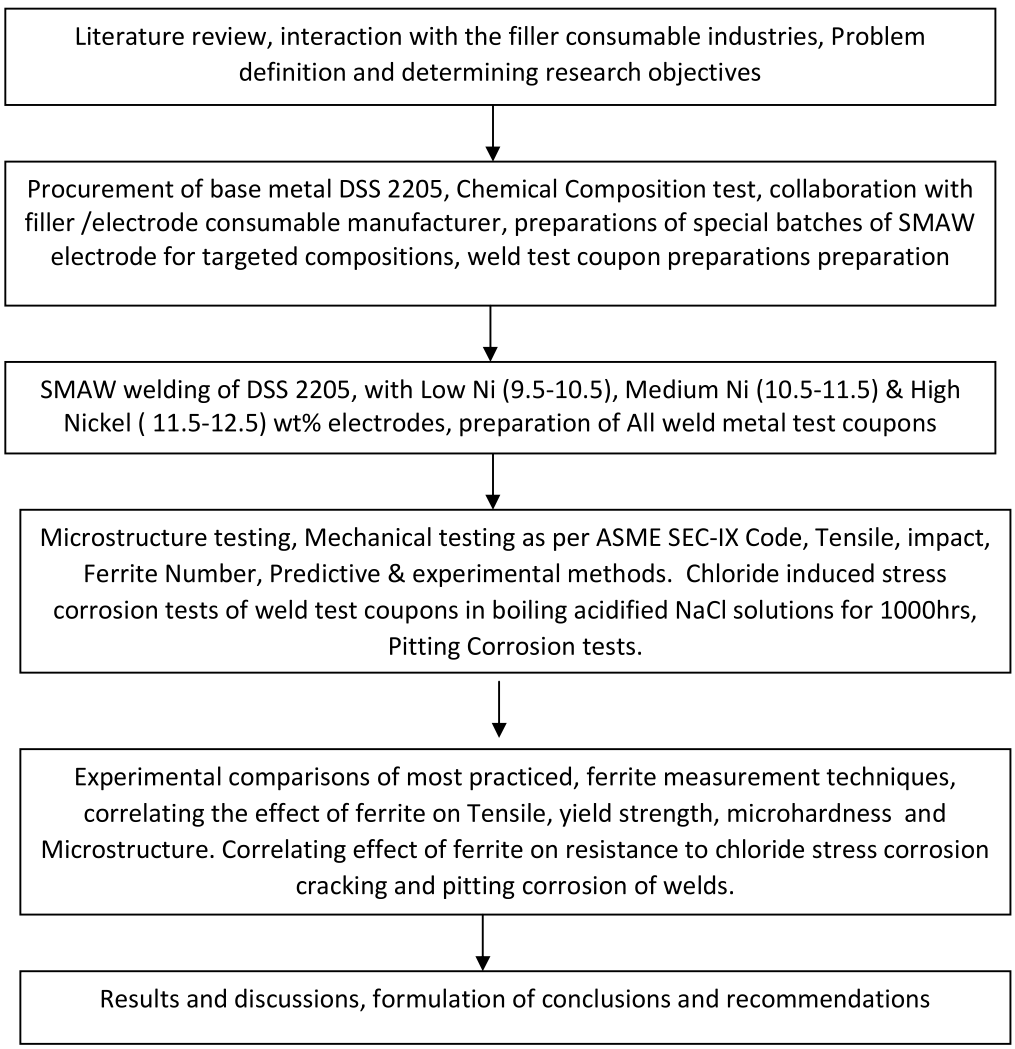
CHAPTER – 3

EXPERIMENTAL WORK

The experimental procedure involved in this research work has been presented in this chapter. This chapter begins with the chemical composition analysis of the candidate metal and electrode composition followed by process parameters employed for welding, metallographic examination and various mechanical Testing as per ASTM / ASME BPVC SEC IX Codes and Corrosion testing as per ASTM Standards of the all weld test coupons prepared as per ASME BPVC Sec IX Code. The detailed step by step methodology adopted while carrying out the research work is given below.



3.1 Procurement of Candidate Materials

OUTO KUMPU Certified Test Certificate of Standard UNS 32205 / 1.4462 confirm with ASTM A 240-06, ASME SA-240 2004 A05, EN 10051 / ASTM A 480.

Table 3.1 Chemical Composition of UNS S31803 modified (UNS S 32205) as per PMI Spectroscopy as per Base Metal Validation report attached in Annexure 6

C	Si	Mn	P	S	Cr	Ni	Mo	Cu	Nb	N
0.023	0.37	1.51	0.018	0.001	22.37	5.72	3.21	0.14	0.011	0.177

Microstructure according to ASTM A 923-A Satisfactory

PRE: Cr +3.3Mo + 16 N = 35.7

Corrosion according to EN ISO 3652-2C

Heat Treatment "Materials Temperature 1100 °/Quenched (Forced air + water)

(**As per OUTOCOMPU Supplier Test Certificate**)

Total 04 Cut plates of dimension 1000 mm (L) X 150 (W) X 25 (T) procured from the reputed Steel suppliers.

Table 3.2 Physical Properties of Duplex Stainless steels Source [9,10]

Properties	Unit	20 °C	100 °C	200 °C	300 °C
Density	g/cm2	7.8			
Modulus of elasticity	GPa	200	194	186	180
Poisson ratio	-	0.3	-	-	-
LTE	X 106/°C	-	13.0	13.5	14.0
Thermal conductivity	W/m°C	15	16	17	18
Thermal capacity	J/Kg°C	500	530	560	590
Electric resistivity	µ Ohm	0.80	0.85	0.90	1.00

3.1.1 Mechanical Properties

Table 3.3 As per ASTM & EN Mechanical property limits for Duplex SS Plate*

	ASTM				EN			
Grade	UNS No.	Y.S 0.2% Mpa(Ksi)	T.S in Mpa(Ksi)	% E.L 2”. %	EN No	Proof Strength Rp0.2 Mpa(Ksi)	Tensile Strength Rm Mpa(Ksi)	EL As %
2205	S32205	450(65)	655(95)	25	1.4462	460(67)	640(93)	25

3.1.2 Impact Toughness

Table 3.4 Min values according to EN 10028, Transverse direction, J*

UNS S32205 Test Temperature	Specimen size(mm)	Readings			Average
20 °C	10X10X55	62	65	60	62.3

*** As per Test report attached in Annexure 6**

3.2 Procurement of sample SMAW Electrodes

Weld Consumable Test support was provided by Ms. GEE Ltd, Kalyan (West) Thane Mumbai, India, They have provided total three lots of samples of Electrode Size 3.15 X 350 mm length. SMAW electrode, each 10 kg wt., starting from a standard commercial AWS E2209-16 alloy design, Nickel was varied in composition as mention in table No.3.5 as per Test Certificates provided by Manufacturers.

3.2.1 Specification of AWS E 2209-16 Commercial (GRINOX-2209-16)

Electrode Specifications

AWS/SFA 5.4: E2209-16, IS 5206: E 22 15 3 LR 26

ASTM A 182 gr. F51 UNS S31803 DIN 1.4462

Description

A rutile coated SMAW electrode is specially designed and formulated with three different Nickel targets (9.5-10.5 % , 10.5-11.5 & 11.5-12.5 % Nickel), referred to herein after as Low Ni, Medium Ni and High Ni, in standard size 3.15 X 350 mm length, for welding commercially available UNS S32205 duplex stainless steels.

It was expected that using this boosted Nickel (9.5-10.5 %, 10.5-11.5% & 11.5-12.5% Ni) electrode, deposit austenitic-ferritic duplex weld metal having a ferrite content approximately 14% to 30%, or 20 FN to 40 FN below that of specified values by the recommended ferrite specification limit i.e 35 % to 60 % Ferrite. (30 to 70 FN)

ASME IX QUALIFICATION: QW-432 F-NUMBER 5, QW-442 A-NUMBER 8

Table 3.5 Chemical compositions of the undiluted weld metals carried out on weld pad as per ASME SEC II C.

SMAW Electrode with Nickel content	C	Mn	Si,	S	P	Cr	Ni	Mo	Cu	N
Standard 2209 Electrode E2209	0.031	1.08	0.59	0.007	0.025	22.38	9.15	3.35	0.096	0.18
Low Ni 9.5-10.5 % wt.	0.019	0.99	0.61	0.005	0.027	22.29	9.80	3.19	0.068	0.17
Medium Ni 10.5-11.5 %wt.	0.021	0.99	0.58	0.007	0.028	22.35	10.20	3.16	0.075	0.16
High Nickel 11.5-12.5 %wt.	0.017	1.11	0.62	0.008	0.028	22.33	12.55	3.20	0.069	0.18

3.3 Preparation of Weld Test coupon

Three Test Coupons of UNS S32205 Duplex Stainless Steel Material Weld Test Coupons respectively of sizes 500 mm (L) X 150 mm (W) 25 mm (T), 700 mm (L) X 150 mm (W) 25 mm (T) & 500 mm (L) + 200 mm (L) X 150 mm (W) 25 mm (T) with single 60 degree V-groove prepared using SMAW GRINOX E2209-16 Electrodes of varying Nickel contents. Target values of nickel being used in experiments was Low Ni (9.5-10.5 %) Medium Ni (10.5-11.5%) and High Ni (11.5-12.5 %)

The test pieces were welded by ITW India Pvt. Ltd professional 2G qualified welder in the flat position as consumables designed was suitable for exclusively for welding in flat position. The welding was started after applying backing to electrodes as per the requirements given in the consumable standard.

The interpass temperature controlled ~ 150 °C Max for each successive pass in accordance with the consumable standard.

