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Characterisation of Weldability and Metallurgical Stability of Dissimilar Metal Welds

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ABSTRACT

Many industrial sectors require the joining of dissimilar metals, including the power plant industry where the different parts of a power plant are made of different materials due to differing requirements. The locations where the dissimilar steels or alloys are joined are termed dissimilar metal welds (DMWs). It is generally known that a significant percentage of DMWs can fail prematurely in service as a result of both primary system loads and constraints introduced due to differences in the coefficient of thermal expansion across the joint, as well as metallurgical compatibility issues.

Somers Forge Ltd and TWI introduced a novel TransForge fabrication technique which incorporates single-pass EBW and forging, followed by post-forge heat treatment, to produce a DMW joint between two thick sections of metallurgically dissimilar materials. This technique needs to be evaluated in order to assess the process that potentially allows welding with much lower distortion than achievable with conventional arc welding techniques and offers the potential to be of high integrity. Two DMW joints (A508/304L and A508/IN600) produced using this fabrication technique were evaluated using microhardness testing and tensile testing and the microstructure characterised.

Microstructural studies revealed that there are many microstructural zones in the vicinity of the interface, running parallel to the interface. In each of the joints examined, there are different microstructural features at the interface in different parts of the joints. Tensile testing at room temperature indicated that the tensile properties across the weld, at different parts of the joints exhibiting different microstructures, are comparably strong and ductile (or more so) compared to the bulk alloys, showing the viability of the fabrication process. In addition, A508 and 304L steels—which previously have only been joined with the use of electrodes/fillers in fusion DMW—have been directly joined successfully, demonstrating good weldability of this pair of dissimilar alloys when utilising the TransForge fabrication technique.

The most common type of DMW is, however, still fusion welding, particularly in the energy sector. Increasingly, newer 9Cr-1Mo steels such as P92 are used in power plants in order to allow the use of higher steam pressure and pressure, and thus the behaviour of P92 in DMW joints are increasingly studied. However, there tends to be some discrepancy between the failure modes of in-service components and those of accelerated creep-tested joints in labs. It is hypothesised that this may be due to the difference in the magnitudes and states of the applied stress and the durations of exposure to high temperature between the two situations. Precipitate growth is a potentially important factor that is affected by stress and high temperature that may in turn influence the failure modes of DMW, so the behaviour of precipitate growth under different stresses and durations of exposure to high temperature may have a role in DMW failure. To test this, the three DMW specimens from P92/IN182 joint were subjected to 154, 129, and 116 MPa of stress, at temperature of 630°C, which resulted in times-to-fracture of 109, 1453, and 5862 hours, respectively. Separately, additional joint specimens were subjected to thermal ageing for the same durations at the same temperature. The difference between these two sets of specimens are that the crept samples experienced mechanical stress during the exposure to the high temperature, whereas the thermally aged specimens did not.

It was found that while both the Laves phase and the Type I interfacial carbides (which occurs near the interface between a ferritic steel and a nickel alloy filler in fusion DMW) were affected by the applied stress, they are affected differently. Applied stress in crept samples likely accelerated the growth of Type I carbides in the minor axis direction, while the major axis dimensions of the Type I carbides appear to not be affected. High mechanical stresses in creep (as observed in the short duration creep sample) increased the number density of Laves particles, indicating greater Laves nucleation and formation, but may have decreased the coarsening rate. Type IV failure in the longer duration creep may be explained by this Laves behaviour in the fine-grained heat affected zone (FGHAZ).

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LIST OF ABBREVIATIONS

BSE	backscattered electrons
BCC	body-centred cubic
BCT	body-centred tetragonal
BPVC	Boiler and Pressure Vessel Code
BM	bulk metal
CALPHAD	CALculation of PHase Diagrams
CDZ	carbon denuded/depleted zone
CEZ	carbon enriched zone
CSEF	creep-strength enhanced ferritic (steel)
DICTRA	Diffusion-Controlled TRAnsformations
DMW	dissimilar metal weld
EDM	electrical discharge machining
EBSD	electron backscatter diffraction
EBW	electron beam welding
EDX	energy dispersive X-ray
FCC	face-centred cubic
FEG	field emission gun
FGHAZ	fine-grained heat affected zone
FEA	finite element analysis
FZ	fusion zone
GTAW	gas tungsten arc welding
HAZ	heat affected zone
ICHAZ	intercritical heat affected zone
LBW	laser beam welding
MMA	manual metal arc
PMZ	partially mixed zone
PFHT	post-forge heat treatment
PWHT	post-weld heat treatment
PDZ	precipitate depleted zone
PAG	prior austenite grain
PAGB	prior austenite grain boundary
SEM	scanning electron microscope
SE	secondary electrons
SMAW	shielded metal arc welding
SAW	submerged arc welding
TWI	The Welding Institute
TEM	transmission electron microscope
TIG	tungsten inert gas
UTS	ultimate tensile stress
wt%	weight percent (percentage by mass)

NOMENCLATURE

Symbol	Description	Units
A	nanoindentation contact area	m^2
C	concentration of an atomic species	m^{-3}
D	diffusion coefficient or diffusivity	m^2s^{-1}
E	Young's modulus	Pa
E_{eff}	effective Young's modulus	Pa
E_i	nanoindenter Young's modulus	Pa
F	number of degrees of freedom	-
G	Gibbs free energy	Jmol^{-1}
h	nanoindenter displacement	m
i, k	arbitrary labels identifying atomic species	-
J	diffusion flux	$\text{mol m}^{-2}\text{s}^{-1}$
K	strength coefficient	Pa
n	strain-hardening exponent	-
N	number of datapoints in each nanoindentation load-displacement curve	-
p	phase	-
P	load	N
P_{thermo}	absolute pressure	Pa
S_{thermo}	entropy	JK^{-1}
S	stiffness	Pa m^{-1}
T	absolute temperature	K
T_m	absolute melting temperature	K
U	internal energy of a system	J
V	volume	m^3
x	mole fraction of an atomic species	-
$\mathbf{x}$	vector containing values of inverse method optimisation parameters x_j	-
x_j	value of a material property j being optimised in the inverse method	Varies
x_j^L	lower limit of x_j set for the optimisation using the inverse method	Varies
x_j^U	upper limit of x_j set for the optimisation using the inverse method	Varies
β	correction factor related to the shape of nanoindenter	-
ϵ_e	elastic strain	-
ϵ_p	plastic strain	-
ϵ_T	total strain	-
μ	chemical potential	Jmol^{-1}
ν	Poisson's ratio	-
ν_i	nanoindenter Poisson's ratio	-
σ	stress	Pa
σ_y	yield stress	Pa
φ	number of phases in equilibrium	-
χ	number of components in a system	-

1 INTRODUCTION

Due to increasing worldwide concern for climate change, there is a strong push to limit and ultimately decrease greenhouse gases emissions, particularly carbon dioxide. In 2014, energy production is the leading source of carbon emissions in the UK, contributing 31% of the total [1]. Thus, one important way to achieve this is to increase the efficiency of power plants, i.e. generating more energy but using less fuel, which in turn will reduce carbon dioxide emissions in fossil fuel power plants. Here, efficiency can be increased thermodynamically by maximizing the peak temperature and pressure of the steam cycle.

However, this introduced new challenges as the power plant components need to be able to withstand such extreme temperature and pressure, but still being economical to fabricate. More specifically, the alloys utilized in these components are required to have sufficient service life in their respective service environments while the minimizing costs of production and maintenance. Thus, different parts of a power plant are made of different materials due to differing requirements. The high-temperature parts require materials with good corrosion and creep resistance at the high service temperatures and pressures, most suitable of which are austenitic metals, such as nickel alloys or austenitic stainless steels. They are relatively expensive, so they cannot be used throughout the power plant. Instead, the less expensive ferritic steels are used in the less critical (lower temperature and pressure) areas. All of these components are interconnected, so welding them together is necessary to create the full steam vessel. The weld joints between the different types of alloys are termed dissimilar metal welds (DMWs).

DMWs are thus commonly employed in power plants. However, peculiar issues that arise in DMWs are generally known to cause premature failures in service in power plants, i.e. the welds have well below the expected creep life of the base materials involved. Microstructural changes, atomic interdiffusion between alloys, as well as differentials in coefficient of thermal expansion, in high temperature service environment are well-known issues.

One well-known issue is the discrepancy between the locations of failure reported in service welds and the locations of failure in accelerated creep tests. Ex-service DMWs using nickel-based weld repeatedly point to Type II premature failure due to the formation of creep cavities around interfacial precipitates called the Type I carbides. But accelerated creep tests tend to cause Type IV cracking. Hence it is thought that for a given system of DMW at a given temperature, the location of failure may depend on the applied stress and the duration of creep. The reasons for this is not clear, but it has been suggested that a contributing factor may involve the effect of applied mechanical stress on precipitate development in a DMW system, but this has not been investigated widely, thus offering a gap in the knowledge in an important area.

Somers Forge Ltd and TWI introduced a novel TransForge fabrication technique which incorporates single-pass EBW and forging, followed by post-forge heat treatment, to produce a DMW joint between two thick sections of metallurgically dissimilar materials. This approach allows welding with much lower distortion than achievable with conventional arc welding techniques and offers the potential to be of high integrity. It is possible that this technique can increase the weldability of dissimilar alloys, i.e. more diverse pairs of dissimilar alloys can be joined directly without producing defects such as pores, voids or cracks, while achieving the level of strength and ductility of at least one of the bulk alloys. The manufacturing process will be faster and more cost-effective using this technique. If successful, this technique could be used to manufacture, for example, a transition joint that can replace the current design of nozzle-to-safe-end fusion DMW in nuclear power plants. The technique could also be utilised for the fabrication of forging tools used in the metallurgical sector. This novel fabrication technique has yet to be deeply characterised, so this can be the focus of this project.

The overall aim of this project is to investigate further the formation of detrimental microstructures within dissimilar metal weld (DMW), and to relate this formation to welding and post-weld heat treatment (PWHT) industrial practices as well as to extended high temperature ageing conditions.

In consultation with industrial partners, the objectives of this project are as follows:

1. To characterise materials from the TransForge project, examining the weldability of the materials and the feasibility of the innovative fabrication of dissimilar weld joints.
2. To characterise the microstructural evolution of DMW between P92 and IN82/IN182 under creep and thermal ageing, and investigate the effect of applied mechanical stress on microstructural evolution of DMW.

The thesis consists of the following chapters:

2. **Literature Review**, where the progress of previous research is outlined and sets the current situation regarding DMW in industry.
3. **Research Methodology**, which outlines the samples preparation, examination, characterisation techniques, and modelling techniques.
4. **Characterisation of A508/304L TransForge Joint**, where the microstructural and mechanical characterisation of a joint made using the TransForge fabrication method is presented.
5. **Characterisation of A508/IN600 TransForge Joint**, where the microstructural and mechanical characterisation of another joint made using the TransForge fabrication method is presented.
6. **Microstructural Evolution of Dissimilar Metal Weld Between P92 and IN82/IN182 Under Creep and Thermal Ageing**, where a particular fusion DMW that underwent creep or thermal ageing are characterised, allowing for comparison between specimens that were exposed to prolonged high temperature with applied stress and those without.
7. **Conclusions and Future Work**, where the main conclusions are detailed and directions for future work are presented.

2 LITERATURE REVIEW

2.1 Introduction

To provide the background and context to the research, it is important to understand the current research on metallurgy of alloys in power plants and the microstructural evolution occurring in typical fusion DMW under high temperature condition. A novel DMW fabrication method, TransForge, is then introduced as a way to address the issues associated with typical fusion DMW.

In order for materials to be considered fit for use in power plants, they must have acceptable strength, toughness, creep resistance (i.e. resistance to plastic deformation of materials under load at temperatures higher than $0.4T_m$, where T_m is the absolute melting temperature), and corrosion and oxidation resistance in their respective operating conditions. With these criteria in mind, these materials are expected to have a predicted life (without failure) on par with that of the power plant itself; if not, then at least be easily repaired or replaced. Materials for use in components in high temperature power plant environments are sometimes designed or selected based on the alloy's 100,000-hour creep rupture strength [2].

Typically, steels are used in power plants because of the relatively ample supply of iron and the general familiarity of iron in engineering applications, with its versatility to obtain a range of physical, chemical, and mechanical properties through process manipulations such as heat treatment; however, nickel-based alloys are also increasingly being used in power plants due to their excellent creep and corrosion resistance and other properties. For this project, nickel alloys are used as filler weld metals, as well as bulk materials in TransForge.

2.2 Steels

2.2.1 Plain carbon steel

All steels contain varying amounts of iron as the main element, and carbon as an alloying element. The simplest type of steel contains only these two elements, with the carbon content generally in the

range of 0.03–0.20 wt%. The phase diagram of iron-carbon binary system in equilibrium is shown in Figure 2.1. This plain carbon steel exists in the form of one or more of the following three stable phases in equilibrium, depending on temperature and carbon content:

- **Ferrite**, with a body-centred cubic (BCC) crystal structure. In Figure 2.1, the ferrite structure is shown to occur in the α and δ phases. Though these two phases exist in different temperature ranges, their crystallographic structures are identical.
- **Austenite**, with a face-centred cubic (FCC) crystal structure, occurring as the γ phase in the phase diagram.
- **Cementite**, also known as iron carbide, with an orthorhombic crystal structure and a chemical composition of Fe_3C . This is actually a pseudo-equilibrium phase, but is stable enough to warrant inclusion in the phase diagram. At low temperatures, cementite tends to exist in alternate layers with ferrite, in a lamellar fashion, to create a structure called pearlite.

The phases reported in phase diagrams are only those that occur in equilibrium; there are other phases that do occur in real alloys but do not appear in the phase diagram because these phases are metastable, i.e. it will degrade over time before the alloy reach equilibrium, but degradation is slow enough for the phase to exist in service. The most important metastable phase is the martensite, which is formed when steel in the austenite phase is quenched (rapid cooled) to room temperature. Without time to diffuse into cementite layers, the carbon is trapped in the ferrite lattice, forming a body centred tetragonal (BCT) structure. A similar phase called bainite is formed when a slightly slower cooling rate is applied.

Carbon, as an element with small atomic diameter, fills in the interstitial space between the metal atoms in the lattice until it reaches its relatively low solubility limit as a solid solution; the rest precipitates into carbides. Both the solid solution and the carbides act as a strengthening mechanism in steels; carbon in the solid solution matrix provides lattice strains that impede dislocation movements, while the carbides act to stop or pin dislocation movements.

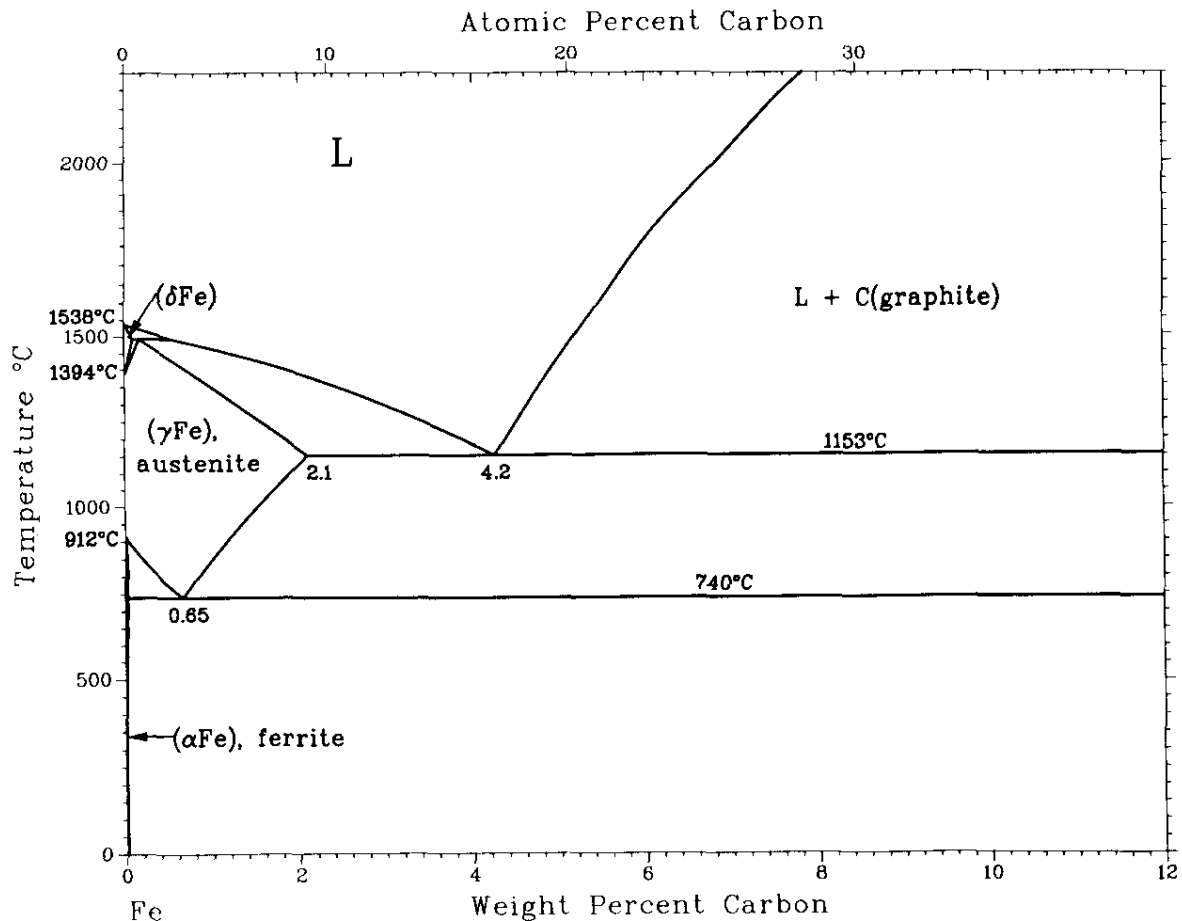


Figure 2.1: Equilibrium iron-carbon binary system phase diagram. Courtesy of ASM International [3].

Iron readily combines with oxygen to form oxides (Fe_2O_3 , FeO or Fe_3O_4), so plain carbon steel has low oxidation resistance. The iron oxide layer that forms on the surface can flake off, which exposes more iron to oxidation. Hence rust prevention methods such as coating or inclusion of other alloying elements are done to protect the steel.

2.2.2 Alloying elements and phases in steel

While plain carbon steel is strong and ductile, it is generally not adequate enough for most modern engineering applications. Hence, other alloying elements such as chromium, manganese, nickel, molybdenum, vanadium, copper, niobium, tungsten, cobalt and silicon are added to enhance steel to desired properties and design applications. The influence of alloying elements includes several important aspects: enhancing mechanical or chemical properties of the alloy matrix (by e.g. solid solution strengthening or corrosion resistance improvement), promoting certain type of crystal structure (FCC, BCC etc.) of the alloy matrix, or becoming the source elements for precipitates (which

would also enhance mechanical properties). Heat-resistant alloys in particular depend on precipitate phases such as carbides and intermetallic phases, whose stability at high temperatures is an important factor in alloy design due to their effects on mechanical properties in high temperature conditions.

The roles of each element in steel are described below:

- **Nitrogen**, like carbon, is also used for strengthening steels. Its role is generally analogous to that of carbon: nitrogen atom fills in the interstitial space in the metal lattice and also precipitates into nitrides or carbonitrides in the presence of some alloying elements, leading to possible improvement of mechanical properties.
- **Chromium** is among the most important alloying element in steels and is used to enhance the corrosion resistance of steel (including at high temperature) and to form carbides. The increase in corrosion resistance is due to the formation of a slow-moving and self-repairing film of chromium oxide on the surface, which acts as protective barrier that prevents further oxidation of the steel. Chromium can also form carbides, such as $M_{23}C_6$. The importance of chromium means that steels for steam vessels almost always contain some level chromium, with stainless steel alloys for higher temperature applications having greater amounts of chromium content.
- **Nickel** is mainly used to promote the stability of the austenite phase so that it can be conserved at lower temperatures. The solid solution strengthening properties of nickel also generally increases strength, ductility and toughness.
- **Manganese**, like nickel, is an austenite stabilizer at low temperature, though at high temperatures it will stabilize ferrite. As a result, manganese can replace some of the nickel, though greater amount is needed to achieve the same effect. Manganese also increases the solubility of nitrogen in steels, which means that a greater manganese content can lead to higher nitrogen contents in steels (especially austenitic stainless steels).

- **Molybdenum** is used to increase resistance to corrosion, and also increases mechanical strength due to solid solution strengthening. It also tends to promote the ferritic microstructure in steels. Molybdenum may also lead to the formation of secondary phases in ferritic and austenitic steels, such as the intermetallic Laves phase $(\text{Fe,Cr})_2(\text{Mo,W})$.
- **Tungsten** can be used as a supplement to or substitution of molybdenum in some alloy systems because the roles are similar, especially with regards to high temperature stability of carbides such as M_6C [4, 5].
- **Vanadium** and **niobium** strongly promote the formation of carbides and carbonitrides, the most common of which is the fine so-called MX-type carbonitrides, which enhances the creep strength and other mechanical properties of various high performance steels [6, 7].

As can be seen, the inclusion of carbon, nitrogen, and other alloying elements may lead to the formation of several carbides and carbonitrides in the steels, some of which are previously mentioned.

These precipitates are:

- M_3C (cementite), where M is usually Fe, can be found even in plain carbon steel, as mentioned previously, though in steels with chromium content this phase is less stable and does not form in equilibrium.
- MX, where 'M' is usually vanadium and/or niobium and 'X' is carbon and/or nitrogen, has an FCC crystal structure. The presence of this precipitate greatly improves the mechanical properties of the creep-strength enhanced ferritic (CSEF) steels.
- M_6C (η -carbide) has an FCC structure. M is predominantly molybdenum, though it can also be iron, chromium, nickel or silicon.
- M_{23}C_6 , where M is predominantly chromium, but may also be iron and other elements. This chromium-rich carbide commonly exists in equilibrium in steels with significant chromium content.
- M_7C_3 , like M_{23}C_6 , is also a chromium-rich carbide.

