CHAPTER 2

MATERIALS AND EXPERIMENTAL METHODS

2.1 INTRODUCTION

This chapter details the outline of the test materials Type 446 stainless steel used and the experimental methods followed during the course of the investigation. This chapter is divided into two sections. The first section describes materials selection, mechanical testing, materials characterization, and high-temperature erosion. Optimization of these parameters using response surface methodology and verification using an artificial neural network. Test details like geometry and fabrication of tensile testing, hardness measurement, and high-temperature erosion testing have been discussed. Process of characterization using different techniques like optical microscopy, scanning electron microscopy, transmission electron microscopy, optical profilometry, surface roughness analyser, and X-ray diffraction are also described. The second part of the study is focussed on hot-corrosion and surface modification using USSP treatment to increase the erosion-corrosion resistance of the material.

2.2 MATERIALS

The Type 446 stainless steel was procured from MIDHANI (Mishra Dhatu Nigam), Hyderabad, India, in the form of cylindrical bars of 40mm diameter, in solution annealed condition. The nominal composition determined by spark emission spectroscopy is listed in Table 2.1.

Table 2.1: Chemical composition of Type 446 stainless steel.

Elements	C	S	P	Si	Mn	Cr	Ni	Mo	N
wt%	0.068	0.0039	0.021	0.38	0.43	24.07	0.15	0.03	0.1430

Coupons of 20 mm diameter were sectioned to characterize the shot peening and corrosion properties. The samples of 25x25x5 mm^3 were prepared to carry out the erosion studies post-USSP and hot corrosion conditions.

2.3 ULTRASONIC SHOT PEENING (USSP) TREATMENT

USSP treatment using Stress Voyager (SONATS) equipment for surface modification by ultrasonic shot peening (USSP) is shown in Fig 2.1.

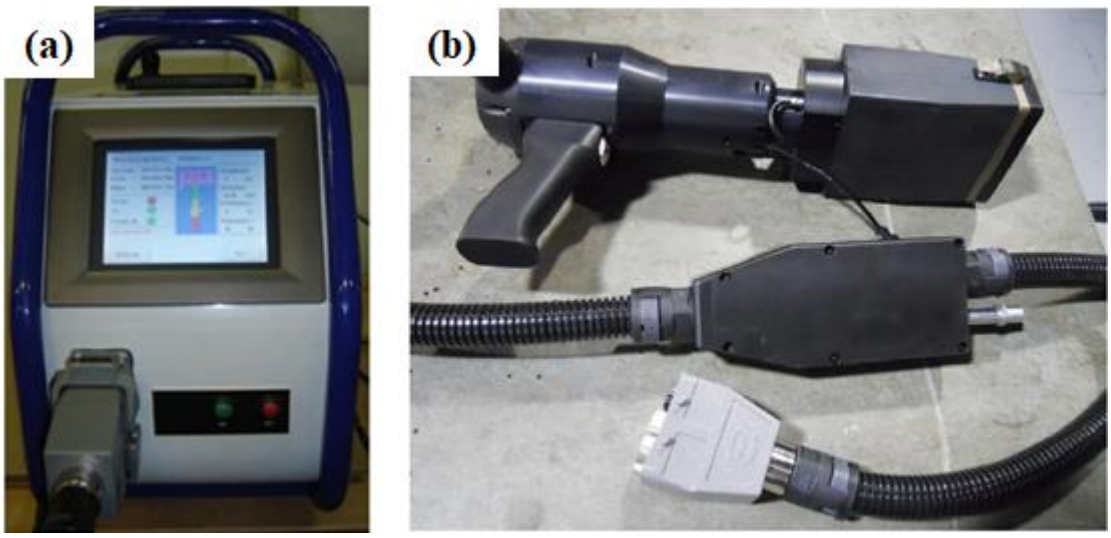

Fig. 2.1: (a) The central unit and **(b)** the peening head of the ultrasonic shot peening device.