Interpass temperatures was measured and recorded after each pass using temperature indicator crayons and optical thermometer.

The welding conditions used, such as current, voltage, travel speed, weld run sequence, and weld bead size, in accordance with the limits specified in the relevant consumable test standard.

Backing strip of the same material was used. The backing strip tack Welded to the test piece on the reverse side as per figure.

The plates of the test piece were restrained in such a way that a sufficiently flat test piece is produced for extraction of specimens.

The dimension of the Test Set up as shown in the table as per ISO 15792-1:2000(E) Welding consumables — Test methods — Part 1: Test methods for all-weld metal test specimens in steel, nickel and Nickel alloys [77]

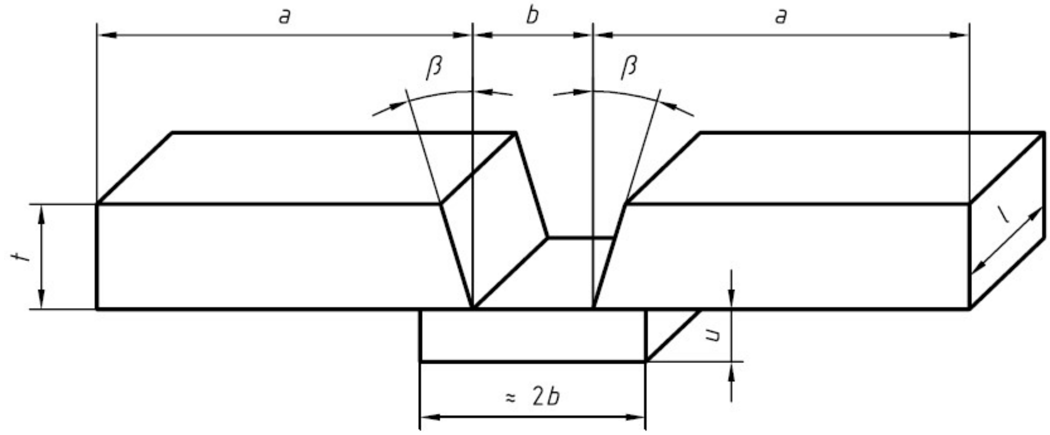


Fig. 3.1 Schematic of All weld metal test coupon preparation set up. Source[77]

Table No. 3.6 Dimension of all weld test coupon set up. (in mm)

Type	t	a	b	u	β Degree	l
1.4	25	≥150	20	≥6	$10\ _{0}^{+2.5}$	≥150

Coupon lD - E2209(10Ni22Cr), E2209(11Ni22Cr) & E2209(12Ni22Cr) Welding Process - SMAW, Filler Wire - E22O9-16 (GRINOX-2209), Welding Position – Flat

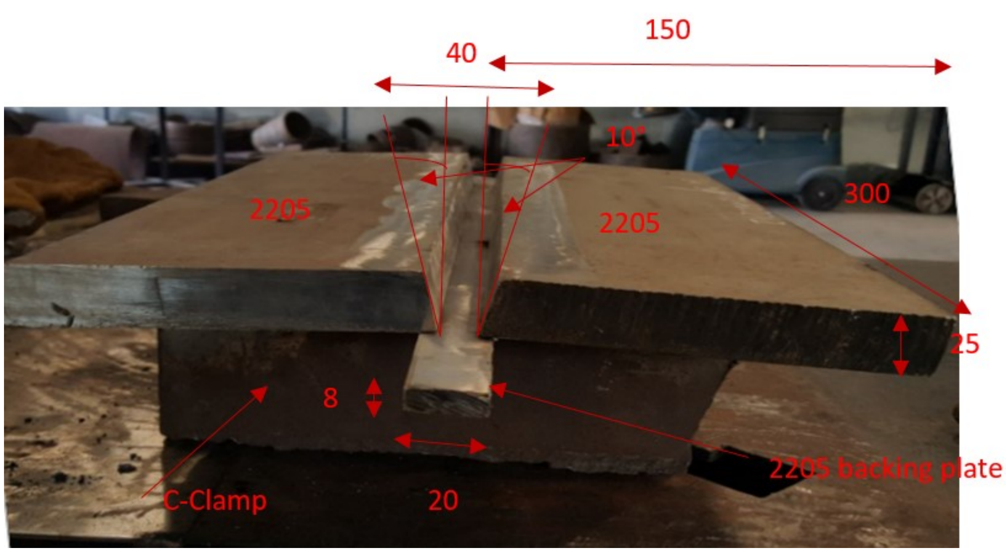


Fig. 3.2 Weld Test Coupon prepared for welding

Preparation of Test Set up

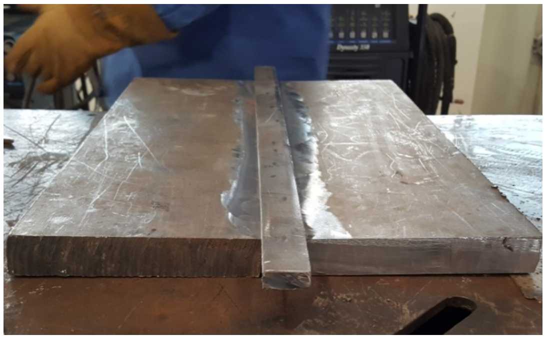


Fig. 3.4 Backing Strip Weld

Fig. 3.5 C-Clap Restraint (Front view)

Fig. 3.6 (Side View)

Fig. 3.7 Wire Brush finished test Piece

Fig. 3.8 Fill Pass completed

Fig. 3.9 Root pass completed

Fig. 3.10 SMAW Electrode GRINOX 2209 E 2209-16   Size 3.15 X 350 mm

Fig. 3.11 Application of digital thermometer to control less than 150 °C inter pass temperature.

Fig. 3.12 Application of templistic to control less than 150 °C inter pass temperature.

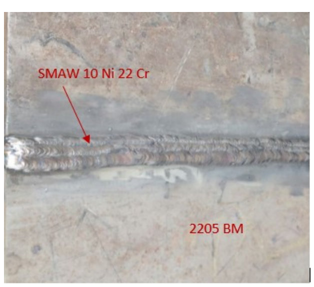


Fig. 3.13

Weld test Coupon ID

10 Ni 22 Cr

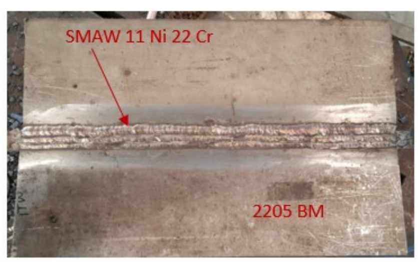


Fig. 3.14

Weld test Coupon ID

11 Ni 22 Cr

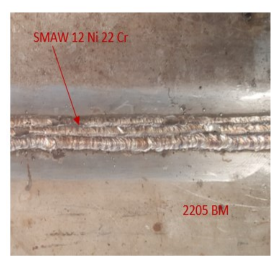


Fig. 3.15

Weld test Coupon ID

12 Ni 22 Cr

3.4 Welding Procedure

3.4.1 SMAW process

All Weld Test Coupons were prepared using Shielded Metal Arc Welding (SMAW) Process with International quality Miller Make Welding machine by an AWS Qualified and experienced welder.

3.4.2 Welding Machine specifications:

The Welding Machine specification as under:-

Miller Make XMT 350 OS Autoline

Volts @ 50/60 Hz Cycle 400

Amps 29.7/20.5

Kilowatt 11.2/13.6

Rated Output

DC Volts 32/34 DC Ampere 300/350

Max OCV 75 Duty Cycle 60%

3.4.3 Setting up process parameters

Process Parameters

Voltage : 20 V (recommended Range of 18-22 V)

Current: 110 A (80-120 A)

H.I : 0.5-2.5 KJ/mm Average 1.0 KJ/mm

Intepass Temperature: below 150 °C

Travellingspeed:160-170mm/min

Backing of Electrodes: 250 °C for 2 hrs.

Preheating temperature of plates: None

PWHT. Not required.

Weld Position: Flat

WEP: 10 Degree groove.

Bead deposition: Stringer bead technique

Heat input and inter pass temperature are the two main welding parameters majorly influencing the cooling rate

3.4.4 Calculations of H.I

Heat input (KJ/mm) = (V * A) / (S * 1000)

Where V = voltage (volt)

A = Current (ampere)

S = travel Speed (mm/s)

3.4.5 Weld process data recording sheet.

3.4.5.1 DATA SHEET Test Coupon Preparation E2209-16 (9 Ni 22 Cr)

Sr. No	Layer	Pass	Current	Voltage	Time	Length	T-Speed	H/Input
	No	No	Amp	Volts	Sec	mm	mm/min	KJ/mm
1	1	1	117	28	238	560	141	2.3
2		2	118	24	231	560	145	2.0
3	2	1	117	23	258	560	130	2.1
4		2	118	24	246	560	137	2.1
5	3	1	119	26	185	560	182	1.7
6		2	117	25	185	560	182	1.6
7		3	118	24	190	560	178	1.6
8	4	1	118	25	189	560	178	1.7
9		2	117	25	209	560	161	1.8
10		3	119	26	185	560	182	1.7
11		4	120	25	185	560	182	1.6
12	5	1	119	24	189	560	178	1.6
13		2	118	25	185	560	182	1.6
14		3	119	25	189	560	178	1.7
15		4	119	25	185	560	182	1.6
16	6	1	120	24	185	560	182	1.6
17		2	119	24	185	560	182	1.6
18		3	118	25	190	560	180	1.6
19		4	119	24	189	560	178	1.6
20	7	1	118	26	209	560	177	1.7
21		2	119	24	185	560	182	1.6
22		3	119	25	185	560	182	1.6

