

**INVESTIGATION OF POST-WELD HEAT  
TREATMENTS AND HIGH-TEMPERATURE  
BEHAVIOUR OF STEEL WELDS OF  
T91 AND SUPER304H**

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T91 AND SUPER304H**

by

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## Certificate

This is to certify that the thesis entitled “**Investigation of Post-Weld Heat Treatments and High-Temperature Behaviour of Steel Welds of T91 and Super304H**” is being submitted by **Mr. Ranjeet Kumar** to the Indian Institute of Technology Delhi for the award of the degree of **DOCTOR OF PHILOSOPHY** is a record of the bonafide research work carried out by him under our supervision and guidance. The matter presented in this thesis or any part thereof has not been submitted to any other University or Institute for the award of any degree or diploma.

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**Ranjeet Kumar**

A handwritten signature in cursive script that reads "Ranjeet".

## Abstract

Welding is inevitable to make permanent and leak-proof joints to specifically establish power plant structures. However, welding introduces inherent heterogeneity in microstructures, mechanical properties and residual stresses across the weldment, which appears to be the source of many premature failures. Hence, it is critical to systematically examine the weld microstructure heterogeneity and its influence on their eventual behaviour during service conditions. On the other hand, post-weld heat treatments (PWHTs) such as post-weld direct tempering (PWDT) and post-weld normalizing and tempering (PWNT) are being developed to overcome the problem of weld heterogeneity. However, the temperature and time of PWHTs are critical to achieve a better combination of homogeneous microstructures and mechanical properties. Considering this, the current research focused on a careful examination of various PWHTs and their subsequent effect on the weld microstructure heterogeneity.

Further, microstructural degradation during high-temperature exposure (HTE) has been studied to understand the material behaviour and possibly prevent premature failures. A lab-scale welding setup with preheating and uniform rotation speed has been designed and developed to achieve defect-free smooth weld beads. Using the setup, welding was performed considering firstly similar materials (i.e., T91 grade steel with T91) and secondly, considering dissimilar material (T91 steel with Super304H) welds.

The impact of PWDT on microstructure evolution, mechanical properties and residual stress was first investigated, considering the effects of Mn + Ni content on lower critical temperature ( $Ac_1$ ). The welded tubes were heat-treated (PWDT) at three temperatures (720 °C, 760 °C and 775 °C), chosen below their  $Ac_1$  ( $\sim 791 \pm 5$  °C), for varied times. The results demonstrate that PWDT temperatures closer to the  $Ac_1$  temperature (i.e., 760 °C and 775 °C) significantly impact hardness, tensile strength and % elongation.

Further, investigations reveal that the tempering at 775 °C – 30 minutes results in achieving optimum mechanical properties (attributed to fine  $M_{23}C_6$  type precipitate size) with minimum residual stresses. Additionally, longer tempering time (~ 120 minutes) at 760 °C and 775 °C coarsen the  $M_{23}C_6$  types of precipitates that initiate de-cohesion at the weak precipitate/matrix interface.

Even after PWDT, microstructure heterogeneity (such as variation in grain size and precipitates size and their distribution) remains across weldment (WM: weld metal, CGHAZ: coarse grain heat affected zone and FGHAZ: fine grain heat affected zone). This indicates that the individual weld zones may exhibit distinct behaviour during service. To simulate in-service conditions (as experienced by power plant weld materials/components) on the weldment microstructural degradation, the PWDT (760 °C for 120 minutes) heat-treated weld was exposed to HTE at 775 °C for varied times (5, 100, 500 and 1000 h). Among the weld zones, FGHAZ specifically demonstrated peculiar behaviour. Significant coarsening of  $M_{23}C_6$  precipitates and abnormal grain growth have been noticed, possibly due to the enhanced diffusion through the higher grain boundary areas resulting from the finest grain size in this welded zone.

Additionally, the grain growth may have also been accompanied by the recovery of substructures of laths/packets/blocks and unpinning of prior austenite grain boundaries (PAGBs) from the coarse precipitate. Due to this, after HTE, FGHAZ's hardness deterioration was the highest of all the zones. Moreover, the tensile strength and toughness of the weldment after 1000 h of HTE were reduced by 39 % and 57 %, respectively, compared to the PWDT condition and failure that occurred at FGHAZ. Hence, these results suggest that the selective microstructure degradation in FGHAZ, even below the  $Ac_1$  temperature, could be the cause of T91 steel welds premature failure during service. In contrast, after PWNT, the weld microstructure appears stable, where the selective grain growth or precipitate coarsening in

FGHAZ is inhibited even under the same HTE condition. This is attributed to the increase in the grain size, in the FGHAZ region, from  $5 \pm 2 \mu\text{m}$  to  $13 \pm 4 \mu\text{m}$ , with an enhancement in martensitic morphology (laths, blocks and packets). The microstructure changes after PWNT had also been demonstrated through their creep performance, where the impression creep rate was substantially lower (i.e.,  $\sim 0.5 \times 10^{-8}/\text{sec}$ ) for PWNT as compared to the creep rate ( $\sim 5.3 \times 10^{-8}/\text{sec}$ ) for PWDT. Similarly, the impression creep rate for PWNT with 1000 h HTE condition is  $\sim 8.3 \times 10^{-8}/\text{sec}$  compared to the creep rate observed (i.e.,  $\sim 21 \times 10^{-8}/\text{sec}$ ) for PWDT with 1000 h HTE condition. Hence, for T91 welds, PWNT outperforms PWDT; further, it was examined to be established for dissimilar metal welds (DMWs).