USSP treatment was given on a flat surface of square-shaped samples of 5 mm thickness and 25x25 mm^2 surface area. The ultrasonic shot peening system comprises an auditory assembly with a piezoelectric transducer, booster, and sonotrode. Acoustic assembly generates mechanical vibration and transfers it to hard balls of 100C6 grade steel to put them in rapid motion. Ultrasonic waves with 20 kHz frequency are emitted by the piezoelectric transducer and amplified. Peak to peak vibration amplitude remained constant (80 μm) during the USSP. When the balls are resonated, the surface of the sample to be treated is impacted by a large number of flying balls over a short period of time. The impact directions of the balls onto the sample surface are random and the surface of the sample is plastically deformed at a high strain rate. The processing parameters for USSP treatment and for subsequent characterization of their microstructure and erosion-corrosion resistance are present in Table 2.2.

Table 2.2: Processing parameters for ultrasonic shot peening.

Ultrasonic frequency (kHz)	**Vibration amplitude (μm)**	**Ball diameter (mm)**	**Processing duration (sec)**
20	80	3	60
20	80	3	120
20	80	3	180

2.4 MICROSTRUCTURAL CHARACTERIZATION

The microstructure of as-received samples and shot-peened were characterized using optical microscopy, scanning electron microscopy and transmission electron microscopy. X-ray diffraction was used to analyze the constituent phases in steel, and to assess the phase stability after USSP treatment.

2.4.1 Optical Microscopy

Samples to be characterized using optical microscopy were manually polished using emery paper from 1/0 to 4/0. Subsequently, using cloth mounted on a rotary polishing wheel. Alumina suspension (Buehler Micro polish 0.3μ) was used as a colloidal solution in water. The polished samples were etched at room temperature using Glyceregia (15cc HCl +10cc Glycerol + 5cc HNO_3). The microstructures were examined using Metalux-3 optical microscope at different magnification.

2.4.2 Scanning Electron Microscopy (SEM)

Scanning electron microscope (FESEM Quanta 200 FEG) study with the EDS spectrum of the specimens, subjected to various experimental conditions, was carried out to examine the surface morphology, corrosion scale, and cross-sectional end results. The hot corroded specimens were washed in hot distilled water and subjected to acetone cleaning in an ultrasonic bath to remove loose salt particles and were finally dried. Further, the hot corroded samples were sectioned in two equal halves, perpendicular to the treated surface and the resulting section was mechanically polished to examine the depth of the oxide scale formed on the surface. The products resulting from hot corrosion of the specimens and the distribution of different elements were analyzed using energy dispersive spectroscopy (EDS).

The erosion mechanism of Type 446 stainless steel resulting from high-temperature air erosion tests at different temperatures and impact angles were characterized using SEM. The size and angularity of the abrasives used in the erosion test were characterized using (Zeiss EVO/18) SEM.

2.4.3 Transmission Electron Microscopy (TEM)

Phase characterization of the un-shot peened and shot-peened samples, and determination of grain refinement in the affected region was carried out using TEM (TECNAI 20 G^2) at 200 kV. TEM foils of the USSP treated surface region were prepared, sectioning thin slices from the USSP treated region using a slow speed precision cutter. These slices were thinned down by mechanical polishing from the side opposite to the treated surface up to a thickness of ~50 µm. Discs of 3 mm diameter were punched out from the thinned slice and TEM foils were prepared by electrolytic thinning from the side opposite to the treated surface, in the electrolyte containing 90% methanol and 10% per-chloric acid, using twin jet polisher (TenuPol-5).

2.4.4 X-Ray Diffraction (XRD)

X-ray diffraction studies of differently treated samples were carried out using RIGAKU X-ray diffractometer for characterization of phases of un-shot peened and shot peened samples. Cu-Kα radiation of wavelength 1.5402Å with Ni filter was used in 2θ range from 20°to 90° for characterization of phases, evaluation of grain size, lattice strain, lattice parameter and dislocation density of the non-USSP and USSP treated samples. The products formed during hot corrosion resulting from exposure of two salt coated samples in the high-temperature furnace were analyzed using XRD.

2.5 MECHANICAL TESTING

2.5.1 Hardness Testing

Microhardness profile of the eroded sample along the depth of scar was measured by sectioning the sample through the center of the scar, using Leco microhardness tester (LM248AT) with Vickers diamond indenter applying a load of 50 gm for 10-sec dwell.