23		4	118	24	189	560	180	1.6
24	8	1	119	25	185	560	182	1.6
25		2	120	24	189	560	180	1.6
26		3	120	24	185	560	185	1.6
						AVG	173	1.71
							T-Speed	H/Input
							mm/min	KJ/mm

3.4.5.2 DATA SHEET Test Coupon Preparation 10 Ni 22 Cr

Sr. No	Layer	Pass	Current	Voltage	Time	Length	T-Speed	H/Input
	No	No	Amp	Volts	Sec	mm	mm/min	KJ/mm
1	1	1	117	24	238	560	141	2.0
2		2	118	22	231	560	145	1.8
3	2	1	117	20	258	560	130	1.8
4		2	118	20	246	560	137	1.7
5	3	1	119	20	185	560	182	1.3
6		2	117	24	185	560	182	1.5
7		3	118	22	190	560	178	1.5
8	4	1	118	21	189	560	178	1.4
9		2	117	22	209	560	161	1.6
10		3	119	24	185	560	182	1.6
11		4	120	21	185	560	182	1.4
12	5	1	119	18	189	560	178	1.2
13		2	118	19	185	560	182	1.2
14		3	119	21	189	560	178	1.4
15		4	119	22	185	560	182	1.4
16	6	1	120	24	185	560	182	1.6
17		2	119	21	185	560	182	1.4
18		3	118	20	190	560	180	1.3
19		4	119	21	189	560	178	1.4
20	7	1	118	21	209	560	177	1.4

21		2	119	21	185	560	182	1.4
22		3	119	22	185	560	182	1.4
23		4	118	22	189	560	180	1.4
24	8	1	119	22	185	560	182	1.4
25		2	120	24	189	560	180	1.6
26		3	120	24	185	560	185	1.6
						AVG	173	1.49
							T-Speed	H/Input
							mm/min	KJ/mm

3.4.5.3 DATA SHEET Test Coupon Preparation 11 Ni 22 Cr

Sr. No	Layer	Pass	Current	Voltage	Time	Length	T-Speed	H/Input
	No	No	Amp	Volts	Sec	mm	mm/min	KJ/mm
1	1	1	117	23	238	600	155	1.7
2		2	118	21	231	600	152	1.6
3	2	1	117	21	258	600	145	1.7
4		2	118	21	246	600	151	1.6
5	3	1	119	21	185	600	161	1.6
6		2	117	24	185	600	162	1.7
7		3	118	20	190	600	166	1.4
8	4	1	118	21	189	600	168	1.5
9		2	117	21	209	600	165	1.5
10		3	119	22	185	600	168	1.6
11		4	120	21	185	600	170	1.5
12	5	1	119	18	189	600	169	1.3
13		2	118	22	185	600	177	1.5
14		3	119	22	189	600	165	1.6
15		4	119	21	185	600	166	1.5
16	6	1	120	22	185	600	182	1.5
17		2	119	20	185	600	182	1.3
18		3	118	22	190	600	170	1.5
19		4	119	21	189	600	178	1.4
20	7	1	118	21	209	600	177	1.4

21		2	119	21	185	600	170	1.5
22		3	119	20	185	600	169	1.4
23		4	118	21	189	600	166	1.5
24	8	1	119	22	185	600	160	1.6
25		2	120	22	189	600	175	1.5
26		3	120	22	185	600	170	1.6
						AVG	167	1.52
							T-Speed	H/Input
							mm/min	KJ/mm

3.4.5.4 DATA SHEET Test Coupon Preparation 12 Ni 22 Cr

Sr. No	Layer	Pass	Current	Voltage	Time	Length	T-Speed	H/Input
	No	No	Amp	Volts	Sec	mm	mm/min	KJ/mm
1	1	1	110	19	238	500	155	1.3
2		2	110	18	231	500	152	1.3
3	2	1	105	18	258	500	145	1.3
4		2	110	20	246	500	151	1.5
5	3	1	115	20	185	500	161	1.4
6		2	110	19	185	500	162	1.3
7		3	105	20	190	500	166	1.3
8	4	1	110	18	189	500	168	1.2
9		2	117	19	209	500	165	1.3
10		3	115	19	185	500	168	1.3
11		4	115	18	185	500	170	1.2
12	5	1	119	19	189	500	169	1.3
13		2	110	20	185	500	177	1.2
14		3	119	21	189	500	165	1.5
15		4	119	20	185	500	166	1.4
16	6	1	110	22	185	500	182	1.3
17		2	119	20	185	500	182	1.3
18		3	118	22	190	500	170	1.5
19		4	119	21	189	500	178	1.4
20	7	1	110	21	209	500	177	1.3

21		2	119	21	185	500	170	1.5
22		3	115	20	185	500	169	1.4
23		4	118	21	189	500	166	1.5
24	8	1	110	22	185	500	160	1.5
25		2	110	22	189	500	175	1.4
26		3	110	22	185	500	170	1.4
						AVG	167	1.36
							T-Speed	H/Input
							mm/min	KJ/mm

3.5 Test Plan for Weld Coupon

3.5.1 Test Plan for Weld Coupon Prepared by E 2209-16 (9 Ni22Cr)

Coupon ID. : E2209(9 Ni22Cr)

Material : DSS 2205

Welding Process : SMAW

Filler Wire : E2209-16 (GRINOX-2209)

Test Coupon : 700L * 300W * 25T mm

Heat treatment : NA

Welding Position : Flat

Type of testing : *RT (Radiography) : No indication – Acceptance criteria and as per below table 3.11*

PQR No. E2209(10Ni22Cr)							
Sr. No	**Heat Treatment**	**Types of Test**	**Orientation**	**Location**	**No. of Test Spec.**	**Test Temp. (°C)**	**Acceptance Criteria**
1	NA	Chemical Analysis	Transverse	T/2	1 No. per location	RT	C%,Mn%,Si%,S%,P%,Cr%,Ni%,Mo%,Cu%,N% : to report
		Tensile Test	Transverse	Full Thickness	2 Nos.	RT	UTS: To report % EL & % RA to be reported
		Hardness Test	Transverse	WELD,BM,HAZ	3 Readings per Location	RT	To report
		Tensile Test	Longitudinal	All weld	2 Nos.	RT	UTS, Y.S : To report % EL & % RA to be reported
		FN	Transverse	WM	1	RT	Ferrito scope ,volume fraction by microstructure examination
		Macro Examination	Transverse	On the cross section of weld	1 No.	RT	Free from defect
		Impact	Transverse	T/2	1 Set (3 Nos.)	-46°C	Value in J: to report, LE : to report

3.5.2 Test Plan for Weld Coupon Prepared by E 2209-16 (10Ni22Cr)

Coupon ID.	:	E2209(10Ni22Cr)
Material	:	DSS 2205
Welding Process	:	SMAW
Filler Wire	:	E2209-16 (GRINOX-2209)
Test Coupon	:	700L * 300W * 25T mm
Heat treatment	:	NA
Welding Position	:	Flat
Type of testing	:	*RT (Radiography) : No indication – Acceptance criteria and as per below table 3.12*

PQR No. E2209(10Ni22Cr)							
Sr. No	**Heat Treatment**	**Types of Test**	**Orientation**	**Location**	**No. of Test Spec.**	**Test Temp. (°C)**	**Acceptance Criteria**
1	NA	Chemical Analysis	Transverse	T/2	1 No. per location	RT	C%,Mn%,Si%,S%,P%,Cr%,Ni%,Mo%,Cu%,N% : to report
		Tensile Test	Transverse	Full Thickness	2 Nos.	RT	UTS: To report % EL & % RA to be reported
		Hardness Test	Transverse	WELD,BM,HAZ	3 Readings per Location	RT	To report
		Tensile Test	Longitudinal	All weld	2 Nos.	RT	UTS, Y.S : To report % EL & % RA to be reported
		FN	Transverse	WM	1	RT	Ferrito scope ,volume fraction by microstructure examination
		Macro Examination	Transverse	On the cross section of weld	1 No.	RT	Free from defect
		Impact	Transverse	T/2	1 Set (3 Nos.)	-46°C	Value in J: to report, LE : to report

3.5.3 Test Plan for Weld Coupon Prepared by E 2209-16 (11Ni22Cr)

Coupon ID. : E2209(11Ni22Cr)

Material : DSS 2205

Welding Process : SMAW

Filler Wire : E2209-16 (GRINOX-2209)

Test Coupon : 500L * 300W * 25T mm & 200L * 300W * 25T mm

Heat treatment : NA

Welding Position : Flat

Type of testing : *RT (Radiography) : No indication – Acceptance criteria and as per below table 3.13*

PQR No. E2209(11Ni22Cr)							
Sr. No	**Heat Treatment**	**Types of Test**	**Orientation**	**Location**	**No. of Test Spec.**	**Test Temp. (°C)**	**Acceptance Criteria**
1	NA	Chemical Analysis	Transverse	T/2	1 No. per location	RT	C%,Mn%,Si%,S%,P%,Cr%,Ni%,Mo%,Cu%,N% : to report
		Tensile Test	Transverse	Full Thickness	2 Nos.	RT	UTS: To report % EL & % RA to be reported
		Hardness Test	Transverse	WELD,BM,HAZ	3 Readings per Location	RT	To report
		Tensile Test	Longitudinal	All weld	2 Nos.	RT	UTS, Y.S : To report % EL & % RA to be reported
		FN	Transverse	WM	1	RT	Ferrito scope ,volume fraction by microstructure examination
		Macro Examination	Transverse	On the cross section of weld	1 No.	RT	Free from defect
		Impact	Transverse	T/2	1 Set (3 Nos.)	-46°C	Value in J: to report, LE : to report

