

Materials and Experimental Details

This chapter describes the details of the base metal (BM) and filler metals used in the welding experiment, their microstructure characteristics, and chemical composition. The details related to the variable and non-variable welding parameters used for the joining of the P92 and 304L steel is discussed in this chapter. The details related to the PWHT performed after the welding is also provided. The specimens prepared for the metallographic and mechanical test is also discussed at the end of this chapter.

3.1 Base metals

The nominal composition of the P92 and 304L steel used in present investigation is listed in Table 3.1. The heat treatment condition of the BM is: (1) P92: 1040 °C for 40 min (normalizing, air-cooled) and 760 °C for 2 h (tempering, air-cooled); (2) 304L: 1100 °C for 20 min (solution annealing, air-cooled). The normalizing produces a 100% martensitic microstructure. This martensitic microstructure is then tempered and air-cooled to soften the brittle and hard martensite produced after normalizing process. The 304L was supplied in solution annealed condition in the form of 10 mm thickness plates. The nickel-based (ERNiCrMo-3, ERNiFeCr-2 and ERNiCr-3) and ferritic-martensitic (Thermanit MTS 616) filler metal was used as a welding consumable. The chemical composition of the filler metal is given in Table 3.2. The microstructure of the P92 steel obtained using optical microscope and field emission scanning electron microscope (FESEM) is shown in Figure 3.1(a-d). The tempering heat treatment after normalizing produces the tempered martensite structure with prior austenite grains (PAGs) and sub-grains boundaries. The microstructure of the P92 steel mainly consists of prior austenite grain boundaries (PAGBs), lath/sub-grain boundaries, and precipitates ($M_{23}C_6$ and MX). These $M_{23}C_6$ and MX precipitates provide precipitation strengthening to the P92 steel. The PAG is further subdivided into the lath martensite packets, which consist of lath martensite blocks. The microstructure of the P92 steel contains tempered martensite and secondary phase particles ($M_{23}C_6$ and MX). The lath martensite strips decorated inside the PAGs are visible from the microstructure of the P92 BM. The precipitation strengthening by the MX and $M_{23}C_6$ type precipitate particles and solid solution strengthening by W and Mo elements provide strength to the P92 steel. The fine dispersion of the MX and $M_{23}C_6$ type precipitate particle inside the matrix and the solid solution of the W and Mo elements in the base metal matrix provide a barrier to the dislocation movement. The microstructure of the as-received 304L is mainly austenite with twins inside the polygonal austenite grains (Figure 3.2(a-c)).

Table 3.1 Base metal composition (%wt.)

Element (%wt.)	C	Mn	P	S	Si	Cr	W	Mo	V	Fe	Nb	Cu	Ni	Ti
P92	0.11	0.5 8	-	0.00 3	0.4 8	9.0 9	1.8 7	0.4 2	0.2 4	Re m.	0.0 5	0.0 3	0.3 0	0.0 02
304L	0.01 8	1.12	0.02 4	0.01 1	0.4 2	18.0 5	-	-	-	Re m.	-	-	8.0 6	-

Table 3.2 Filler metal composition (%wt.)

Element (%wt.)	ERNiCrMo-3 (Inconel 625)	ERNiFeCr-2 (Inconel 718)	ERNiCr-3 (Inconel 82)	Thermanit MTS 616 (P92 steel)
C	0.012	0.05	0.016	0.11
Mn	0.37	0.03	3.09	0.6
P	0.008	0.002	0.010	-
S	0.001	0.0007	0.010	-
Si	0.091	0.06	0.168	0.2
Cr	20.67	17.39	19.70	8.8
W	-	-	-	1.6
Mo	9.23	2.93	-	0.5
V	-	-	-	0.2
Fe	0.36	20.10	0.132	Rem.
Nb	3.96	5.2	2.45	0.05
N ₂	-	-	-	-
N	-	-	-	0.05
Ni	64.89	52.70	73.90	0.7
Ti	0.12	0.99	0.300	-
Al	0.08	0.4	0.08	-
B	-	-	-	-
Zr	-	-	-	-
Cu	0.01	0.02	0.010	-

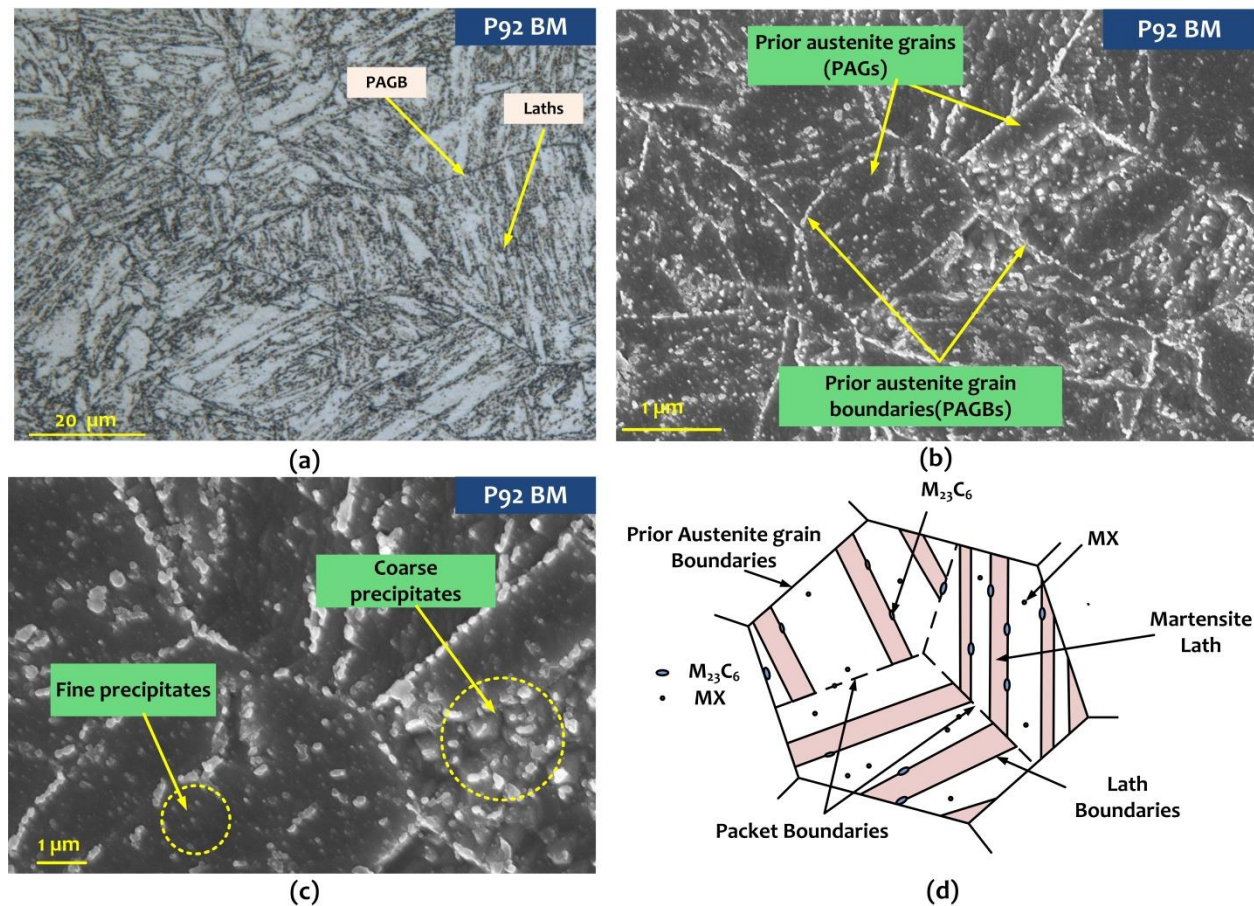


Figure 3.1: (a) Optical microstructure of the P92 BM (b, c) FESEM microstructure of the P92 BM at low and high magnification and (d) schematic diagram of microstructure of the P92 BM