Also, the microhardness profile of the USSP treated surface was determined from the surface along the thickness by sectioning the samples.

2.5.2 Tensile Testing

Room temperature and high-temperature tensile tests were performed using a 100 kN (Instron 5982) Universal Testing Machine (UTM) with the cross-head displacement of 1mm min^{-1}. The sample used for testing was a cylindrical shape with a gauge length of ~15mm and a gauge diameter around 4.5mm (Fig. 2.2), prepared over lathe machine by turning operation. Results obtained from the data acquisition system serve the purpose of the study which is mentioned in the later section of this work. Yield strength has been calculated by 0.2% offset method. The results are the outcome of the average of the five samples tested under the same conditions for the repeatability.

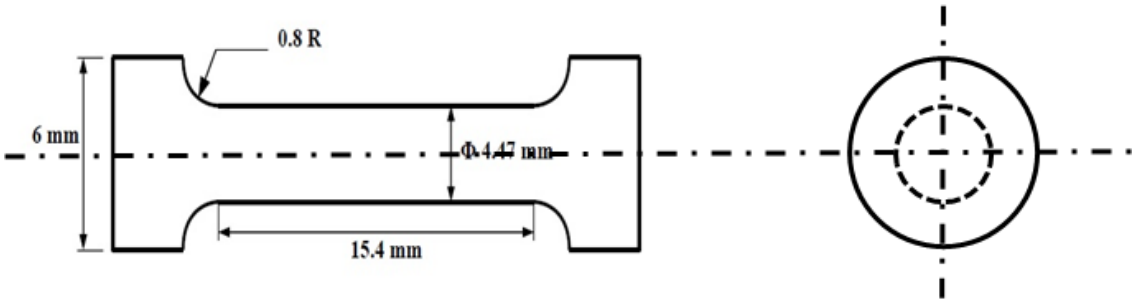

Fig. 2.2: Tensile Specimen Dimensions

2.5.3 Surface Roughness Measurement

The roughness of the eroded surface under variable impact conditions was determined using surface roughness tester (Mitutoyo, Model no. SJ410). The 3D-profilometer was used to evaluate the surface roughness of the sample before and after the erosion test using Multifunction Tribotester (Rtec, USA). The multifunction

tribometer holds attachment for optical profilometry with Nikkor objective lens with 4X and 10X magnification.

2.6 PRE-HOT CORROSION OF EROSION SPECIMENS

Two salt mixture (75% Na_2SO_4 + 25% NaCl) coating was used to study low-temperature hot corrosion on Type 446 stainless steel samples. Samples were polished and cleaned in an ultrasonic bath using acetone before salt deposition. The samples were pre-heated to a temperature of 150-200℃ for 15 minutes so as to remove the moisture from the aqueous salt solution. The salt mixture was coated using the spray gun deposition technique. The set-up of salt coating and air brush are shown in Fig. 2.3. The specimens were weighed before and after the salt deposition, and the weight of the coating per unit area was determined.