3.5.4 Test Plan for Weld Coupon Prepared by E 2209-16 (12Ni22Cr)

Coupon ID.	:	E2209 (12Ni22Cr)
Material	:	DSS
Welding Process	:	SMAW
Filler Wire	:	E2209-16 (GRINOX-2209)
Test Coupon	:	500L * 300W * 25T mm
Heat treatment	:	NA
Welding Position	:	Flat
Type of testing	:	*RT (Radiography) : No indication – Acceptance criterai and as per below table 3.14*

PQR No. E2209(12Ni22Cr)

Sr. No	Heat Treatment	Types of Test	Orientation	Location	No. of Test Spec.	Test Temp. (°C)	Acceptance Criteria
1	NA	Chemical Analysis	Transverse	T/2	1 No. per location	RT	C%,Mn%,Si%,S%,P%,Cr%,Ni%,Mo%,Cu%, N% : to report
		Tensile Test	Transverse	Full Thickness	2 Nos.	RT	UTS: To report % EL & % RA to be reported
		Hardness Test	Transverse	WELD,BM,HAZ	3 Readings per Location	RT	To report
		Tensile Test	Longitudinal	All weld	2 Nos.	RT	UTS, Y.S : To report % EL & % RA to be reported
		FN	Transverse	WM	1	RT	Ferrite scope and point count method*
		Macro Examination	Transverse	On the cross section of weld	1 No.	RT	Free from defect

*Point count method could not be applied due to Non –continuous & discrete grains of austenite in ferrite matrix.

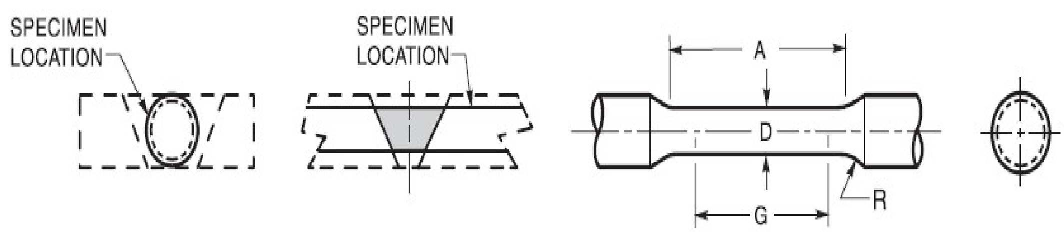
3.6 Mechanical Testings

3.6.1 All Weld Tensile Test

The objective of the all weld Tensile Test is to evaluate the welding consumables to assess mechanical properties obtained with it. Although, this result might vary from those obtained in actual production joints because of variances in welding procedure such as electrode diameter and size, deposition techniques, width of weave, welding position and material composition etc.

As per ISO 15792-1:2000(E) Welding consumables — Test methods — Part 1: Test methods for all-weld metal test specimens in steel, nickel and Nickel alloys [77]

Equivalent to ISO Standard for Welding Consumable test method AWS B4.0.[78]



Dimensions					
	Standard Specimen	Small-size specimens proportional to standard specimen			
Nominal Diameter	in (mm) 0.500 (13)	in (mm) 0.350 (9)	in (mm) 0.250 (6)	in (mm) 0.160 (4)	in (mm) 0.113 (3)
G. gage length	2.000 ± 0.005 (50 ± 0.127)	1.400 ± 0.005 (35 ± 0.127)	1.000 ± 0.005 (25 ± 0.127)	0.640 ± 0.005 (16 ± 0.127)	0.450 ± 0.005 (12 ± 0.127)
D. diameter	0.500 ± 0.010 (13 ± 0.25)	0.350 ± 0.007 (9 ± 0.18)	0.250 ± 0.005 (6 ± 0.127)	0.160 ± 0.003 (4 ± 0.08)	0.113 ± 0.002 (3 ± 0.05)
R. radius of fillet, min.	3/8 (10)	1/4 (6)	3/16 (5)	5/32 (4)	3/32 (2.4)
A. length of reduced section, min.	2-1/4 (60)	1-3/4 (44)	1-1/4 (32)	3/4 (20)	5/8 (15)

Fig. 3.16 Standard Dimension for preparation of all weld test specimen. Source[78]

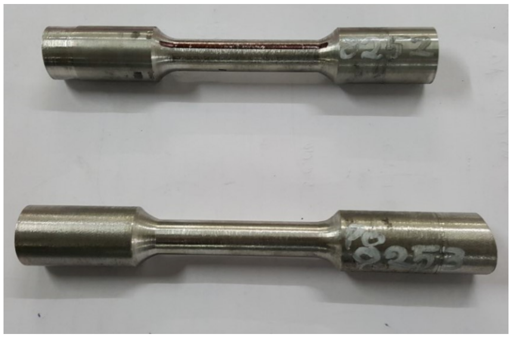
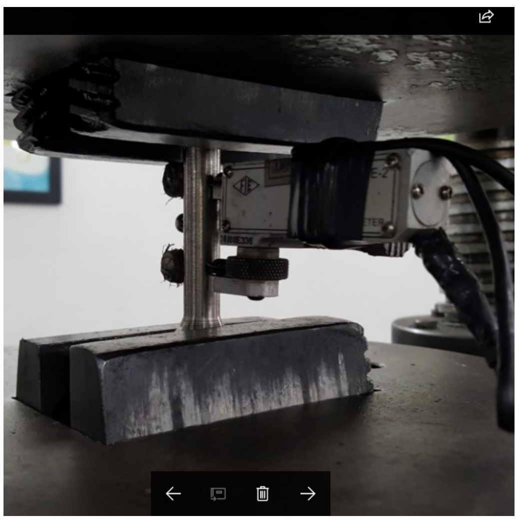


Fig. 3.17 Prepared specimen for All weld Test

Fig. 3.18 Initial gauge length measurement ~ 50 mm

Longitudinal sample as per ASME SEC IX : Ø12.5 X 60



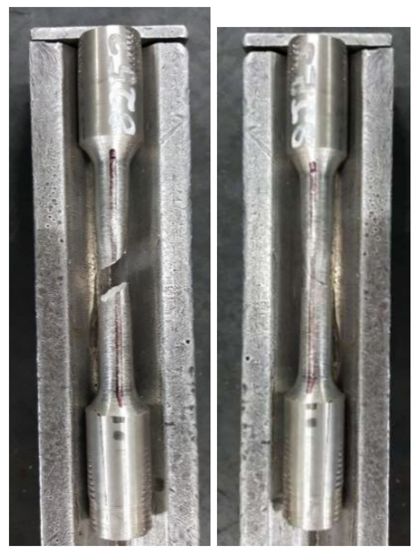


Fig. 3.19 Specimen under Tensile load of UTM Machine with 40 Ton capacity

Fig. 3.20 Fracture within Gauge length at 91740 N

Fig. 3.21 Final Diameter 7.32 mm measurement.

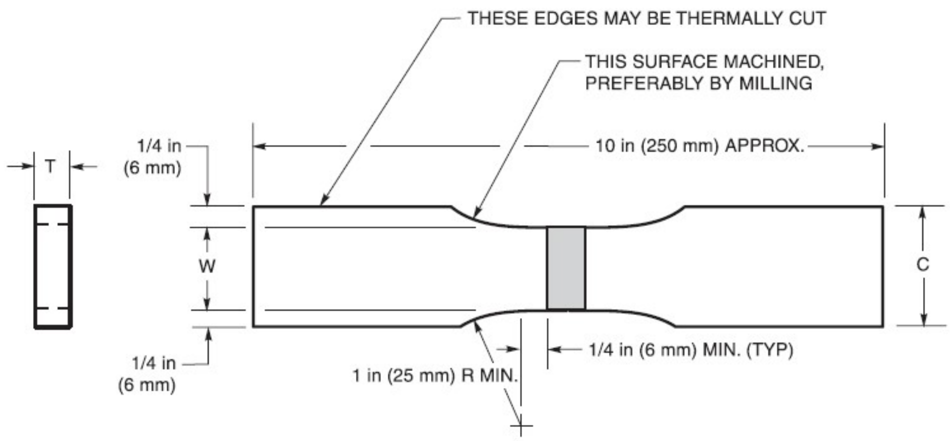
Fig. 3.22 Final Length Measurement : 64.87 mm

3.6.2 Transverse Rectangular Tension Test Specimen (Plate)[78]

The purpose of the Transverse Tensile Test is to determine the Mechanical strength of the welded joint. As fracture in Base metal indicates that Weld metal is stronger than base metal. Tension tests provide quantitative data of the mechanical properties like, Yield Strength at 0.2 % Proof Load, Ultimate Tensile strength, Percentage Elongation, reduction in area etc. that can be compared and analysed for use in the design and analysis of weld joint structures.

Fracture surfaces may also give information on the presence of some defects, discontinuities whose adverse effect may result in incomplete fusion, incomplete joint penetration, porosity, inclusions, and cracking.

Transverse Tensile Test as was prepared as per ASME SEC IX Ed 2015 as shown in figure 3.23 below. The purpose of this specimen was to evaluate the yield strength, Tensile strength of the weld joint in transverse directions.



T	W
<1 in (25 mm)	1.50 in ± 0.01 in (38 mm ± 0.25 mm)
≥1 in (25 mm)	1.00 in ± 0.01 in (25 mm ± 0.25 mm)

Fig 3.23 (a). Source Standard Methods for Mechanical Testing of Welds AWS B4.0:2007ANSI, May 2, 2007,7th Edition, Supersedes ANSI/AWS B4.0-98 Pg No. 13.