DMW of T91 and Super304H material induces an extremely heterogeneous weldment. The studies found failures either from FGHAZ on the T91 side due to rapid microstructure deterioration or Super304H side due to sensitization/intergranular corrosion (IGC). To mitigate the heterogeneity across welds, PWNT was employed to homogenize the microstructure in both the materials and compared with PWDT. It was found that after PWDT, there was no impact on grain size across the weldment. Additionally, it resulted in a sensitization phenomenon on the Super304H side. On the other hand, PWNT heat treatment eliminated the critical FGHAZ of T91 side and as well reduced the sensitization on the Super304H side.

Furthermore, the microstructural analysis revealed that PWNT heat treatment reduced grain size heterogeneity and decreased the precipitates' size on both sides of the WM. Moreover, the optimal combination of mechanical properties (0.2 % proof stress, tensile strength, % elongation and toughness were 384 MPa, 691 MPa, 22 % and  $132 \text{ MJ/m}^3$ , respectively) was found in the case of PWNT heat treatment, which is attributed to uniform grain size and fine precipitates. Furthermore, DMW resulted in higher tensile residual stresses on the T91 side, while the Super304H side exhibited compressive residual stress. PWDT and PWNT could reduce residual stresses, although PWNT is more effective due to complete austenitizing.

## सार

बिजली संयंत्र संरचनाओं को विशेष रूप से स्थापित करने के लिए स्थायी और रिसाव-सबूत जोड़ों को बनाने के लिए वेल्डिंग अनिवार्य है। हालांकि, वेल्डिंग माइक्रोस्ट्रक्चर, यांत्रिक गुणों और वेल्डमेंट में अवशिष्ट तनावों में निहित विषमता का परिचय देता है, जो कई समयपूर्व विफलताओं का स्रोत प्रतीत होता है। इसलिए, सेवा शर्तों के दौरान उनके अंतिम व्यवहार पर वेल्ड माइक्रोस्ट्रक्चर विषमता और उसके प्रभाव की व्यवस्थित जांच करना महत्वपूर्ण है। दूसरी ओर, वेल्ड विषमता की समस्या को दूर करने के लिए पोस्ट-वेल्ड हीट ट्रीटमेंट (PWHTs) जैसे पोस्ट-वेल्ड डायरेक्ट टेम्परिंग (PWDT) और पोस्ट-वेल्ड नॉर्मलाइज़िंग एंड टेम्परिंग (PWNT) विकसित किए जा रहे हैं। हालांकि, सजातीय माइक्रोस्ट्रक्चर और यांत्रिक गुणों के बेहतर संयोजन को प्राप्त करने के लिए PWHTs का तापमान और समय महत्वपूर्ण है। इसे ध्यान में रखते हुए, वर्तमान शोध ने विभिन्न PWHTs की सावधानीपूर्वक जांच और वेल्ड माइक्रोस्ट्रक्चर विषमता पर उनके बाद के प्रभाव पर ध्यान केंद्रित किया।

इसके अलावा, भौतिक व्यवहार को समझने और संभवतः समय से पहले विफलताओं को रोकने के लिए उच्च तापमान जोखिम (HTE) के दौरान माइक्रोस्ट्रक्चरल गिरावट का अध्ययन किया गया है। प्रीहीटिंग और एकसमान रोटेशन स्पीड के साथ लैब-स्केल वेल्डिंग सेटअप को दोष मुक्त वेल्ड बीड्स प्राप्त करने के लिए डिजाइन और विकसित किया गया है। सेटअप का उपयोग करते हुए, पहले T91 के साथ T91 ग्रेड स्टील और दूसरी बात, T91 स्टील के साथ Super304H वेल्ड पर विचार करते हुए वेल्डिंग का प्रदर्शन किया गया।

कम महत्वपूर्ण तापमान ( $A_{c1}$ ) पर Mn + Ni के प्रभावों पर विचार करते हुए, सबसे पहले माइक्रोस्ट्रक्चर विकास, यांत्रिक गुणों और अवशिष्ट तनाव पर PWDT के प्रभाव की जांच की गई। वेल्डेड ट्यूबों को अलग-अलग समय के लिए तीन तापमान (720 °C, 760 °C और 775 °C) पर उनके  $A_{c1}$  (~ 791 ± 5 °C) के नीचे चुना गया था। परिणाम प्रदर्शित करते हैं कि PWDT तापमान  $A_{c1}$  तापमान (यानी, 760 °C और 775 °C) के करीब कठोरता, तन्य शक्ति और % बढ़ाव पर महत्वपूर्ण प्रभाव डालता है।

इसके अलावा, जांच से पता चलता है कि 775 °C - 30 मिनट पर परिणामस्वरूप न्यूनतम अवशिष्ट तनाव के साथ इष्टतम यांत्रिक गुणों को प्राप्त किया जाता है। इसके अतिरिक्त, 760 °C और 775 °C पर लंबे समय तक (~ 120 मिनट),  $M_{23}C_6$  प्रकार के अवक्षेपों को मोटा कर देता है जो कमजोर अवक्षेप/मैट्रिक्स इंटरफ़ेस पर डी-कोहेशन की शुरुआत करता है।