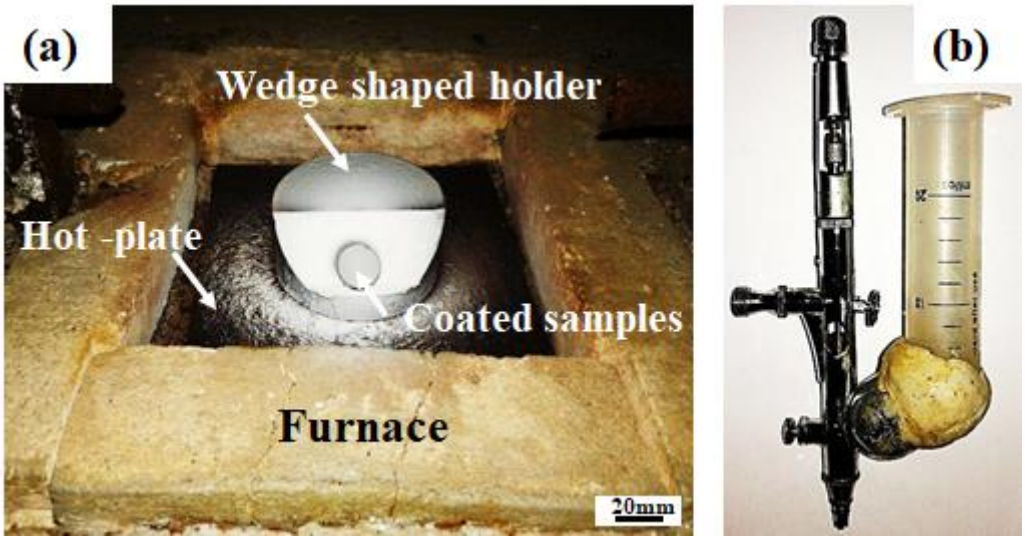

Fig. 2.3: Salt coating set-up **(a)** Hot plate for salt coating and **(b)** Air brush (Model-BD203)

An amount of 4-4.5 mg/cm^2 of salt mixtures was deposited separately on each sample for hot corrosion study. Hot corrosion tests were performed keeping the as-sprayed samples in silica crucible in an electric resistance heating furnace at 550, 650 and 750°C

with temperature control of ±2ºC using the Sandvik digital controller. The specimens were subjected to isothermal heating for the total duration of 20h. The weight of the silica crucible was stabilized by initially pre-heating the empty crucible at 850℃ for 10h. The final weight gain of the salt coated samples was measured by measuring the difference of weight before and after exposure taking into account the weight of the spalled scale, using digital balance (Mettler Toledo) with a sensitivity of 0.1mg.

2.7 SOLID PARTICLE EROSION

Solid particle erosion behaviour of Type 446 stainless steel in as received conditions and hot corroded conditions were evaluated by air-jet erosion tester (Model: TR-471-800, M/s DUCOM Instruments Bengaluru, India). The schematic of the test rig is shown in Fig. 2.4. The standard size of the test samples was cut from 40 mm diameter cylindrical bar using a milling machine and precision cutter. The samples were freed from all the side furs and ground using 1/0 to 4/0 SiC grit papers and polished using alumina suspension (Ra ≈0.2µm) as per ASTM G-76-95 standard at room temperature. Flux rate (gm min^{-1}) was measured by collecting the mass of erodent discharged through the nozzle for 10 minutes. Double – disc method was used to calibrate the impact velocity of the erodent. The operating parameters for the erosion test are given in Table 2.3. The room temperature during the test was recorded between 30 to 35℃, using a hygrometer (Testo 623). The flux rate of 4.2 ± 0.5 gm min^{-1} was selected to investigate the effect of the inter-particle collision caused due to high particle feed rate. Angular shape alumina particle was used as the erodent having particle size ~50 µm. The physical properties of the alumina sand are listed in Table 2.4. The specimens were cleaned in an ultrasonic bath using acetone and weighed using an analytical balance to an accuracy of 0.1mg

before and after the test. The material was eroded for a period of 30 min although each sample was removed after every cycle to determine the weight loss and erosion rate (ER). The total weight loss was measured for a net period of 60 minutes. The erosion rate is expressed as:

$$\text{Erosion Rate (g/g)} = \frac{\text{weight loss of target material}}{\text{weight of the erodent particle impinging on the target surface}}$$

The erosion rate (g/g) versus time (min) was plotted to fit the data point.

Table 2.3: Operating parameters for solid particle erosion testing.

Operating Conditions	
Nozzle diameter	1.5 mm
Standoff Distance	10 mm
Test gas	Dry compressed air
Test duration	1 hrs (as per cycle time)
Test temperature	Room temp, 350°C, 550°C and 650°C
Impact angle	30°, 60°, 90°
Abrasive flow rate	4.5 ± 0.5 g/min
Impact Velocity	100 m/sec

Table 2.4: Physical properties of Aluminium Oxide erodent.