- M_2C is a carbide with M being predominantly molybdenum, tungsten, and/or chromium [8]. This carbide forms in low-alloy steels. In steels with higher molybdenum and chromium, $M_{23}C_6$ forms more favourably.
- Z-phase is a nitride consisting of $Cr(V,Nb)N$ that forms in steels with significant nitrogen content. Greater chromium content allows more favourable formation of the Z-phase [9]. The Z-phase forms and grows at the expense of MX carbonitrides, and is detrimental to the creep strength of CSEF steels.
- Laves is an intermetallic consisting of $(Fe,Cr)_2(Mo,W)$ that forms in steels with significant molybdenum and/or tungsten content. The formation and growth of Laves depletes molybdenum and tungsten from the matrix, thus reducing solid solution strengthening.

2.2.3 Ferritic/bainitic steels

Table 2.1: Limiting compositions of common ferritic/bainitic steels designed for use in power plants.

Element	Amount (wt%)			
	P22	P91	P92	A508
Al	—	0.04 max	0.04 max	0.025 max
B	—	—	0.001-0.006	0.003 max
C	0.05-0.15	0.08-0.12	0.07-0.13	0.25 max
Ca	—	—	—	Ca 0.015 max
Cr	1.9-2.6	8.0-9.5	8.5-9.5	0.25 max
Cu	—	—	—	Cu 0.2 max
Fe	Bal	Bal	Bal	Bal
Mn	0.3-0.6	0.3-0.6	0.3-0.6	1.2-1.5
Mo	0.87-1.13	0.85-1.05	0.3-0.6	0.45-0.60
N	—	0.03-0.07	0.03-0.07	—
Nb	—	0.06-0.10	0.04-0.09	0.01 max
Ni	—	0.4 max	0.4 max	0.4-1.0
P	0.025 max	0.02 max	0.02 max	0.025 max
S	0.025 max	0.01 max	0.01 max	0.025 max
Si	0.5 max	0.2-0.5	0.5 max	0.4 max
Ti	—	—	—	0.015 max
V	—	0.18-0.25	0.15-0.25	0.05 max
W	—	—	1.5-2.0	—

Ferritic/bainitic steels are commonly employed in most industries because they have better properties than plain carbon steels while having relatively low concentrations of alloying elements, so generally have lower cost to produce compared to some other types of steels (e.g. stainless steels). The limiting compositions of ferritic/bainitic steel alloys of interest to this project are outlined in Table 2.1.

2.2.3.1 P22

P22 steel, also known as 2.25Cr-1Mo, has been widely used in temperatures up to 560°C in powerplants for decades. Unlike the other ferritic steels considered in this project, it is a bainitic high-strength low-alloy steel. Chromium gives the oxidation resistance, while the molybdenum increases the strength of steel. There also has been extensive research of DMW utilising this metal as the ferritic part of the joint. As a result, most reports on DMW of ferritic steels involve P22 as a bulk metal, so they form a significant part of the literature review of DMW, even though this alloy will not be used in samples for this project.

2.2.3.2 P91

Demands to increase the temperature and pressure of power plant operations in order to improve efficiency led to efforts to design a better steel alloy. Eventually, the CSEF steels containing 9-12wt% chromium were developed. The reason why this chromium composition range is desired is that it maximises the chromium content of the steel (which maximises the corrosion and creep resistance etc.), while at the same time still allows the alloy to fully transform to FCC crystal structure (γ -austenite) at certain high temperatures. This ability is illustrated in the iron-chromium phase diagram in Figure 2.2, which shows that fully austenitic γ phase is only possible up to around 12 wt% Cr. The CSEF grade most commonly used in power plants is P91 [10].

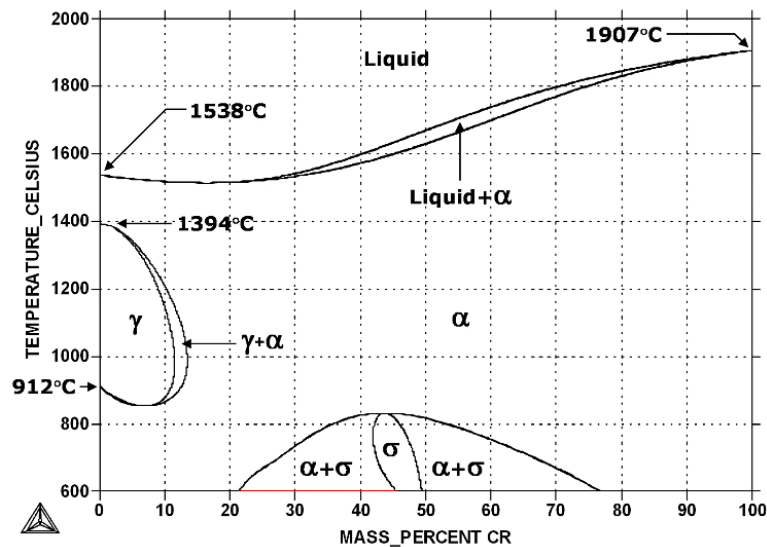


Figure 2.2: Iron-chromium phase diagram [11].

In service, P91 is preferred to have tempered martensite microstructure, which is the reason why the full transformation to austenitic phase is desired. This is obtained during the fabrication of the joint using stages of heat treatments. It is first normalised to around 1050-1100°C for full austenite transformation, followed by quenching to form martensite. Afterwards, the steel is tempered at 730-780°C for a number of hours to decrease residual stresses and to encourage the formation of precipitates such as $M_{23}C_6$ and MX in order to increase creep rupture strength to be useful in service [12]. In welding applications, the steel is post weld heat treated (PWHT) to re-temper the weld-induced martensite. Even with this, however, the heat-affected zone (HAZ) in P91 is reported to be problematic in welds [13-15].

The formation of precipitates are generally beneficial because their placement in prior-austenite grain boundaries (PAGB) and lath boundaries contribute to creep strength, as long as they do not coarsen excessively. The Laves formation is somewhat detrimental due to the removal of molybdenum from the alloy matrix, which decreases solid solution strengthening.

Unfortunately, with the presence of chromium, the MX phase is not thermodynamically stable at high temperature, causing it absorb chromium and transform to Z-phase. The vanadium nitride (VN) is the type of MX more prone to transform into Z-phase due to favourable crystal structure [16]. Z-phase coarsens into large particles, so its formation would strongly lead to a net decrease in creep strength

due to the replacement of fine and dispersed MX phase (especially VN) with a much coarser phase. Hence the development of Z-phase thought to have a strongly detrimental effect on the creep strength. This is especially a concern for CSEF alloys with relatively high chromium concentrations, because there would be more Cr available to feed into the Z-phase growth [17]. Nonetheless, for P91 or other 9%Cr steels the formation of modified Z-phase is relatively small in the pertinent time scales; only low amount of modified Z-phase is observed even after over 100,000 hours of creep at 600°C [18].

As for the $M_{23}C_6$ and Laves precipitates, there are still potential to coarsen—by Ostwald ripening—and thus decrease the creep resistance [19]. $M_{23}C_6$ and Laves precipitates particles that coarsen beyond 0.5 μm are detrimental [20]. The fine MX phase is less of a concern because of its coarsening resistance, though again must be prevented from excessively transform to Z-phase. Nevertheless, the main microstructural degradation mechanisms of 9%Cr heat resistant steels during long-term creep and/or aging are Laves phase precipitation and recovery of the matrix rather than precipitation of Z-phase [18].

2.2.3.3 P92

P92 steel is an improved version of modified P91 steels, with the most notable change being the addition of 1.8 wt.% tungsten and a small amount of boron while the molybdenum content is slightly decreased. Like P91, P92 is strengthened by tempered martensite structure stabilized by $M_{23}C_6$ carbide and MX carbonitride [20, 21]. However, in P92 the creep strength is further improved because the incorporation of tungsten leads to greater solid solution strengthening and hardenability [22]. The molybdenum content is reduced to around 0.5wt% in order to suppress the increasing tendency to the formation of delta ferrite.

The major difference in precipitate characteristics in P92 compared to P91 is that the composition of the Laves phase is now $(\text{Fe,Cr})_2(\text{Mo,W})$, which is different from P91 due to incorporation of tungsten in P92. The incorporation of tungsten further stabilises this phase, which leads to a greater amount

appearing in P92 [23]. The addition of small amount boron leads to improved coarsening resistance of the $M_{23}C_6$ phase and perhaps also promotes grain growth in FGHAZ, which improves welding properties [24], though it is essential to limit the boron content in order to avoid the formation of BN particles [25].

2.2.3.4 A508

A508 Grade 3 (a.k.a SA508 Gr.3, SA508 Cl.3, 16MND5, etc.) is a Mn-Ni-Mo low-alloy bainitic steel used in the nuclear power plant industry, mainly for structural and other relatively low-intensity (moderate temperature and pressure) environments. According to ASME Boiler and Pressure Vessel Code (BPVC), A508 steel is to be utilised in nuclear reactors up to 371°C/700°F [26].

The microstructure in service is mainly tempered bainite after being quenched and tempered (at 650-700°C) in the fabrication process. Various precipitates can form during the tempering process, the most abundant of which is cementite, forming as long-rod and spherical particles in the inter-lath region. Another important carbide is the needle-like β - Mo_2C carbide, which are nano-sized (0.02 μ m thick 0.3 μ m long) and forms in bainitic ferrite laths. The carbides, mostly cementite, are shown to create voids that allow the initiation of fracture [27]. Other possible phases that may occur during tempering include MC-type carbides (such as MoC) and KSI-carbides ($Fe_{11}Mo_6C_5$ or Fe_2MoC) [28, 29].

Sulphur, which exists in all steels to some degree as an undesired impurity, may combine with manganese to form the MnS inclusion, which has been shown to be the primary cause of large void nucleation in nuclear pressure vessel steels [30], alongside cementite mentioned above.

2.2.4 Austenitic stainless steels

All stainless steels have a chromium content of at least ~12 wt%, higher than those of ferritic steels as reported before, in order to have the oxidation resistance that makes the steel 'stainless'. Some also have high nickel content, which then causes the austenitic crystal structure to be maintained at low temperatures, instead of transforming to ferrite (or martensite). Austenitic stainless steels are more

resistant to corrosion and oxidation, as well as having superior creep resistance. Due to relatively large amount of these alloying elements, however, the cost of production is higher compared to ferritic steels, so the usage of these alloys is limited to critical components at very high temperature conditions. Sometimes stainless steels are also used as filler metals in DMW, though disadvantages such as the large gap in coefficient of thermal expansion between them and the ferritic base, and other issues, mean that other filler metals (especially as nickel-based alloys) are preferred.

The stainless steel used in this project is 304L, the composition of which is outlined in Table 2.2.

Table 2.2: Limiting composition of austenitic stainless steel 304L.

	Amount (wt%)								
	C	Cr	Fe	Mn	N	Ni	P	S	Si
304L	0.03	16.00-18.0	Bal	2	0.1	8.00-12.00	0.045	0.03	0.75

304L is an austenitic grade stainless steel containing 18Cr-10Ni and low carbon (hence 'L') content. The low carbon content is intended to minimise the precipitation of chromium carbide (of the $M_{23}C_6$ type) at the temperature range of $\sim 500^{\circ}\text{C}$ - 900°C , which is commonly encountered in welding operations, which can lead to intergranular corrosion (phenomenon known as sensitisation). Typically, no other precipitates types form, so unless there is $M_{23}C_6$ present, 304L is a single-phase alloy.

Efforts to eliminate the need for austenitic stainless steels, to be replaced by improved CSEF steels, in order to cut costs as well as to decrease the amount austenitic steel-to-ferritic steel weld joints in power plants have so far been unsuccessful due to the unexpected failures of CSEF steels in service [15], so austenitic stainless steels will continue to be used for the foreseeable future.

2.3 Nickel-based alloys

Nickel-based alloys are common in applications involving high temperature and high stress, such as the power plant industry, due to good mechanical properties and corrosion resistance at high temperature, surpassing that of stainless steels in most conditions. An important and common

austenitic nickel-based family of alloys is the Inconel range, which are predominantly composed of nickel and chromium as the primary and secondary elements, respectively (with Ni >50 wt% and Cr >15 wt%). Even more so than austenitic stainless steels, the high cost of production limits their use to only critical components and sometimes as coating of steels for corrosion resistance.

Nickel-based alloys are also widely used as filler materials in DMW joints because their coefficient of thermal expansion is between the ferritic steel and the austenitic stainless steels, but closer to the ferritic one, as well as other beneficial properties. This is important because the interfacial region between the ferritic bulk and the weld will already be the weakest point in the joint; so the fact that the distortions experienced by the two alloys are similar after processes such as welding helps to prevent this distortion phenomenon from being an additional factor that further contributes to failure. There are also other advantages of using nickel-based alloy filler, such as decreased carbon migration between the dissimilar alloys.

2.3.1 Inconel 82/182

Hot cracking has been a major issue for the integrity of weldments in high temperature applications, especially the nuclear power industry. Research conducted from the late 1940s for weldments with better integrity eventually gave rise to welding metals that became Inconel Welding Electrode 182 (AWS A5.11 ENiCrFe-3) and Inconel Filler Metal 82 (AWS A5.14 ERNiCr-3) [31]. These weld metals are part of the 600 Series Ni-alloy filler metals. They are the most commonly used filler metals for DMW applications, in austenitic-ferritic steels joints as well as to join them to high-nickel alloys. They are also considered to be among the best weld materials for creep or high temperature applications.

The difference between the 182 and 82 is that the 82 filler is used in gas tungsten arc welding (GTAW), also known as tungsten inert gas (TIG) welding, whereas 182 electrode is used for shielded metal arc welding (SMAW), also known as manual metal arc (MMA); otherwise the properties of these welds are similar. The limiting compositions of these metals are listed in Table 2.3. It is common to utilise

both of these alloys in a single weld joint, certainly when the joint fabrication procedure involves both TIG and SMAW welding.

Table 2.3: Limiting compositions of nickel-based weld metals commonly used in DMW.

Element	Amount (wt%)	
	In 182 (ENiCrFe-3)	In 82 (ERNiCr-3)
C	0.10 max	
Cr	13.0-17.0	18.0-22.0
Cu	0.50 max	
Fe	10.0 max	3.0 max
Mn	5.0-9.5	2.5-3.5
Nb+Ta	1.0-2.5	2.0-3.0
Ni+Co	59.0 min	67.0 min
P	0.03 max	
S	0.015 max	
Si	1.0 max	0.50 max
Ti	1.0 max	0.75 max
Others	0.50 max	0.50 max

Klueh and King [32] reported that, in long-term high temperature exposure, the formation of precipitates causes the weld metal to increase in strength but reduce ductility, but it was only recently that Siressha et al. [33] identified these precipitates as $M_{23}C_6$, Ni_3Ti (γ' intermetallic phase), and NbC . Siressha et al. [33] also showed that in a Charpy impact test, these precipitates only appear at the fracture surface in aged welds and not in the as-welded condition. While both the as-welded and aged welds exhibit microvoid coalescence, the precipitates that form in aged welds can accelerate the microvoid coalescence by allowing more decohesion between the particles and the matrix, thus contributing to premature failure. It should be noted, however, that despite these significant changes in strength and ductility, the ductility and toughness is still relatively high.

2.3.2 Inconel 600

IN600 (Ni-15Cr-8Fe) is a standard nickel superalloy used for its good corrosion resistance in the chemical and nuclear industries, due to its particularly high Ni content (minimum 72 wt%), the increase

of which has been shown to increase stress corrosion cracking resistance [34, 35]. According to Special Metals [36], the only precipitated phases present in the microstructure are chromium carbides (in the form of $M_{23}C_6$ and M_7C_3). The chromium carbides can form on the grain boundaries (intergranular) and within grains (intragranular). Intermetallic phases like γ' , while common in most nickel-based alloys, are *not* present in IN600 at any temperature due to the lack of Al and Ni.

2.4 Interface between ferritic bulk alloy and austenitic weld in fusion dissimilar metal welding

The austenitic to ferritic weld joints are the most relevant DMW joints in this project. Since most of the failure in these DMWs occur in the vicinity of the ferritic-weld interface as well as the ferritic bulk, the examination of the interface between ferritic bulk alloy and the weld—as well as adjacent regions in either side—are prioritised.

It is important to understand the properties of the DMW in as-welded state for the weld metal and ferritic metal, as these will affect the microstructural evolution of the DMW joint during the subsequent heat treatments. During the heat treatment, DMW experiences microstructural evolution when exposed to high temperature for a prolonged period of time, with particular attention paid to carbon migration/diffusion and interfacial precipitate formations and their consequences. These in turn affects the failure modes of DMW.

2.4.1 Properties of as-welded joint

The most common form of DMW is fusion welding, which involves melting the weld metal onto the bulk metal [37-39]. The distinct microstructural zones that appears in fusion DMW is illustrated schematically in Figure 2.3.

In the fusion zone (FZ), any melted portion of the bulk metal is completely and homogeneously mixed with the weld metal, so the composition is usually macroscopically uniform throughout. But near the boundary, however, the melted fluid experiences some sort of stagnation and does not homogeneously

mix with the bulk metal alloy; this is the partially mixed zone (PMZ). Next to it, on the other side of the weld line/interface, the bulk metal is heated to a high temperature due to the conduction of heat from the weld, which causes microstructural changes, but below the solidus, so no melting occurs; this is known as the heat affected zone (HAZ).

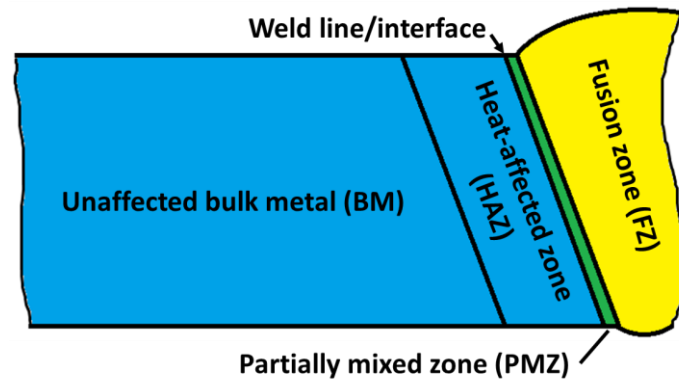


Figure 2.3: Schematic illustration of microstructural zones in the ferritic side of fusion DMW joints.

2.4.1.1 Properties of weld metal in the as-welded condition

As mentioned, the mixing between the weld and bulk metals in PMZ is incomplete and heterogeneous, so the composition in this zone varies as a function of position; the composition gradually changes from that of the bulk metal to that of the weld metal, as shown in Figure 2.4. This composition gradient plays a significant role in the microstructural evolution and thus service performance. While the width of PMZ can vary as a function of welding variables such as heat input and filler feed rate, the zone always exists in fusion DMW [40, 41], regardless of the alloys involved; in general, however, the width of PMZ in steel-nickel welds is found to be less than 50 μm .

The weld metal typically solidifies in directional manner from the bulk metal out, with a dendritic structure growing in the FCC $\langle 100 \rangle$ crystallographic direction [42, 43]. The fact that the weld metal has an FCC structure causes the weld crystal that initially solidified on the bulk ferritic steel to orientate differently with respect to the BCC bulk metal (because the un-melted ferritic steel material that is close to the melting point next to the PMZ would have a δ -ferrite BCC crystal structure). As the bulk metal cools and transforms to FCC austenite, however, some of the weld crystals adjacent to the bulk

metal rearrange to fit into the FCC crystal structure, leading to an apparent shift in the boundary between the bulk and weld metals, i.e. the FCC crystal structure ‘expands’ into the bulk metal.

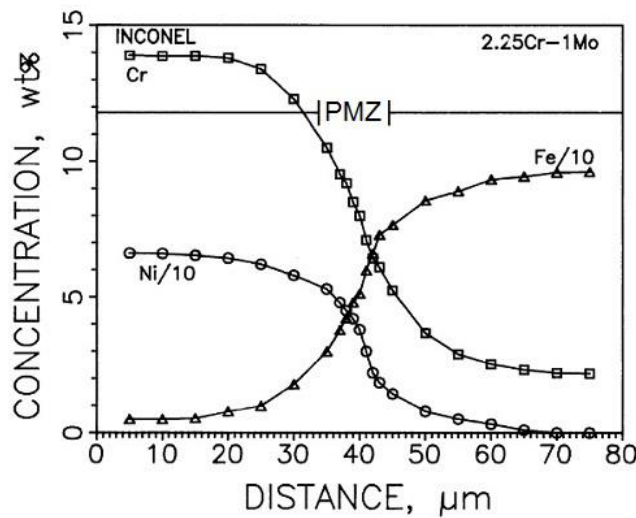


Figure 2.4: Distribution profile of major alloying elements in the vicinity of PMZ in DMW joint, between the Inconel 182 weld metal and P22 steel. The zero position of the horizontal axis is arbitrary. Courtesy of [44].

2.4.1.2 Properties of ferritic bulk metal in as-welded condition

The HAZ shown in Figure 2.3 needs to be broken down into more detailed classifications in order to be useful for analysis. This is shown in Figure 2.5, where the iron-carbon phase diagram aids the identification of phases at various locations in the vicinity of the weld, as a function of the maximum local temperature caused by the welding process. The diagram shows the following regions:

- Coarse-grained heat affected zone, or CGHAZ. The upper part of this zone, while not melted at all, has experienced the highest temperatures during welding, during which both the δ -ferrite (fine-grained) and γ -austenite (coarse-grained) phases appear. The δ -ferrite, although fine-grained, is detrimental to the local strength because it does not form martensite upon fast cooling (recall that the ideal microstructure of P91/P92 is tempered martensite), since no allotropic phase transformation can occur between δ -ferrite and α -ferrite [45]. The lower temperature part of CGHAZ contains only the γ -austenite phase (because it is within the austenite-only region), but the grains here are also coarse because this high temperature environment encourages their growth by the complete dissolution of carbide particles that

would otherwise restrict the growth. When the zone is cooled quickly after the weld pass near the region, martensite laths form.