PWDT के बाद भी, माइक्रोस्ट्रक्चर विषमता वेल्डमेंट में बनी हुई है। यह इंगित करता है कि अलग-अलग वेल्ड जोन सेवा के दौरान विशिष्ट व्यवहार प्रदर्शित कर सकते हैं। वेल्ड माइक्रोस्ट्रक्चरल डिग्रेडेशन पर इन-सर्विस स्थितियों (जैसा कि पावर प्लांट वेल्ड / घटकों द्वारा अनुभव किया गया है) का अनुकरण करने के लिए, PWDT (120 मिनट के लिए 760 °C) हीट-ट्रीटेड वेल्ड को विभिन्न समय (5, 100, 500 और 1000 घंटे) के लिए 775 °C पर HTE के संपर्क में लाया गया था। वेल्ड क्षेत्रों में, FGHAZ ने विशेष रूप से अजीबोगरीब व्यवहार का प्रदर्शन किया।  $M_{23}C_6$  अवक्षेप के महत्वपूर्ण मोटेपन और असामान्य वृद्धि को देखा गया है, संभवतः इस वेल्डेड ज़ोन में बेहतरीन आकार के परिणामस्वरूप उच्च ऊर्जा सीमा क्षेत्रों के माध्यम से बढ़े हुए प्रसार के कारण।

इसके अतिरिक्त, ग्रेन की वृद्धि के साथ लथ/पैकेट/ब्लॉक के सबस्ट्रक्चर की रिकवरी और मोटे अवक्षेप से पूर्व ऑस्टेनाइट ग्रेन बाउंड्री (PAGBs) को खोलना भी हो सकता है। इसके कारण, HTE के बाद, FGHAZ की कठोरता अवनति सभी क्षेत्रों में सबसे अधिक थी। इसके अलावा, PWDT स्थिति और FGHAZ में हुई विफलता की तुलना में, HTE के 1000 घंटे के बाद वेल्ड की तन्य शक्ति और क्रूरता क्रमशः 39% और 57% कम हो गई थी। इसलिए, इन परिणामों से पता चलता है कि FGHAZ में चयनात्मक माइक्रोस्ट्रक्चर गिरावट, यहां तक कि  $Ac_1$  तापमान से भी नीचे, सेवा के दौरान T91 स्टील वेल्ड की समयपूर्व विफलता का कारण हो सकता है।

इसके विपरीत, PWNT के बाद, वेल्ड माइक्रोस्ट्रक्चर स्थिर दिखाई देता है, जहां FGHAZ में चयनात्मक ग्रेन की वृद्धि या अवक्षेपण एक ही HTE स्थिति के तहत भी बाधित होता है। यह ग्रेन के आकार में वृद्धि के लिए जिम्मेदार है, FGHAZ क्षेत्र में,  $5 \pm 2$  माइक्रोन से  $13 \pm 4$  माइक्रोन तक, मार्टेंसिटिक मॉर्फोलॉजी (लैथ, ब्लॉक और पैकेट) में वृद्धि के साथ। PWNT के बाद माइक्रोस्ट्रक्चर में बदलाव को उनके क्रीप प्रदर्शन के माध्यम से भी प्रदर्शित किया गया था, जहां क्रीप रेट ( $\sim 5.3 \times 10^{-8}$  / सेकंड) की तुलना में PWNT के लिए इम्प्रेशन क्रीप रेट काफी कम था (यानी,  $\sim 0.5 \times 10^{-8}$  / सेकंड)। पीडब्ल्यूडीटी के लिए। इसी तरह, 1000 घंटे HTE स्थिति वाले PWNT के लिए इम्प्रेशन क्रीप रेट  $\sim 8.3 \times 10^{-8}$  / सेकंड है, जबकि 1000 घंटे HTE स्थिति वाले PWDT के लिए क्रीप रेट (यानी,  $\sim 21 \times 10^{-8}$  / सेकंड) देखा गया है। इसलिए, T91 वेल्ड के लिए, PWNT, PWDT से बेहतर प्रदर्शन करता है; इसके अलावा, इसे असमान धातु वेल्ड (DMW) के लिए स्थापित करने की जांच की गई।

T91 और Super304H का DMW, एक अत्यंत विषम वेल्ड को प्रेरित करता है। अध्ययनों में या तो FGHAZ से T91 की ओर तेजी से माइक्रोस्ट्रक्चर बिगड़ने या सुपर304H की ओर से संवेदीकरण /

इंटरग्रेनुलर जंग (IGC) के कारण विफलता पाई गई। वेल्ड में विषमता को कम करने के लिए, PWNT को दोनों सामग्रियों में माइक्रोस्ट्रक्चर को समरूप बनाने और PWDT के साथ तुलना करने के लिए नियोजित किया गया था। यह पाया गया कि पीडब्ल्यूडीटी के बाद, वेल्डमेंट में ग्रेन के आकार पर कोई प्रभाव नहीं पड़ा। इसके अतिरिक्त, इसके परिणामस्वरूप Super304H की तरफ एक संवेदीकरण घटना हुई। दूसरी ओर, PWNT ताप उपचार ने T91 पक्ष के महत्वपूर्ण FGHAZ को समाप्त कर दिया और साथ ही Super304H पक्ष पर संवेदीकरण को कम कर दिया।

इसके अलावा, माइक्रोस्ट्रक्चरल विश्लेषण से पता चला है कि PWNT हीट ट्रीटमेंट ने ग्रेन के आकार की विषमता को कम कर दिया और WM के दोनों किनारों पर अवक्षेप के आकार को कम कर दिया। इसके अलावा, यांत्रिक गुणों का इष्टतम संयोजन (0.2% प्रूफ तनाव, तन्य शक्ति, % बढ़ाव और क्रूरता क्रमशः 384 MPa, 691 MPa, 22 % और 132 MJ/m<sup>3</sup>, क्रमशः) PWNT ताप उपचार के मामले में पाया गया था, जो कि है एकसमान ग्रेन के आकार और महीन अवक्षेप के लिए जिम्मेदार। इसके अलावा, DMW ने T91 पक्ष पर उच्च तन्यता अवशिष्ट तनाव का परिणाम दिया, जबकि Super304H पक्ष ने संकुचित अवशिष्ट तनाव प्रदर्शित किया। PWDT और PWNT अवशिष्ट तनाव को कम कर सकते हैं, हालाँकि PWNT पूर्ण austenitizing के कारण अधिक प्रभावी है।