Physical Properties	
Crystal Phase	Alpha
Specific Gravity	3.95 g cm^{-3}
Particle Shape	Sharp, angular
Vickers Hardness	1600 HV
Average particle size	41.78 µm

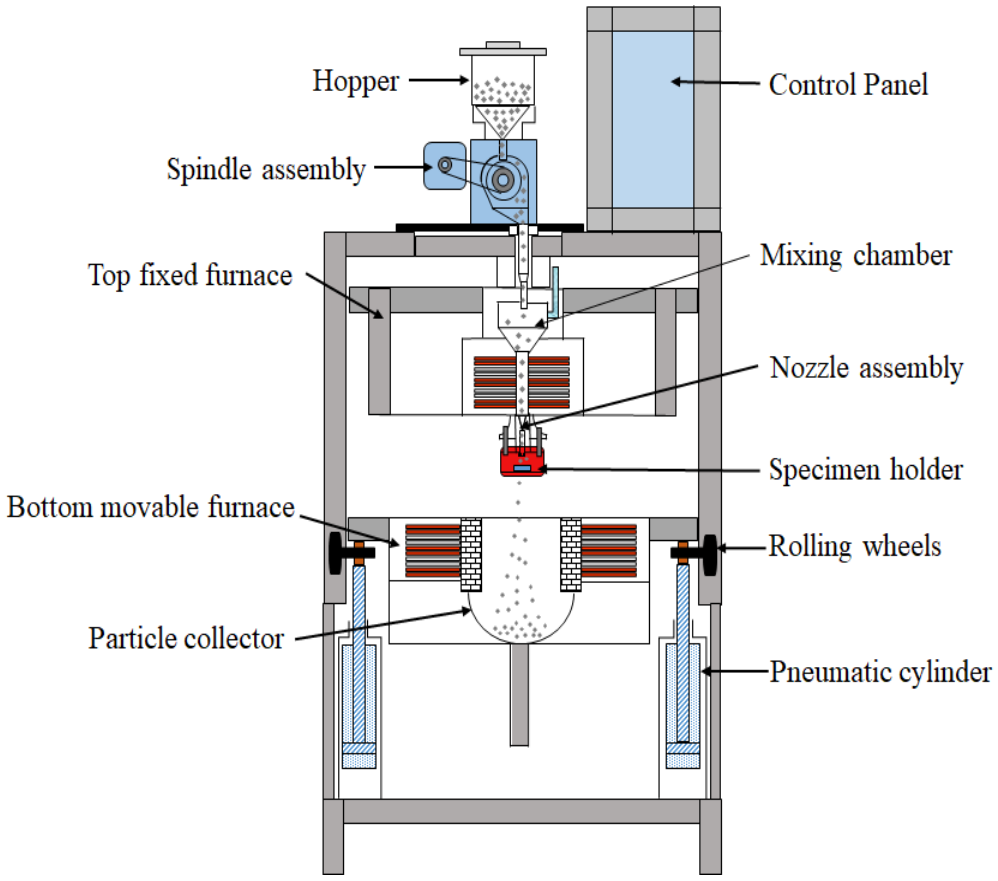

Fig. 2.4: Schematic diagram of the erosion test rig.

2.8 MODELLING AND OPTIMIZATION

A second-order polynomial model is developed using response surface methodology (RSM), and the effect of the main and interactive parameters on erosion is optimized. A well trained artificial neural network (ANN) with a back propagation algorithm is used to verify the optimized parameters for erosion rate generated by RSM.

2.8.1 Response Surface Methodology

RSM is a collection of mathematical and statistical techniques that are used for modelling, analysis, and optimization, where the response is influenced by several variables. This is an economical and effective method of optimization which reduces the experiments to a fairly low number. Although RSM employs either linear or square polynomial functions to describe and explore the experimental conditions, second-order models are generally used in RSM as they are flexible and also provide a better approximation to the true response surface.

2.8.2 Artificial neural network (ANN)

ANN is inspired by human brain functions and consists of highly interconnected processing elements in groups called neurons. The pattern of interconnecting these neurons is called “architecture”. The performance of the neural network depends on the number of hidden layers and some neurons in hidden layers, i.e., network structure. Therefore, a computer program “NEURONET” is developed using PYTHON 3.7 for training the data using three layers, and the result is inferred using the backpropagation neural network.