- Fine-grained heat affected zone, or FGHAZ. The transition between CGHAZ to FGHAZ is gradual. Like in the lower CGHAZ, the region fully transforms to γ -austenite phase; however, the grains fail to coarsen significantly like in CGHAZ because the carbide phases do not dissolve completely at this lower temperature (and thus impede grain growth) meaning the austenite grains here are relatively fine. Hence, when the zone is cooled quickly after the weld pass near the region, the martensite laths in this region are smaller than in CGHAZ (and also smaller than in the bulk metal, because recall that the parent P91/P92 material was essentially 'normalised' to the lower CGHAZ during its fabrication heat treatment). Since the carbides are not fully dissolved, the metal matrix has relatively lower carbon content, meaning that the martensite that forms would be less distorted (i.e. less tetragonality) compared to CGHAZ and bulk metal.
- Intercritical heat affected zone, or ICHAZ. This region partially transforms to austenite, with small γ -austenite grains forming and growing within the original tempered martensite structure of the bulk metal. Like in FGHAZ, the austenite grains here have relatively low carbon content. The part of the region that does not transform to austenite retains the martensite structure from the previous fabrication heat treatment, but has been over-tempered.
- Over-tempered region. This is the region exposed to a temperature that is too low for any austenite to form, but above tempering temperature (approximately 760°C for P91/P92), so it is over-tempered. This causes dislocation annihilation and also greater local carbon diffusion, which leads to excessive ferrite recovery and carbide coarsening, lowering the local strength of the material.

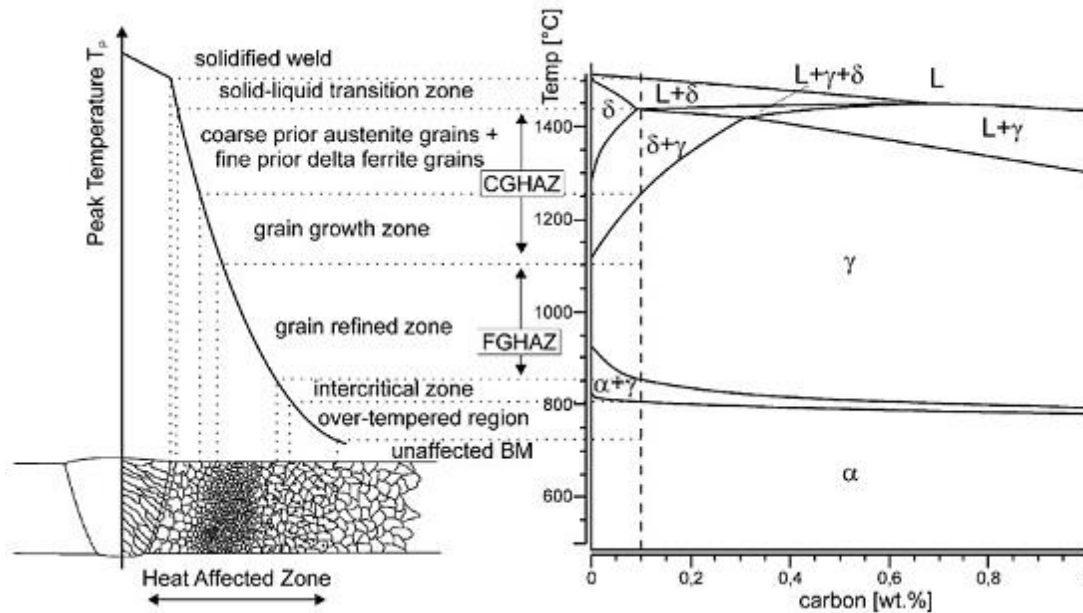


Figure 2.5: Microstructural regions/zones in P91 steel in the vicinity of the weld, shown to be linked to the maximum local temperature during the welding process by using the iron-carbon phase diagram [46].

2.4.2 Microstructural evolution during ageing

As there are compositional gradients between the different alloys of a DMW joint, there would be chemical potential gradients of elements across the weld lines, which will be the driver for the diffusion process. Hence, diffusion of these materials across the weld lines, termed interdiffusion, are expected to occur when exposed to high temperature for an extended period of time, and higher temperature leads to an increased diffusion rate. This is especially true for carbon (and nitrogen), because they are small and occupy the interstitial spaces within the matrix of either alloy, so they have greater freedom to move and diffuse.

2.4.2.1 Carbon diffusion and migration

As mentioned, carbon has a relatively fast diffusion rate. Since carbon is known to have significant influence on the microstructure and thus mechanical properties of metals, the diffusion of carbon is of fundamental importance when dealing with DMW; as such, it has been extensively studied.

The constitutive law for diffusion states that elements diffuse down a chemical potential gradient. Chemical potential gradient in turn is usually proportional to the concentration gradient. Hence,

elements tend to diffuse from areas of high concentration to areas of low concentration. Assuming steady state diffusion in one dimension, this is described by Fick's first law:

$$J_k = -D \frac{\partial C_k}{\partial x} \quad 2.1$$

where J_k is the diffusion flux of species k , in amount of substance per unit area per unit time; D is the diffusion coefficient or diffusivity, in area per unit time; C_k is concentration of species k in amount per unit volume; and x is the position along the diffusion gradient, the dimension of which is length.

However, chemical potential gradient is also heavily influenced by the presence of different elements within the alloy system. This is especially true for carbon, whose chemical potential was demonstrated to be affected by the presence of silicon [47]. Silicon sufficiently increases the chemical potential of carbon to cause it to diffuse up its concentration gradient.

Chromium, manganese, and molybdenum have also been found to decrease the chemical potential of carbon. The role of chromium is especially important, since ferritic and austenitic alloys contain significantly different levels of chromium, which leads to very different chemical potentials between the two alloys. It is shown, for example, that the level of chromium in a high-alloy steel directly impacts the decarburisation level in a low-alloy steel joined to it [48]. It was also found that carbon diffusion from ferritic steel to austenitic weld metal is less pronounced when P91 is used as the ferritic component, compared to using P22, partly because the chromium level difference across the weld interface is less when using P91 (P91 has higher chromium level than P22) [33]. This phenomena has since been observed in other ferritic-austenitic joints as well [49, 50].

Metals with an FCC crystal structure (e.g. the austenitic alloys) generally have shorter diffusion penetration distance compared to BCC metals (e.g. the ferritic alloys). FCC metals with high nickel content are especially effective in reducing the diffusion distance, due to reduced atomic mobility in the nickel matrix, so carbon diffusion in nickel-based alloys is significantly less extensive than in

austenitic steels [51, 52]. This means that it is advantageous to utilise nickel-based alloys as weld metals in order to minimise carbon migration.

2.4.2.2 *Carbon enriched and denuded zone*

When carbon diffuses through the interface between the two alloys against its concentration gradient, the carbon content at the low-alloy side of the interface would be depleted, whereas on the other side, the carbon content is augmented. This creates the carbon denuded/depleted zone (CDZ)—also referred as decarburized layer—in the low alloy part of the interface, and a carbon enriched zone (CEZ) in the austenitic side of the interface.

Figure 2.6 illustrates this phenomenon in the interface between ferritic P22 steel and an Inconel filler weld that had been in service. It shows a decreased carbon concentration in the ferritic side close to the interface; on the other side of the interface, the carbon concentration spikes to such a high level that it significantly exceeds the carbon solubility of the weld metal. This causes carbide precipitates to form in the CEZ. The precipitates that usually form in this zone are identified to be $M_{23}C_6$ and M_6C carbides [41, 53, 54]. On the other side, the CDZ exhibits the dissolution of carbide to supply the carbon for diffusion (because carbides themselves are immobile, so carbon needs to be freed from the carbides before it can diffuse), which also leads to local grain growth due to the removal of the pinning effect of carbide precipitates on the grains.

The choice of alloys in DMW affects the extent to which carbon migration, and thus CDZ and CEZ, occurs. For the ferritic side, it is found that P91 experiences less decarburised region compared to P22, as mentioned earlier. For the austenitic weld side, it has been found that the decarburized layer in the ferritic side of the joint is far less severe when using a nickel-based weld metal, compared to an austenitic stainless steel weld metal, because the diffusion is reduced to the extent that the decarburized layer does not appear or is very small [53, 55, 56].

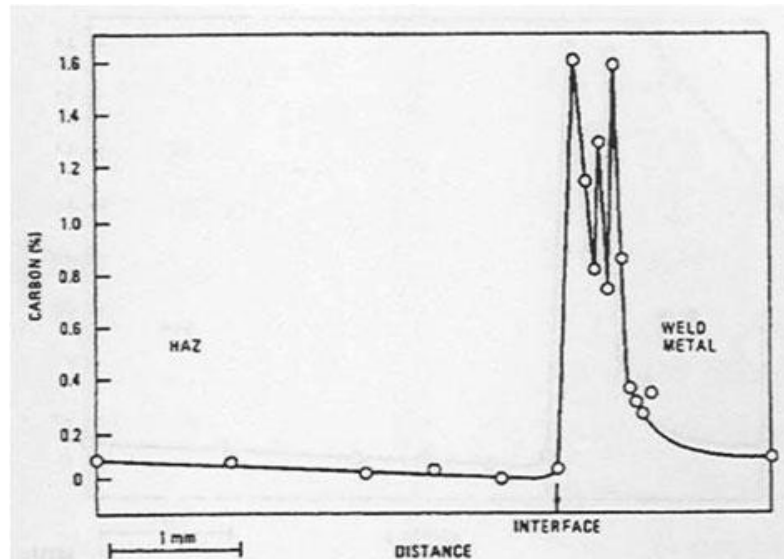


Figure 2.6: Carbon concentration profile in the weld interface between P22 steel and an Inconel weld metal that has been in service. Courtesy of [57].

2.4.2.3 Precipitation formation near weld line

One phenomenon unique to the ferritic-austenitic system that is not observed in, say, ferritic-ferritic DMW, is the formation of carbide bands adjacent to the weld interface in the ferritic side during thermal ageing. The formation of these carbides is strongly influenced by the welding fabrication process, high temperature conditions, as well as compositions of both alloys in the joint.

These interfacial carbide bands are recognised to contain two distinct morphologies [58, 59], examples of which are shown in Figure 2.7:

- Type I carbides – these carbides form in a single line along (but not on) the weld interface in the ferritic side of the steel. They initially form in discrete spherical shape, but gradually become more lenticular (whose dimensions can be defined by major and minor axes) [60], which allows them to join or coalesce with each other and thus form a continuous line of carbides.
- Type II carbides – a wide band of fine lath-like structures

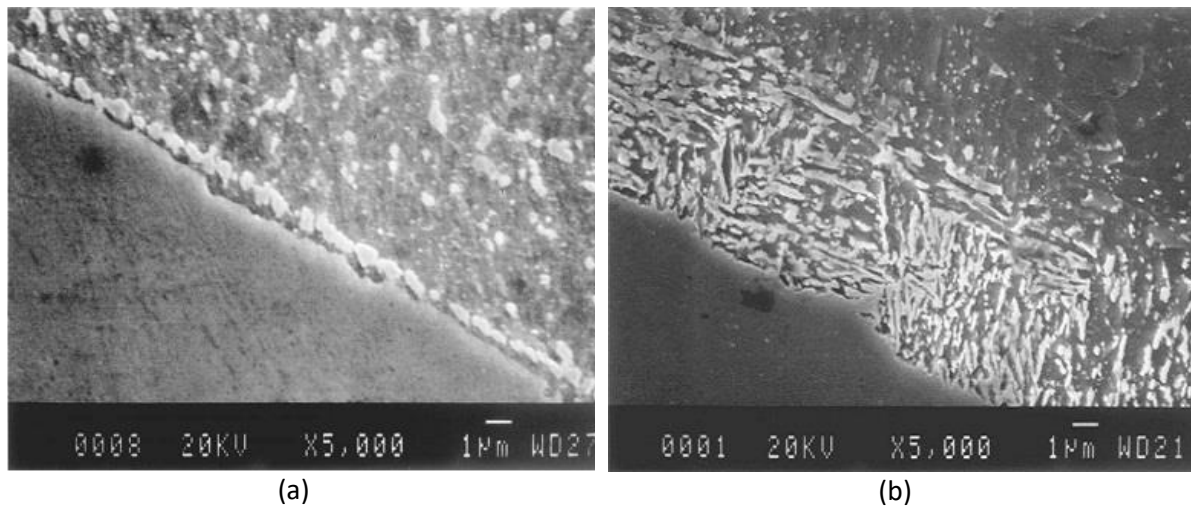


Figure 2.7: Scanning electron micrographs of the two carbide morphologies that are observed at the DMW interface between the P22 steel and Inconel 182 after aging: (a) Type I carbide and (b) Type II carbide [60].

Type I carbides are the most frequently observed, and it is the predominant type carbide that appears in DMW using nickel-based alloy fillers. Type II carbides are rarely observed in DMW using nickel-based alloy fillers, mostly only appearing in DMW using stainless steel fillers due to the wider interfacial martensite region in stainless steel welds. Hence, investigations regarding nucleation, growth, and morphological changes of Type I carbides are given priority and have been studied substantially.

Regarding combinations of alloy systems and their impact on the precipitates, Type I carbides in welds of P22 steel using 316 stainless steel filler are observed to contain two groups of carbides: the chromium-rich $M_{23}C_6$ carbide and the molybdenum-rich M_6C carbide [61]. When welding Inconel 625, however, only $M_{23}C_6$ is observed [62]. Type I carbides have also been observed in 9Cr-1Mo steels (i.e. P91/P92), though these carbides appear to be smaller than in P22 joints [63], and the precipitates that form there may include the Laves phase as well as $M_{23}C_6$ carbides in a joint with IN625 [64].

2.4.2.4 Failure modes of DMW

The microstructural evolution during welding fabrication and ageing discussed above has significant effects on the mechanical performance of DMW, making the DMW joint weaker than the unaffected bulk metals. One of the most important measurable mechanical properties relevant to this is the hardness of the material. The carbon migration that results in the formation of the CEZ and CDZ directly affects the local hardness of the materials. It is well known that CEZ has high hardness relative

to the bulk alloy due to the formation of large amount of precipitate (carbide) phases, while the CDZ has relatively low hardness due to the depletion the carbide phases; the changes in hardness values correspond directly to the changes in precipitate volume fractions. An example of this is illustrated in Figure 2.8, where there is initially a high hardness in the HAZ after welding, but after PWHT and especially creep testing, the HAZ exhibits a lower hardness due to carbon depletion, whereas the local hardness at the interface increases due to carbon enrichment at the interfacial area such as the PMZ.

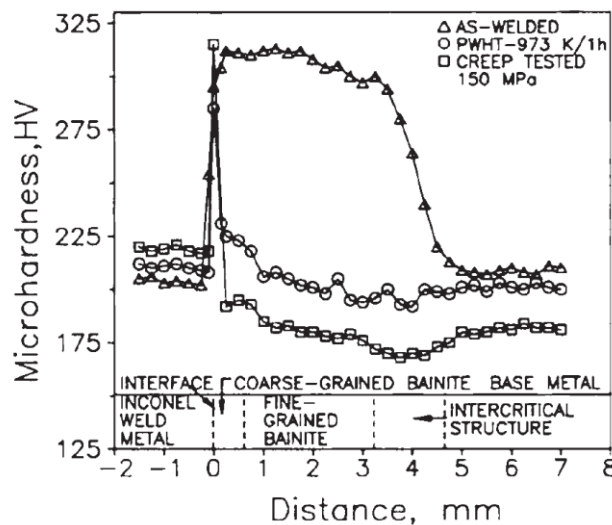


Figure 2.8: Microhardness profiles across interfacial region of weld made between P22 steel and alloy 800H with Inconel 182 filler in as welded condition, after application of PWHT at 700°C for 1 h, and after creep testing at 150 MPa at 550°C (failure occurring at ~1000 h). Courtesy of [44].

The failure of DMWs in high-temperature environments primarily involves creep. Damage accumulation due to creep is almost always most severe at the weld line/interface or on the ferritic side, not in the weld itself, if proper weld procedures are followed. There are four types of damage that can contribute to failure (not to be confused with Type I/II carbides mentioned in a previous section), depending on location relative to the weld as illustrated in Figure 2.9:

- Type I, which is cracking in the weld itself. Since austenitic weld metals are more creep resistant than the ferritic bulk metal, this rarely occurs in practice if the weld is carried out correctly.
- Type II, which is cracking on, or close to (within microns of), the weld interface
- Type III, which is cracking in the CGHAZ

- Type IV, which is cracking that reportedly occurs in either/both the FGHAZ and ICHAZ

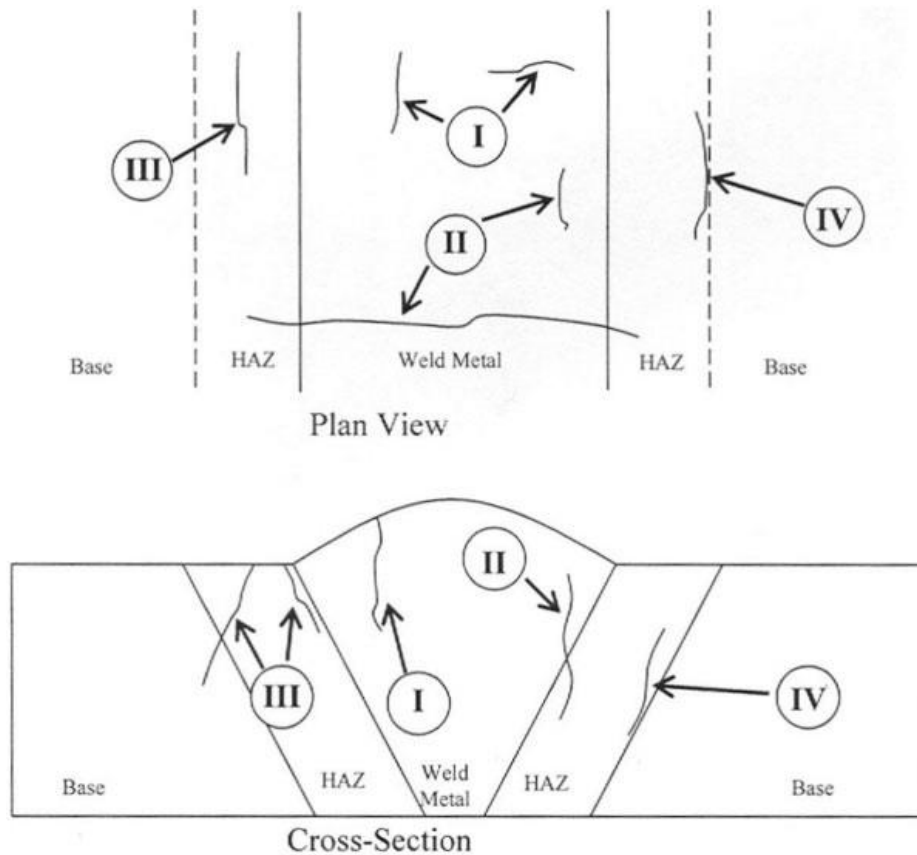


Figure 2.9: Classification of cracking in weldments according to Scheller et al. [65]. Courtesy of [66].

Mechanical compatibility issues such as differences in the coefficient of thermal expansion and metallurgical compatibility issues can result in premature failure [41-43, 67]. There have been numerous investigations into the factors and mechanisms that contribute to cracking problem in ferritic-austenitic DMWs [41, 55, 68, 69], which are summarised below:

- Differences in the coefficients of thermal expansion between ferritic steels and austenitic welds lead to different magnitudes of thermal expansion between the weld and the bulk metal in high temperature operation, causing stress to develop. This may contribute to high residual stress following welding procedure as well as cyclic fatigue during service. This is especially true for welds using austenitic stainless steels, but it is far less severe in welds using nickel-based fillers as they have similar thermal expansion characteristics as the ferritic alloy.

- Carbon depletion in the ferritic material. The carbon migration results in the formation of a decarburized CDZ band, which reduces the local hardness and strength to below design levels. Furthermore, the adjacent CEZ is particularly hard and strong, so additional strain is forced to be 'absorbed' by the CDZ.
- The Laves phase may coarsen (leading to the leeching of molybdenum and tungsten from the matrix which decreases solid solution strengthening), and the strengthening MX phases may transform into the detrimental Z-phase, during prolonged high temperature exposure, particularly in FGHAZ where the small grains provide larger surface area of PAGB (where the Laves phase forms) per unit volume of material.
- The Type I interfacial carbides may induce the nucleation, growth, and coalescence of microscopic void that would create crack surface, especially as they grow in size during ageing. This leads to the Type II damage failure. An example is shown in Figure 2.10 where it can be seen that the voids nucleate around the Type I particles to form the interface cracking.

For a given system of DMW in a given temperature, the location of failure may depend on the applied stress and the duration of creep, as shown schematically in Figure 2.11. At high stresses, failures commonly occur in the bulk metal or the weld metal; however, when the stress is reduced, there is a threshold beyond which the dominant failure shifts to the FGHAZ/ICHAZ, causing type IV creep cracking. The premature failure in this regime occurs sooner than what can be predicted from the performance of the bulk metal or weld metal material under the same conditions, termed the Type IV shortfall.

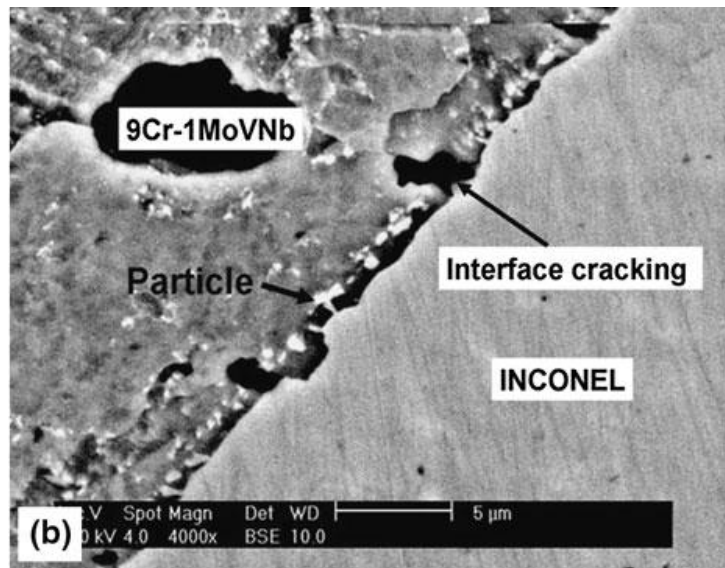


Figure 2.10: Creep cavities are associated with interface particles of DMW joint between 9Cr-1Mo-VNb and Inconel 182, creep tested at 180 MPa [63].

