CHAPTER 3

EXPERIMENTAL DETAILS

3.1 MATERIALS

The material used for this study was 2” pipes of DSS (UNS 31803) and SDSS (UNS 32750) with 5.54 mm thickness and 150 mm length. The material composition was selected in such a way to get low and high PREN values. A typical chemical composition of the base materials is given in Table 3.1 and microstructure is shown in Figure 3.1. The mechanical properties of base material are listed in Table 3.2

Table 3.1 Base metal chemical composition

Material Grade	Cr	Mo	Ni	N	C	PREN	Remarks
UNS S31803	22.9	3.03	7.92	0.15	0.017	35.15	DSS-Low PREN
UNS S31803	22.9	3.04	7.63	0.17	0.019	36.30	DSS-High PREN
UNS S32750	25.1	3.75	8.86	0.21	0.028	41.40	SDSS-High PREN
UNS S32750	25.1	3.71	8.9	0.2	0.016	40.36	SDSS-Low PREN

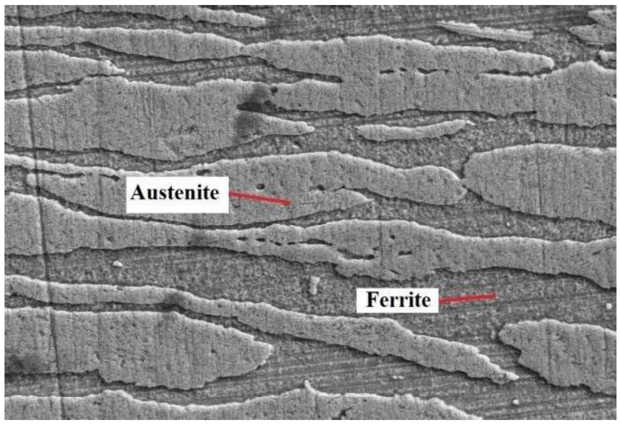

Figure 3.1: Typical base material microstructure

Table 3.2- Base material properties

Grade	Tensile Strength (MPa)	Hardness (VHN)	Impact Toughness [J] at -46 ^{O}C
DSS-Low PREN	750	255	127
DSS-High PREN	762	260	109
SDSS-Low PREN	806	301	140
SDSS-High PREN	830	310	119

The filler metal compositions are given in Table 3.3. A standard Sandvik made filler metal of 2 mm diameter was used for all welding trials.

Table 3.3- Filler metal chemical composition

Base Metal	Filler Grade	C	Si	Mn	P	S	Cr	Ni	Mo	N
DSS	22.8.3.L	$\leq$0.02	0.5	1.6	$\leq$0.02	$\leq$0.015	23	9	3.2	0.16
SDSS	25.10.4.L	$\leq$0.02	0.3	0.4	$\leq$0.02	$\leq$0.015	25	9.5	4	0.25

3.2 WELDING

Duplex Stainless Steel and Super Duplex Stainless Steel pipes were welded using Gas Tungsten Arc Welding (GTAW) process with Direct Current Electrode Negative (DCEN) polarity. The experiments were conducted in two ways.

a) By varying welding heat input

b) By varying shielding gas/back purging gas composition and inter-pass temperatures.

The general welding specifications for first part of the study are given in Table 3.4.

Table 3.4- Welding specifications

Welding Position	5G (pipe fixed horizontally and weld around without rotating the pipe)
Groove design	Single V groove 70^{O} groove angle 1 mm root face and 2.5-4 mm root gap
Welding current(A)	80-150

Arc voltage (V)	10-12
Welding speed (mm/min)	40-80
Number of weld layers	4-5
Inter pass temperature (^{O}C)	100-140
Gas flow rate (L/min)	13-18
Heat input (kJ/mm)	0.75-1.25

Experiments were conducted to study the effect of shielding gas, purging gas and interpass temperature on mechanical and corrosion properties. During experiments, one of the above parameter was varied while other parameters were kept constant as given in Table 3.5 and Table 3.6.

Table 3.5- Welding parameters to study effect of shielding/purging gas and interpass temperature for DSS weldments

Exp. No.	**PREN**	**Heat Input (kJ/mm)**	**Shielding gas**	**Purging gas**	**Interpass temperature (^{O}C)**
1	35.15	0.75~1.25	Ar+2%N	Ar+2%N	120
2	35.15	0.75~1.25	Ar+5%N	Ar+5%N	120
3	35.15	0.75~1.25	Ar+2%N	Ar+2%N	160
4	35.15	0.75~1.25	Ar+2%N	Ar+5%N	160

Table 3.6- Welding parameters to study effect of shielding/purging gas and interpass temperature for SDSS weldments

Exp. No.	PREN	Heat Input (kJ/mm)	Shielding gas	Purging gas	Interpass temperature (^{O}C)
1	41.4	0.75~1.25	Ar+2%N	Ar+2%N	120
2	41.4	0.75~1.25	Ar+5%N	Ar+5%N	120
3	41.4	0.75~1.25	Ar+2%N	Ar+2%N	160
4	41.4	0.75~1.25	Ar+5%N	Ar+5%N	160

From literature, we noted that shielding gas effect on both DSS and SDSS are similar. Hence, we have taken low PREN from DSS and high PREN from SDSS to cover both low and high PREN.