Fig 3.23. (b) Preparation of Test Specimen for Transverse Tensile Testing as per AWS B4.0 [78]

Fig 3.24 Transverse Tensile Specimen before fracture

Fig 3.25 Specimen under Tensile load Press capacity 60 Ton Capacity

Fig 3.26 Transverse Tensile Specimen After fracture Dimension

Fig 3.27 Fractured specimen shows fracture from the P.M.

3.7 Micro hardness test [78,83,84]

The Micro hardness of weld, HAZ and base metal was checked to verify sufficient ductility of weld as per ASTM E-384, EN ISO 9015-1:2011 using Vickers hardness tester, all weld metal test coupons. Specimens were measured for micro hardness at different 12 locations with 500 gmf load according to the prescribed method in the standard

Acceptance criteria

The harness shall no exceed 290 VHN for DSS.

Instrument Utilized : Vickers micro Hardness tester

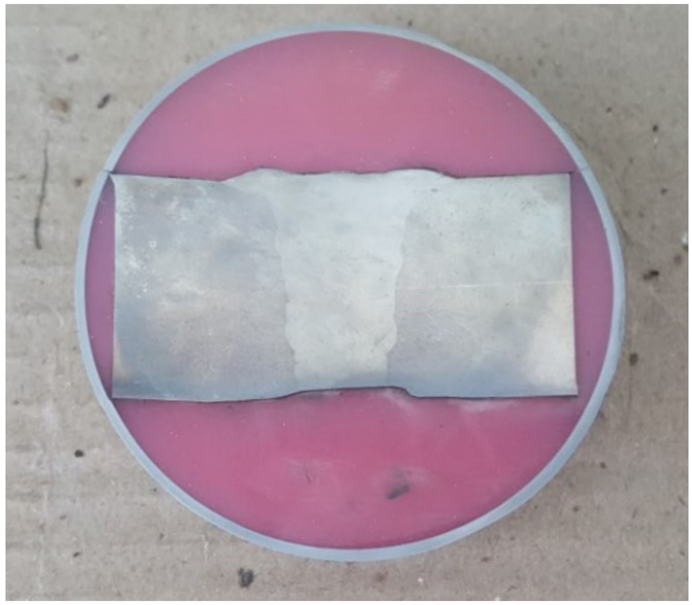


Fig 3.27 (a) Specimen for Micro hardness measurement

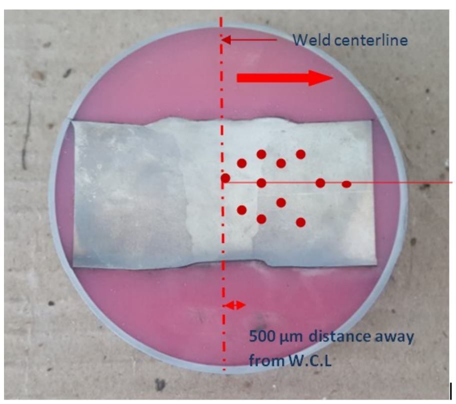


Fig 3.28 micro hardness at 12 locations away from weld centre line

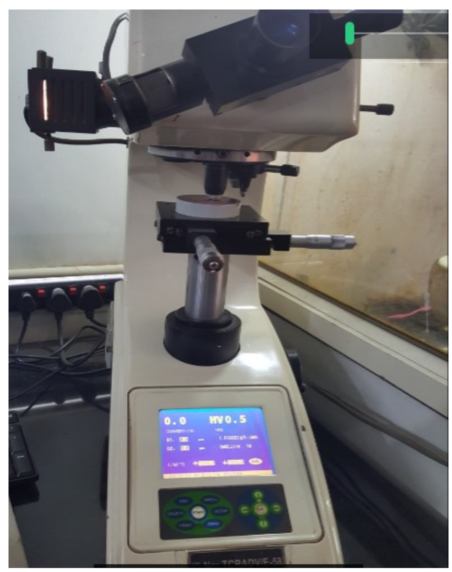


Fig 3.29 Specimen being measured for micro hardness at different 12 locations with 500 gmf load according to AS PER E-384, EN ISO 9015-1:2011

3.8 Macro examinations[95]

It is a qualitative visual examination to evaluate a weld fusion, penetration and probable defects.

Macrostructure Examination

Instrument utilized: Digital Camera/Stereo Microscope

Test Method: ASTM-E-381-01

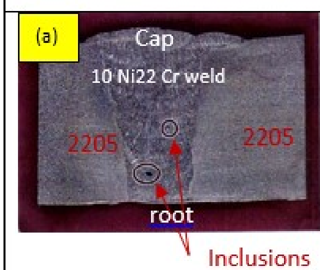
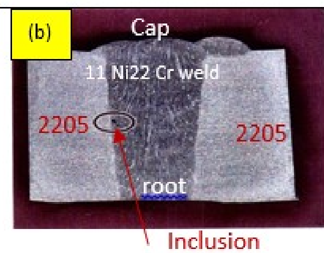
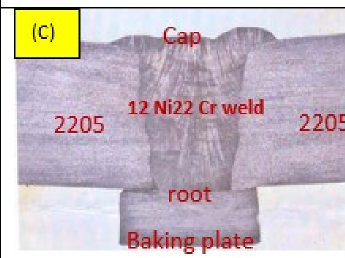
<table>
<tr><td>Weld Coupon prepared by 10NiCr22</td><td>Weld Coupon prepared by 11NiCr22</td><td>Weld Coupon prepared by 12NiCr22</td></tr>
<tr><td>
</td><td>
</td><td>
</td></tr>
<tr><td>Macrostructure shows good fusion however inclusion is observed.</td><td>Macrostructure shows good fusion however inclusion is observed</td><td>Weld joint sample etched with 50% HCL Macrostructure shows good fusion without any significant defect</td></tr>
</table>

Fig 3.30. Macro examination of Weld Specimens

Macro etching will show: (1) variations in structure such as grain size, dendrites, and columnar structure; (2) variations in chemical composition such as segregation, coring, and banding; and, (3) the presence of discontinuities such as laps, seams, cracks, porosity, bursts, pipe and flakes

One face of cross section of three specimens were made smoothed and etched with suitable enchants, most common for macro etching iron and steel is a 1:1 mixture, by volume, of concentrated hydrochloric acid (HCl) and water also known as muriatic acid.

Acceptance criteria

Visual examination of the cross sections should have complete fusion and free from any cracks, Slag inclusions, cracks or other weld defects.

3.9 Metallography [80]

Metallography of the welded duplex stainless steel UNS S 2205 is carried out for the analysis of Austenite Ferrite volume fraction measurement in the microstructure and to check whether the intermetallic phases are present in to the weld. Polishing was

Carried out in successive stages starting from 120 grit, 240 grit, 320 grit, 400 grit to 600 grit finish using emery papers, followed by lapping with 6 µm and 1µm size diamond pastes.

Subsequently, the polished area was electrolytic etched with 10% Oxalic acid solution or 20% NaOH to reveal the microstructures. Microstructural observation were carried out as per ASTM E 562-2011 with Optical Microscope at 400X magnification (Magnification 50X-1000X).

3.10 Chemical Analysis [79]

Chemical analysis of the weld joint sample is carried out by Optical Emission Spectrometer Method As per ASTM E-1086-08, IS 9879-98 with Spectramax X Instrument at Weld Metal zone at T/2 location with 02 spots.

Total 10 elements as percentage Wt. Carbon, Sulphur, Phosphorous, Mangenese, Silicon, Chromium, Nickel, Molybdenum, Copper and Nitrogen is revealed.

3.11 Impact Test [78]

ASTM A 370 "Standard Test Methods and Definitions for Mechanical Testing of Steel Products" and ASTM E 23 "Standard Methods for Notched Bar

Impact Testing of Metallic Materials are the methods selected for fracture toughness testing".

Specimens was carefully removed from a weldment so that the results of the test are representative of the performance of the welded joint.

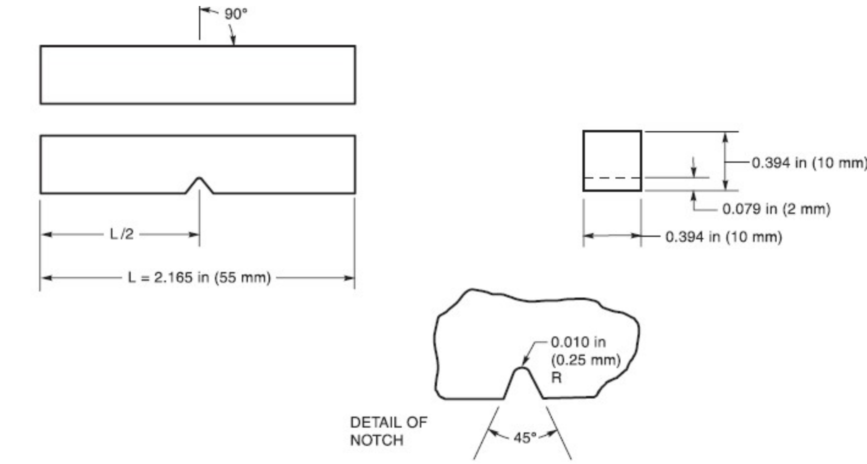
Fracture toughness testing provides a measure of resistance to unstable crack extension (i.e., fracture initiation), ductile tearing, or both. [ASTM A 370]

TEST: V Notch Charpy Impact Test

TEST METHOD: ASTM A 370-14

Sample Size (mm) 10 X 10 X 55 mm

Test Temperature (°C)   -46



NOTE—Dimensional Tolerances shall be as follows:

Notch length to edge	90° ± 2°
Adjacent sides shall be at	90° ± 10 minutes
Cross section dimensions	±0.003 in (0.076 mm)
Length of specimen (*L*)	+0, −0.100 in (+0, −2.5 mm)
Centering of notch (*L*/2)	±0.039 in (1 mm)
Angle of notch	±1°
Radius of notch	±0.001 in (0.025 mm)
Notch depth	±0.001 in (0.025 mm)
Finish requirements	63 microinches (1.5 micrometers) R_a on notched surface and opposite face; 125 microinches (3 micrometers) R_a on other two surfaces

Fig 3.31 Standard dimension for the CVN Specimen. Source[84]

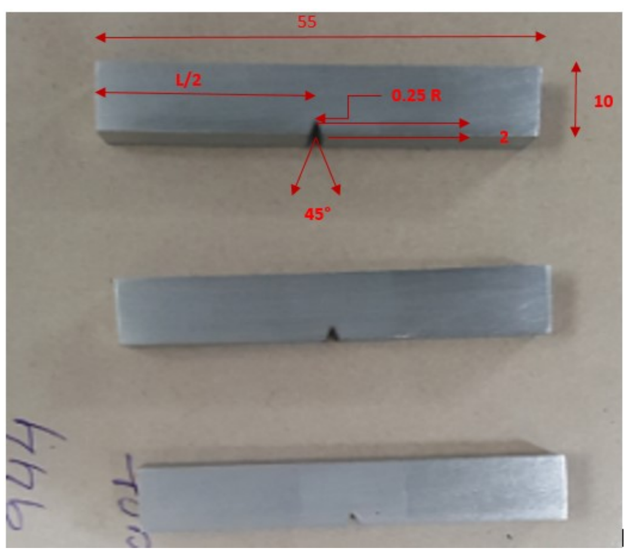


Fig 3.32 One Set (3 specimens) for V Notch Charpy Impact Test as per ASTM A 370-14

Fig 3.33 Test samples set (3 specimens)

Fig 3.34 Test temp -46 °C maintained by Liquid N2, Temperatures monitored by thermocouple

Fig 3.35 Impact Test Machine Capacity 350 Joule

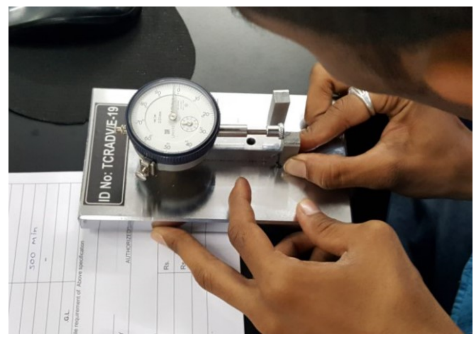


Fig 3.36 Measurement of Lateral Expansion by dial gauge.

3.12 Ferrite measurement [80]

3.12.1 Volume Fraction by Systematic Manual Point Count (ASTM 562 practice E)

Measurement of ferrite is done with the Point count method and with Ferritscope.

Metallographic Point Counting ASTM E562 is the "Standard Practice for Determining Volume Fraction by Systematic Manual Point Count." This specification may be applied to any micro constituent or phase, which is metallographically identifiable.

The principles governing this method are clearly defined in the specification. A two-dimensional metallographic sample is prepared and examined at an appropriate magnification. A grid is then superimposed over the image and the operator counts the number of points, which fall within the desired phase or micro constituent. Statistical analysis reveals the fraction of points, which fall within the desired phase, and the volume fraction is then calculated. When correctly implemented, this technique is an excellent method for determining the volume fraction of a desired phase or micro constituent. However, accuracy is often influenced by many factors, including the following:

Homogeneity
Quality of Sample Preparation
Grid Density
Magnification of the Substrate
Operator interpretation of microstructure.

Preparation includes metallographic polishing to a 0.05 micron finish and the application of a suitable etching technique. Etching techniques are modified to a specific micro constituent. Additionally, this technique is destructive in nature, requiring that a sample be extracted from the component or substrate. It was also limited to the number of fields examined and the location of the removed sample.

3.12.2 Magnetic permeability (e.g.: Feritscope)[96, 97]

The Fischer Feritscope was developed as a hand-held device, which utilized magnetic permeability as a method to assess ferrite content. As depicted the Feritscope® was designed to be portable and provide the operator with a user-friendly interface, which readily provides ferrite content on the ferrite number as well as ferrite percentage.

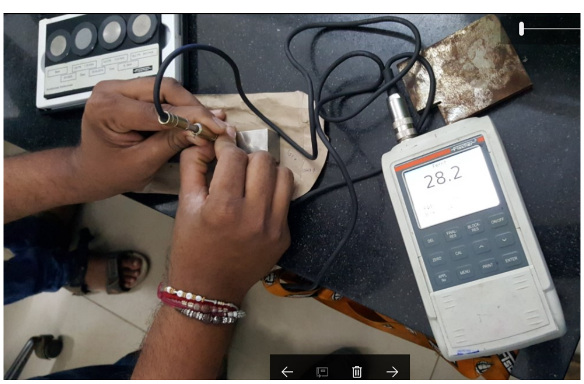


Fig 3.37 Instrument utilized was Fischer Ferritscope® Germany Make: Fischer 2531

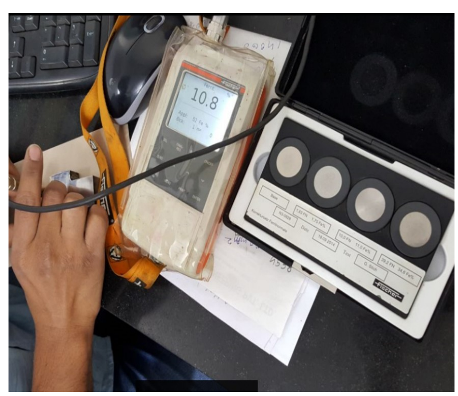


Fig 3.38 Calibrated Fischer Ferritscope® Germany Make: Fischer 2531

3.12.3 Ferrite Measurement by WRC 1992 diagram[1]

Ferrite prediction and measurement is very important in duplex stainless steel. In DSS ferrite measurement and prediction can be done by WRC 1992 diagram.

Schaeffler and WRC constitution diagrams introduced a non-destructive method to relate alloy composition to the amount of ferrite present in an alloy

There are certain useful diagram like Schaeffler, WRC-Delong diagrams and WRC 1992 diagram.

WRC-1992 diagram is now latest and more accurate for higher alloys and some special grades like Manganese-austenitic or duplex, austenitic-ferritic alloys.

Despite long use, the Schaeffler Diagram is now obsolete because it does not take into account effect of Nitrogen as Austenite former and because it has failed to provide consistent result among several measures[1]

These limitations associated with the Schaeffler Diagram is taken care of in the development of 1973 WRC-DeLong Diagram, which can be used to predict ferrite level. The main differences are that the DeLong Diagram includes nitrogen (N) in the Ni equivalent (% Ni + 30 x % C x 30x % N + 0.5 x % Mn) and shows Ferrite Numbers in addition to "percent ferrite." Ferrite Numbers at low levels may approximate "percent ferrite."[1]

WRC-1992 Diagram has replaced the WRC-DeLong Diagram in the ASME

Code with publication of the 1994-95 Winter Addendum.[1]

Its Ni equivalent (% Ni + 35 x % C + 20 x % N + 0.25 Cu) and

Cr equivalent (% Cr + % Mo + 0.7 x % Cb) quite differ from those of Schaeffler and WRC-DeLong[1]

The various alloying elements are expressed in terms of nickel or chromium equivalents (i.e. elements which, like nickel, tend to form austenite and elements like chromium which tend to form ferrite).

By plotting the total values for the nickel and chromium equivalents on these diagrams, with help of filer metal and base metal composition, considering dilution of 15 % or 20 % from base metal and 70 % or 60 % contribution from filler metal, a point can be found that indicates the main phases present in the stainless steel in terms of percentage ferrite and ferrite number respectively.

The final weld metal composition, and hence properties, depend on the amount of dilution that occurs during welding.

Weld metal dilution is normally expressed as a percentage of the final weld metal composition, the effect depending on a number of factors such as the joint design, the type of welding technique adopted and the welding process.

With the manual metal arc process, dilution in the vicinity of ±25% can occur. This will obviously be high in the root pass and least in fill-in and cap passes where two or more runs per layer are used.

Suppose we want to weld UNS S32205 Duplex Steel (22 Cr, 5.5 Ni , 1.5 Mn, 3.3 Mo, 0.17 N and 0.03 C) with High Nickel (11.5-12.5 %wt) electrode (22.3 Cr; 12.50 Ni, 1.11 Mn, 3.2 Mo, 0.18 N and 0.017 C), and we assume 30% dilution (the base metal contributes total 30% of the union (15 % + 15 %) and the electrode the other 70%).

The composition of the resultant weld metal can be predicted as below.

The UNS S32205 plate is represented by point B (Cr equivalent 25.3%; Ni equivalent 9.95%) and the High Nickel (11.5-12.5 %wt) electrode by point A (Cr equivalent 25.53%; Ni equivalent 16.94%). Any resultant weld metal from this mixture of A and B will be on the line that joins them. As we have assumed 30 percent dilution, point C will give the resultant microstructure (i.e. FN 22).