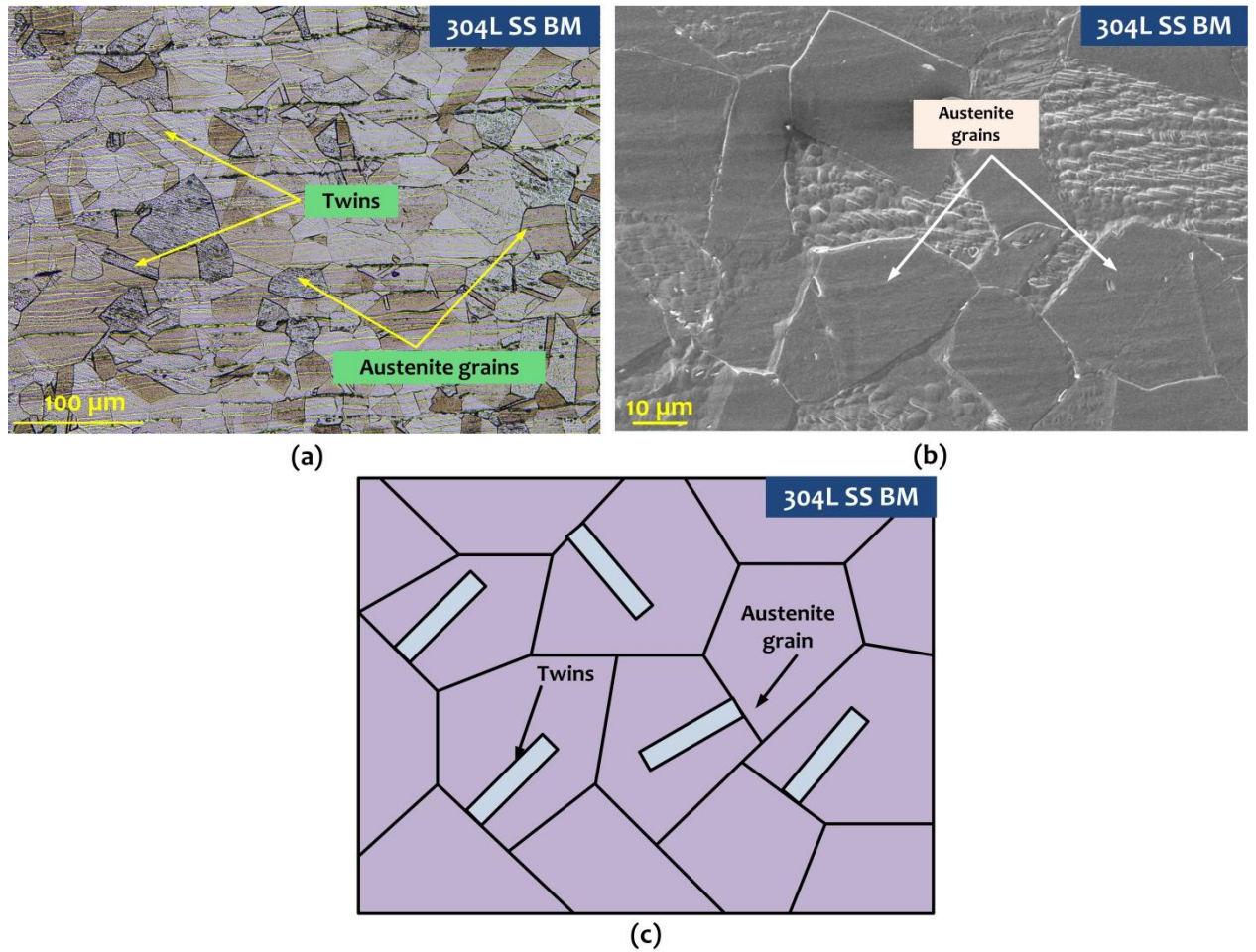


Figure 3.2: (a) Optical microstructure of the 304L BM (b) FESEM microstructure of the 304L BM and (c) schematic diagram of microstructure of the 304L BM

3.2 Groove geometry

For the welding experiments, the rectangular plates with dimensions $150 \times 55 \times 10 \text{ mm}^3$ were machined from P92 and 304L BM. The selection of the groove geometry depends on the thickness of the metal to be joined (Figure 3.3 and Table 3.3). The standard V-groove and narrow groove configuration was prepared on edges of both plates, as depicted in Figure 3.4(a, b) and Figure 3.4(c, d). The root gap and root height of 1.5 mm was maintained.