# Table of Contents

<b>Certificate .....</b>	<b>i</b>
<b>Acknowledgements .....</b>	<b>iii</b>
<b>Abstract.....</b>	<b>v</b>
<b>Table of Contents .....</b>	<b>xi</b>
<b>List of Figures.....</b>	<b>xvi</b>
<b>List of Tables .....</b>	<b>xxiii</b>
<b>Glossary .....</b>	<b>xxiv</b>
<b>Symbols .....</b>	<b>xxv</b>
<b>Chapter 1 Introduction.....</b>	<b>1</b>
<b>Chapter 2 Literature review .....</b>	<b>5</b>
2.1 Power plants and key high-temperature materials .....	5
2.2 Welding of reheaters/superheaters steel tubes .....	7
2.3 Modified 9Cr-1Mo steel (SA213-P/T91).....	8
2.3.1 Evolution of the second phase in T91 .....	10
2.3.2 Summary .....	12
2.4 Super304H .....	12
2.4.1 Time-temperature dependence of second phases in Super304H .....	14
2.4.2 Summary .....	14
2.5 Similar welding of T91 .....	14
2.5.1 Microstructure evolution after welding .....	14
2.5.2 PWHT and their influence on microstructure .....	17
2.5.3 Hardness variation across weldment.....	21
2.5.4 Mechanical properties of welds .....	23
2.5.5 Fractography of welds.....	25

2.5.6 Summary .....	25
2.6 Dissimilar welds of T91 and Super304H.....	26
2.6.1 Microstructure of DMW .....	28
2.6.2 Hardness variation .....	30
2.6.3 Mechanical properties .....	31
2.6.4 Fractography .....	33
2.6.5 Summary .....	33
2.7 Residual stress distribution across weldment .....	34
2.7.1 Similar welds .....	34
2.7.2 Dissimilar welds.....	36
2.7.3 Summary .....	37
2.8 Impression creep properties .....	37
2.9 Factors contributing to power plant weld component failures.....	40
2.9.1 Microstructure degradation at high temperatures .....	42
2.9.2 Type IV cracking .....	45
2.9.3 Sensitization and associated failures.....	47
2.9.4 Summary .....	50
2.10 Overall summary.....	50
<b>Chapter 3 Scope and objectives .....</b>	<b>52</b>
<b>Chapter 4 Materials and Methodologies.....</b>	<b>54</b>
4.1 Introduction.....	54
4.2 Initial materials .....	54
4.3 Welding setup .....	56
4.3.1 Working principle .....	56
4.4 Welding.....	57

4.4.1 Similar welding of T91 .....	58
4.4.2 Dissimilar welding of T91 and Super304H .....	60
4.4.3 Post-weld examination.....	60
4.5 Heat treatments .....	62
4.5.1 PWHTs of similar welds of T91 with T91.....	62
4.5.2 PWHTs of dissimilar welds of T91 and Super304H .....	64
4.6 Microstructural characterization .....	64
4.6.1 Sample preparation .....	65
4.6.2 Spectroscopy .....	65
4.6.3 Optical microscope .....	66
4.6.4 Scanning electron microscope (SEM) .....	66
4.6.5 High-resolution transmission electron microscope (HR-TEM).....	67
4.6.6 Electron probe micro analyzer (EPMA) .....	67
4.6.7 Electron backscattered diffraction (EBSD) .....	68
4.7 Mechanical Characterization .....	69
4.7.1 Microhardness.....	69
4.7.2 Tensile test .....	69
4.7.3 Impression creep test.....	70
4.8 Residual stress measurement .....	73
4.8.1 $\sin^2\psi$ based X-ray diffraction method (I-XRD) .....	74
4.8.2 $\cos \alpha$ based XRD method.....	77
<b>Chapter 5 Influence of post-weld direct tempering on microstructure, mechanical properties and residual stress of similar welds of T91 .....</b>	<b>79</b>
5.1 Introduction.....	79
5.2 Experimental results and discussion .....	80

5.2.1 Initial microstructure.....	80
5.2.2 Weld macrostructure.....	81
5.2.3 Hardness and mechanical properties of the weldment.....	82
5.2.4 Microstructure evolution of weldment.....	85
5.2.5 Composition analysis of the second phase.....	87
5.2.6 Fractography .....	88
5.2.7 Residual stresses .....	92
5.3 Summary.....	95
<b>Chapter 6 Influence of post-weld direct tempering and post-weld normalizing and tempering on the microstructural changes, mechanical properties and impression creep properties of the T91 steel weldment under high temperature exposure .....</b>	<b>96</b>
6.1 Introduction.....	96
6.2 Experimental results and discussion .....	97
6.2.1 High-temperature degradation of PWDT heat-treated condition.....	97
6.2.2 Effect of PWNT on the HTE of FGHAZ.....	115
6.3 Summary .....	127
<b>Chapter 7 Influence of modified post-weld heat treatments on microstructural heterogeneity, sensitization, mechanical properties and residual stresses of dissimilar metal welds .....</b>	<b>129</b>
7.1 Introduction.....	129
7.2 Experimental results and discussion .....	130
7.2.1 Initial microstructure.....	130
7.2.2 As-welded microstructures .....	131
7.2.3 Microstructure evolution after PWDT .....	133
7.2.4 Microstructure evolution after PWNT .....	135
7.2.5 Second phase compositional analysis .....	137