At still lower stresses and longer creep duration, however, the failure location may no longer correspond to Type IV cracking. In a recent study by Laha et al. [63], creep rupture properties of DMWs using Inconel 182 weld metal, with P22, 9Cr-1Mo, or P91 on the ferritic side of the joint were investigated. It was demonstrated that, as the creep stress was decreased (and thus increasing the duration of creep) at a fixed temperature of 550°C, the failure site shifted from deep in the ferritic bulk metal to ICHAZ in ferritic side (indicating Type IV damage). At still lower stress, the failure shifted close to the weld interface, which is at the location of Type I carbides (indicating Type II damage). This is illustrated in Figure 2.12, where there is a rupture life threshold between failure at the interface and Type IV failure, with the performance of the DMWs experiencing shortfall when the failure occurs at the interface.

There has been some difference in observations about where failure would predominantly occur under long term service conditions. A review by Abson and Rothwell [69] shows that weldments made with steels such as P91 and P92 as bulk metal would experience Type IV cracking in general. A review of DMWs by DuPont [41], however, indicated that when nickel-based weld metal is used, the failure would occur near the weld interface as a result of formation of creep cavities around the Type I carbides.

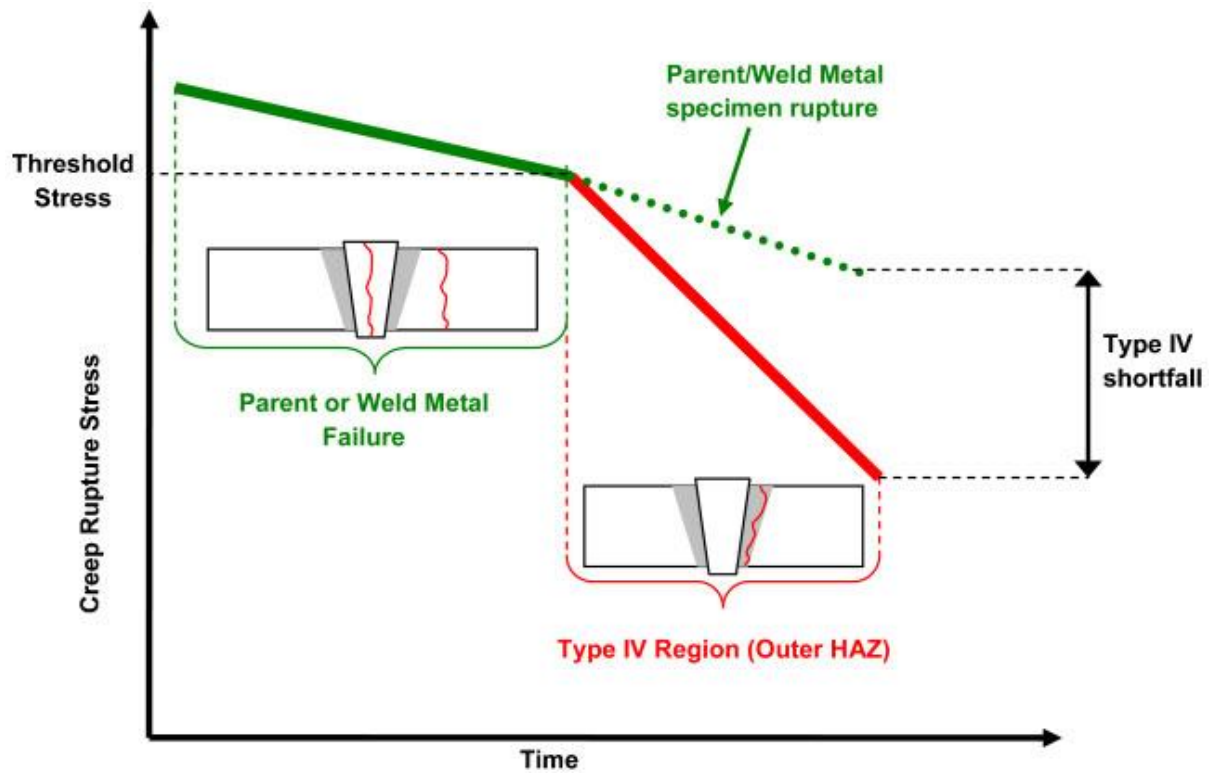


Figure 2.11: Schematic representation of the reduction in creep rupture strength beyond the onset of type IV cracking in welds involving 9-12%Cr ferritic bulk metals (such as P91) at a given temperature [69].

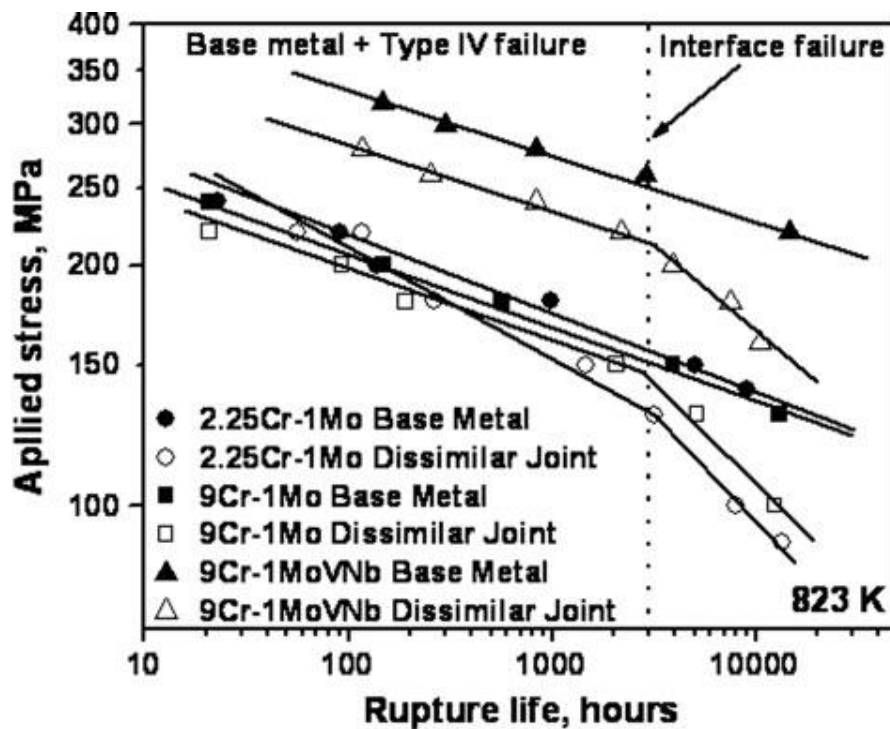


Figure 2.12: Comparison of the creep rupture lives of different DMW joints made with ferritic bulk metals and IN182 weld metal [63].

Ex-service DMWs using nickel-based weld repeatedly point to Type II premature failure due to the formation of creep cavities around Type I carbides, as interfacial carbides are sometimes detected on the crack surface [39, 70]. But accelerated creep tests tend to cause Type IV cracking [71, 72]. This is

consistent with the observations of Laha et al. [63] and DuPont [41]. All these imply that in accelerated creep tests, the stresses are too excessive so that the time scales involved are usually insufficient for microstructural evolution to take place that would accurately reflect the microstructural evolution of DMWs in long service durations. Perhaps another failure regime at even lower stresses should be added to Figure 2.11 to account for phenomenon illustrated in Figure 2.12. This is important to be kept in mind when conducting and examining accelerated creep tests of DMWs.

2.5 Effect of creep stress/strain on precipitation behaviour

It is known that precipitation processes are positively affected by increasing stress as well as temperature, but it is thought that the effect of applied stress is only merely apparent, when in fact precipitation behaviour is strain dependent [73-75]. Elastic and plastic strain can contribute to the nucleation and coarsening of precipitates [76]. *Elastic strain* contributes to a more favourable condition for **nucleation** because the resulting additional interaction energies contribute to the total precipitate energy, which lowers the required transformation energy and critical nucleation energy. *Plastic strain* also contributes to nucleation, mainly due to dislocation multiplication and movement, which provides favourable nucleation sites. *Elastic strain* may affect the **growth and coarsening** of precipitates by contributing interaction energy that affects the orientation of precipitates. *Plastic strain* also contributes to coarsening because the higher dislocation density promotes more effective pipe diffusion routes, where the dislocations in the matrix act as a channel for rapid transit; and that the movement of dislocations would increase the mobility of solid-solution elements in the matrix that could form precipitates, which results in the solute dragging effect, leading to more rapid coarsening.

All these indicate that the effect of strain on nucleation and coarsening rates of precipitates is only significant when the strain is particularly large, i.e. when the stresses are high, leading to significantly high deformation when compared to stress-free situations. Therefore, strain is likely to have a negligibly small effect on nucleation and coarsening of precipitates in practical service conditions,

which generally involves only very small creep deformation, while accelerated creep tests—particularly those with very high stresses—may unrepresentatively alter the precipitate behaviour.

A number of studies have observed the effect of strain on microstructural evolution on bulk alloys, particularly on P91 [75] and P92 [74, 77-79] and various other high-chromium ferritic steels [73], chiefly by comparing the microstructures in the grip (i.e. mechanically unstressed location) and gauge (i.e. mechanically stressed location) parts of a creep specimen. The precipitates of interest, which were $M_{23}C_6$, Laves phases, and to a lesser extent MX, were shown to have enhanced nucleation and growth under creep strain.

So far, studies for this kind of behaviour in welded samples (similar or dissimilar) remain limited, for the simple reason that one cannot make a comparison between the grip and gauge portions in order to evaluate weld behaviour (because the weld interface cannot appear on both locations unless the weld interface is parallel to the uniaxial stress application); however, according to one study, the behaviour of certain precipitates—Type I carbides—appear not to be affected by mechanical loading, as shown in Figure 2.13 [60]. The authors for this study offered no reasoning for this, though the likely reason is that only a small stress was applied (30–80 MPa) which would have led to minimal strain/deformation in the interfacial region, which means that the precipitate behaviour in that vicinity was not affected significantly by the applied stress.

This is just one study, and in this respect, it only focuses on the morphology and growth of Type I carbides. Effects on other precipitates such as those in FGHAZ, which may have a role in inducing Type IV cracking, was not investigated. This is a gap in literature that could be investigated for this project.

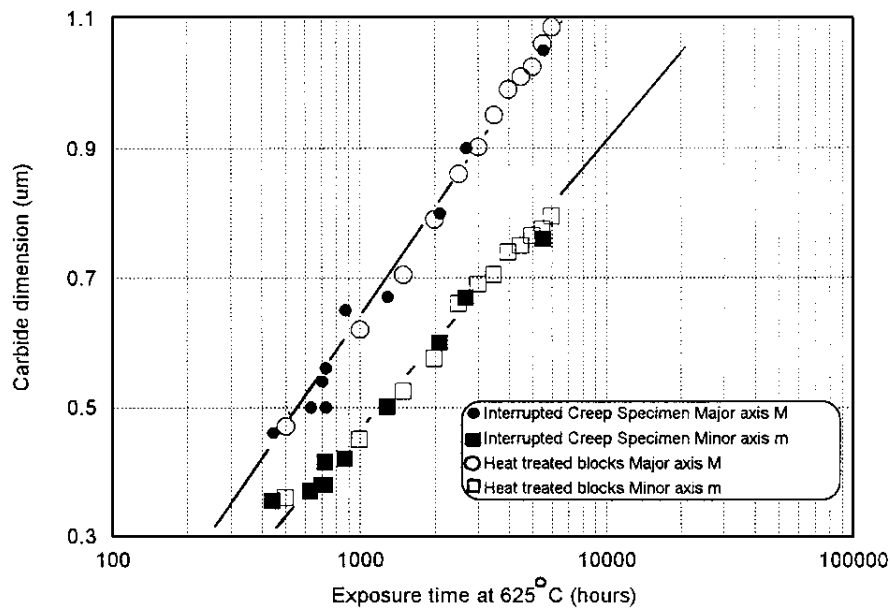


Figure 2.13: Variation in Type I carbide size in both the major axis and minor axis with respect to time for aged samples (heat treated blocks) and for uniaxial cross weld creep specimens, at 625°C [60].

2.6 TransForge fabrication method background and development

Fusion welding, commonly in the form of arc welding such as GTAW, SMAW, or submerged arc welding (SAW), is typically used to join components of dissimilar alloys; however, this introduces challenges unique to DMW in addition to the drawbacks that would be present in similar metal welding. Mechanical compatibility issues such as differences in the coefficient of thermal expansion (which may contribute to high residual stress) and metallurgical compatibility issues (such as that which result from the difference in chemical composition of the dissimilar alloys) can result in premature failure [41-43, 67]. High energy density fusion welding processes, such as electron beam welding (EBW) and laser beam welding (LBW), have been gaining popularity in industry recently due to their potential to reduce these issues to some extent, in addition to providing practical benefits such as the possibility of automation [80, 81].

Alternatively, solid state welding, such as friction welding (rotary or linear), friction stir welding, and diffusion bonding can be used to join dissimilar alloys. As the alloys remain in the solid state, much of the complications associated with fusion DMW are not present and these techniques may produce excellent joints [82, 83]; however, these processes may be constrained by joint geometry (as in the case of friction welding or friction stir welding), economical considerations (diffusion welding takes a

significant time) or process difficulty (friction stir welding is so far only commercially viable for soft materials, e.g. Al/Mg, and but not for joints with exclusively hard materials) [82]. Forge welding, another solid-state joining process, may be a viable option, but there is scarce literature available for the application of forge welding for DMW. A study investigating forge welding between stainless steel and aluminium alloy shows that, at least microstructurally, the weld interface can be as good as diffusion bonding, though weld quality is sensitive to several forging parameters and conditions (temperature, deformation amount and forging speed) [84]. So high energy density fusion welding processes such as EBW remain a competitive DMW technique [85].

TransForge aims to demonstrate the feasibility of combining EBW and forging to join two dissimilar metals in order to create an interface piece between two respective dissimilar metal pipes (i.e. creating a transition joint), so that autogenous welding (i.e. without filler or interlayer) can be performed without compromising the stringent quality requirement of the nuclear power plant industry. The EBW provides a strong fusion joint, and the forging consolidates the joint, removing any unfused locations as well as providing flexibility to further shape the component close to the desired finished shape (e.g. a disk). The forged joint would have no distinct heat affected zones because the whole component is heated to a high temperature during the forging process.

TransForge follows a previous successful project called ForgeMan which investigated the use of EBW for similar material joints. The ForgeMan project was conceived as an alternative method to manufacture RPVs used in nuclear power plants, presently fabricated as forged monoblocks. The goal of that project was to study the feasibility of joining smaller pieces with a novel thick-section EBW. Instead of making one very large forged component, with the aim of splitting the fabrication of RPV into smaller pieces, which would then be joined together using EBW.

The advantage of using EBW compared to other fusion welding processes is that it has a high penetration depth which allows for thick sections to be welded, it can also be performed quickly with a great degree of automation. Conventional arc welding cannot be performed on thick sections in a

single pass and may require the use of filler for thick sections. The other highly automatable autogenous welding technique with deep penetration, laser welding, shows promising results with respect to mechanical performance compared to conventional fusion welding methods (specifically GTA welding) and being porosity-free [86], but the thickness of the material may be limited because the weld depth to width ratio is limited to approximately 10:1 (versus 40:1 for EBW). So for welds that require depth in excess of 10 mm, EBW is by far the more cost-effective method, because penetrations of up to 250 mm in steel can be achieved with a high power (~100kW) EBW in a single pass [87]. The high energy density of EBW enables deep penetration with a relatively narrow fusion zone and thus minimizes residual stresses and distortion and also minimizes the mixing between dissimilar metals. In addition, a vacuum environment is required during the EBW process, which ensures that any gaps at the unfused centre would not contain air (as trapped air could present complications during the subsequent forging process and also potentially creates unwanted voids). This is thus another advantage of incorporating the EBW process before proceeding to forging, especially for larger components.

The drawback of the EBW process is that it requires a vacuum chamber to perform, which may represent a constraint on the size of the component, as well as a (small) increase in manufacturing time due to the chamber evacuation during the process. EBW operations are done in a high-vacuum environment, typically 10^{-3} – 10^{-5} atm.; however, local vacuum or 'out of chamber' EBW is being developed in order to perform EBW without a vacuum chamber [88, 89].

The novel TransForge fabrication technique incorporates single-pass EBW and forging, followed by post-forge heat treatment, to produce a DMW joint between two thick sections of metallurgically dissimilar materials. This approach will allow welding with much lower distortion than achievable with conventional arc welding techniques and offers the potential to be high integrity. The manufacturing process will be faster and more cost-effective using this technique. If successful, then this technique could be used to manufacture, for example, a transition joint that can replace the current design of

nozzle-to-safe-end fusion DMW. The technique could also be utilised for the fabrication of forging tools used in the metallurgical sector.

2.7 Knowledge gaps

The review of research suggests creep stress/strain may have an effect on precipitation behaviour. In DMW between ferritic steels and austenitic alloys,

- A study suggested that no effect of creep stress on a specific type of precipitation (Type I carbides) in DMW exposed to a fixed high temperature, perhaps due to low stress levels exerted. It is possible that strain that develops under larger stress in creep conditions may affect precipitation behaviour at the interfacial region between the two alloys, which in turn affect the integrity of the weld itself.
- Type IV failure is associated with higher creep stress, whereas lower creep stresses tend to cause failure closer to the weld interface in DMWs with nickel-based weld metals. Laves phase coarsening in FGHAZ has been suggested as a cause for Type IV failure. The high stress itself have may a role in inducing Type IV failure, perhaps by influencing precipitation behaviour in FGHAZ.

In order to examine whether creep stress/strain have an effect on precipitation behaviour, and thus weld integrity, microstructural evolution of crept and thermally aged samples experiencing similar high temperature in similar durations from the same DMW joint need to be compared.

The TransForge fabrication method outlined in the previous section has the potential to be a fast and cost-effective way to produce thick section DMW joints while maintaining high integrity. Being a novel method of producing DMW joints, it has yet to be deeply reviewed and thus offers a wide scope for evaluation on its integrity in terms of mechanical and microstructural properties.

3 RESEARCH METHODOLOGY

3.1 Introduction

This section firstly describes the type of materials to be examined as part of this project. It is then followed by descriptions of the methods of preparing the samples for microstructural examinations. Afterwards, the techniques for samples examinations and characterisations are described. Finally, the computational technique used for predicting the phases that may appear in a given alloy under given conditions, which helps supplement the microstructural investigation, is outlined.

3.2 Materials supply

There are two groups of samples provided for this project. They are from TWI and Doosan Babcock.

3.2.1 TWI – TransForge samples

The intention of the TransForge process is to bypass problematic fusion DMW between dissimilar metal pipes by creating a transition joint that is produced offsite (the ring) and then simply incorporated into the desired location by similar metal welding. The transition joint made of two dissimilar alloys is produced by using EBW followed by forging, machining etc.

3.2.1.1 *TransForge fabrication method summary*

A summary of the TransForge fabrication process is illustrated in Figure 3.1.

1. A pair of solid blocks or cylinders made of dissimilar alloys are electron beam welded in vacuum (5×10^{-4} mBar) at the outer edges all around the interface, with each edge welded in a single pass. The incorporation of EBW in the process means that there may be some fusion welding that occurs in the interface, except at the centre of the interface between the two blocks where it may be deep enough to be beyond the reach of electron beam welding.
2. The joined blocks are heated to approximately 1000°C. The actual forging temperature(s) depends on the alloys being joined.

3. While still glowing hot, the blocks are forged/pressed together.
4. The fused blocks are further forged into a desired shape (disk/cylinder shape for this work) in stages.
5. A hole is pierced through the centre of the disk, creating a ring-shaped component (Figure 3.2), where one side (top) is made of an alloy dissimilar to the one at the other side (bottom).
6. The component can be further machined, inspected, given heat treatment, etc.

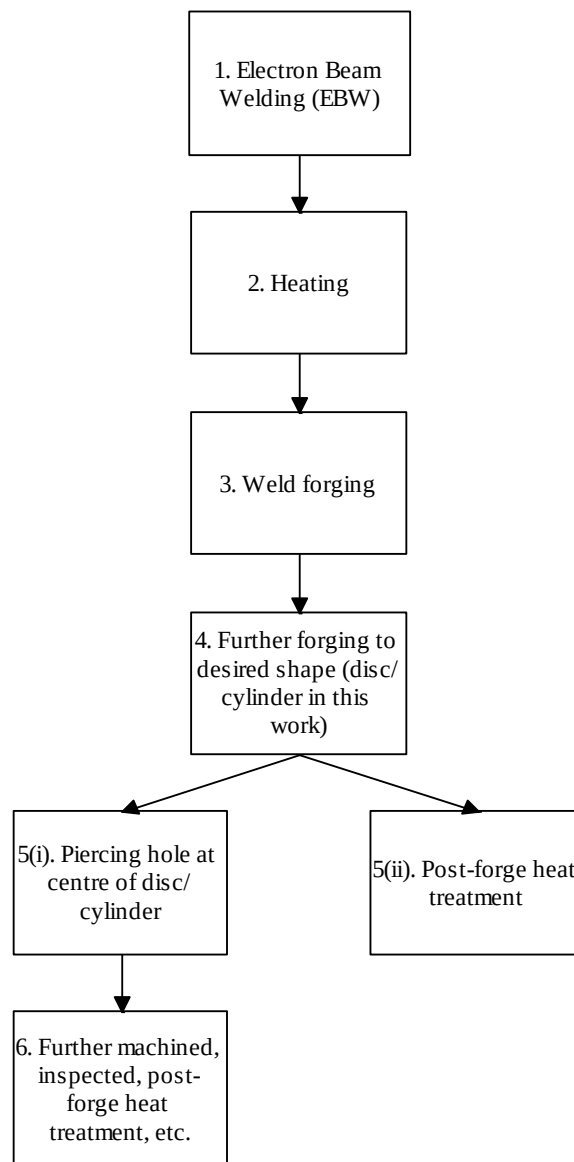


Figure 3.1: Flow chart of the TransForge fabrication process.

Step 5(i) in Figure 3.1 is for the sole purpose of creating a ring with different alloy on each side, as shown in Figure 3.2; however, this step (Step 5(i)) of the fabrication process was not necessary for the demonstration purposes of this work, which only analysed the specimen produced in steps 1-5(ii).