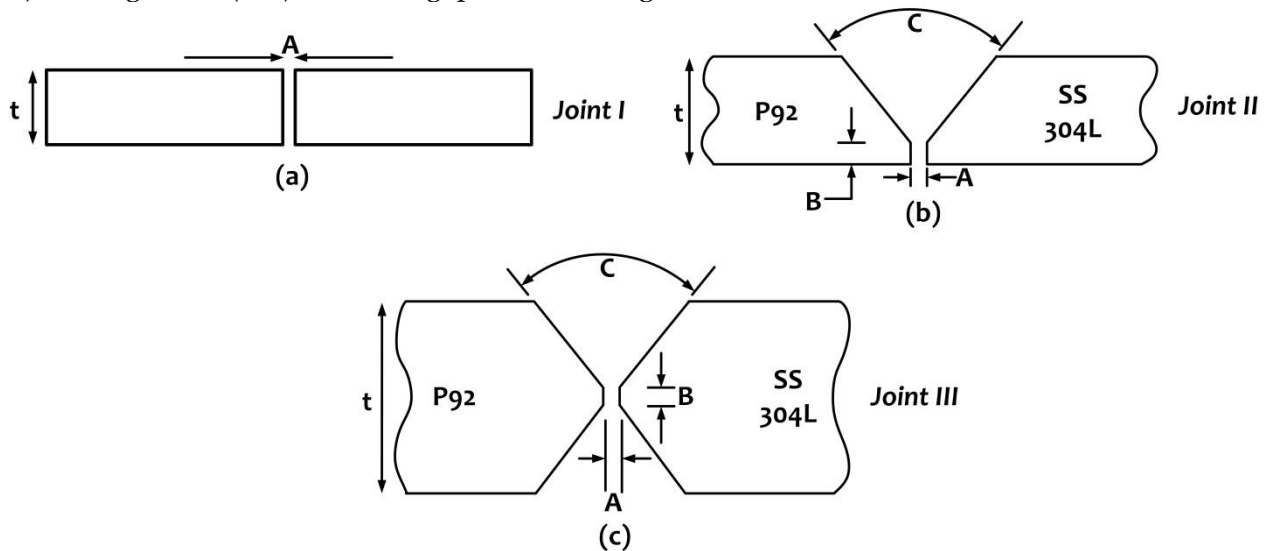


Figure 3.3: (a-c) Butt joint configuration for welding

Table 3.3 Groove geometry configuration

Material Thickness (t)	Preferred Joint Design	Root Opening (A)	Land Thickness (B)	Included Weld Angle (C)	Approx. Weight of Weld Metal Required
mm	-	mm	mm	degree	kg/m
1.6	I	0-1.6	N/A	None	0.03
2.4	I	0-2.4	N/A	None	0.06
3.2	I	0-3.2	N/A	None	0.09
6.3	II	1.6-3.2	(0.8-2.4)	60-75	0.45
9.5	II	0.8-4.0		60-75	0.89
12.7	II			60-75	1.41
12.7	III			60-75	0.89
15.9	II			60-75	2.08
15.9	III			60-75	1.22
19.1	II			60-75	2.83
19.1	III			60-75	1.79

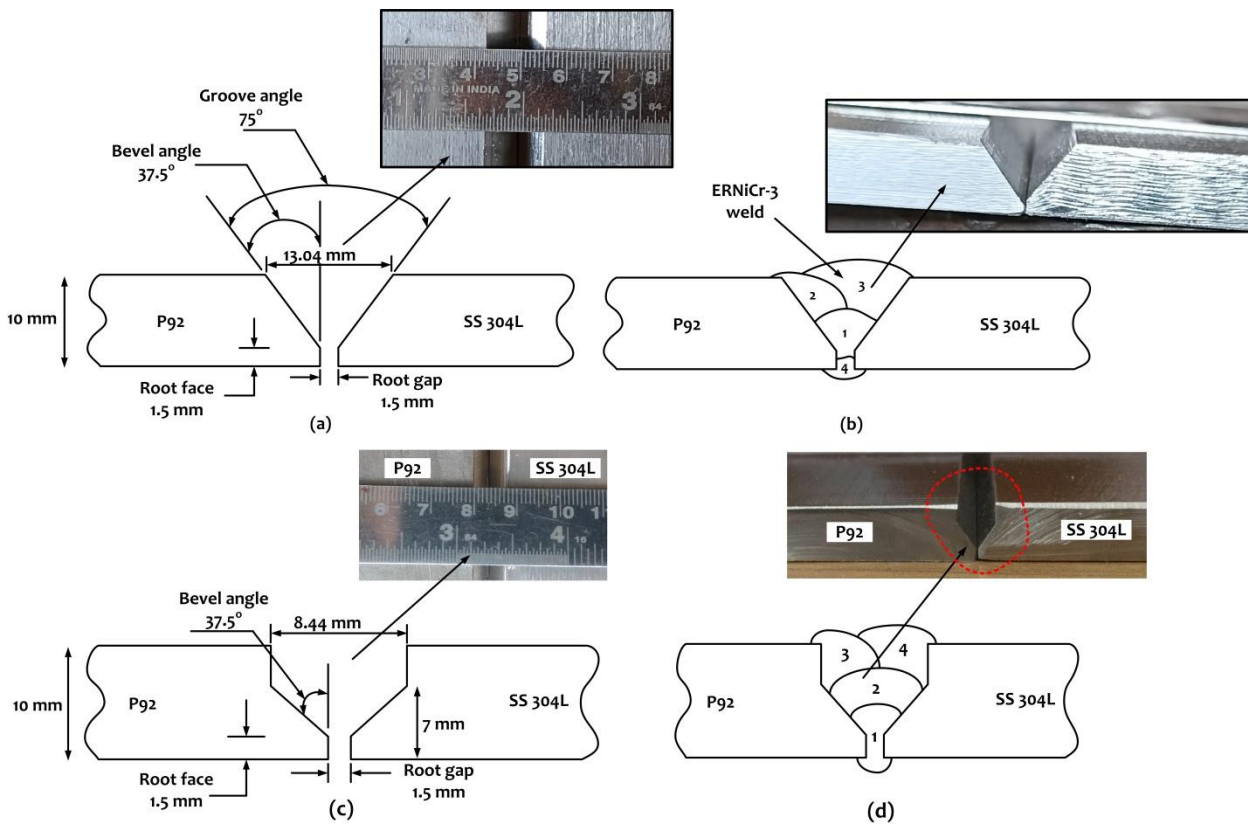


Figure 3.4: (a, b) Schematic diagram of V-groove geometry and (c, d) Schematic diagram of narrow groove geometry

3.3 Welding procedure

The manual gas tungsten arc welding (GTAW) machine (Make: Lincoln, Model: Precision TIG 225) was used for welding operation. The plates were cleaned with acetone before welding to remove the foreign impurities and get a defect-free weld. Both the plates to be welded were tack welded at three different locations to ensure the proper alignment during the welding, as shown in Figure 3.5(a). The tack weld prevents the misalignment and ensures the appropriate gap between the two plates to be welded. After tacking, the P92 plate was preheated up to the temperature of 200 °C to avoid cold cracking and to control the hydrogen diffusion into the molten pool, as shown in Figure 3.5(b). The preheating temperature of 200 °C and interpass temperature of 250 °C are recommended for P92 steel (Taylor, 2010). The temperature was monitored with the help of an infrared (IR) temperature gun. After preheating,

a welding pass was performed by a qualified welder, as shown in Figure 3.5(c). The top and rear view of the welded plate is shown in Figure 3.5(d, e). The welding was performed using direct current straight polarity (DCEN) to achieve through thickness-penetration. The pure argon (purity: 99.995 %) was used as a shielding gas, and thoriated tungsten electrode (EW-Th-2) of 3.2 mm diameter was used for GTAW. The shielding gas flow rate of 15 l/min was maintained during the welding. The non-variable welding parameters are listed in Table 3.4. The welding was completed in four passes. The welding current, arc voltage, average travel speed, and corresponding heat input for each pass considering 60% arc efficiency are listed in Table 3.5 to Table 3.8. The heat input for each welding pass was calculated by using Eq. (3.1) (Ye et al., 2015), considering arc efficiency as 60% for the GTAW welding process.

$$Q = \frac{\eta VI}{s} \quad (3.1)$$

Where “ η ” is the heat transfer efficiency, “ V ” is arc voltage in volts (V), “ I ” is welding current in amperes (A), and “ s ” is average travel speed in mm/s.