3.3 EXPERIMENTATIONS

3.3.1 Non-Destructive Testing

After completion of welding, welded specimens were subjected to Non Destructive Testing (NDT) such as Penetrant testing (PT) to find out surface defects and Radiographic testing (RT) for finding internal defects. These tests are conducted mainly to locate the defects and discard the defective portion and make sure that the sound test samples are prepared.

Test samples were prepared as per American Society for Testing and Materials (ASTM) and American Society of Mechanical Engineers (ASME) standards and are referenced in the applicable section.

3.3.2 Metallography

After successful completion of welding, the weld specimen were cut and polished up to 1200 grit fineness which was followed by cloth polishing with 0.05 µm alumina powder. Later the specimens were etched with 20% sodium hydroxide to reveal the microstructures. Microstructures were viewed under optical microscopes (Make: Olympus, Model: BX Series Upright Metallurgical Microscope BX53M). Figure 3.2 illustrate the Optical microscope used in this work for viewing the microstructure.

Figure 3.2: Optical microscope

Ferrite content was determined in base metal, HAZ and weld region through point count method in accordance with ASTM E562 standard. The elemental composition of each phase was checked by Energy Dispersive Spectroscopy (EDS) method. Figure 3.3 shows the SEM-EDS microscope (Make: JEOL, Model JSM-7600F) used in present study.

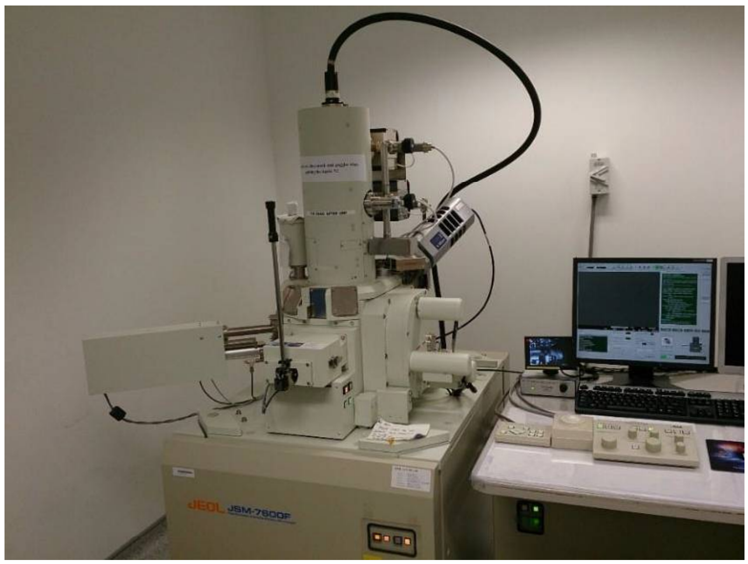

Figure 3.3: SEM-EDS equipment

3.3.2 PITTING CORROSION TEST

3.3.2.1 ASTM G48 test

Pitting behavior of the welded samples was studied using ASTM G48 gravimetric test [14], which is a common method used for measuring pitting corrosion rate of DSS in ferric chloride solution. After welding, specimens were cut into 50 mm x 25 mm size for testing and the initial weight was measured by digital weighing machine (Make: Contech, Model: CAH-1003). The specimens were immersed in

6 % of ferric chloride solution for a period of 24 h at a constant test temperature of (22$\pm$1) ^{O}C and (28$\pm$1) ^{O}C for DSS and (35$\pm$1) ^{O}C and (40$\pm$1) ^{O}C for SDSS. After 24 h period of immersion, specimens were rinsed with water, dipped into acetone in an ultrasonic cleaner and air-dried. Subsequently, the test specimens were examined for visible pits and weighed to obtain the weight loss due to corrosion attacks. Digital weighing machine used for measuring the weight in this work is shown in the Figure 3.4.

Figure 3.4: Digital weighing machine

3.3.2.2 Potentiodynamic Polarization test

Corrosion behavior was also studied through potentiodynamic polarization technique in 1.0 mol/L NaCl solution at (22$\pm$1) ^{O}C and (35$\pm$1) ^{O}C temperature for DSS and SDSS respectively with a scan rate of 0.5 mV/s from -1200 mV (SCE). The area of the specimen exposed to the solution was 100 mm^2. Some part of the exposed surface was insulated with adhesive tape so that exposed surface would be the cross-sectional area of the pipes. An electrochemical cell was used with specimen as a working electrode, platinum as a counter and calomel as a reference electrode. The potentiostatic measurements were also carried out to evaluate pitting corrosion of the weldments as per ASTM G150 standard. The anodic potential applied was about 750 mV (SCE) until the occurrence of stable pitting. The temperature of the solution was increased at the rate of 1 ^{O}C/min. Figure 3.5 shows the image of corrosion testing setup (Make: Gamry Reference 600 Potentiostat).