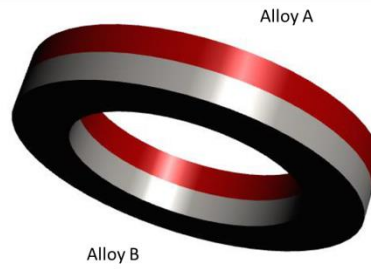


Figure 3.2: Transition joint ring created using TransForge process (not part of this investigation). This is the finished product after Step 5(i) in Figure 3.1.

3.2.1.2 Materials

Three alloys have been used to demonstrate the TransForge process: A508 Grade 3 (bainitic steel - supplied by Carrs Stainless Steels), 304L (austenitic stainless steel - supplied by Tecphy Division Firminy), and IN600 (nickel superalloy - Heat 260027 - supplied by Acciaierie Valbruna). The joint combinations are 1) A508/304L and 2) A508/IN600. The compositions of these alloys are outlined in Table 3.1.

Table 3.1: Chemical Composition of A508, 304L and IN600.

Element	Amount (wt%)		
	A508	304L	IN600
Al	0.003	0.009	-
B	0.0001	7 PPM	-
C	0.24	0.029	0.07
Ca	0.0001	-	-
Co	-	0.03	0.01
Cr	0.11	18.57	15.6
Cu	0.039	0.21	0.01
Fe	Bal	Bal	8.74
Mn	1.42	1.62	0.53
Mo	0.58	0.11	-
N	0.007	0.084	-
Nb	0.004	0.02	-
Ni	0.61	10.45	74.37
P	0.015	0.019	
S	0.01	0.001	0.001
Si	0.24	0.54	0.3
Sn	-	0.007	-
Ta	-	0.03	-
Ti	0.001	0.01	-
V	0.002	0.08	-

3.2.1.3 Electron Beam Welding

To make a joint, a pair of blocks of dimension 150x150x175mm of the dissimilar alloys were joined using EBW in a vacuum (5×10^{-4} mBar) at the four edges. The welding parameters for the different joints are summarized in Table 3.2 with the only difference between the two material combinations being the welding speed; with the welding speed for the A508/IN600 joint being faster than that of the A508/304L joint.

The joint was welded on one edge and then rotated 180° to weld the opposite edge. The third edge was welded after rotating 90°, and then the last edge was welded after rotating further 180°.

In the case of the A508/304L joint, the 304L specimen was demagnetized prior to the welding process; however, EBW of dissimilar metal joining is complicated by the Seebeck effect, where thermoelectric fields produced by temperature gradients deflect the beam from its intended path (i.e. along the interface between the two alloys, perpendicular to the outer surface) [90]. So, in order to keep the electron beam perpendicular to the outer surface and parallel to the interface, the electron beam height was offset +1mm and slightly tilted into the A508 side for each joint.

Table 3.2: Electron beam welding parameters used for the fabrication of A508/304L and A508/IN600 TransForge welds

Parameter	Joint	
	A508/304L	A508/IN600
Voltage (kV)	150	
Working Distance (mm)	230	
Beam Current (mA)	80	
Welding Speed (mm/min)	125	200

A cross section of the A508/IN600 after electron beam welding is shown in Figure 3.3. It shows that the weld penetration extends about 2/3 to the centre from each edge, leaving an unfused region at the centre of the joint between the two blocks.

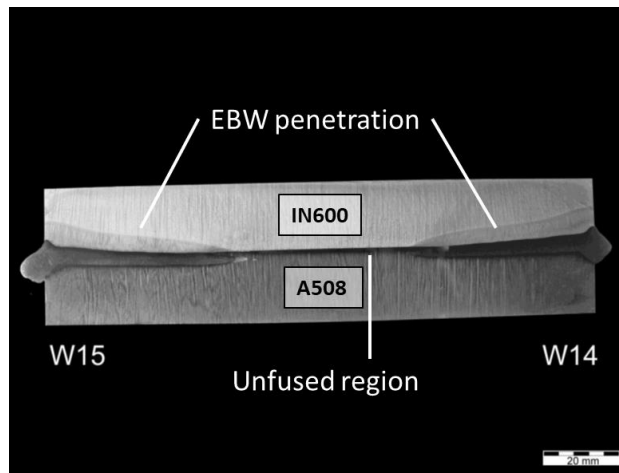


Figure 3.3: Cross section of A508/IN600 after electron beam welding the outer edges of the interface.

3.2.1.4 Forging

The joined blocks are heated up to approximately 1000°C. The blocks are then forged/pressed together. Forging two metals with different high temperature strength would result in a different deformation magnitude between the fused alloys, producing a lip flow of A508 around the IN600 or 304L, as shown schematically in Figure 3.4, because A508 is the weaker alloy at this temperature.

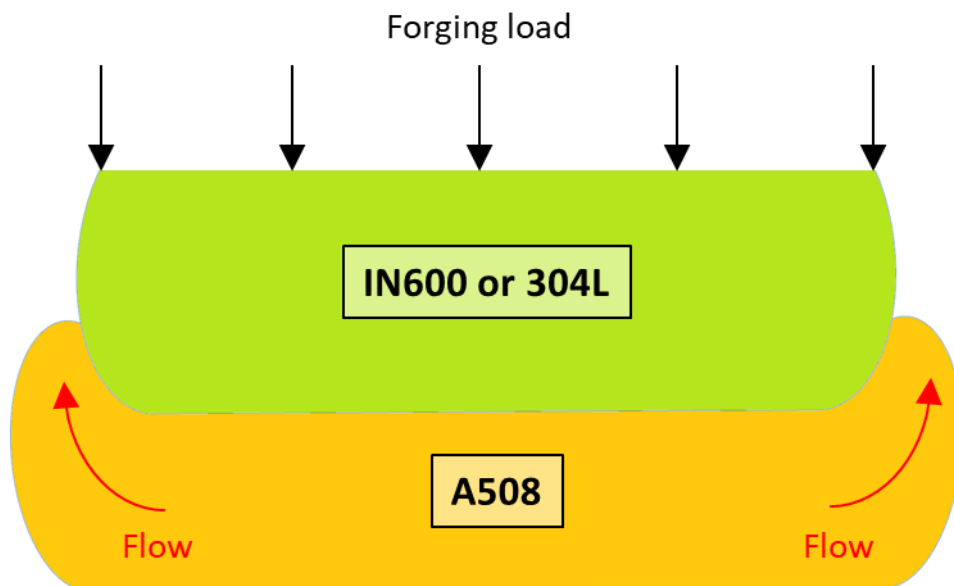


Figure 3.4: Schematic of the fused block of A508/IN600 or A508/304L, showing that difference in deformation and flow between the two alloys in each pair at high temperature forging cause a lip of A508 to form around the opposite alloy block.

This fused block is further forged into a desired shape (disk/cylinder shape for this work) in stages. Unfortunately the data regarding force etc. is not available as the forging was performed manually, and the data has not been logged.

3.2.1.5 *Post-forge Heat Treatment*

After fabrication, the samples were given a post-forge heat treatment (PFHT), consisting of austenitisation/solution treat at 980°C followed by water quenching and then tempering at 680°C, followed by air cooling.

Overview images of both the TransForge joints after forging and heat treatment are shown in Figure 3.5. It can be seen that, due to the difference in high temperature strength between the alloys, there is a difference in the deformation of each alloy, resulting in a lip of A508 to flow around the austenitic alloy in both cases.

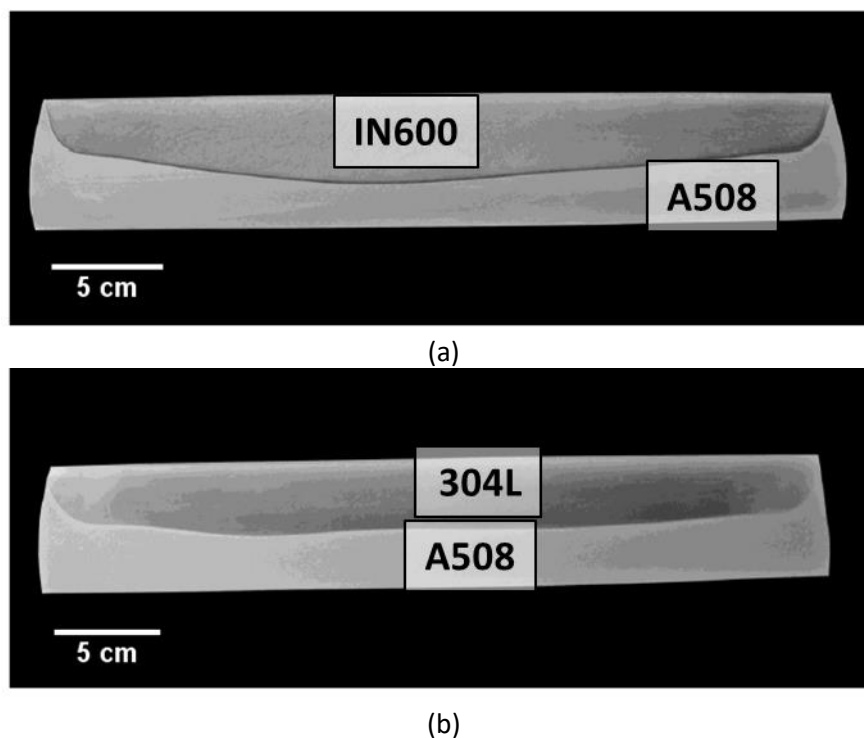


Figure 3.5: Overview of TransForge joints after forging and post-forge heat treatment for the (a) A508/IN600 joint and (b) A508/304L joint.

3.2.2 Doosan Babcock – fusion P92/IN182 DMW samples

3.2.2.1 Alloys and pipe geometry and samples received

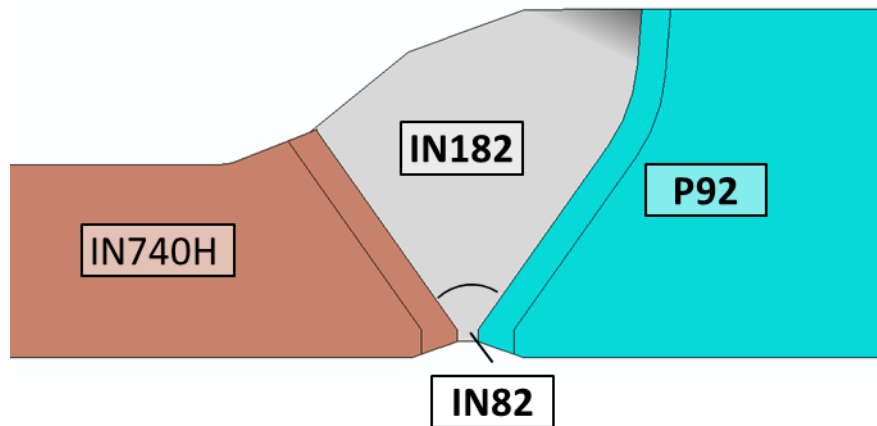


Figure 3.6: Configuration of Doosan Babcock P92/IN182/740H weld, viewed as the 2D sketch to be revolved to form a pipe model.

The conventional DMW weld was constructed using single sided vee groove (pipe) weld configuration, as shown in Figure 3.6. First, the root of the weld is made with gas tungsten arc welding (GTAW) in a single pass with Inconel 82 filler. Then a shielded metal arc welding (SMAW) multipass weld with Inconel 182 is used to close the weld. Afterwards, post-weld heat treatment (PWHT) was performed at around $770 \pm 10^\circ\text{C}$ for about 8 hours to temper the martensite that formed in the heat affected zone (HAZ) of P92, before controlled cooling to 425°C (at the cooling rate of 178°C/H), and then cooled in still air.

The compositions of the alloys used in the pipe mockup are reported in Table 3.3 and Table 3.4. Composition of alloy 740H is also available, but is not reported here because this alloy is not examined, as the focus of analysis is on the interface between the P92 steel and IN182 weld metal, where the review of literature suggests is the likely location of failure.

Table 3.3: Chemical composition for ferritic steel P92, as reported by Vallourec Group, which supplied the alloy to Doosan Babcock.

Element	Al	B	C	Cr	Fe	Mn	Mo	N	Nb/Cb	Ni	P
Amount (wt%)	0.004	0.0021	0.1	8.79	88.01	0.46	0.47	0.052	0.052	0.12	0.015
	S	Si	Ti	V	W	Zr					
	0.005	0.15	0	0.2	1.568	0					

Table 3.4: Chemical composition for nickel-based electrode IN182, as reported by Bohler Welding Group, which supplied the electrode to Doosan Babcock.

Element	C	Co	Cr	Cu	Fe	Mn	Nb	Ni	P	S	Si	Ti
Amount (wt%)	0.02	<0.01	16	0.01	5.85	5.75	2.15	69.4	<0.01	0	0.45	0.1

3.2.2.2 As-manufactured (PWHT samples)

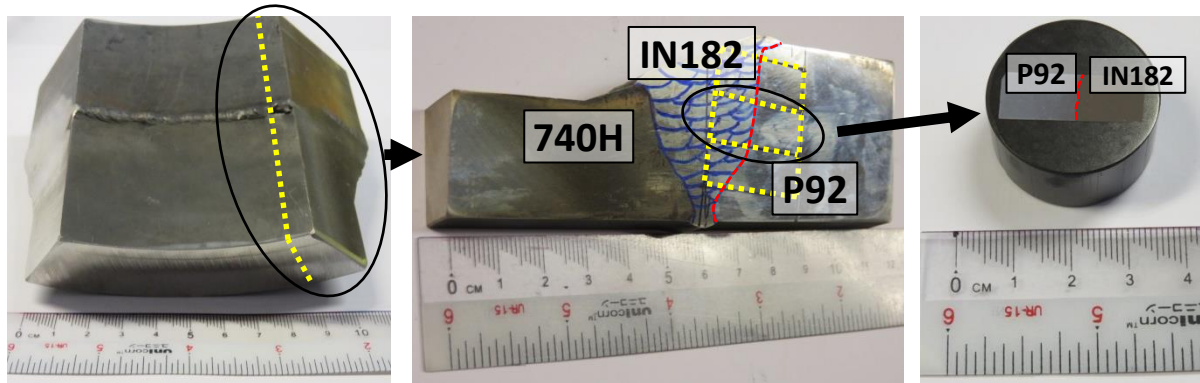


Figure 3.7: Diagram showing how the PWHT sample was obtained from the as-received pipe mockup sample. The yellow dotted line indicates where cuts were made, and the red dashed line indicates the interface of interest (between P92 and IN182). The blue lines in IN182 were added indicate the individual weld beads in the multipass weld.

A segment from the wedge is cut using electrical discharge machining (EDM) cutting, then small pieces (up to three per segment) in the interfacial area between P92 and IN182 were further cut using waterjet cutting, as shown in Figure 3.7. Care was taken to avoid the root weld at the bottom because the weld material there is different (IN82 instead of IN182). These pieces are then mounted, polished, and analysed. As this sample has been tempered during the post weld heat treatment (PWHT), it is labelled as sample T.

3.2.2.3 Crept samples

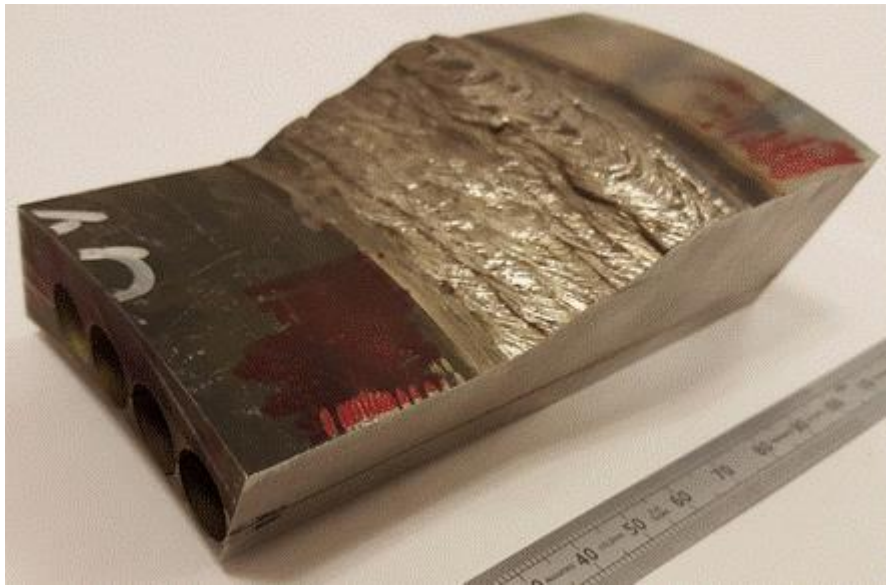


Figure 3.8: The locations (holes) in the pipe mockup from which the creep samples were obtained.

Doosan Babcock also provided crept samples that are tested to failure, obtained from the pipe mockup as shown in Figure 3.8. In the creep tests, the three *pipe* weld joint specimens were subjected to 154, 129, and 116 MPa of stress, at temperature of 630°C, which resulted in time-to-fracture of 109, 1453, and 5862 hours, respectively. These samples are labelled as **Creep-109h**, **Creep-1453h**, and **Creep-5862h**, respectively. More information on the crept samples are available in the Appendix (Table 0.1).

3.2.2.4 Heat treatment

A few of the PWHT samples were further heat treated (thermally aged) at the same conditions as the crept sample (630°C) for 109h, 500h, 1000h, 1453h, 3000h, and 5000h in order to investigate the effect of creep stress on microstructural evolution, especially on precipitate development, by creating samples that underwent the same high temperature conditions as the crept samples but without the applied mechanical stress. These samples are labelled as **HT-109h**, **HT-500h**, **HT-1000h**, **HT-1453h**, **HT-3000h**, and **HT-5000h**, respectively.

To prevent the surface oxidation that occurs during heat treatment from interfering with the observation, the cut has been made at the middle of the thermally aged samples, and the surface along the cut was examined.

3.3 Metallographic preparation

To examine material microstructure, samples are hot mounted in the black conductive resin, as shown in Figure 3.9. The faces of the samples to be examined are then successively grounded on P240, P400, P800, and P1200 grit silicon carbide papers, in order. Afterwards, the faces are polished using 6 μm and then 1 μm diamond abrasive particles. Final polishing is done using 0.025 μm colloidal silica suspension.

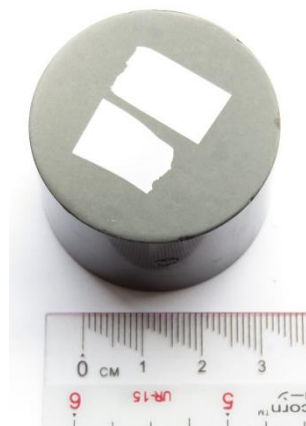


Figure 3.9: Sectioned samples mounted in a black conductive resin (hot mounted).

Etchants are used to expose the microstructures of the polished samples. It works by preferentially attacking some features of the alloys, such as grain boundaries and precipitates, which then results in surface topography defined by microstructure that can be examined with a microscope.

Since there will always be different types of alloys present in a single DMW, there will be different levels of corrosion resistance within a single sample surface, with the austenitic stainless steels and nickel alloys generally being more corrosion resistant than the ferritic steel. As a result, no single etchant can be used to examine all the alloys in a single sample face. Therefore, the etchant(s) suitable for the less corrosion resistant alloys in the sample face are applied first, followed by the examination of these alloys, and only then the etchants for the more corrosion resistant alloys are applied.

The following are the etchants used for this project:

- **Nital** is used to reveal σ grain boundaries and constituents in the A508 steel. It is a mixture of nitric acid and ethanol (or methanol), most commonly in 1:50 volumetric ratio to produce 2% nital. This etchant is applied by swabbing it onto the surface for up to 60 seconds.
- A solution of **1 ml of hydrofluoric acid, 2 mL of nitric acid, and 100 mL distilled water** is used to reveal constituents such as carbides and grain boundaries in the P92 steel. It is swabbed onto the surface for up to 30 seconds.
- **Aqua regia** is for austenitic stainless steel (304L) and nickel-based alloys (IN600 and IN82/IN182) to reveal grain boundaries, carbides, and dendritic structures. It is a mixture of hydrochloric acid and nitric acid in a 3:1 molar ratio. It is heated up to 50°C and then swabbed for up to 10 seconds.

Application of aqua regia, which is used to etch more corrosion-resistant alloys, onto the surface dissolves substantial amount of ferritic steel, damaging surface of that alloy. Thus, the samples must be re-polished before the ferritic steel can be re-examined.

3.4 Microstructural characterisation

Metallographic examinations of the samples are done using both optical and scanning electron microscopy with energy-dispersive x-ray analysis (SEM/EDX) and electron backscattered diffraction (EBSD).

3.4.1 Optical microscopy

Optical microscopy is done using Nikon Eclipse LV100ND digital optical microscope to examine etched samples under visible light, whose digital images can be captured and recorded into a computer. The equipment contains five objective lenses, with 5X, 10X, 20X, 50X, and 100X magnifications.

The limitation is that optical microscopy has absolute limited resolution, only down to 1 μm , and has a poor depth of field (i.e. unable to uniformly focus on surfaces with rough topography). In addition, it does not give direct chemical or crystallographic information of the features in the alloys.

3.4.2 Scanning electron microscopy

Scanning electron microscopy (SEM) uses electrons to illuminate the surface of the sample, performed in a vacuum chamber. A beam of electrons is fired from an electron gun and is typically accelerated through voltages of 1-30 kV. The electron beam is then narrowed into the sample surface using magnetic condenser lenses. When the electrons hit the surface of the sample, some knock out existing electrons on the surface to scatter, some others penetrate a small distance into the surface before ballooning out and scattering, while others cause electrons in the sample to become excited. The three types of emissions produced are inelastically scattered electrons, elastically scattered electrons, and characteristic X-rays. Some electrons may be absorbed into the sample, which is why it is important for the sample to have a conductive connection to the ground to prevent buildup of charge that may interfere with the electron scattering. The depth of penetration of electrons in the surface can be controlled by adjusting the acceleration voltage. These emissions are collected by three corresponding types of electron detectors placed close to the specimen. The detectors then input information into a CPU to construct an image of the sample surface.