7.2.6 Comparison of as-welded and post-weld heat-treated microstructures .....	138
7.2.7 Quantification of grain size and precipitate size.....	141
7.2.8 Effect of PWHTs on mechanical properties .....	143
7.2.9 Fractography .....	145
7.2.10 Residual stress distribution in dissimilar welds .....	147
7.3 Comparison of similar and dissimilar welds of T91 and Super304H.....	149
7.4 Summary.....	150
<b>Chapter 8 Summary and Conclusions .....</b>	<b>151</b>
8.1 Summary.....	151
8.2 Conclusions.....	153
8.3 Future work.....	154
<b>REFERENCES.....</b>	<b>155</b>
<b>PUBLICATIONS AND CONFERENCE .....</b>	<b>171</b>
<b>CURRICULUM VITAE.....</b>	<b>174</b>

## List of Figures

Figure 2.1: (a) Schematic of super-critical steam power plant [25] and (b) Photograph of reheater/superheater tubes [3].	6
Figure 2.2: Power plant welding techniques (a) SMAW and (b) TIG welding with its components [27].	8
Figure 2.3: Micrographs illustrating (a) Optical, (b) SEM, (c) Schematic of fine features and (d) EDS of precipitates of T91 steel [37].	9
Figure 2.4: TEM images showing (a) pinning effect of $M_{23}C_6$ on sub-grain boundaries [40] and (b) MX carbonitrides in a 9% Cr steel for the tempered condition [43].	11
Figure 2.5: Micrographs illustrating (a) Grains and carbides [20] and (b) STEM image of Cu-rich phase and its distribution [48] in Super304H.	12
Figure 2.6: Time-temperature precipitation diagram of different phases such as $M_{23}C_6$ and $\sigma$ phase for Super304H [49].	13
Figure 2.7: (a) Welding HAZs and relation to the calculated equilibrium phase diagram of T91 [38], (b) Overview of the weldment; (c) CGHAZ, (d) FGHAZ, (e) IC-HAZ [40].	15
Figure 2.8: SEM micrographs of sub-zones of P91 steel weldments after PWDT at 760 °C for 120 minutes: (a) WM, (b) CGHAZ, (c) FGHAZ, (d) IC-HAZ [67].	18
Figure 2.9: SEM micrographs of sub-zones of P91 steel weldments after PWNT (a) WM, (b) CGHAZ, (c) FGHAZ, (d) IC-HAZ [51].	19
Figure 2.10: (a) Schematic of microstructural evolution of P91 weldments during PWNT [50], (b) Variation in grain size and (c) Precipitate size, of sub-zones [51].	20
Figure 2.11: Schematic shows the location of hardness measurement and (b) Hardness profile across the weldment of welded pipe for different heat treatment conditions [70].	22
Figure 2.12: (a) Fractured tensile specimen and their location; fracture surface morphology of (b) As welded, (c) PWDT and (d) PWNT [37].	24
Figure 2.13: (a) Longitudinal cross-section view of the DMW of Super304H and T92, (b) HAZ of Super304H, (c) WM: ERNiCrFe-7A, (inset: SEM-BSE micrograph of fine Nb and Mo precipitates at the inter-dendritic regions), (d) CGHAZ close to the fusion line, (e) FGHAZ, (f) ICHAZ, after PWDT. Note: [white coloured arrow: indicates the PAGBs]; [fresh tempered martensite-red colored arrows]; [over tempered martensite: yellow-colored arrows] [82].	29
Figure 2.14: Microhardness profiles across the welds after PWDT at 750 °C for 120 minutes [89].	30

Figure 2.15: Impact strength of T92/S304H dissimilar materials joints after PWDT at 760 °C for 60 minutes [75].	32
Figure 2.16: Tensile fracture of weld joints (a) Surface morphology of WM (ERNiCr-3) (b) Central fiber zone of (WM) ERNiCr-3 [83].	33
Figure 2.17: (a) Residual stress distribution across T91 weldment along longitudinal and transverse direction [93] and (b) Influence of PWDT on residual stresses distribution of weldment [95].	35
Figure 2.18: Residual stress distribution along dissimilar weldments in longitudinal and transverse directions [97].	36
Figure 2.19: Graphs illustrating (a) Impression depth with time at different stresses at 600 °C and (b) Corresponding minimum creep strain rate (MSR) obtained for T91 steel [106].	38
Figure 2.20: Stress dependence of the steady-state creep rate for different zones of T91 weldment at 600 °C [107].	39
Figure 2.21: Deformation region beneath the impression creep regions in (a) Optical micrograph of 316 LN stainless steel [108] and (b) Superposed maps of local misorientation angle and grain boundaries (>10 deg—black line) of Super304H [110].	40
Figure 2.22: Schematic illustrating different factors contributing to the power plant components failures where the main contributors are (a) Creep-rupture failure [86], (b) SCC in Super304H [114], (c) Distortion and cracks formation due to the presence of residual stress and (d) Microstructure degradation in terms of precipitation coarsening [112].	42
Figure 2.23: SEM images of microstructure evolution for different times and temperatures of annealing (a) Initial state quenched at 1040 °C and tempered at 730 °C for 30 minutes, (b) Annealed 3 h/800 °C, (c) Annealed 24 h/800 °C and (d) Annealed 168 h/800 °C [116].	43
Figure 2.24: (a) Changes of Cr/Fe and (b) Mo/Fe ratio in $M_{23}C_6$ particles depending on P91 steel thermal ageing duration and temperature [119].	44
Figure 2.25: Ageing of Super304H at 650 °C for 5000 h (a) SEM and (b) TEM [22].	44
Figure 2.26: (a) Evolution of cracks in T91 weldments [121] and (b) Montage showing the type IV damage in a cross weld sample [72].	45
Figure 2.27: Microstructural variation after 90 % creep test (a) CGHAZ, (b) FGHAZ, (c) BM [73] and (d) Creep cavities formation along the PAGBs [121].	47
Figure 2.28: Surface creep cavity morphologies of the Super304H steel crept under 650 °C /195MPa for (a) 3000 h, (b) 4000 h and (c) 4578 h in a longitudinal direction [131].	49
Figure 2.29: Illustrating grain boundaries initiating micro-voids and cracks at (a) 850 °C and (b) 950 °C [138].	49