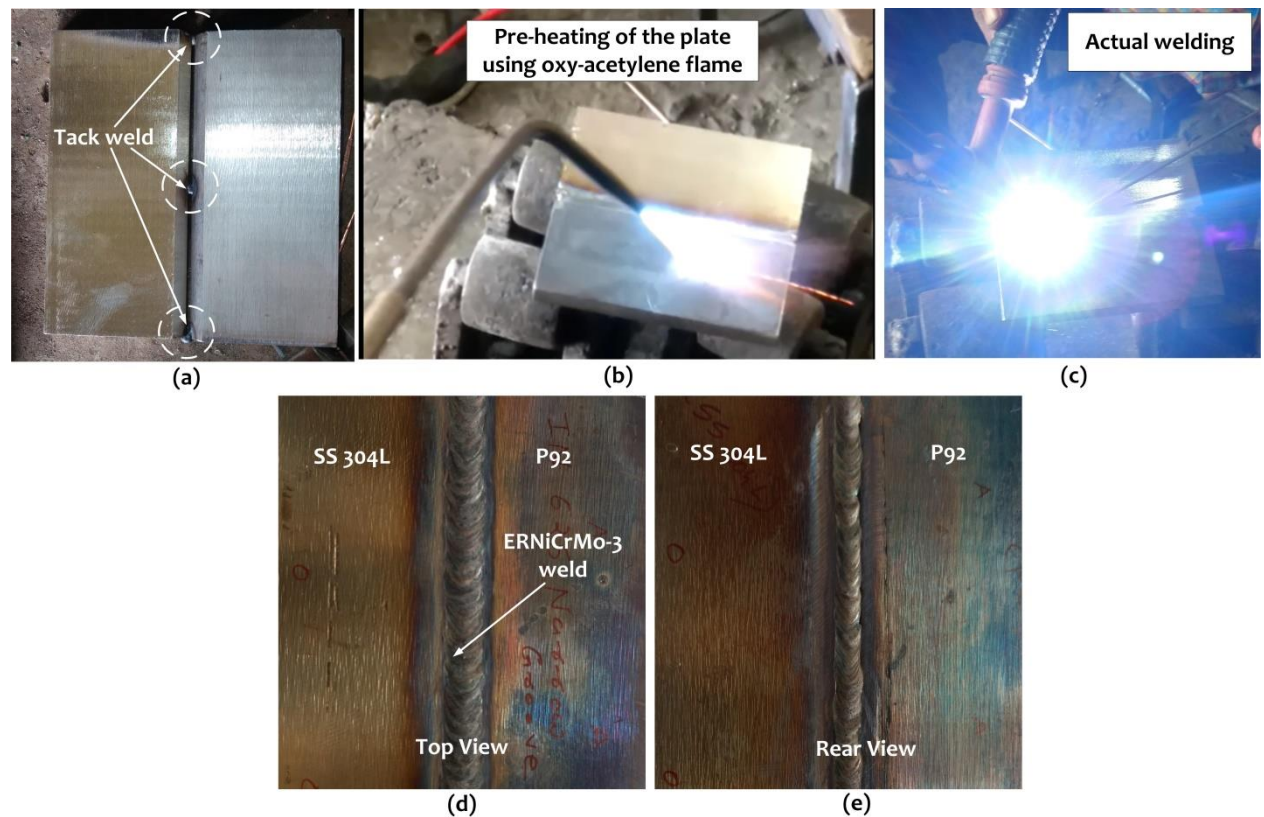


Figure 3.5: (a) Tack welding (b) preheating of the plate (c) GTAW welding using filler metal and (d, e) top and rear view of the welded plate

Table 3.4 Non-variable welding parameters

Electrode	EWTh-2 (2 % Thoriated)
Electrode diameter	3.2 mm
Electrode Tip Angle	60°
Polarity	DCEN
Arc Gap	2 mm
Shielding Gas	Argon
Shielding Gas Purity	99.995 %
Shielding Gas Flow Rate	15 l/min
Filler Wire Diameter	2.4 mm

Table 3.5 Welding parameters for P92-ERNiFeCr-2-304L DWJ

Table 3.5 Welding parameters for 192 ERNiCr2-304L DWJ					
Pass number	Welding Parameters			Average heat input per unit length per pass (kJ/mm)	Total heat input per unit length of the weld (kJ/mm)
	Welding current (A)	Arc voltage (V)	Average travel speed (mm/min)		
V-groove Design Parameters					
First pass	155	~16.7	~85	1.096	4.149
Second pass	145	~16.2	~80	1.057	
Third pass	145	~16.2	~80	1.057	
Fourth pass	145	~16.2	~90	0.939	
Narrow-groove Design Parameters					
First pass	155	~16.7	~98	0.951	3.621
Second pass	145	~16.2	~95	0.890	
Third pass	145	~16.2	~95	0.890	
Fourth pass	145	~16.2	~95	0.890	

Table 3.6 Welding parameters for P92-ERNiMoCr-3-304L DWJ

Pass number	Welding Parameters			Average heat input per unit length per pass (kJ/mm)	Total heat input per unit length of the weld (kJ/mm)
	Welding current (A)	Arc voltage (V)	Average travel Speed (mm/min)		
V-groove Design Parameters					
First pass	140	15.9	85	0.942	3.562
Second pass	130	15.5	80	0.907	
Third pass	130	15.5	80	0.907	
Fourth pass	130	15.5	90	0.806	
Narrow-groove Design Parameters					
First pass	140	15.9	98	0.818	3.11
Second pass	130	15.5	95	0.764	
Third pass	130	15.5	95	0.764	
Fourth pass	130	15.5	95	0.764	

Table 3.7 Welding parameters for P92-Thermanit MTS 616-304L DWJ

Table 3.7 Welding parameters for 1.52 thermally treated 5042 DWS					
Pass number	Welding Parameters			Average heat input per unit length per pass (kJ/mm)	Total heat input per unit length of the weld (kJ/mm)
	Welding current (A)	Arc voltage (V)	Average travel Speed (mm/min)		
V-groove Design Parameters					
First pass	130	15.4	85	0.847	3.158
Second pass	120	14.8	80	0.8	
Third pass	120	14.8	80	0.8	
Fourth pass	120	14.8	90	0.711	
Narrow-groove Design Parameters					

First pass	130	15.4	98	0.735	2.754
Second pass	120	14.8	95	0.673	
Third pass	120	14.8	95	0.673	
Fourth pass	120	14.8	95	0.673	

Table 3.8 Welding parameters for P92-ERNiCr-3-304L DWJ

Pass number	Welding Parameters			Average heat input per unit length per pass (kJ/mm)	Total heat input per unit length of the weld (kJ/mm)
	Welding current (A)	Arc voltage (V)	Average travel Speed (mm/min)		
V-groove Design Parameters					
First pass	135	15.8	85	0.90	3.37
Second pass	125	15.2	80	0.86	
Third pass	125	15.2	80	0.86	
Fourth pass	125	15.2	90	0.76	
Narrow-groove Design Parameters					
First pass	135	15.8	98	0.784	2.944
Second pass	125	15.2	95	0.72	
Third pass	125	15.2	95	0.72	
Fourth pass	125	15.2	95	0.72	

3.4 Experimental details of Buttering

The P92 and 304L steel plates were utilized for the experiment. The plates had a thickness of 12 mm and dimensions of 140×55 mm². To carry out the buttering process on the P92 steel plate, ERNiCr-3 filler metal was employed. The V-groove configuration for this study is illustrated in Figure 3.6(a, b). The root gap, root height, and groove angle were 2 mm, 1.5 mm, and 75°, respectively. The buttering on the P92 steel plate was conducted using ERNiCr-3 filler metal by the GTAW technique, with the specific conditions listed in Table 3.9. A butter layer thickness of 10-11 mm was chosen for the experiment (Figure 3.6(c)). The butter layer consists of 4 layers, each consisting of three welding passes performed adjacent to one another along the workpiece. On average, each layer of the buttering process adds 3 mm to the thickness of the butter layer. After buttering, the buttered plate underwent heat treatment at 760 °C for 90 min to alleviate thermal and mechanical residual stresses. After the heat-treatment, the buttered plate was machined at the edges to create a V groove (Figure 3.6(d)). The machining was performed to achieve a 7 mm thickness from the interface of the P92 steel to the ERNiCr-3 butter layer. After machining, both the buttered P92 plate and the 304L plate were clamped in the fixture, and welding was performed using the ERNiCr-3 filler with the GTAW process. The welding was completed in 7 passes. The weld parameters are listed in Table 3.10. The top and root region of the deposited metal is displayed in Figure 3.6(e, f).