SEM has two main advantages over optical microscopy: higher spatial resolution, due to electron's shorter wavelength compared to visible light photons, and greater field of depth. In addition, magnification and focus work independently of each other, unlike optical microscopy.

SEM works were done using the tungsten-filament JEOL JSM-6490LV SEM as well as JEOL 7100F FEG-SEM.

3.4.2.1 *Secondary electron*

The secondary electron (SE) mode is the primary mode in SEM. The detector collects electrons that have inelastically scattered at the surface of the sample that resulted from the inelastic interaction of the primary electron beam with the sample. Like optical microscopy, this reveals the surface topography of the sample, which means that it is most useful for examination of etched samples but is far less useful for flat polished samples. Typically the acceleration voltage is set to 20 kV and the working distance to 10 mm.

As Type I particles in ferritic steels may play an important role in crack nucleation close to the weld line in service welds, Type I particles were investigated quantitatively. These particles were observed using SEM-SE on etched surfaces of P92. Type I particle size was quantified using the ImageJ software. Parker and Stratford [60] suggested that it is important to measure Type I particles in terms of elliptical major and minor axes, instead of assuming circular shapes, due to the carbides sometimes developing into lenticular shapes. So the particle sizes are characterised in terms of the mean area, mean major axis dimension, and mean minor axis dimension. Five measurements (repeats) were taken in different locations near the weld line to establish confidence limits for these measurements.

3.4.2.2 *Backscatter electron*

Backscattered electrons (BSE) are the beam electrons that reflect off the sample surface, similar to how light reflect off a mirror. This elastic interaction between the sample surface and the electrons occurs in a region somewhat below the surface of the sample, typically in a balloon-shaped interaction volume. Because the interaction volume is larger than the width of the original electron beam, the BSE mode cannot give image resolution as high as SE images can. The backscattering efficiency depends on the atomic number, so regions with heavier elements such as tungsten will appear brighter than those dominated with lighter elements such as iron. It can also be used to contrast the surface topography, but in lower expected resolution compared to the SE mode. Hence, this mode is most useful for examining grain structure (e.g. dendritic structure in weld) as well as the presence or

distribution of secondary phases that have different average (weighted) atomic numbers from the alloy matrix. Like the SE mode, typically the acceleration voltage is set to 20 kV and the working distance to 10 mm, but the sample surface has to be polished smoothly.

The development of Laves phase in P92 steel specimens was examined as this type of precipitate can be readily distinguished in SEM-BSE, and may be relevant to Type IV creep failures. Brightly coloured particles that appeared in SEM-BSE were confirmed to be Laves phase by checking that these are rich in W and/or Mo. For the quantification of the Laves phase, a sampling area of 420 μm^2 was taken for each measurement. From this, the Laves particle size was quantified using the ImageJ software, from which the mean diameter, area fraction, and number density was calculated. According to Dimmler et al. [91], no truncation correction of equivalent diameter is necessary when quantification of Laves particle sizes is performed solely using BSE images. Five measurements (replicates) were taken in different locations within the each region (bulk, CGHAZ, and FGHAZ) of each specimen to establish confidence limits for these measurements.

3.4.2.3 *X-ray Emission*

Sometimes the electrons from the electron beam collide with the existing electrons in the sample. If the electrons from a low energy shell get collided, then it gets knocked out of the shell. The electrons in the higher energy shell then drops into the newly-created vacancy, releasing x-ray energy whose magnitude depends on the energy difference between those high- and low-energy shells. The energy difference between the shells are discrete, and they are distinct for different elements (as well as different pair of orbital shells in question), so the x-ray energy detected can be used to identify and distinguish the different elements present in the sample surface. This method is called energy-dispersive X-ray (EDX, also known as energy-dispersive X-ray spectroscopy, EDS). EDX is used to identify the relative amount of different elements present locally (by wt% and at%) as well as to map the distribution of elements on the surface of samples.

3.4.2.4 *Electron backscatter diffraction*

Crystallography of phases on smooth (finely polished) sample surfaces can be observed using electron backscattered diffraction (EBSD) in SEM. The surface of the sample is tilted (typically at 70° from horizontal), so that when the electron beam hits the surface, the electrons are diffracted onto a phosphor screen in patterns characteristic to the crystal structure and orientation of the material at that location. This pattern can be used to determine and discriminate different phases (by referencing a library of crystallographic data of phases), measure the crystal orientation, measure grain boundary misorientations, etc. EBSD mapping of the sample surface can be obtained by scanning the surface with the electron beam in a grid fashion and obtain the diffracted patterns at each grid point.

An EBSD detector fitted to JEOL 7100F FEG-SEM and controlled with AZTEC software was used to obtain EBSD results. EBSD and EDX mapping were obtained simultaneously in order to relate the chemical composition with the crystallography of the phases. The acceleration voltage is set to 15 kV.

3.5 Hardness testing

Hardness testing is a non-destructive test used to determine a material's local resistance to indentation, which is type of permanent deformation and is thought to directly relate to the materials hardness, which in turn has a degree of relationship with the material's yield stress.

3.5.1 Microhardness testing

In a microhardness testing, a diamond-tipped indenter presses a small controlled load onto the surface of a polished sample, which creates an indent. The local hardness of the material is then calculated using the load magnitude as well as the size of the indent. Successive indentation along a straight line can create a hardness profile across the face of the sample. For this project, Vickers hardness tests are implemented using Buehler MMT-7 Digital Microhardness Tester, typically using 50-200 gf load with 5 second dwell time. In the case of creating microhardness profiles, the offset between each indents within the profile is at least 50µm in order to ensure that there is sufficient distance to avoid strain field interaction between different indents.

3.5.2 Nanoindentation testing

3.5.2.1 Nanoindentation setup

While microhardness testing is useful to obtain the local hardness measurement in most situations, the indents created are relatively large and may well include differing microstructural regimes within a single indent, averaging out and masking the local microhardness variations. It is for this reason that nanoindentation testing was also performed on some of the samples, as nanoindentation allows smaller indents to be made. Nanoindentation testing was performed using Micro Materials Ltd. NanoTest™ NTX with a Berkovich indenter.

Like the microhardness profiling, there needs to be sufficient distance between indents in order to avoid strain field interaction. The offset for the nanoindentations were at least 20 times the maximum depth of the indent, in accordance with ASM International [92]. To satisfy this requirement, the testing was depth-controlled, with the maximum depth h of each indent of 1 μm and the offset between each indents of 30 μm . This consistent offset of 30 μm was chosen as it would capture distinct microstructural zones near the interface between two alloys, while still fulfilling the minimum offset requirement.

3.5.2.2 Determining elastic-plastic material properties from nanoindentation data

Depth sensor in nanoindentation tester enables the measurement of the depth of the indenter and the corresponding load exerted by the indenter onto the surface while the testing occurs. A load-displacement relation can be plotted for each indent, as shown in Figure 3.10, and from this elastic-plastic properties could be extracted. The elastic property Young's modulus, E , can easily be obtained from the plot by considering the stiffness S (the slope of the unloading force–displacement curve that is measured at the maximum load). The relationship between S , the contact area A and the effective modulus, E_{eff} is

$$S = \beta \frac{2}{\sqrt{\pi}} E_{eff} \sqrt{A} \quad 3.1$$

where

$$\frac{1}{E_{eff}} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i} \quad 3.2$$

describes the effective elastic modulus, which consists of the indenter modulus, E_i , the indenter Poisson's ratio, ν_i , and, the elastic modulus and Poisson's ratio of the specimen, E and ν , respectively. As the diamond tip of the Berkovich indenter is very stiff, E_i is assumed to be infinite, so

$$\frac{1}{E_{eff}} \approx \frac{1 - \nu^2}{E}. \quad 3.3$$

β is a correction factor and is not constant, but assumed to be ≈ 1.034 here for the Berkovich indenter.

Elastic-plastic properties can also be obtained using the inverse method, which involves optimising the input parameters (E , ν , yield stress σ_y , and strain-hardening exponent n) into Finite Element Analysis (FEA) simulations so that these simulations would produce results that best fit the P - h plot obtained experimentally. This inverse approach was described by Iracheta [93] in more detail, with the only difference being that only a single objective function is used, but for the reader's sake it is reproduced here with minor modifications.

The nanoindentation testing using a Berkovich indenter was numerically simulated using the ABAQUS. Due to symmetry of the Berkovich indenter, only half of the problem geometry has been modelled as illustrated in Figure 3.11.

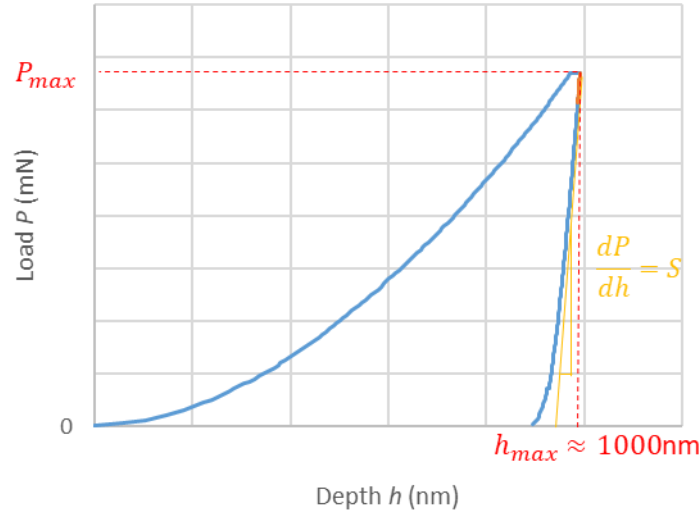


Figure 3.10: Schematic of load-displacement (P - h) plot from nanoindentation testing.

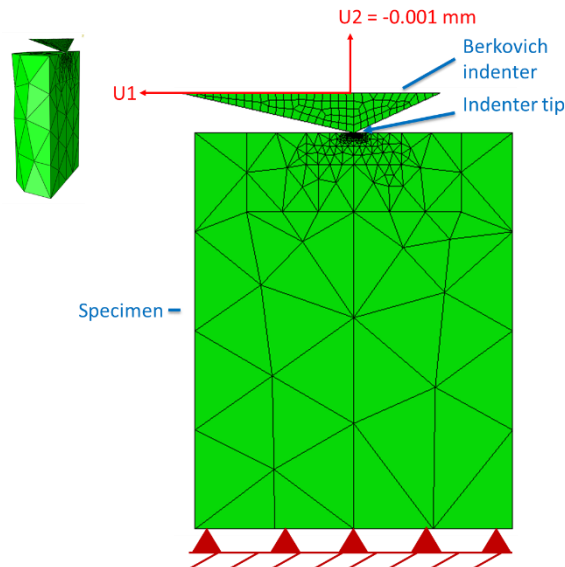


Figure 3.11: Finite element model of the nanoindentation testing using Berkovich indenter

The Berkovich indenter was modelled as a 3D discrete rigid body using 3-node 3D rigid triangular facet elements (R3D3). The specimen was modelled as a 3D deformable body using tetrahedral elements (C3D4) due to the ability to approximate complex boundaries, such as the plasticised material around the indenter. The indenter is only allowed to translate in the vertical direction, to a depth of $U_2 = 10^{-3}$ mm (i.e. the maximum depth h of 1 μ m) and constrained in other axes ($U_1 = U_3 = U_{R1} = U_{R2} = U_{R3} = 0$). The output from the simulation is the ‘reaction force’ experienced by indenter tip against the specimen, equivalent to load P . The data of load P vs the displacement h of indenter in the U_2 direction were then used to construct the predicted/simulated P - h curve.

The model assumes a rigid indenter pressed onto an isotropic, elastic-plastic, rate independent, semi-infinite body under quasi-static and isothermal conditions. The material constitutive model was defined using a Swift's power law hardening rule. Thus, the elastic-plastic true stress-true strain behaviour was assumed to be

$$\sigma = \begin{cases} E\varepsilon_T & \text{for } \sigma \leq \sigma_y \\ K\varepsilon_T^n & \text{for } \sigma > \sigma_y \end{cases} \quad 3.4$$

where σ is the stress at the corresponding total strain $\varepsilon_T = (\varepsilon_e + \varepsilon_p)$, ε_e and ε_p are the elastic and plastic strain components, E is the Young's modulus, K the strength coefficient and n the strain hardening exponent. In order to ensure continuity at $\varepsilon_p = 0$,

$$\begin{aligned} \sigma_y &= E\varepsilon_y = K\varepsilon_y^n \\ K &= \sigma_y \left(\frac{E}{\sigma_y} \right)^n \end{aligned} \quad 3.5$$

Therefore the complete material constitutive relationship when $\sigma > \sigma_y$ is modelled by

$$\sigma = \sigma_y \left(1 + \frac{E}{\sigma_y} \varepsilon_p \right)^n \quad 3.6$$

As a result, three material properties (Young's modulus (E) the initial yield stress (σ_y) and the strain hardening exponent (n)) are sufficient to describe the stress–strain relationship of a material.

The inverse analysis involved an iterative optimisation procedure to find the set of material properties that generated, using FEA, the predicted P - h curve that best fits the corresponding experimental curve. The optimisation model has been defined as follows:

$$f(\mathbf{x}) = \sum_{k=1}^N (P_k^{exp} - P(\mathbf{x})_k^{pred})^2 \quad 3.7$$

subject to

$$\mathbf{x} = \begin{bmatrix} E \\ \nu \\ \sigma_y \\ n \end{bmatrix}$$

$$x_j^L \leq x_j \leq x_j^U, j = 1, 2, 3, 4$$

The vector $\mathbf{x}$ contains the optimisation parameters, i.e. the values of the material properties, and $f(\mathbf{x})$ is the objective function to be minimised. $f(\mathbf{x})$ is the normalised error, defined as the sum of square of error of the predicted P values, i.e. the sum of the difference between the experimental (exp) and predicted data (pred) of P at each datapoint on the P - h curve, squared. The physical data extracted from experimental indentation and FE simulated data have been linearly interpolated in order to evaluate the objective function at the specific sample points, k . k goes up to N , which is the number of datapoints available in each experimental P - h curve.

The range of possible solutions has been limited by a set of bound constraints, where x_j^L and x_j^U represent the lower and upper limits of x_j , respectively. For this study, there are three sets of limits imposed on the range, as shown in Table 3.5. Most metals have strain hardening exponent in the range $0.1 < n < 0.6$. Imposing Limits Set 1 resulted in the 'optimised' E being close to 300 GPa, i.e. maximum allowed limit, so the limit of E is increased in Limits Set 2 to 400 GPa (though unfortunately even with this, the E still tends towards the maximum).

Table 3.5: Limits Sets on the range of possible parameter solutions.

Parameter	Limits Set 1		Limits Set 2		Limits Set 3	
	Lower limit	Upper limit	Lower limit	Upper limit	Lower limit	Upper limit
E (GPa)	150	300	150	400	[Determined from Equation 3.3]	
ν	0.28	0.32	0.28	0.32	0.3	
σ_y (MPa)	0	1500	0	1500	0	1500
n	0.1	0.6	0.1	0.6	0.1	0.6

For Limits Set 3, individual E of each indent is determined by calculation using Equation 3.3, where E_{eff} is automatically provided by the nanoindentation machine (which used Equation 3.1 to obtain it), and ν is assumed to be 0.3. Hence only σ_y and n are optimised.

The optimisation method starts from an initial guess material properties defined by the user in vector $\mathbf{x}_0$. The current vector $\mathbf{x}_i$, which is the solution of the optimisation problem $f(\mathbf{x}_i)$ at the i -th iteration, is iteratively modified in MATLAB using a trust-region-reflective algorithm until convergence is reached, as detailed in Figure 3.12. The convergence is reached, terminating the optimisation process, when the solver attempts to take a step norm that is smaller than the defined lower bound on the size of the step (TolX = 1e-8), or that results in a change in the value of the objective function that smaller than the defined lower bound (TolFun = 1e-8).

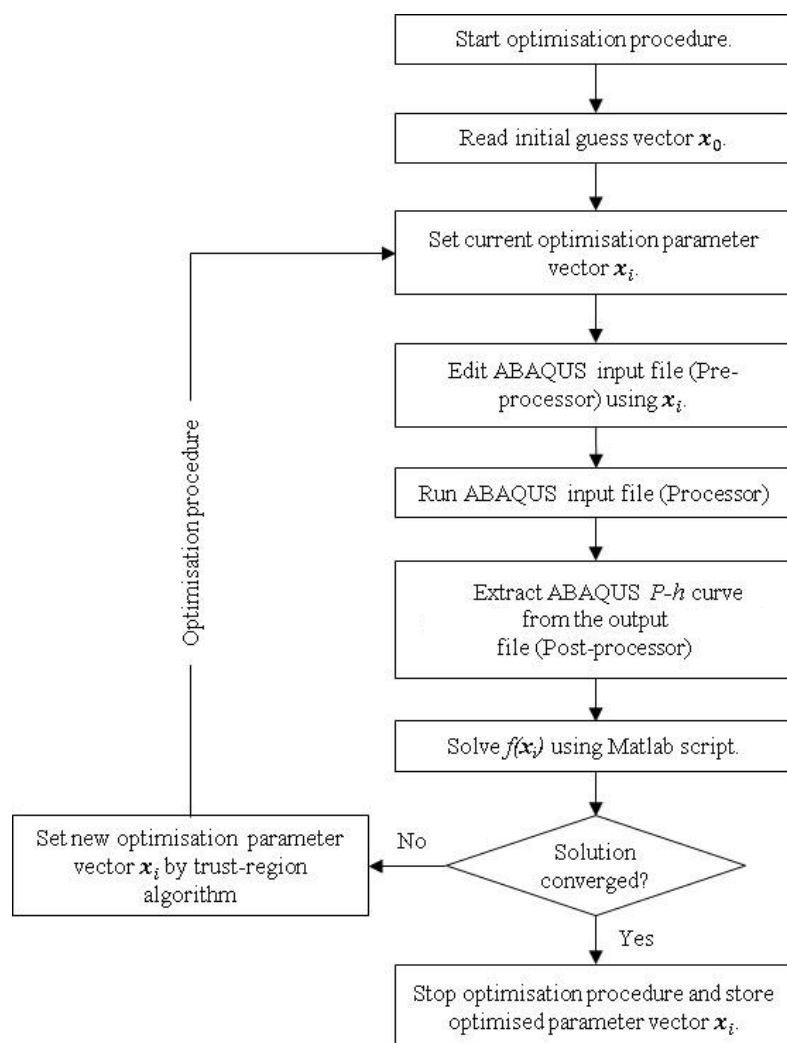


Figure 3.12: Flow chart of the single function optimisation procedure. Modified from [93].

3.6 Tensile testing

Tensile testing was used to measure bulk cross-weld mechanical properties such as yield stress (0.2% offset method), ultimate tensile stress (UTS), and ductility (elongation). These were then compared to

the values of the base metals (the bulk portion of each alloy) in order to demonstrate whether the joint could perform at least as well as the base materials. The samples were machined into tensile specimens with the dimensions specified in Figure 3.13 (the geometry was chosen due to the small transverse dimension of the forged joint), in accordance with ASTM E8/E8M-16a [94]. The tests were performed with 0.5 mm/min (strain rate $<10^{-3}\text{s}^{-1}$) extension rate, slow enough to ensure quasi-static behaviour, until fracture at room temperature (18°C) using an Instron Servohydraulic Test Machine. The fracture surfaces were observed using JEOL JSM-6490LV scanning electron microscope in the secondary electron mode in order to examine the fracture behaviour of the specimens.

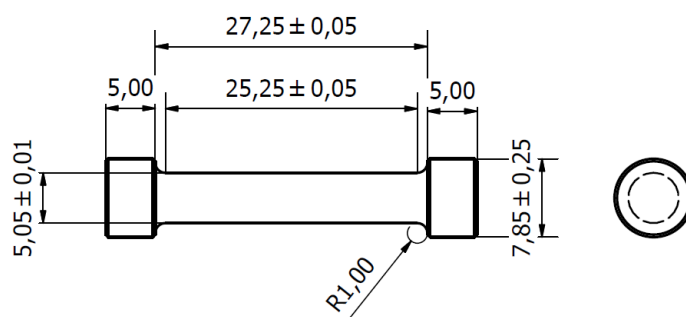


Figure 3.13: Tensile testing specimen dimensions (mm).

3.7 Thermodynamic equilibria phase calculations

In addition to experimental methods, it is also advantageous to undertake mathematical or computational modelling as it allows reasonable predictions and parametric analysis about the behaviour of materials without having to conduct large amounts of experiments.

One important type of calculations involves predicting the phases that may appear in a given alloy under given conditions, that is to *calculate* what phases are likely to occur under given the conditions (such as composition and temperature). This is used to predict the phases in equilibrium (i.e. the phases formed after a long period of time).

These calculations can be made using commercially available software packages of the CALPHAD (CALculation of PHase Diagrams) family. This technique utilizes minimization of Gibbs free energy to determine which phases are likely to appear in a given condition, as defined by several state variables

[95]. This is because within any material, only a phase or phases that has/have the lowest Gibbs free energy, compared to other possible phases of the alloy, can materialise. The CALPHAD method combines the equilibrium thermodynamic properties of all phases of a system in order to calculate the thermodynamic properties of the system as a whole, which can be used to generate a phase diagram. *ThermoCalc* software was used to conduct these calculations.

The formula for the Gibbs free energy, G , is the following:

$$G = U + P_{\text{thermo}}V - TS_{\text{thermo}} \quad 3.8$$

where U is the internal energy of the system, P_{thermo} is absolute pressure, V is volume, T is absolute temperature, and S_{thermo} is entropy.

Each phase has its own characteristic Gibbs free energy, obtained experimentally. The Gibbs free energies of each possible phases that can occur in the material at a given temperature can be plotted as a function of the composition as shown in an arbitrary binary system (i.e. an alloy consisting of 2 elements, here A and B) in Figure 3.14. Here, the composition is defined by the mole fractions of the two components, x_A and x_B , which is assumed to give rise to two possible phases: liquid and solid phases; the temperature is 1100°C. In this case, at this temperature, the liquid phase has a lower free energy at all molar compositions, and thus only liquid phase can appear at any composition at this temperature in equilibrium.