Figure 4.1: Schematic diagram illustrating the important steps involved in the preparation and characterization of welds. ....	54
Figure 4.2: Photographs of initial material used for (a) Similar welding of T91 and (b) Dissimilar welding of T91 and Super304H. ....	55
Figure 4.3: (a) Schematic of welding setup and (b) Actual welding setup.....	56
Figure 4.4: (a) Schematic of tube edge prepared and arrangement of tubes and (b) Actual picture of tube arrangement on welding setup. ....	59
Figure 4.5: Photographs of welded joints of (a) Similar welds of T91 and (b) Dissimilar welds of T91 with Super304H. ....	61
Figure 4.6: Thermo-Calc used for calculating critical temperature for (a) As-received T91 and (b) Filler material (WM). ....	63
Figure 4.7: PWDT performed on similar welds of T91 at different temperatures and times. .	63
Figure 4.8: Schematic of welded tube and cutting of samples for characterization. ....	65
Figure 4.9: Schematic illustrating the location of hardness indent across the weldment. ....	69
Figure 4.10: Schematic illustrating (a) Welded tubes and flat plate removed location and (b) Wire cut dog-bone geometry from the flat plate as per ASTM E8/E8M-16a [138]......	70
Figure 4.11: (a, b, c) Illustrate the sequence of cutting off the impression creep test sample and (d) Shows the indenter and sample position. ....	71
Figure 4.12: (a) Photograph of impression creep tester and (b, c) Photographs of an impression on the sample with top and side view. ....	72
Figure 4.13: Schematic illustrating the radiation diffraction within a crystal structure [139].	73
Figure 4.14: Photographs of portable residual stress measurement system I-XRD and (b) Axial system for residual stress measurements $\sin^2\psi$ method.....	74
Figure 4.15: (a) Photographs illustrating the portable instrument of the $\text{Cos } \alpha$ method, (b) Diffraction and formation of the Debye-Scherrer ring and (c) Deviation in the Debye-Scherrer ring due to stresses. ....	77
Figure 5.1: As-received T91 (a) Optical micrograph, (b) EBSD-IPF maps (sample normal), (c) SEM micrograph (inset higher magnification SEM micrograph to show precipitates) and (d) EDS spectra of precipitate marked in Figure 5.1 (c). ....	80
Figure 5.2: Stereo-micrograph showing the demarcation of different regions such as weldment, HAZs and BM in as-welded conditions. The different regions are highlighted as A = WM, B = CGHAZ, C = FGHAZ, D = ICHAZ and E=BM. ....	81

Figure 5.3: (a) Hardness profile across the WM, different HAZs and BM regions and (b) Tensile stress-strain behaviour for as-welded and various PWDT conditions of the T91 samples.....	84
Figure 5.4: As-welded, PWDT at 760 °C – 120 minutes and PWDT at 775 °C – 30 minutes SEM micrographs illustrating (a, b, c) WM, (d, e, f) CGHAZ and (g, h, i) FGHAZ. ....	86
Figure 5.5: SEM micrographs highlighting the point of interest (yellow circle) for EDS analysis and corresponding EDS spectra of WM and HAZ regions for (a, b) As-welded, (c, d) PWDT at 760 °C – 120 minutes and (e, f) PWDT at 775 °C – 30 minutes condition.....	87
Figure 5.6: Fractography after (a, b) as-welded, (c, d) PWDT at 720 °C – 120 minutes, (e, f) PWDT at 760 °C – 30 minutes, (g, h) PWDT at 760 °C – 120 minutes, (i, j) PWDT at 775 °C – 30 minutes and (k, l) PWDT at 775 °C – 120 minutes; The numbers in figures indicate following features as 1=dimples, 2=cleavage facets, 3=precipitates, 4=de-cohesion, 5=secondary crack propagation and 6=primary crack. ....	89
Figure 5.7: Precipitate size from fracture surface of as-welded and the different PWDT conditions.....	90
Figure 5.8: SEM fractographs with EDS spectra on the marked (red colour) locations of T91 weld samples after tensile testing for (a) As-welded, (b) PWDT at 720 °C – 120 minutes, (c) PWDT at 760 °C – 30 minutes, (d) PWDT at 760 °C – 120 minutes, (e) PWDT at 775 °C – 30 minutes and (f) PWDT at 775 °C – 120 minutes conditions.....	91
Figure 5.9: Residual stress behaviour using $\text{Sin}^2\psi$ and $\text{Cos } \alpha$ methods across weldments after welding.....	94
Figure 5.10: The influence of PWDT on residual stress distribution using the $\text{Cos } \alpha$ method. ....	94
Figure 6.1: (a, c, e) SEM and (b, d, f) EBSD-grain boundary maps illustrating the precipitates and PAGBs with lath/block/packet boundaries distribution, respectively, for WM, CGHAZ and FGHAZ after PWDT; (Note: In grain boundary maps, lime green colour represents laths, black colour represents PAGBs, red colour represents blocks/packets and blue colour possibly represents laths/packets/PAGBs). ....	99
Figure 6.2: SEM micrographs illustrating the microstructural variations, mainly coarsening of $\text{M}_{23}\text{C}_6$ precipitates, in the WM, CGHAZ and FGHAZ during HTE at 775 °C for (a, b, c) 5 h; (d, e, f) 100 h; (g, h, i) 500 h and (j, k, l) 1000 h. ....	100
Figure 6.3: Bar graphs comparing the number of precipitates in all the zones for the fixed area after (a) PWDT and (b) HTE for 1000 h.....	101