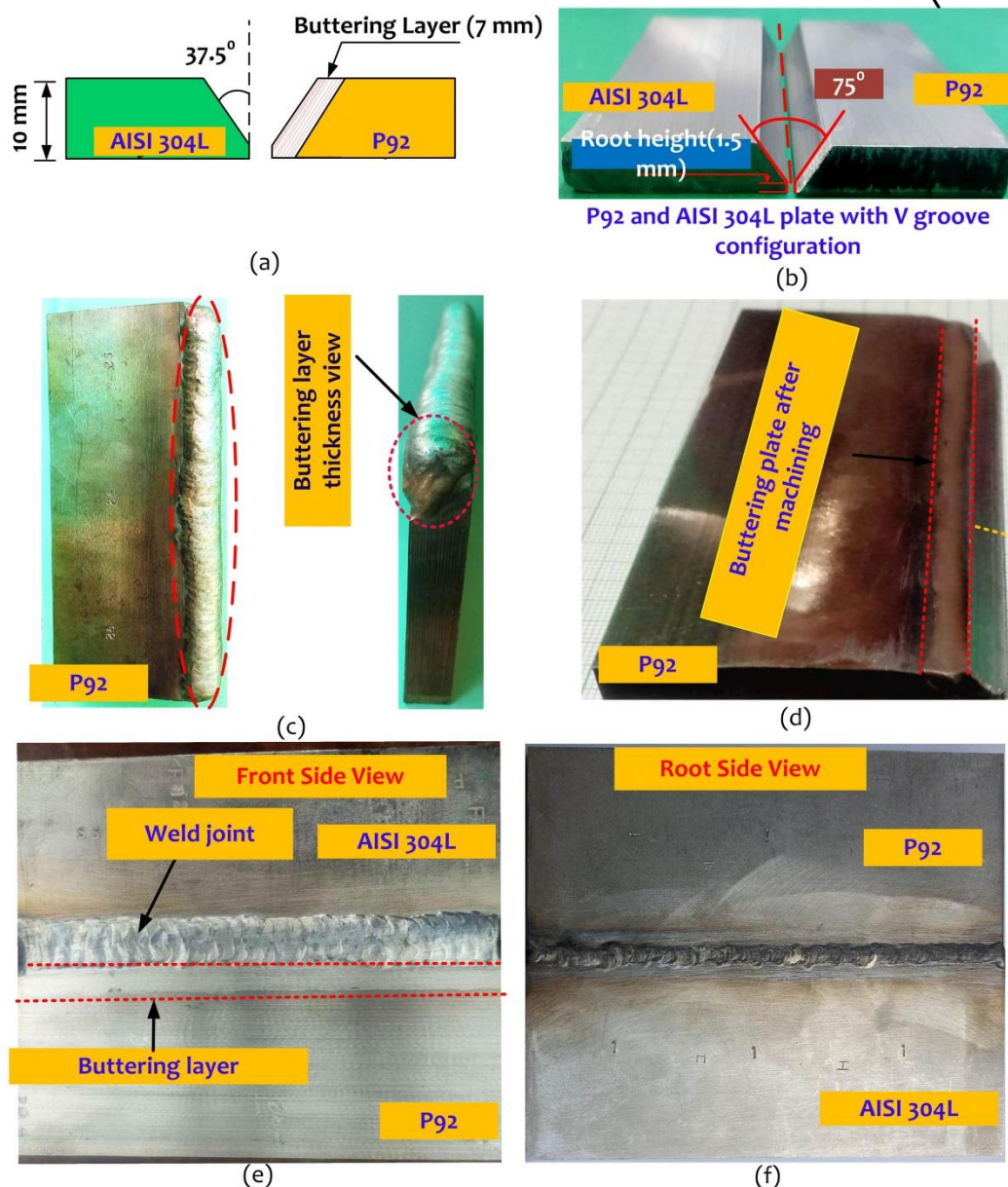


Figure 3.6: (a) Weld groove configuration for P92 and 304L weld joint (b) plate positioning after initial machining (c) plate after deposition of the buttering layer (d) machined surface after buttering and (e, f) welded plate with buttering layer with top and root view

Table 3.9 Parameters used for buttering of P92 plate

Parameter	Unit	Buttering
Welding process	-	GTAW
Welding position	-	1G (Flat)
Joint design	-	Butt Joint Single V groove 75°
Voltage	V	9-10.2
Current	A	105-122
Polarity	-	DCEN
Average welding speed	mm/min	75-120
Shielding gas	-	Argon (99.99%)
Shielding gas speed	L/min	12
Electrode	-	EWTh-2
Electrode diameter	mm	2.4
Heat input	kJ/mm	0.99
Nozzle diameter	mm	6.5 for Root Run 11.5 for Fillup and Capping pass
Preheating	°C	180-200

Inter-pass	°C	180-200
Filler wire	-	ERNiCr-3
Machine name	Fronius	Magic wave 2200

Table 3.10 Parameters used for welding of P92/304L plate

Parameter	Unit	Welding
Welding process	-	GTAW
Welding position	-	1G (Flat)
Joint design	-	Butt Joint Single V groove 75°
Voltage	V	8-9.8
Current	A	100-105
Number of welding passes	-	07
Polarity	-	DCEN
Average welding speed	mm/min	57
Shielding gas	-	Argon (99.99%)
Shielding gas speed	L/min	10
Electrode	-	EWTh-2
Electrode diameter	mm	2.4
Heat input	kJ/mm	1.08
Nozzle diameter	mm	6.5 for Root Run 11.5 for Fillup and Capping pass
Preheating	°C	180-200
Inter-pass	°C	180-200
Filler wire	-	ERNiCr-3
Machine name	Fronius	Magic wave 2200

3.5 Post weld heat treatment

The PWHT was carried out in muffle furnace with an accuracy of ± 2 °C. The welding mainly consists of the following steps: Preheat of the base material (around 200-250 °C), maintaining inter-pass temperature (around 250-300 °C), and post-weld heat treatment (PWHT). Figure 3.7(a, b) represents the temperature history of the as-received base metal and post weld heat treatment process of the P92/304L DWJ. The base plate was preheated to a temperature of 200 °C, and an interpass temperature of 250 °C was maintained with the help of flame heating. The PWHT was carried out at 760 °C, 2h.