Figure 3.5: Corrosion testing setup

3.3.3 MECHANICAL TESTING

3.3.3.1 Tensile test

After successful completion of welding, the specimens were subjected to mechanical testing. The weldments were characterized with tensile test, hardness test and impact test. For tensile test, ASME IX: 2010 standard was followed. The material thickness and width were 5.30 mm and 13.20 mm respectively. Three sets of tests were performed and average was taken in this study. Later, the fractured surfaces were examined under SEM. Universal testing machine (Make: Instron, Model: 5900 Series UTM) used in this work for tensile test is shown in Figure 3.6.

Figure 3.6: Universal testing machine

3.3.3.2 Impact test

Charpy V-notch impact tests were performed on a pendulum type impact tester as per ASTM A370 standard. The specimen dimensions used for impact tests were 5*10*55 mm. All the impact tests were carried out at -46 ^{O}C (This is the critical temperature for DSS and SDSS material). Three sets of tests were performed and mean value was considered for our studies. Figure 3.7 shows the image of the impact test machine (Make: FIE, Model: IT-30) used to conduct impact testing and Figure 3.8 shows standard specimen size used for carrying out impact test.

Figure 3.7: Impact testing machine

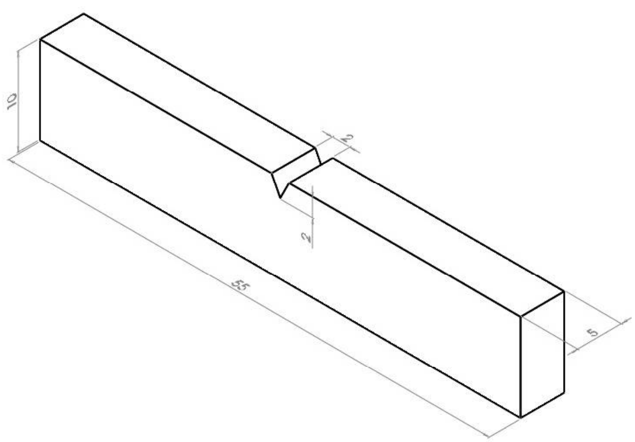

Figure 3.8: Standard specimen for Impact test

3.3.3.3 Hardness test

Hardness measurements were taken on transverse section of weldments where hardness values were measured at weld metal, HAZ and base metal. In addition, hardness was measured along thickness of weldments. Four set of tests were conducted in same region and average value is reported. The ASTM E92 standard was followed with 10 kg of test load. Figure 3.9 shows the hardness test machine (Make: Buehler, Model: Wilson VH1202) and Figure 3.10 shows a hardness test specimen.

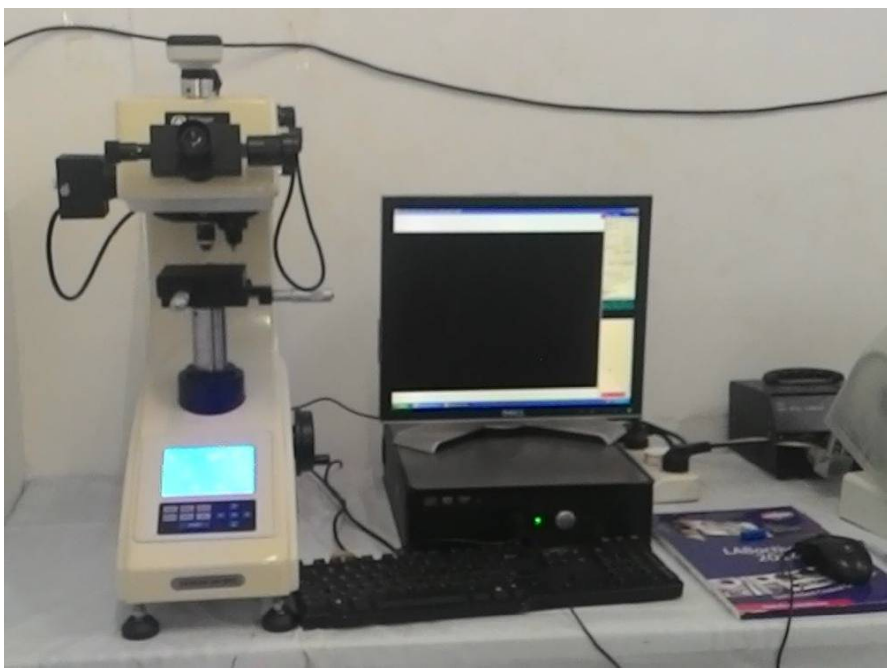

Figure 3.9: Hardness testing setup

Figure 3.10: Sample specimen for Hardness test