Figure 6.4: EBSD grain boundaries maps after 1000 h of HTE of the: (a, b) WM, where (b) shows the features in a magnified manner of box region of (a) and (c, d) CGHAZ, where (d) shows the features in a magnified manner of box region of (c); (Note: In grain boundary maps, lime green colour represents laths, black colour represents PAGBs, red colour represents blocks/packets and blue colour possibly represents laths/packets/PAGBs). ..... 103

Figure 6.5: EBSD grain boundary maps of FGHAZ illustrating grain size variation after HTE of (a) 5 h, (b) 100 h, (c) 500 h, (d) 1000 h and (f) average grain size values in PWDT and HTE conditions; (Note: In grain boundary maps, lime green colour represents laths, black colour represents PAGBs, red colour represents blocks/packets and blue colour possibly represents laths/packets/PAGBs). ..... 105

Figure 6.6: The microstructure variation in FGHAZ region after HTE for 5 h, 100 h and 1000 h conditions showing: (a) Fine grains, (b) Fine- and growing-grains and (c) Grown grains, highlighting the accumulated strain levels through misorientation angles using EBSD-LAM maps; (d) Fine grain unimodal, (e) Fine- and growing-grain bimodal and (f) Grown grain unimodal, grain size distribution plots and (g, h, i) Schematic illustration of the selective grain growth due to the recovery of PAGBs, lath- and block-boundaries, dislocations and precipitates ( $M_{23}C_6$ ) coarsening mechanisms..... 106

Figure 6.7: (a, b) SEM micrographs illustrating the pinning and unpinning phenomenon at grain growth frontier after 100 h HTE condition. .... 108

Figure 6.8: HR-TEM bright-field (BF) micrographs of (a, b) FGHAZ region, (c, d) CGHAZ region and (e, f) EDS of  $M_{23}C_6$  precipitates and matrix, respectively, for HTE of 1000 h condition. Note: white arrows show the  $M_{23}C_6$  precipitates, red arrows show the MX precipitates, yellow arrows show the dislocations, yellow dashed lines show the laths and subgrains boundaries..... 110

Figure 6.9: Microhardness variation illustrates the hardness deterioration in different weld zones, viz. WM, CGHAZ and FGHAZ, from PWDT to HTE for 5 h, 100 h, 500 h and 1000 h conditions. .... 112

Figure 6.10: Tensile stress-strain behaviour of PWDT and HTE for 1000 h condition. .... 113

Figure 6.11: SEM micrographs illustrating the morphology of the precipitates after (a) PWDT, (b) PWNT and after HTE of 100 h, 500 h, 1000 h of PWDT heat-treated condition (c, e, g) and PWNT heat-treated condition (d, f, h). .... 116

Figure 6.12: (a) BSE micrograph of FGHAZ after HTE of 1000 h to show the distribution of precipitates and (b, c) EDS area mapping and line scan of precipitates chemical elements, respectively, as marked in Figure a..... 117

Figure 6.13: EBSD-IPF maps illustrating the influence of HTE on the grain size after PWDT (a, c, e, g) and PWNT (b, d, f, h) heat-treated condition. ....	119
Figure 6.14: HR-TEM bright-field micrographs of FGHAZ after HTE of 1000 h for (a) PWDT and (b) PWNT heat-treated conditions. ....	121
Figure 6.15: Graphs illustrating (a) number density of precipitates, (b) maximum size of precipitates and (c) grain size variation for HTE of PWDT and PWNT heat-treated condition. ....	121
Figure 6.16: Graphs illustrating hardness variation for HTE of PWDT and PWNT heat-treated condition. ....	123
Figure 6.17: Impression creep curves for PWDT and PWNT with and without HTE. ....	125
Figure 6.18: EBSD micrographs beneath of impression (a) IPF-map and (b) LAM map for PWDT-HTE-1000 h condition; (c) IPF-map and (d) LAM map, for PWNT-HTE-1000 h condition. ....	127
Figure 7.1: Micrographs of as-received material of (a) T91 and (b) Super304H. ....	130
Figure 7.2: Micrographs illustrating the microstructural variation across the weldment (a) Stereo micrograph illustrating the different regions of weldment, (b, c) FGHAZ, (d, e) CGHAZ from the T91 side, (f, g) WM and (h, i) HAZ of Super304H, after welding. ....	132
Figure 7.3: Micrographs illustrating the microstructural variation across the weldment (a, b) FGHAZ T91 side, (c, d) CGHAZ T91 side, (e, f) WM and (g, h) HAZ of Super304H side, after PWDT. ....	134
Figure 7.4: Micrographs illustrating the microstructural variation across the weldment (a, b) FGHAZ T91 side, (c, d) CGHAZ T91 side, (e, f) WM, (g, h) HAZ of Super304H side, for PWNT. ....	136
Figure 7.5: Chemical composition of precipitates for (a) T91 side, (b) WM, (c, d) HAZ of Super304H side, after PWDT. ....	137
Figure 7.6: EBSD micrographs (sample normal) illustrating (a, b) Phase map and IPF map of as-welded condition, (c) IPF map after PWDT, (d) IPF map after PWNT, of the weldment. ....	139
Figure 7.7: EBSD-IPF map (sample normal) of FGHAZ for (a) As-welded, (b) PWDT and (c) PWNT from the T91 side. ....	140
Figure 7.8: Grain size distribution on both sides of WM (a) CGHAZ-T91 side, (b) HAZ-Super304H, (c) Average grain size across weldment and (d) Precipitate size variation across weldment, with respect to heat treatment condition. Note: dashed lines are used to guide the reader. ....	142