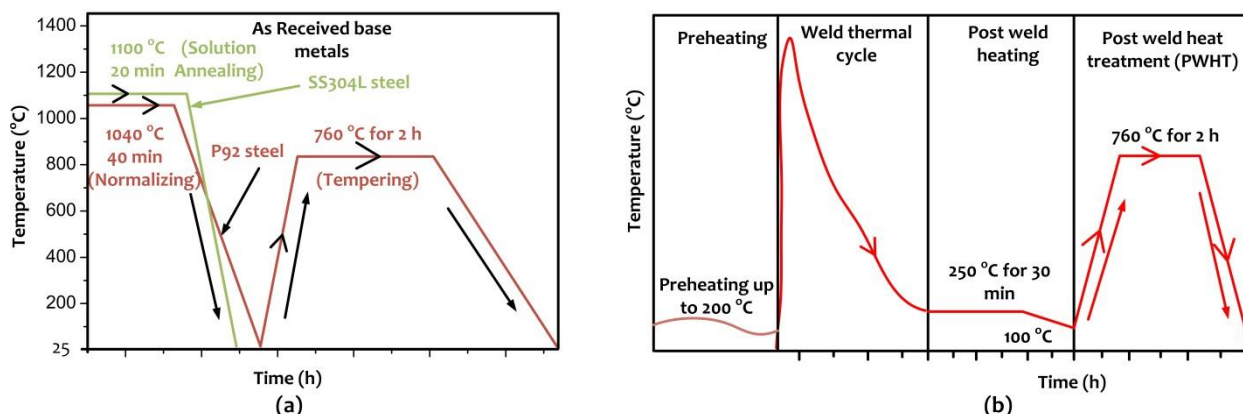


Figure 3.7: (a, b) Schematic diagram showing the temperature history of the base metal before and after welding

3.6 Sample preparation for metallographic examination

For metallographic studies, coupons were prepared by grinding using 80, 120, 180, 220, 320, 400, 600, 800, 1000, 1200, 1500 and 2000 grits of SiC paper, followed by the final polishing with 1µm alumina powder slurry. The guide for the preparation of the metallographic specimen

is available in ASTM E3-11 standard. The nickel-based weld fusion zone was electrolytically etched at current density and voltage of 1 A/cm². The different chemical etchants used for microstructure observations of different metals are listed in Table 3.11. The details of the procedures to be used for etching metals and alloys for microscopic examination are available in ASTM E407-07 standard. The microstructural examinations were conducted using an optical microscope (Make-Leica, Model: DMC4500) and a scanning electron microscope (Make-Carl Zeiss, Model: EVO 18).

Table 3.11 Chemical composition of etchants used to reveal microstructure of weld fusion zone, and P92 and 304L BM

Materials	Etchants
P92 steel BM	1g picric acid + 5 ml hydrochloric acid (concentrated) + 100 ml of ethanol, etching time-40s
304L steel BM	10 ml Nitric acid + 20 ml glycerol + 30 ml hydrochloric acid (concentrated), etching time-50s
ERNiCrMo-3 filler weld fusion zone	10 gm oxalic acid + 100 ml water, time-35s at current density and voltage of 1 A/cm ² and 4V, respectively
ERNiFeCr-2 filler weld fusion zone	10 gm oxalic acid + 100 ml water, time-45s at current density and voltage of 1 A/cm ² and 6V, respectively
ERNiCr-3 filler weld fusion zone	10 gm oxalic acid + 100 ml water, time-45s at current density and voltage of 1 A/cm ² and 9V, respectively
Thermanit MTS 616 filer weld fusion zone	1g picric acid + 5 ml hydrochloric acid (concentrated) + 100 ml of ethanol, etching time-40s

3.7 Sample preparation for the mechanical testing

The specimens for tensile test, Charpy test, and microstructure examinations were extracted from the transverse direction of the P92/304L DWJ with the help of wire cut electric discharge machine (WCEDM), as shown in Figure 3.8(a-d). A 100 kN capacity INSTRON 5980 universal testing machine (UTM) was used to conduct the room temperature tensile test on sub-size specimen. The tensile test was performed at a 1 mm/min loading rate. The Charpy V-notch specimens with dimensions 55 mm × 10 mm × 7.5 mm was prepared to determine toughness of the P92/304L DWJ. The specimens with notch situated at weld center, P92 HAZ, and 304L HAZ were prepared. The Vickers microhardness was determined on as-weld and PWHT specimens by applying a 500 g load for 10 s dwell time. The specimens were divided into two categories: as-weld (AW) specimens and PWHT specimens.

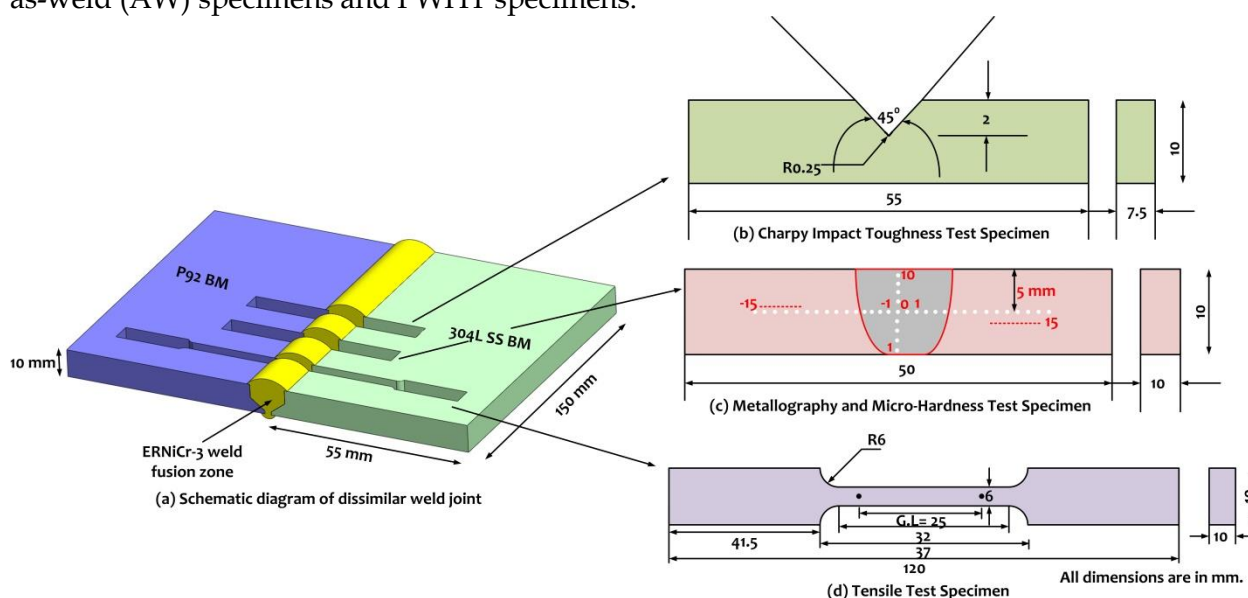


Figure 3.8: (a-d) Schematic diagram of the P92/304L DWJ with filler addition and corresponding specimen dimensions for various metallography and mechanical test

