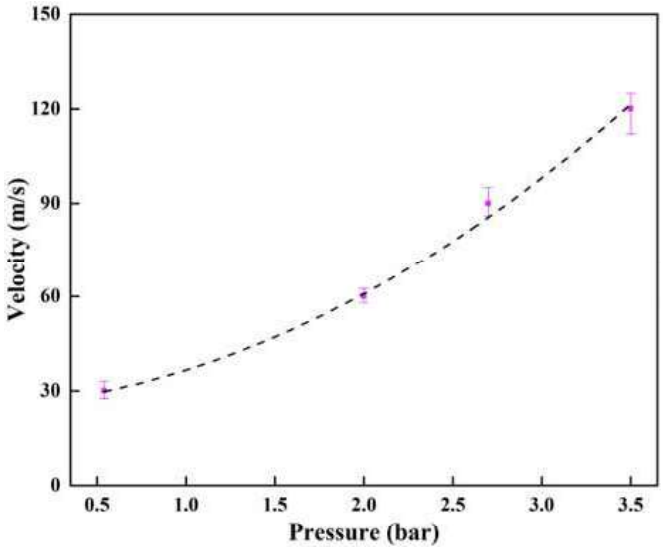

Figure 3.9 Representation of uncertainty in the impact velocity against the pressure.

Table 3.1 Operating parameters for erosion test

Parameters	**Values**
Nozzle diameter	1.5 mm
Standoff distance	10 mm
Test gas	Dry compressed air
Test duration	1 h (10 min cycle time)
Impact angle	15°, 45°, 75° and 90°
Impact velocity	30, 60, 90 and 120 m/s
Erodent discharge rate	5.4 ± 1g min^{-1}

3.9 Corrosion Studies

The corrosion behavior of N and DP steels with different ferrite and martensite volume fractions (MVF) has been investigated using potentiodynamic polarization (PDP), electrochemical impedance spectroscopy (EIS), and gravimetric method using a 3.5%

NaCl solution prepared by dissolving 35 g of analytical grade sodium chloride powder in deionized water to form 1-liter solution. All the tests were conducted at ambient temperature (27 ± 5 °C) utilizing the CS350 EIS Potentiostat /Galvanostat (CORRTEST Instruments Corp., Ltd., Wuhan, China). During electrochemical experiments, the electrolytic solution interacts directly with the laboratory air. Prior to the electrochemical polarization measurements, one of the larger surfaces of each specimen was polished using standard metallographic procedure and subsequently cleaned with acetone and distilled water. The electrochemical cell shown in Fig. 3.8 consisted of Ag/AgCl as the reference electrode, Pt wire as a counter electrode, and the polished surface (1cm^2) of steel specimen as the working electrode when submerged in the solution.

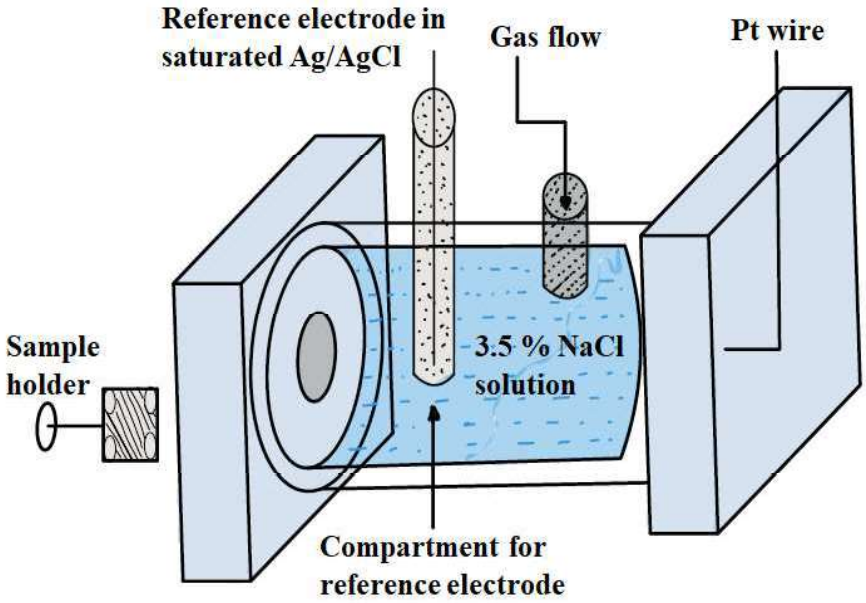

Figure 3.10 Schematic of polarization cell for electrochemical behavior measurement

The 1cm^2 area of the specimen was exposed to the electrolytic solution, which was then subjected to open circuit potential (OCP) measurement for 30 min at a 1 Hz frequency

prior to EIS and PDP tests to attain a steady state. The EIS tests have been conducted on the specimens to acquire information about the surface properties of steels during the immersion period. For EIS measurements, frequency ranges of 100 kHz to 10 Hz and amplitude of 10 mV of sinusoidal AC voltage were selected. Nyquist and Bode plots for each steel were drawn using ZView (CS studio version 5.2) software. Potentio dynamic polarization (PDP) tests were performed within -250 mV (cathodic) and 1000 mV (anodic) of the potential range. All the experiments were carried out three times, and the average results have been reported. Tafel plots have been drawn using the CView (CS Studio, Version 5.2) software from the data obtained from the PDP test, and corrosion potential (E_{corr}), as well as corrosion current (I_{corr}), have been determined from those plots.

An electronic balance (GR 202, A&D Company Ltd., Japan) has been used to measure the corrosion rate using the gravimetric method following the ASTM G1-90 standard for testing. The gravimetric method is generally slower than other techniques, but it allows for the analysis of multiple samples simultaneously. The average corrosion rate (CR) using this method is determined by Eq. (3.1).

$$CR = kW/ATD \qquad \text{Eq. 3.1}$$

Where K is a constant, W (g) is the difference between the initial and final weight of the specimen, D (g cm^{-3}) is the specimen's density, A (cm^2) is the area of the specimen exposed to the corrosive solution, and T (hr) is the immersion time.

3.10 Examination of Eroded and Corroded Surface and Sub-Surface

The surface of normalized steel, DP steels and FMS eroded at different impact velocities and impingement angles have been examined under Scanning Electron Microscope to reveal the prevailing mechanisms of erosion. Further, in order to analyze the extent of deformation, the sub-surface of the worn specimens has also been examined by subjecting the polished and etched transverse section of the worn surface to SEM examination and the salient features have been photographed. The corroded surface of normalised as well as DP steels have also been examined under scanning electron microscopy and the salient features have been captured.