					Synthetic
	Base	Base	Filler	Weld	Base
	Metal 2	Metal 1	Metal	Metal	Metal
C, %	0.03	0.03	0.017	0.021	0.030
Mn, %	1.50	1.50	1.11	1.227	1.500
Cr, %	22	22	22.33	22.231	22.000
Ni, %	5.5	5.5	12.75	10.575	5.500
Mo, %	3.3	3.3	3.2	3.230	3.300
Nb, %	0	0	0	0.000	0.000
Cu, %	0	0	0	0.000	0.000
N, %	0.17	0.17	0.18	0.177	0.170
% of Joint	15	15	70	100	
Creq	25.3	25.3	25.53	25.461	25.3
Nieq	9.95	9.95	16.945	14.8465	9.95
FN	85.3	85.3	13.0	22.5	

Fig 3.39 Considering dilution from BM 15% & 70% from FM

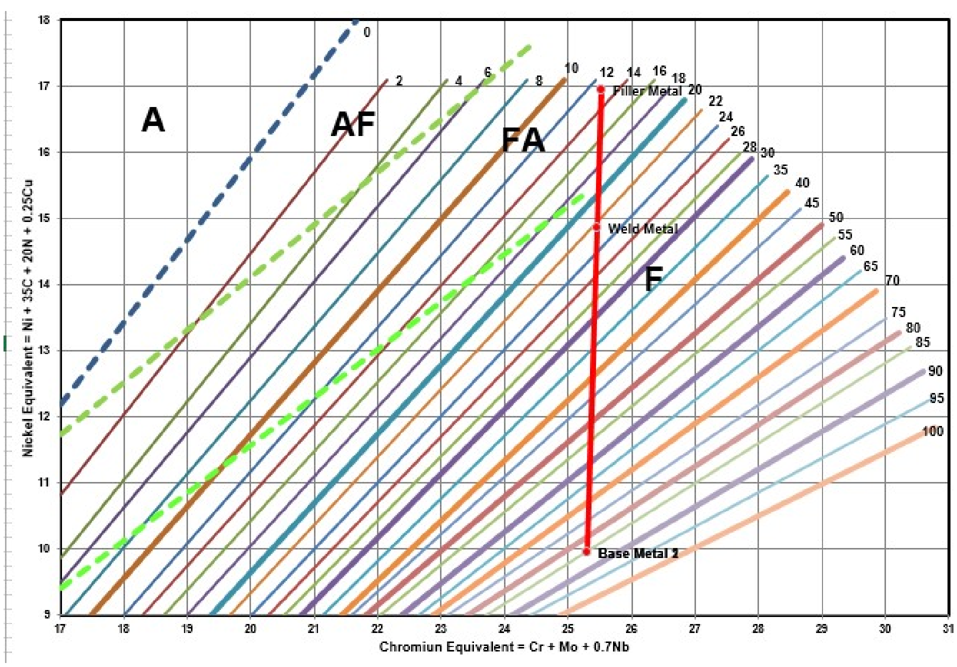


Fig 3.40 WRC -1992 DIAGRAM PREDICTING FN 22

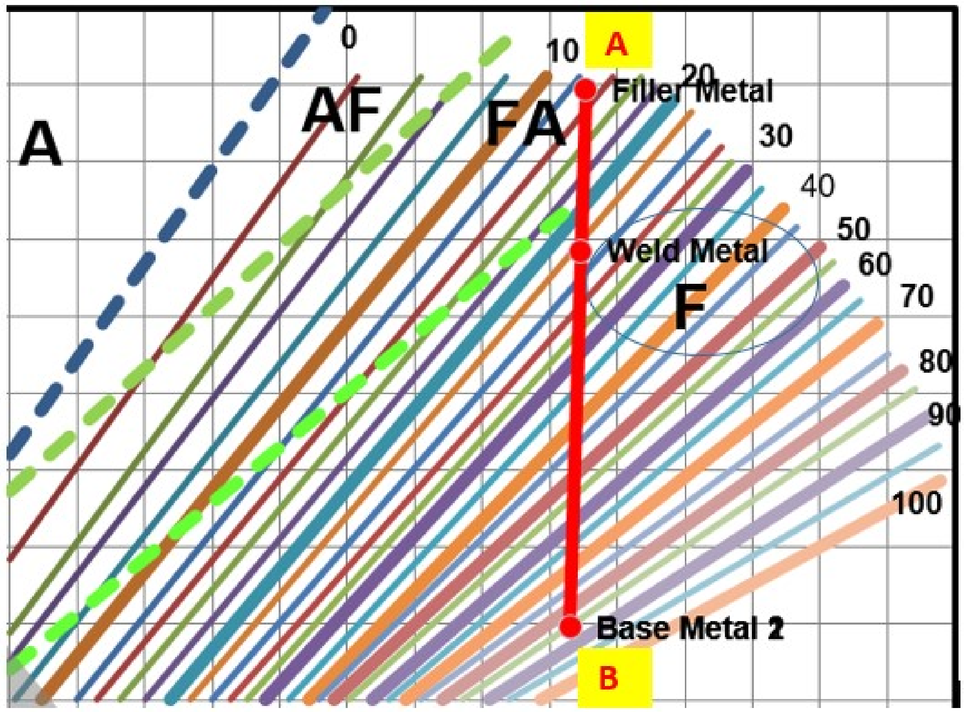


Fig 3.41 Calculation of Ferrite Number from WRC 1992 diagram Enlarged view indicating approx. FN 22

3.13 Corrosion Testing

3.13.1 Pitting corrosion (ASTM G 48 Practice A)[98]

Ordinary methods using weight losses are not recommended for pitting tests because the overall material loss may be very small even though severe pits have developed. Evaluating the number of pits, their depths or localization can give useful information about the mechanisms of initiation and growth, but is not adequate for evaluation of pitting resistance.

It is therefore preferable to simulate conditions, which result in pit initiation to provide a relevant means of assessing resistance to this type of corrosion attack. This is done by exposing the test material to an aggressive environment for a certain time. If corrosion pits are observed the material has failed.

Critical pitting temperature

"The minimum temperature to produce pitting attack at least 0.0025mm deep on the bold surface of the specimen, edge attack ignored."

Method

Pour 100mL of the ferric chloride test solution into the 1000ml test beaker. Transfer the test beaker to constant temperature bath and allow the test solution to come to the equilibrium temperature of interest. Place the specimen in a glass cradle and immerse in the test solution after it has reached desired temperature. Maintain test solution temperature throughout the test. For the test at 50 deg C weight loss shall be reported.

PREn: Cr + 3.3 Mo + 16 N

Acceptable

The acceptance criteria in this test shall be no pitting corrosion shall occur on the surface of the tested material at 25deg C and 30 deg C.

3.13.2 Chloride induced Stress Corrosion Test (Ch-CSSt)[3, 86]

The typical application of fabricated Duplex Stainless steels products is in offshore platform pipe work, pipelines transporting chloride bearing products or sour gas and process vessels for chloride environment demands high resistance to chloride induced Stress corrosion resistance & pitting corrosion resistance.

In the present research work, the standard "U-bend" stressed welded joint test sample for evaluating the stress corrosion crack susceptibility in chloride containing environment is extracted from the same batch of heat of weldments employed for carrying out various mechanical tests. The idea is to correlate the susceptibility to Chloride induced stress corrosion cracking with the Yield strength of the weldment obtained while Ferrite content is being varied in the Weld metal & Heat affected Zone and to derive the relationship among metallurgical feature-mechanical & corrosion behaviour by interpreting and confirming the test results.

To meet the objectives of the research program, ASTM G 123 – 00 (Reapproved 2005) Standard Test Method for Evaluating Stress-Corrosion Cracking of Stainless

Alloys with Different Nickel Content in Boiling Acidified Sodium Chloride Solution is used.

Some standard Duplex stainless steel manufacturer's data sheets[87,88] & NACE data[75, 76] suggests that it is resistance to stress corrosion crack in boiling sodium chloride solution but not in magnesium chloride solution. So ASTM Standard G 36 Practice for Evaluating Stress-Corrosion-Cracking Resistance of Metals and Alloys in a Boiling Magnesium Chloride Solution is not used in the present work. [91].

This test program uses boiling sodium chloride solution (25 % (by mass) sodium chloride acidified to pH 1.5 with phosphoric acid to evaluate wrought stainless steels, including duplex (ferrite-austenite) stainless and an alloy with up to about 33 % nickel. It in the cast or welded Conditions. [86]

The summary of test specimen, test procedure, and solution and evaluation criteria is as under:

3.14.1 Typical: U-Bend" Test Specimens

The methods of fabricating U-bend specimens are also provided in practice G 30 but with a restriction in dimensions in such a way that sufficiently thick Specimen (that shouldn't rupture & crack blast under tightened condition) should pass through a 45/50 Ground-glass joint 1000-mL Erlenmeyer type flask. The legs of the U-bends are sufficiently tightened with the bolt, nut, and flat washer of a material that is resistant to corrosion in the given test solution environment [3].

3.14.2 Test solution:

The solution is prepared by adding 750 g H_2O (750mL) to 250 g NaCl, and adjusting to pH 1.5 with H_3PO_4. Varying quantities of solution may be prepared subsequently can be stored in suitable glassware. [3]

3.14.3 Test Procedure [3]

Extraction of standard size specimen having length transverse to the direction of the weld.

Press bending to 180 degree U bend shape on mechanical press thereby stressing the specimen with anti -corrosive material bolts, nuts, washer & examining at 20 X magnification for the cracks developed due to stressing.

Degreasing the specimen with halogen free solvent, rinsing and drying for long storage placing the specimen in desiccator with silica gel for moisture absorption.

Placing min four duplicate specimen in each of 3 duplicate flasks for comparison and evaluation purpose.