3.8 Residual stress analysis using deep hole drilling technique

The residual stresses of the multi-pass welded P92/304L DWJ were measured using the deep hole drilling (DHD) strain gauge method. The measurement was carried out as per the specifications given in the ASTM E837-13 standard. The DHD technique was used to measure the through-thickness residual stress fields in the weld fusion zone and HAZ. It is considered as a semi-destructive residual stress measurement technique competent in measuring the in-plane biaxial residual stress fields through the thickness. Four basic steps are involved in the DHD technique, as represented in Figure 3.9. In the first step, reference holes of 3 mm in diameter were made at the desired locations through the thickness. Later, the internal diameters of the reference holes ($d(\theta)$) were measured using an air probe along the longitudinal and transverse direction in 1 mm depth increments through the thickness. In the third step, the reference holes were trepanned coaxially using an electro-discharge machining process to relax the present locked-in stresses surrounding the reference hole. After that, the internal diameters of the reference holes ($d'(\theta)$) were remeasured using the air probe at the previous locations through the thickness following similar directions. The diametral deformation ($\Delta d(\theta) = d'(\theta) - d(\theta)$) of the reference hole corresponding to different depth intervals was then used to calculate the in-plane residual stress magnitudes. The normalized strain $\varepsilon(\theta)$ related to the σ_x , σ_y , σ_{xy} and σ_z residual stresses can be calculated using the below Eq. (3.1) (P.K. Taraphdar et al., 2021),

$$\varepsilon(\theta) = \frac{\Delta d(\theta)}{d(\theta)} = -\frac{1}{E} [\sigma_x f(\theta) + \sigma_y g(\theta) + \sigma_{xy} h(\theta) - \mu \sigma_z] \quad (3.1)$$

Where, $f(\theta) = 1 + 2\cos 2\theta$, $g(\theta) = 1 - 2\cos 2\theta$, $h(\theta) = 4\sin 2\theta$, E is young's modulus, and μ is poisons ratio. In the above equation, the x-axis lies with the $\theta = 0^\circ$ angular direction. The stress magnitude σ_z was assumed to be zero. Thus Eq. (3.1) can be rewritten as,

$$\varepsilon(\theta) = \frac{\Delta d(\theta)}{d(\theta)} = -\frac{1}{E} [\sigma_x f(\theta) + \sigma_y g(\theta) + \sigma_{xy} h(\theta)] \quad (3.2)$$

Eq. (3.2) can further be modified for the longitudinal (ε_x) and transverse (ε_y) strains by simply placing $\theta = 0^\circ$ and $\theta = 90^\circ$,

$$\varepsilon_x = \frac{\Delta d_x}{d_x} = -\frac{1}{E} [3\sigma_x - \sigma_y] \quad (3.3)$$

$$\varepsilon_y = \frac{\Delta d_y}{d_y} = -\frac{1}{E} [3\sigma_y - \sigma_x] \quad (3.4)$$

Eq. 3.3 and Eq. 3.4 were used to calculate the longitudinal and transverse residual stress magnitudes, respectively.

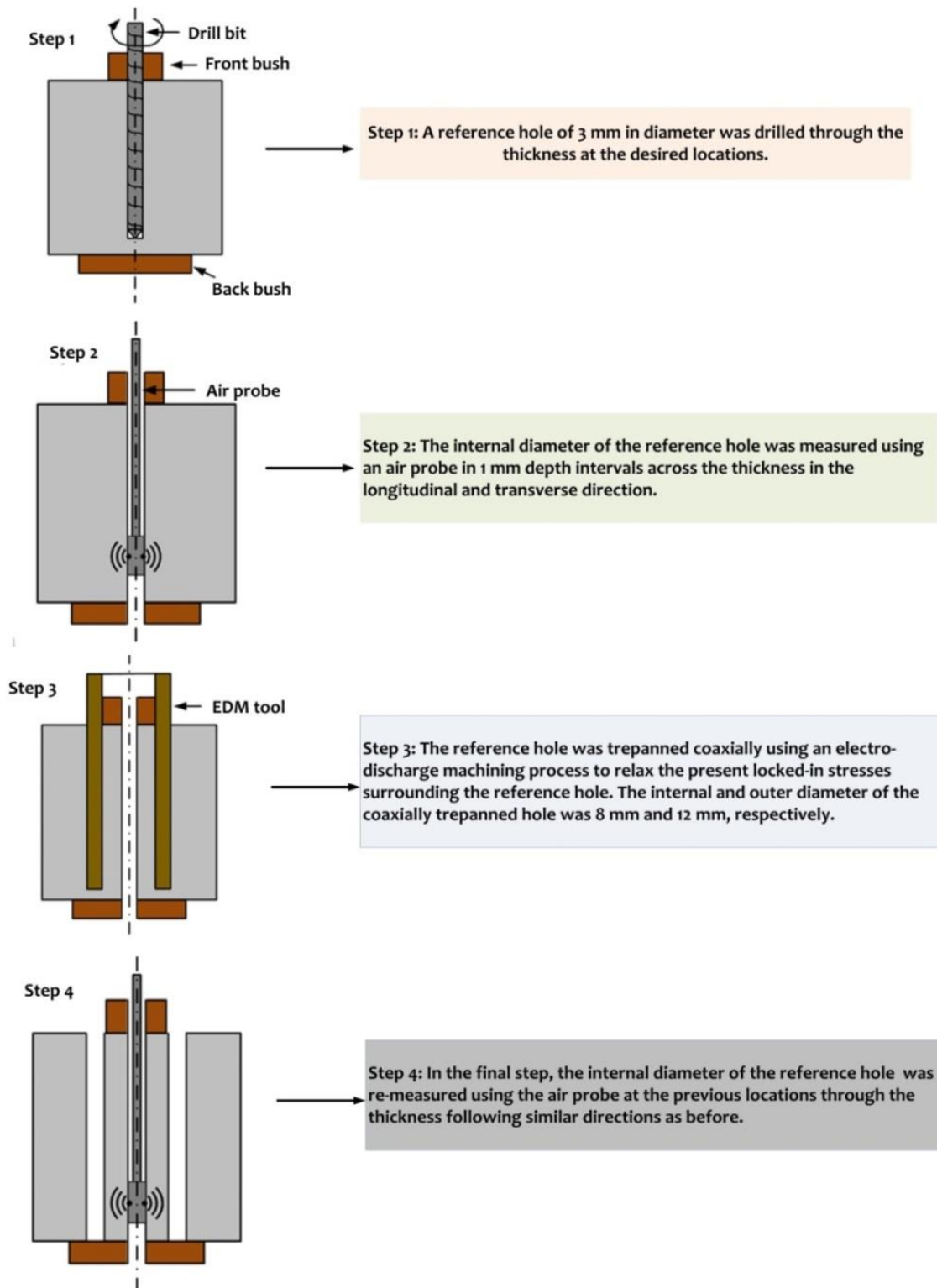


Figure 3.9: Schematic diagram of DHD technique for residual stress measurement

3.9 Sample preparation for creep and high-temperature tensile test

For the preparation of the high-temperature tensile and creep specimen, the rectangular strip was taken from the transverse direction of the dissimilar weld joints, as shown in schematic Figure 3.10. The round shape high-temperature tensile and creep specimens of required dimension, as shown in Figure 3.11(a-b) and Figure 3.12(a-b) were then prepared using a lathe machine. The cylindrical shape specimen for the high-temperature tensile test with a diameter of 3 mm, gauge length of 70 mm, and gripping length of 20 mm was prepared from the transverse direction of the weld according to the ASTM E8 standard. The high-temperature tensile test was conducted using a hydraulic universal testing machine (Make: Shimadzu) of 50

The diagram illustrates the preparation of creep and high temperature tensile specimens from a P92/304L SS weld fusion zone. The main components and dimensions are:

- Weld Fusion Zone:** A 10 mm thick plate with a 55 mm wide weld fusion zone. The weld is made of Thermanit MTS 616 (P92 filler).
- Materials:** P92 (blue) and 304L SS (green).
- Specimen Preparation:**
 - Creep Specimen:** A strip taken from the DWJs for the preparation of the creep specimen, prepared using a lathe machine.
 - High Temperature Tensile Specimen:** A strip taken from the DWJs for the preparation of the high temperature tensile specimen, prepared using a lathe machine.
- Dimensions:** All dimensions are in mm. The main plate is 150 mm wide.