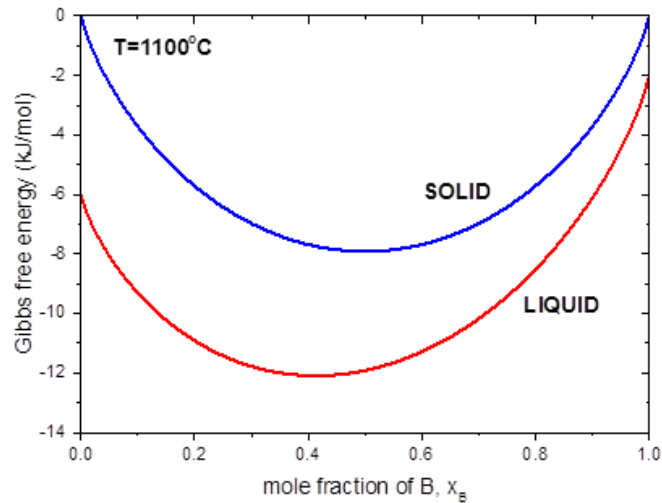


Figure 3.14: A plot of Gibbs free energies of two phases in an arbitrary binary system at 1100°C. Here, at this temperature, the liquid phase has a lower free energy at all molar compositions.

At a slightly lower temperature, 900°C, the Gibbs free energy of each phase changes and this causes the two curves to overlap, as shown in Figure 3.15. When $x > x_2$, the solid phase has the lower associated Gibbs free energy and thus will be the sole phase to appear in equilibrium. When $x < x_1$, only the liquid phase will be in equilibrium and thus only liquid phase will appear. When the composition is between x_1 and x_2 , both the solid and liquid phases will appear in equilibrium.

An important property of a phase is its chemical potential μ_i^p , which is the ability of a component i to change the energy of a given phase p . It is simply the rate of change of the Gibbs free energy of the phase with respect to the component:

$$\mu_i^p = \frac{\partial G_p}{\partial x_i} \quad 3.9$$

A system will be in equilibrium when the total Gibbs free energy is minimized. Any changes to the chemical potential within an alloy would drive change, so at equilibrium the chemical potential must be the same everywhere in the system of interest, though energy itself may vary across the system.

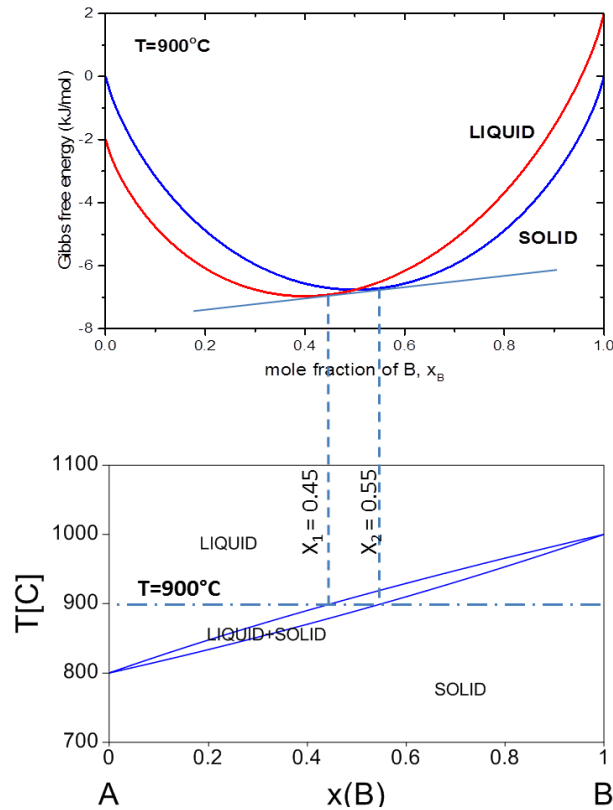


Figure 3.15: A plot of Gibbs free energies of two phases in an arbitrary binary system at 900°C . Minimization of Gibbs free energy here is achieved by the common tangent method. Underneath shows how phase diagram is constructed by referring to the Gibbs free energy curves at each temperature.

All setup of thermodynamic calculations must follow the Phase Rule. According to Gibbs, the number of phases ϕ , the number of degrees of freedom F , and the number of components χ , are related as follows:

$$\phi + F = \chi + 2 \quad 3.10$$

The *phases* refer to homogenous parts of a system, though in practice it may include parts of a system that are not completely homogenous. The *number of components* in this case means the number of constituent elements. The *number of degrees of freedom* refers to the number of conditions/variables that can be varied without changing the state of the system, or that have to be specified in order to fully describe the state of a system. This equation assumes that there are only three variables that can be externally controlled in order to effect phases in equilibrium: temperature, pressure, and composition.

Since the vapour pressure of the metal (in solid or liquid form) is usually negligible compared to the atmospheric pressure or service pressure, the pressure can be considered as a constant in most metallic systems. Thus the pressure variable would usually have negligible effect on the thermodynamics of metallurgical changes, which allows the phase rule to be reduced to:

$$\phi + F' = \chi + 1 \quad 3.11$$

where F' is number of degrees of freedom if pressure is assumed to be constant.

The ThermoCalc software was used to calculate property diagrams such as the molar and mass fractions of phases that appear in the alloys and their mixtures under equilibrium conditions. Two thermodynamic databases were used for these calculations. The TCFE7 database was used for alloys/mixtures containing >50 wt% Fe, while the TCNI8 database was used for alloys/mixtures containing >50 wt% Ni. With the important exception of C, elements of <0.01 wt% content were generally excluded to simplify the calculations if the review of literature suggests that they do not perform any role in the microstructural evolution of the respective alloys/mixtures. The pressure P_{thermo} was set at 1 atm and the system size was set to 1 mole. For the property diagrams, the phase fractions were calculated in the temperature range of 500-1200°C. Sometimes, if the output of the calculation included phases that may occur in equilibrium (i.e. after a long time) but were known to not appear in service timescales, such as graphite, these phases were excluded/suspended in the next iteration of calculations.

The limitation with this modelling method is that this is a calculation for equilibrium conditions and only considered the driving force for phase transformations (thermodynamics) and their results, not the mechanism or time dependency of the phase transformation. So non-equilibrium phases, including metastable phases such as martensite or bainite, will not appear in calculations even though they do appear in real alloys. In addition, the model will output phases that could eventually occur in equilibrium (in infinite amount of time), but may not appear in the alloy straight away after fabrication,

or even within the entire service life of the components. In reality, phase transformations and diffusions across alloys of different compositions may not have enough time to occur to appear in the form as predicted by CALPHAD. Thus, when using performing the computational methods, it is imperative to keep in mind that even if the CAPHAD models and the inputs are accurate, the results may not completely reflect the behaviours of real alloys as observed in experiments or in service. If phases which have sluggish kinetics of formation appear in the simulation results, then they can be omitted from the simulation which is run again to predict a metastable equilibrium. Which phases are to be accepted or rejected depends on which are observed to form from literature surveys.

Also, this model does not take into account the movement (i.e. diffusion) of elements into and out of the system in question. Hence, in terms of diffusion, ***diffusion modelling*** like those used in DICTRA (Diffusion-Controlled TRAnsformations) software need to be used to simulate or model non-equilibrium kinetics of diffusion in the interface between the different alloys. This method has been explored by a previous study using a fusion weld between P92 base and nickel-based IN625 alloy [64]. However, for the purposes of this project, this tool is not so useful because it does not take into account a possible factor in the kinetics of phase transformation, namely elastic stress [96]. Thus the simulation method cannot be applied to model the microstructural development crept samples as being distinct from that of thermally aged samples, as the only difference between these two types of samples are the presence or absence of applied mechanical stress. Hence, this type of modelling would not contribute to the relevant objective of this project, which is to compare the microstructural behaviour of the fusion weld at the interface of a base metal (P92) and weld filler (IN82/182) between thermally aged (i.e. without stress) and crept (mechanically stressed) samples at high temperature.

4 CHARACTERISATION OF A508/304L TRANSFORGE JOINT

4.1 Introduction

A macroscopic investigation of the A508/304L joint is presented in Figure 4.1 and shows some evidence of flow experienced by the lower strength A508 material during the forging process at the centre of the joint, not just at the edge where the lip is formed. It can also be seen that there are differences in the thickness of the transition zone between the centre of the specimen and the outer edge of the specimen. The centre of the TransForge joint contains the thin transition zone region, whereas the transition zone gets thicker nearer to the edge of the joints. A possible reason is that electron beam welding could have caused the transition zone to be thicker at the edges due to the width of the electron beam welding fusion zone itself being thicker at the edges prior to forging, which created a wider mixed zone.

Because the interfacial regions are distinctive in different parts of the joint, more than one location of the transition zone has to be studied in order to fully evaluate the interface between the two alloys. Therefore, the distinct interfacial regions are divided and studied as follows:

- Thin transition zone region, which originates from the centre of the forged A508/304L joint (the region around 'a' in Figure 4.1).
- Medium thickness transition zone region, which has the transition zone intermediate in thickness between the thin and thick transition zones, originating from a region between the thin and thick transition zone regions (the region around 'b' in Figure 4.1)
- Thick transition zone region, which originates from a region close to the outer edge of the forged A508/304L joint (the region around 'c' in Figure 4.1).

As a starting point for microstructural study, cross-weld microhardness profiles in the different regions above are presented to provide an indication of cross-weld microstructural variability. The microstructural studies of the bulk materials are first presented, followed by the microstructure in the

vicinity of the interface between the two alloys, divided into the different regions above. Nano indentation testing was also carried out to determine the cross-weld elastic-plastic properties of the thin transition zone region, by using the inverse method. Thermodynamic simulations of phase equilibria, using the ThermoCalc software, were carried out in order to assist the identification of phases present, such as possible precipitate phases and whether the matrix phases should be predominantly austenitic (FCC) or ferritic (BCC). Tensile testing were carried out to evaluate whether the microstructural features have any impact on the mechanical performance of the joint.

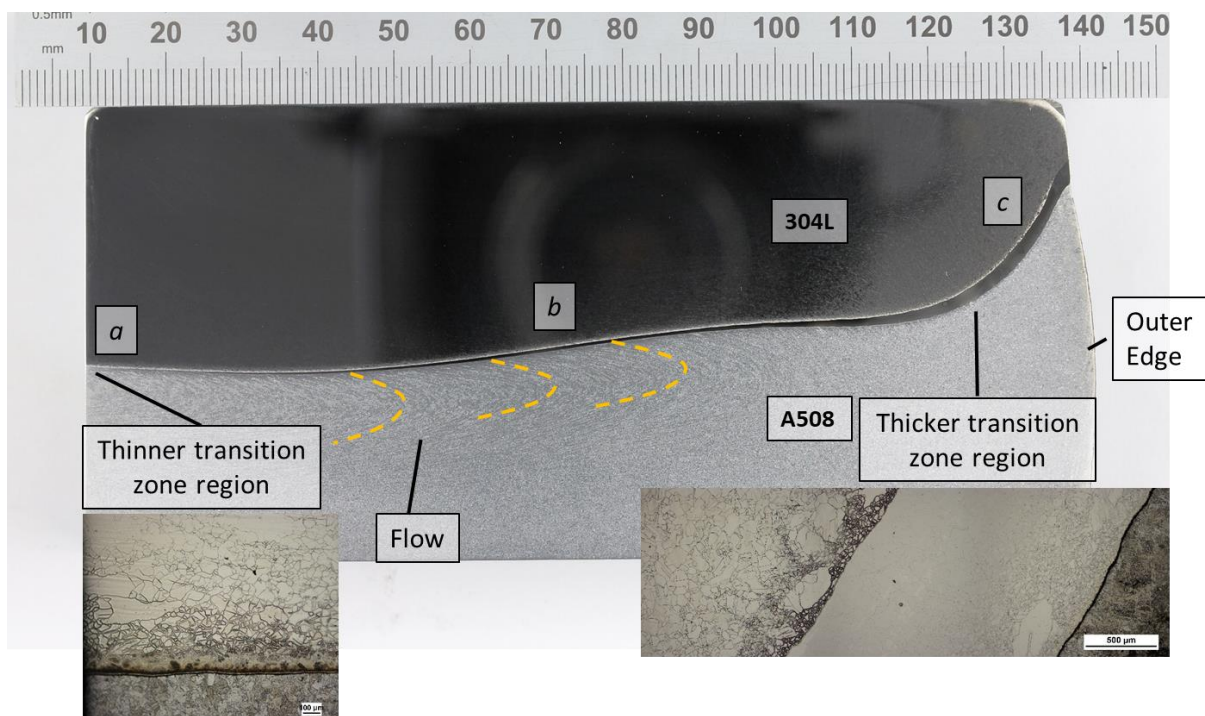


Figure 4.1: TransForge A508/304L joint after forging and post-forge heat treatment. Etched in 2% Nital followed by an electrolytic etch in sulphuric acid. (a) indicates the thin transition zone region, (b) indicates the medium thickness transition zone region, and (c) indicates the thick transition zone region.

4.2 Cross-weld microhardness profiles of A508/304L joint

Hardness values were taken across the interface of the A508/304L joint in three locations (thin, medium, and thick transition zone regions) with 100 μm spacing between indents, and these hardness profiles are presented in Figure 4.2. In addition, bulk alloy microhardness values were measured on 10 points far ($>10\text{mm}$) from the interface, which are presented in Table 4.1.

In all three regions, there is scatter of hardness values throughout A508, likely caused by the presence of MnS inclusions, since the formation and growth/deformation of inclusions would introduce stress

fields in their vicinity [97]. The measured hardness of 304L is less scattered, due to the fact that this is a largely single-phase austenitic alloy.

Figure 4.2 shows that there is great variation in microhardness across the weld in the A508/304L, suggesting that the A508/304L joint has microstructurally distinct zones.

Figure 4.2(a) shows that there is also scatter in the hardness of the bulk A508 material in the thin transition zone region of the A508/304L joint. In the bulk material, the hardness has an average of approximately 247 HV₂₀₀. From -1.5 mm to -0.1 mm there is a trend where the hardness gradually decreases from around 240 HV₂₀₀ to less than 180 HV₂₀₀ at -0.2 mm. The hardness peaks again at 272 HV₂₀₀ at the interface. On the other side of the interface, on the 304L side, the hardness is around 260 HV₂₀₀ before dropping sharply to around 210 HV₂₀₀ at 0.2 mm from interface. Then the hardness drops gradually to an around 180.0 HV₂₀₀. At present, it is unclear why this is slightly higher than the bulk hardness of 304L presented in Table 4.1.

Table 4.1: Microhardness measurements of the bulk material in the A508/304L joints. 10 measurements were taken at points far (>10mm) from the interface in each case.

	Microhardness measurements (HV ₂₀₀)	
	A508	304L
Mean	250.6	167.4
Range	25.7	14.3
Std Dev	8.4	5.6
Std Err	2.7	1.8

Similar to the thin transition zone region, the hardness of the A508 bulk material in the medium thickness transition zone region (Figure 4.2(b)) also drops from an average of around 240 HV₂₀₀ to less than 180 HV₂₀₀ at distance of -1.5 mm to -0.1 mm from the interface. The hardness increases to more than 400 HV₂₀₀ in the transition zone, before dropping to 221 HV₂₀₀ at the interface with the 304L bulk (which is about 0.4 mm from the A508 interface). Into the 304L bulk material, the hardness then gradually decreases to an average of 173 HV₂₀₀, at 1mm from the A508 interface (or 0.5 mm into the 304L bulk).

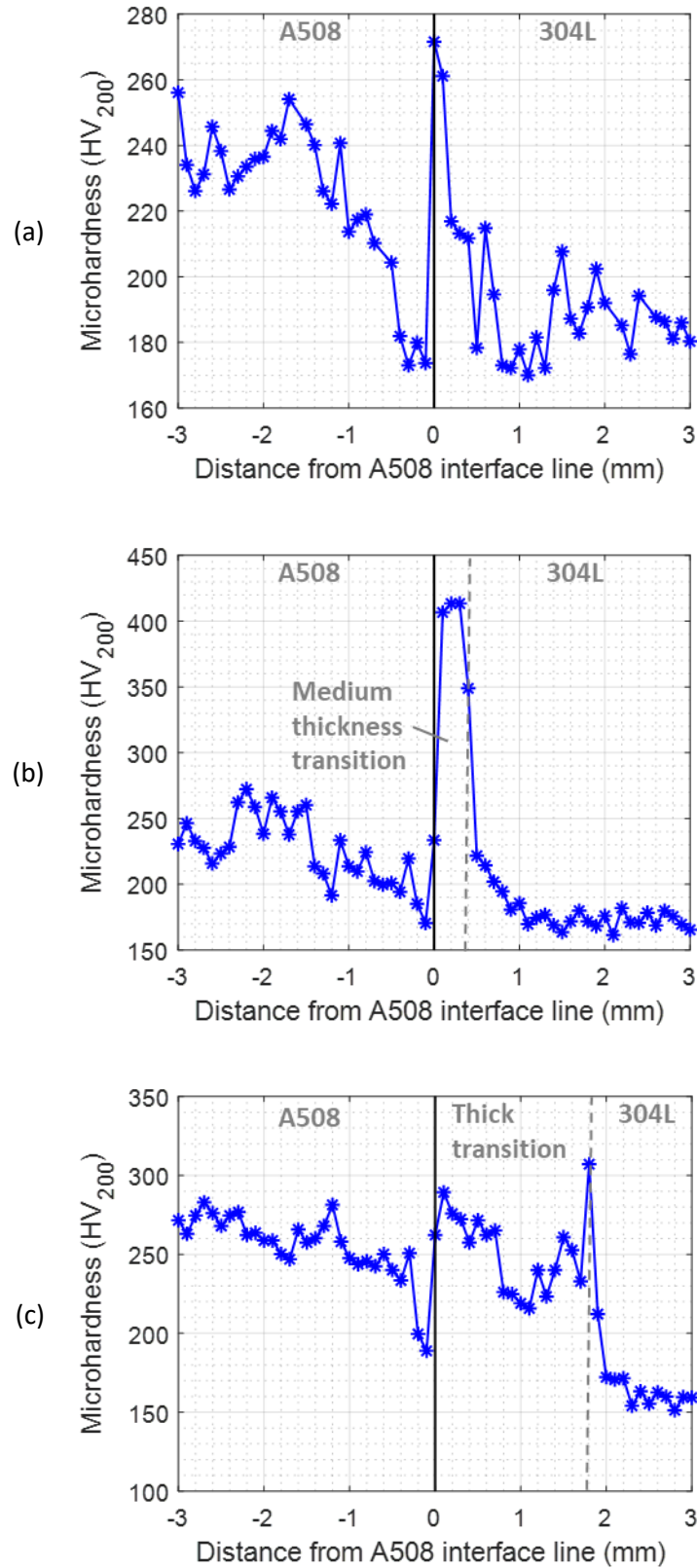


Figure 4.2: Microhardness profiles across interface of the A508/304L joint at (a) thin transition zone region, (b) medium transition zone region and (c) thick transition zone region. The dotted line indicates the boundary between thick transition zone and 304L viewed under optical microscope.

At 265 HV₂₀₀, the hardness of A508 bulk in the thick transition zone region (Figure 4.2(c)) is slightly higher than the other two regions and the bulk material, and there is less scatter overall. Similar to other regions in this joint, the hardness also drops to less than 190 HV₂₀₀ (but more than the other two regions) at distance of -1.5 mm to -0.1 mm from the interface. The hardness then increases to a value of around 270 HV₂₀₀ from the interface into 0.7 mm of the transition zone. From 0.8 to 1.1 mm, the hardness drops to average of 221 HV₂₀₀. The hardness then gradually increase to a peak of 307 HV₂₀₀ adjacent to the interface with the 304L bulk (about 1.8 mm from the interface with A508 bulk). In the 304L bulk, hardness then drops to about 170 HV₂₀₀ from 2.0 mm to 2.2 mm. and subsequently drops further to about 158 HV₂₀₀ in the rest of the 304L bulk.

The gradual change of hardness in the region about 1 mm on the A508 side of the interface, which results in a hardness trough that could be a weak zone, and 0.5 mm in the 304L side, can be seen in all three transition zone thickness regions.

4.3 Microstructure of bulk materials

The microstructure of the bulk materials in the joint, away from the interface between the alloys, were analysed in order to provide the starting point for the microstructural study of the joint. This can then be compared with the microstructure in the vicinity of the interface between the alloys in order to assess the weld integrity.

The microstructure of etched A508 bulk is presented in Figure 4.3. This shows typical tempered bainitic structure with some inclusions visible. A quick EDX analysis of the inclusions, presented in Figure 4.4, yields high concentration of Mn and S, which confirms that the inclusions are of MnS variety.

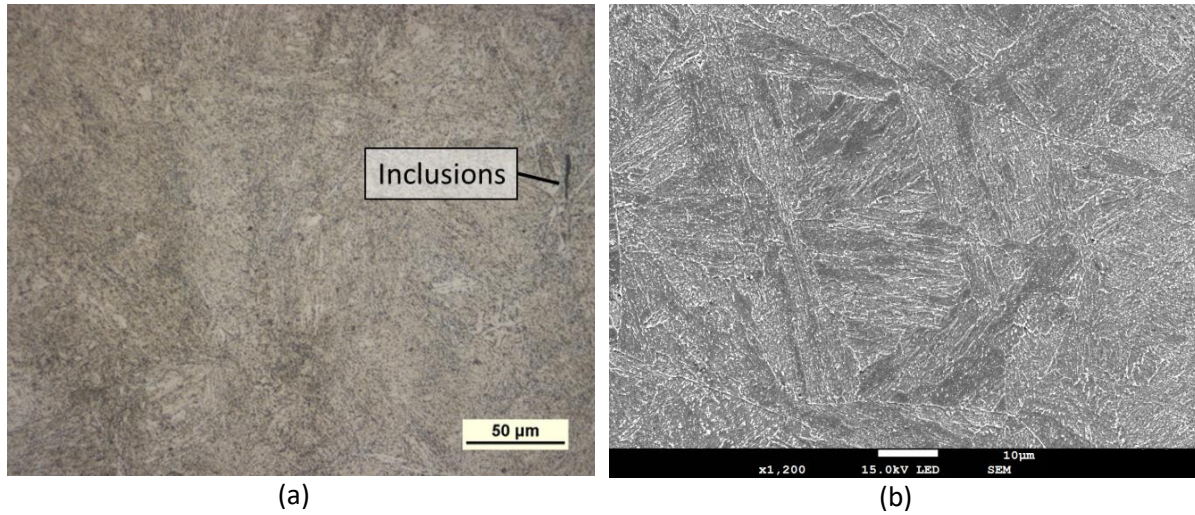


Figure 4.3: (a) Optical micrograph and (b) scanning electron micrograph of A508 bulk from A508/304L joint.