Figure 7.9: (a) Hardness variation across the weldment, (b) Tensile stress-strain behaviour and (c) Photograph illustrating the fracture location; of as-welded, PWDT and PWNT. ....	144
Figure 7.10: Fractographs of (a) As-welded, (b) PWDT and (c) PWNT. The numbers in the figures indicate the following features 1. Dimples, 2. Cleavage facets, 3. Intermetallics, 4. De-cohesion, 5. Tear ridges. ....	145
Figure 7.11: BSE micrographs illustrate (a, b) Carbides on the fractured surface, (c) EDS spectrum of carbide on the fractured surface and (d) EBSD-IPF (sample normal) illustrating the grain size variation in WM for PWDT condition. ....	147
Figure 7.12: (a, b) Axial residual stresses and (c, d) Circumferential residual stresses for the T91 side and Super304H side. ....	148

## List of Tables

Table 2.1: Classification of power plants on the basis of different operating parameters [3] ...	6
Table 2.2: Candidate material for power plant for various steam condition [1].....	7
Table 2.3: Influence of PWDT and PWNT on mechanical properties of T91 welded joints ..	23
Table 2.4: Microstructure characteristics and mechanical properties of HAZs based on experienced temperature and their corresponding transformation reaction [38].....	26
Table 2.5: Mechanical properties of dissimilar welds for different PWDT conditions.....	32
Table 2.6: Residual stress distribution across the weldments.....	35
Table 4.1: Nominal composition (in wt.%) of initial materials and fillers .....	55
Table 4.2: Welding parameters considered for similar welds of T91 steel tubes .....	60
Table 4.3: Radiography results of DMW.....	61
Table 4.4: Illustrating PWHTs conditions for different DMW.....	64
Table 5.1: Tensile properties of as-welded and different PWDT conditions .....	85
Table 5.2: EDS point analysis on the marked locations in Figure 5.8.....	92
Table 6.1: Comparison of mechanical properties of PWDT and HTE 1000 h condition.....	113
Table 6.2: Creep rate of PWDT and PWNT with 1000 h HTE conditions from impression creep (present study) and tensile creep ([7]). .....	125
Table 7.1: Tensile properties of as-welded and different PWDT sample conditions .....	144
Table 7.2: Comparison of similar and dissimilar weld .....	149

## Glossary

<b>Abbreviations</b>	<b>Meaning</b>
PWHT	Post-weld heat treatment
PWDT	Post-weld direct tempering
PWNT	Post-weld normalizing and tempering
HAZ	Heat affected zone
WM	Weld metal
CGHAZ	Coarse grain heat-affected zone
FGHAZ	Fine-grain heat-affected zone
IC-HAZ	Inter-critical heat-affected zone
BM	Base metal
DCEN	Direct current electrode negative
DCEP	Direct current electrode positive
HTE	High temperature exposure
FESEM	Field emission scanning electron microscopy
EDS	Energy dispersive spectroscopy
EBSD	Electron backscattered diffraction
IPF	Inverse pole figure
LAM	Local area misorientation
XRD	X-ray diffraction
DMW	Dissimilar metal welding
PAGBs	Prior-austenite grain boundaries
HR-TEM	High-resolution transmission electron microscopy
IGC	Intergranular corrosion
SCC	Stress corrosion cracking
CE	Carbon equivalent
AWS	American welding society
IIW	International institute of welding

## Symbols

Symbols	Meaning
$P_{cm}$	Composition characterizing parameter
$Ac_1$	Lower critical temperature (°C)
$Ac_3$	Upper critical temperature (°C)
$\sigma_{imp}$	Mean impression stress (MPa)
$v_{imp}$	Impression velocity (mm/sec)
$\sigma_{uni}$	Uniaxial stress (MPa)
$\eta, \beta$	Correlation factors
$\dot{\epsilon}_{uni}$	Uniaxial strain rate (per sec)
$d_{\phi\psi}$	Interplanar spacing along $\phi$ and $\psi$ angles
$\epsilon_{33}$	Strain along $L_3$
$\sigma_{\phi\psi}$	Surface residual stresses for tilt angles $\phi$ and $\psi$ (MPa)
$\epsilon_{\alpha}, \epsilon_{\pi+\alpha}, \epsilon^{-\alpha}, \epsilon_{\pi-\alpha}$	Strains calculated using the Debye-Scherrer ring
$\sigma_{\phi}$	Residual stresses (Cos $\alpha$ method, MPa)
$2\eta$	Semi-Debye ring angle (degree)
$\psi_0$	Angle between sample normal and incident beam (degree)