(a)



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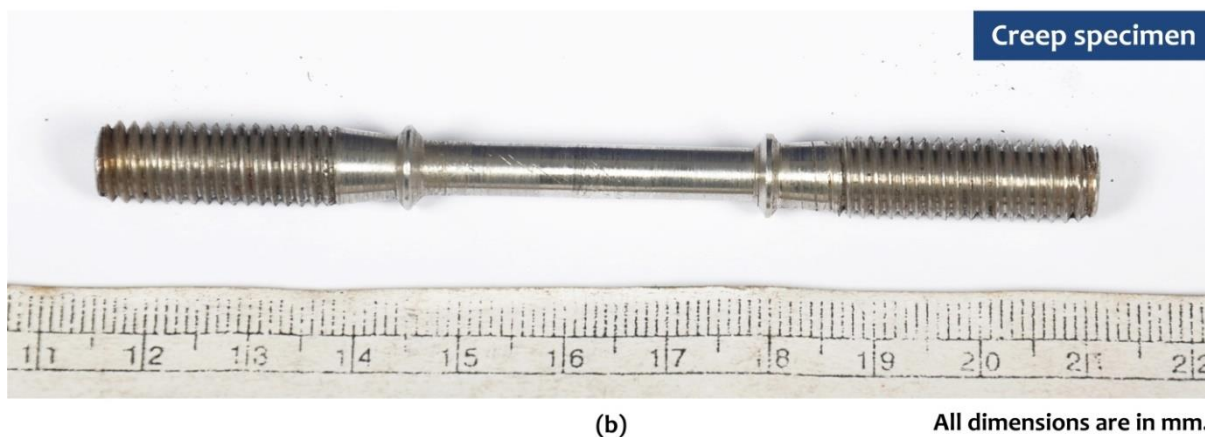
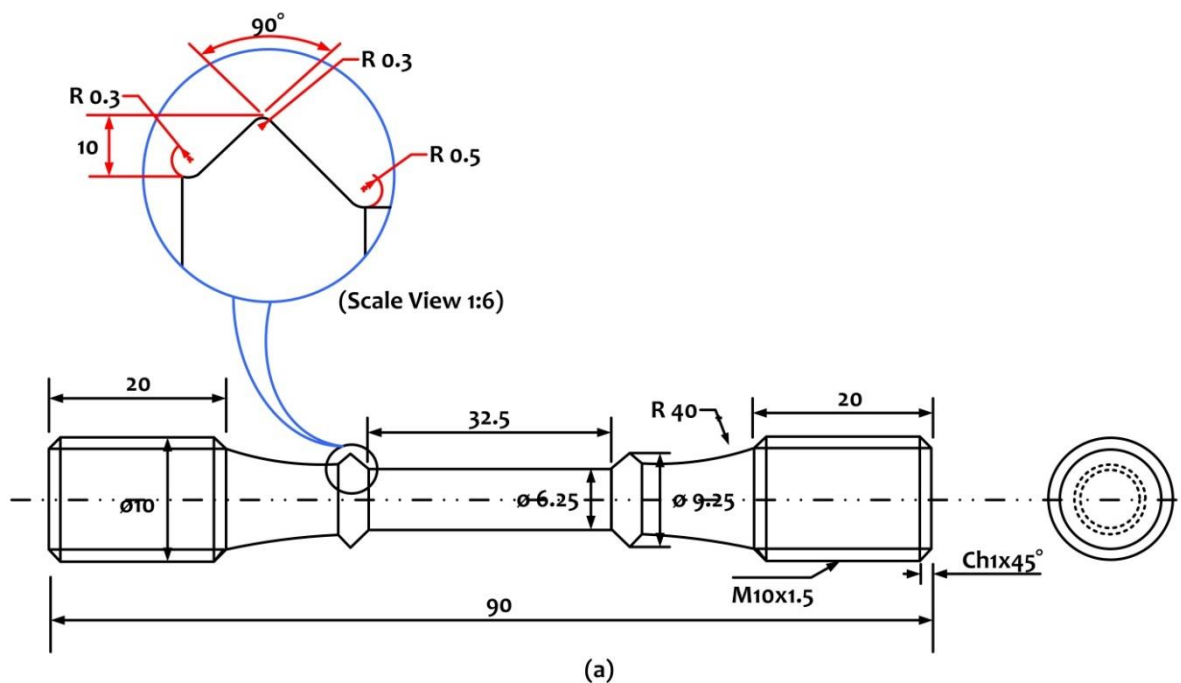


Figure 3.12: (a, b) Creep test specimen

For the metallographic investigation, the fracture high-temperature tensile and creep specimen was sliced into two halves from the transverse direction of the weld. The extracted sample was then hot-mounted with epoxy resin (Figure 3.13(a-b)) for further metallographic procedure. The hot-mounted specimen of dimension 30 mm was then mirror polished using SiC abrasive paper from #80 grit to #2000 and 1 μ m alumina slurry. The DWJs consist of three different metals, so different chemical etchant (Table 3.11) was used to reveal the microstructure features.

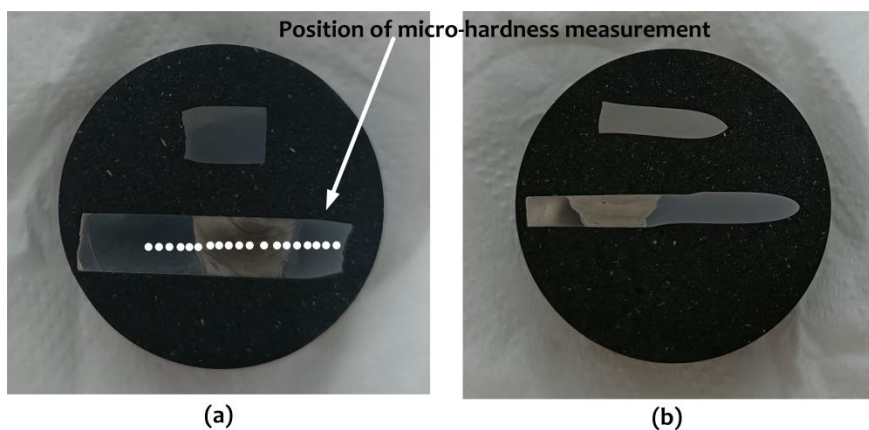


Figure 3.13: (a, b) Hot mounted fractured Creep and high-temperature tensile test specimen