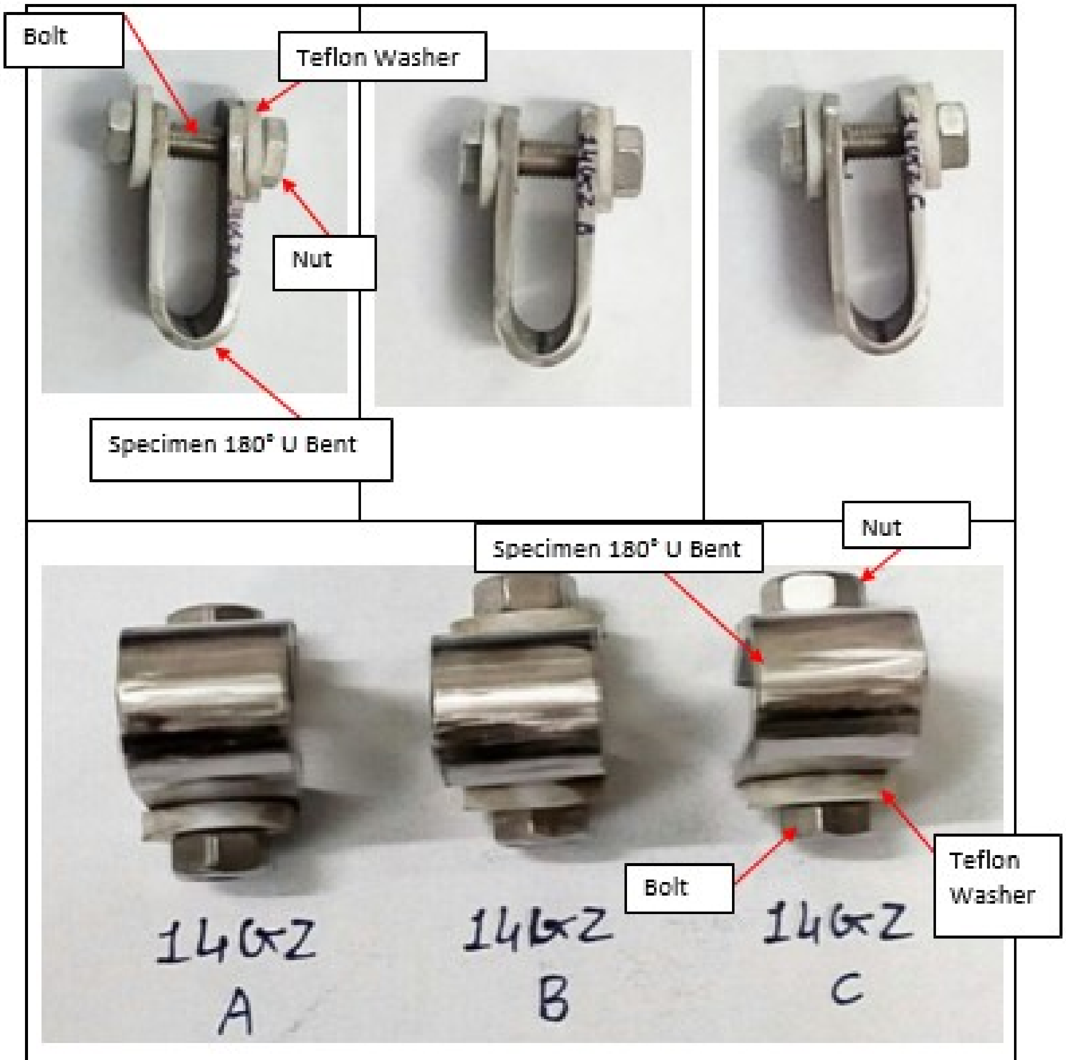
Adding 600 mL of 25 % NaCl solution, pH 1.5, to each flask so that solution volume to sample surface area ratio is 5:1 mL/cm 2 (33 mL/in.2).for each samples contained in the flask.

Placing the flasks on a hot plate and insert the condenser and begins boiling solution around 106 to 110°C.

Periodically remove the specimen after one week, examining the specimen under 20 X magnification for the cracks, test solution pH.

Changing the solution and repeat this process for schedule after 6 h, daily for one week, and weekly for six weeks up to 1000 hrs or (4-weeks). As per mentioned in ASTM G 123 standard.

Rinsing and drying specimen, examining under 20 X magnification for cracks appearance.



Set of 03 Specimens for SMAW E 2209-16 (10 Ni 22 Cr) press bend 180 ° "U" Bent samples (for producing stress) fastened at free ends with anti- corrosive materials bolts, Nuts and Teflon washers.

Fig. 3.42 Set of 03 specimens for SMAW E 2209-16 (10 Ni 22 Cr) press bend 180° "U" bent samples (for producing stress) fastened at ree ends with anti-corrosive materials bolts, Nuts and Teflon washers.

TEST SPECIMENS AS PER ASTM G 123 STANDARD TEST PROCEDURE

Fig. 3.43 Set of 03 Specimens for SMAW E 2209-16 (11 Ni 22 Cr) press bend 180 ° "U" Bent samples (for producing stress) fastened at free ends with anti- corrosive materials bolts, Nuts and Teflon washers.

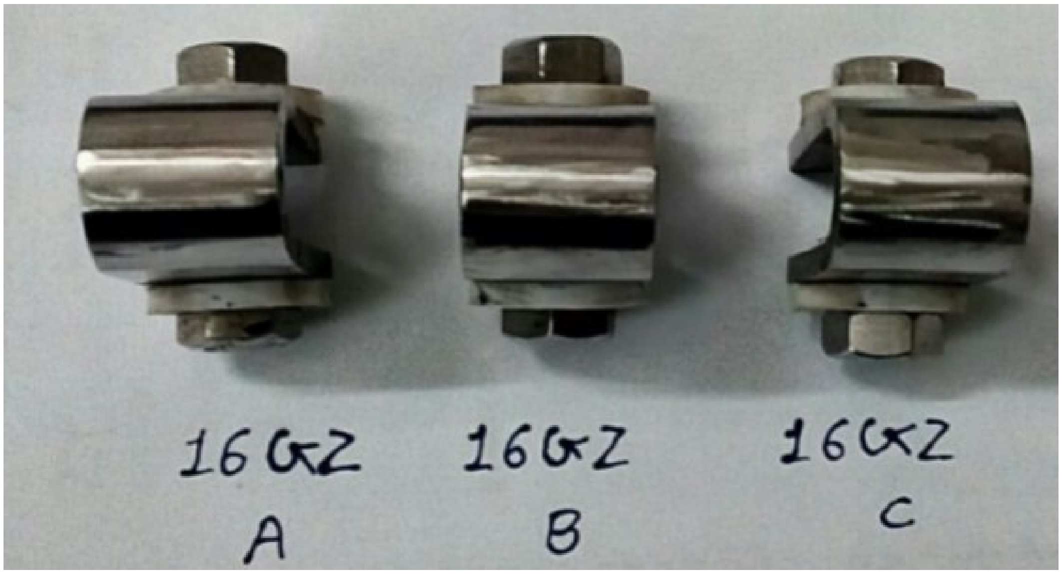


Fig. 3.44 Set of 03 Specimens for SMAW E 2209-16 (12 Ni 22 Cr) press bend 180 ° "U" Bent samples (for producing stress) fastened at free ends with anti-corrosive materials bolts, Nuts and Teflon washers.

ASTM G 123 Test Set up

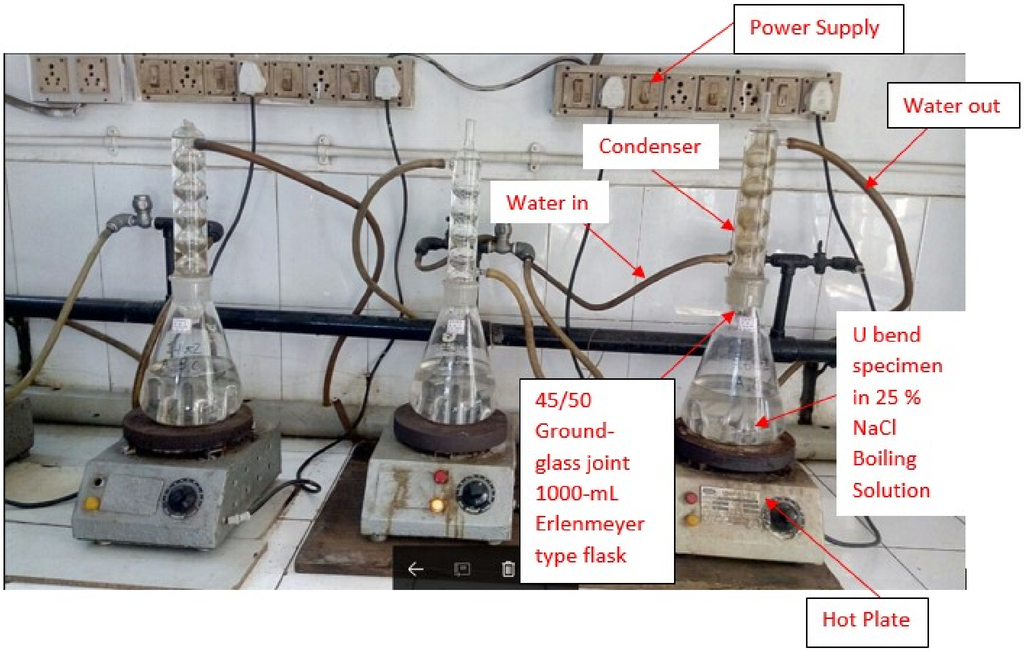


Fig. 3.45 Set of Specimen (03 Nos. Per Heat) immersed in boiling 25 % NaCl Solution (120-150 °C) in each flask on Hot Plate equipped with Condenser arrangement. Cycle time 1000 Hrs. (ASTM G123)