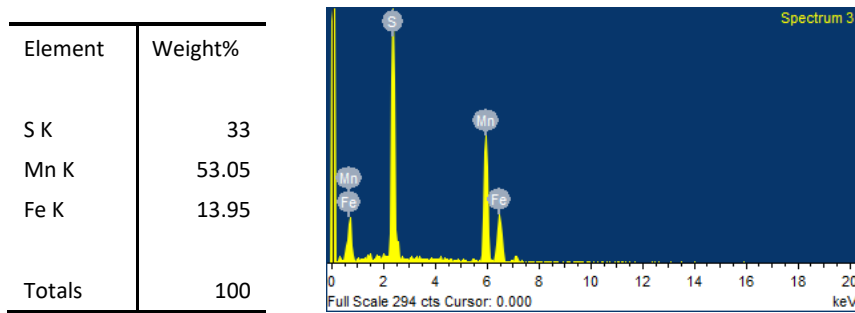


Figure 4.4: EDX analysis of the inclusion present in A508 bulk.

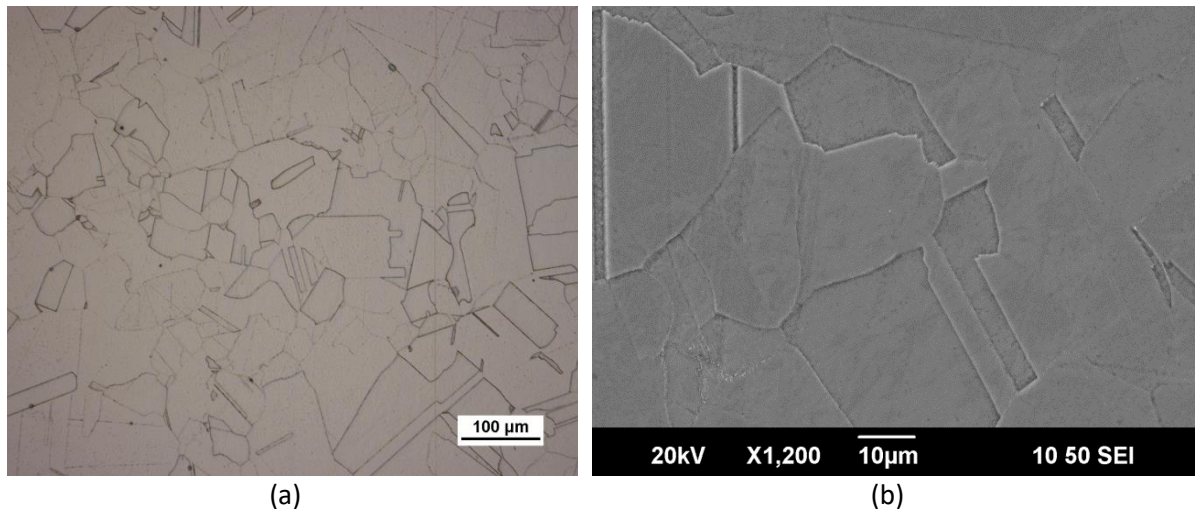


Figure 4.5: (a) Optical micrograph and (b) scanning electron micrograph of 304L bulk from A508/304L joint.

The microstructure of etched 304L bulk is presented in Figure 4.5, which shows normal equiaxed twinned austenitic grain structure, with precipitates not evident, only grain and twinning boundaries. There is no evidence of grain boundary precipitation (i.e. sensitisation), which would not be expected in the L-grade of the 300 series of austenitic stainless steel.

4.4 Microstructure of thin transition zone region of A508/304L

4.4.1 Cross-weld composition profile

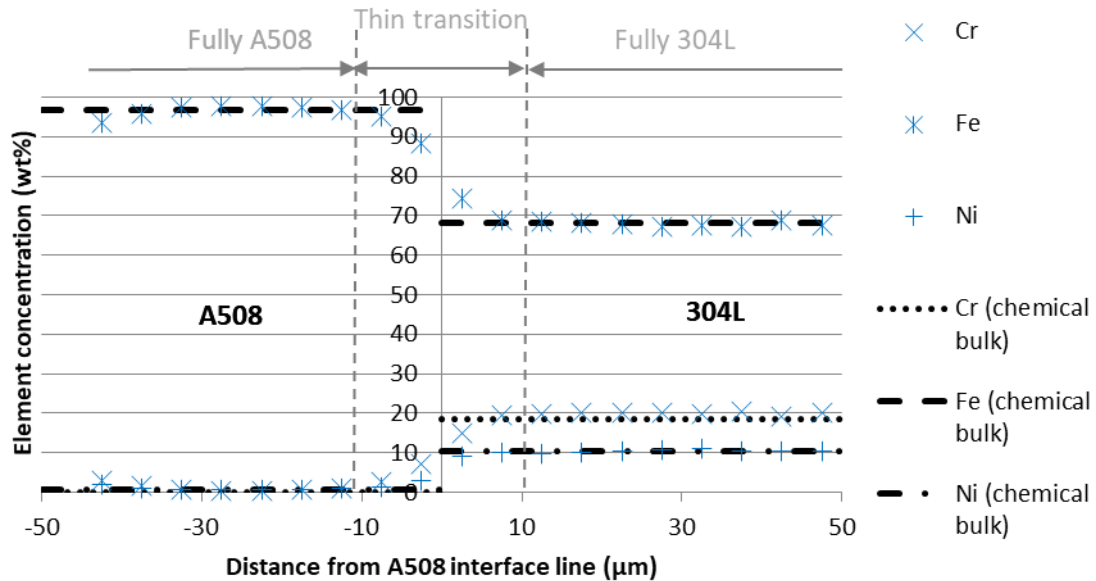


Figure 4.6: Composition profile of major elements (Cr, Fe, Ni) across thin transition zone region of A508/304L joint. The values for the chemical bulk compositions for each alloy originates from the chemical analysis provided by supplier of the bulk alloys.

The composition profile across the interface of the A508/304L joint in the thin transition zone region was measured, with 5 μm spacing between each measurement, using EDX analysis. This is presented in Figure 4.6, showing that the concentration of Fe in A508 is 97 wt% and transitions smoothly to around 68 wt% in 304L, which agrees with the nominal compositions of the alloys provided by the manufacturer. The same is true for Cr and Ni (Cr is 0.2 wt% in A508 and 20 wt% in 304L, while Ni is 0.7 wt% in A508 and 11 wt% in 304L). The change/transition in composition occurs at range of approximately 20 μm (10 μm on either side of joint interface line), with the joint interface line located at the middle of the compositional change. At this location, there is no evidence of EBW effect in the location of EDX measurement as the transition of composition between one alloy to another is too sharp (only 20 μm).

4.4.2 Microstructure of A508 side of interface in A508/304L joint

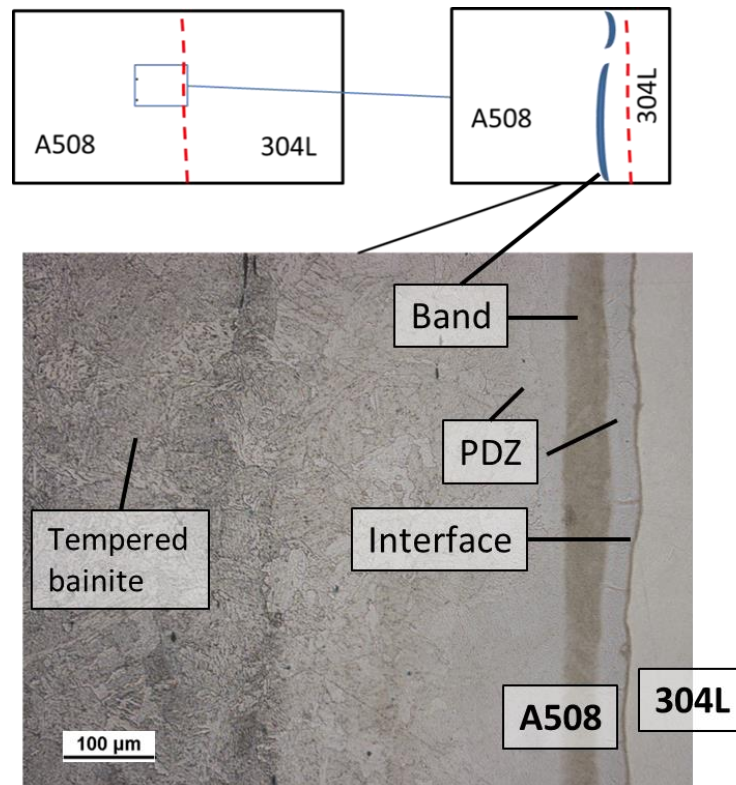


Figure 4.7: A508 side of the interface in the thin transition zone region of A508/304L joint.

The location of examination for microstructure of the A508 in the vicinity of the interface is displayed in Figure 4.7. Figure 4.8, which illustrates the etched A508 side of the interface, shows that the A508 does not etch well near the interface. This could indicate that there is lack of carbides in this vicinity, which may explain why the hardness dropped gradually in the A508 side of the interface for about 400 μm distance. This zone could be called the precipitate-depleted zone (PDZ).

Also noticeable is a semi-continuous band to the left of the interface within the A508 alloy, at around 40 μm from the interface. It is not continuous throughout the interface, appearing only in long separated bands. It is found that this band is slightly Cr- and Ni-rich (and to far lesser extent also Mn-rich) compared to the area on either side of it, as can be seen in EDX mapping in Figure 4.9. Figure 4.10 shows that this band seems to be made of numerous coarse precipitates. Further microhardness testing using 50 g load at three locations within the band shows that this band has high hardness value ($373.3 \pm 9.4 \text{ HV}_{50}$, compared with $\sim 240 \text{ HV}_{50}$ in A508 bulk). Qualitative line EDX analysis, plotted in Figure 4.12, shows that the concentrations of Cr and Ni increase in the band, at the expense of Fe.

Quantitative EDX analyses give these concentrations as approximately 87.9 ± 0.8 wt% Fe, 6.5 ± 0.4 wt% Cr, 3.8 ± 0.4 wt% Ni, and 2.0 ± 0.2 wt% Mn, which are higher than the composition of the bulk A508. It also shows that the band can have a gap within it, with elemental composition within the gap being similar to that adjacent to the band (i.e. in the PDZ). Note the composition profile in Figure 4.6 does not reflect this elevated level of Cr and Ni, likely due to the fact that this band was not present at the site of measurement because of the non-continuous nature of the band.

At this point, it is unclear what this band is, and why it forms there. An EBSD analysis, presented in Figure 4.11, shows that the boundary between the BCC and the FCC crystal structures correspond to the interface line between the A508 and 304L alloy. The band, which lies in the A508 side of the interface line, has matrix with BCC structure like the rest of A508 as well as PDZ, so the peculiar composition of the band did not affect its matrix crystal structure (though dense precipitates form in this vicinity as a result). However, the band does have a smaller block, packet and lath morphologies compared to the surrounding area.

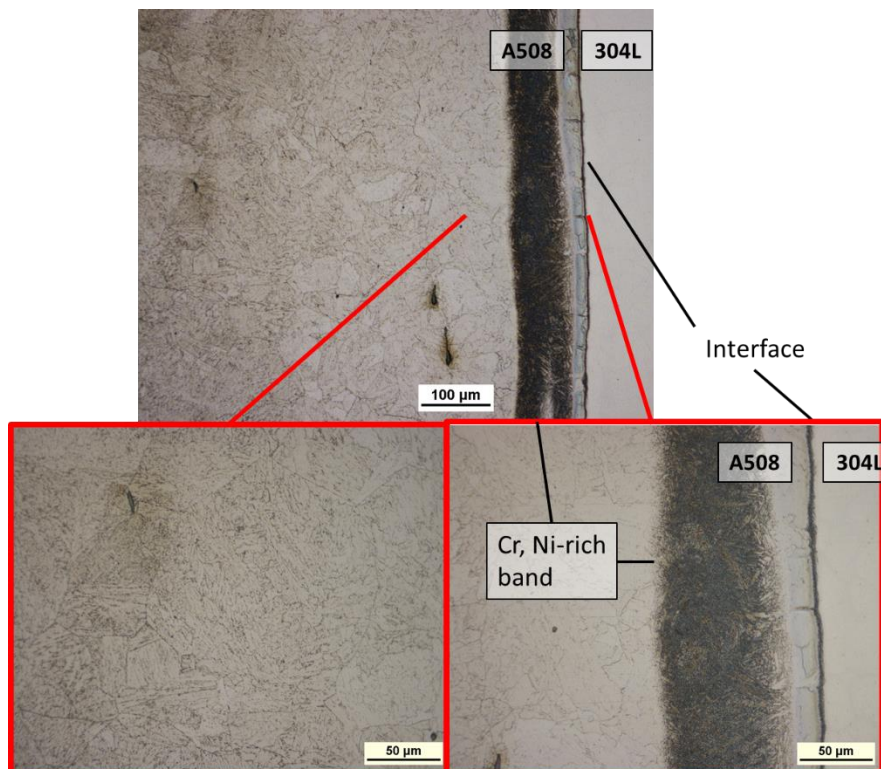


Figure 4.8: Optical micrograph of A508 steel side of the interface in the thin transition zone region of A508/304L joint.

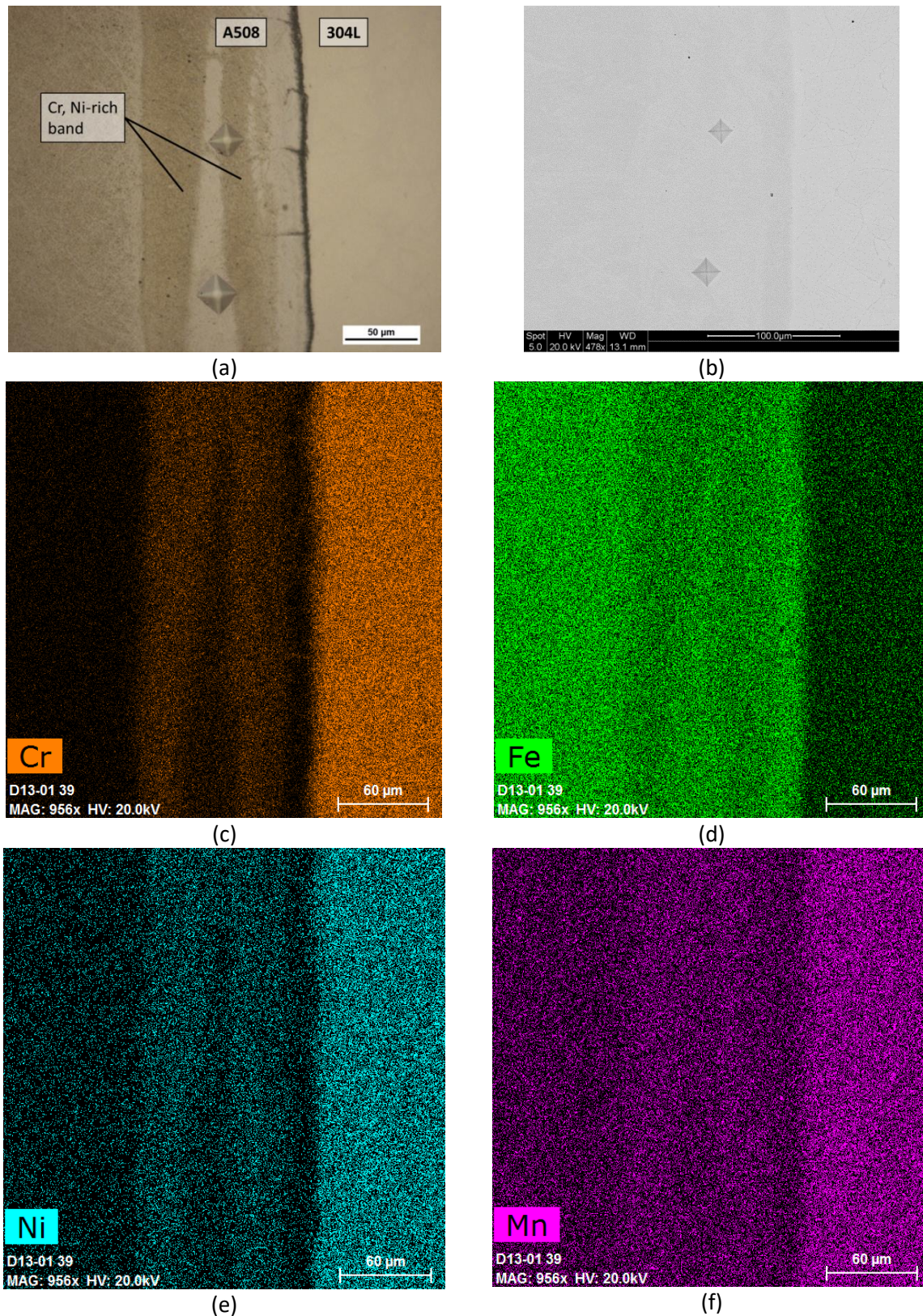


Figure 4.9: Analysis of semi-continuous band in the A508 side of interface of A508/304L joint, in the thin transition zone region. Note that the indents are just present as location marker. a) Optical micrograph of interfacial area and band, (b) BSE image of same location (polished). The corresponding EDX mapping (polished) for (c) Cr, (d) Fe, (e) Ni, and (f) Mn.

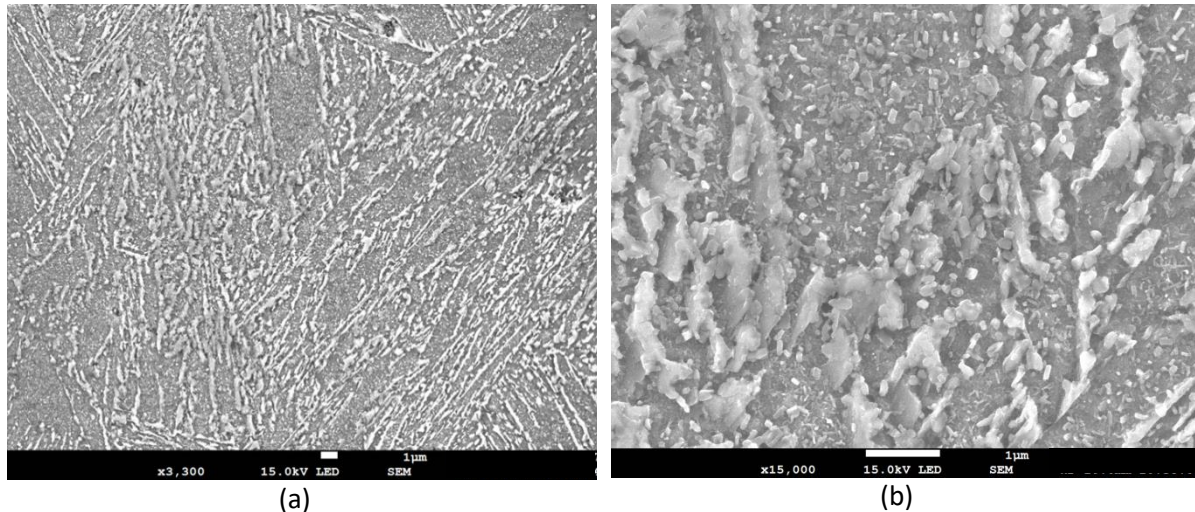


Figure 4.10: Scanning electron micrograph (SE mode) of the semi-continuous band in the A508/304L joint. The images represent different magnifications of the same site.

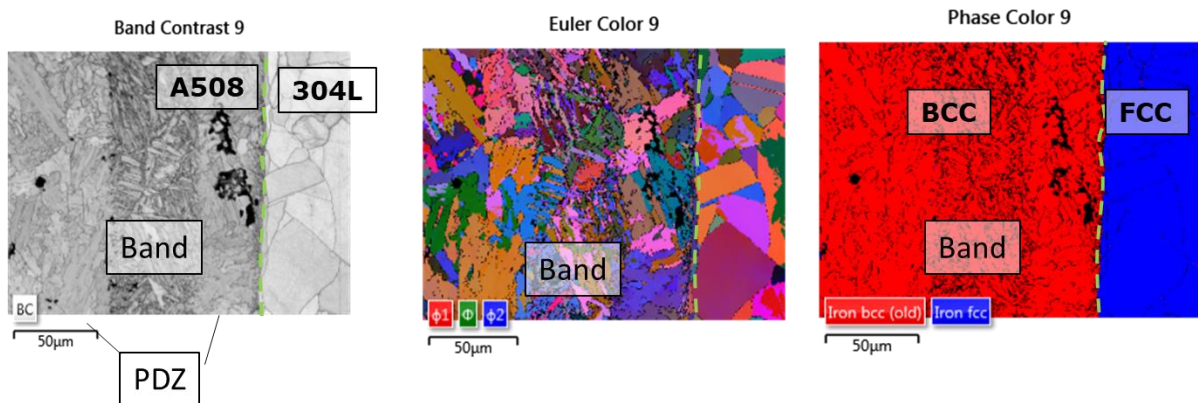


Figure 4.11: EBSD mapping of the semi-continuous band in the A508/304L joint, near the interface with 304L. Black indicates non-indexed solutions.

A more detailed microhardness profile, using 50 g load with 50 µm spacing and is taken at a 30° offset from the interface perpendicular, is shown in Figure 4.13. This plot confirms that:

- The interface has a high hardness value, but the semi-continuous band has even higher hardness values.
- Outside of the semi-continuous band, the hardness in the vicinity of the interfacial line is relatively low, corresponding to the less-etched region, but gradually increases further away from the interface.

It is also notable that the hardness measurement would spike in the vicinity of some inclusions (which from EDX analysis is identified to be likely MnS), indicating some sort of high strain region, and this is likely the source of scatter in the measured hardness of A508 steel mentioned earlier.

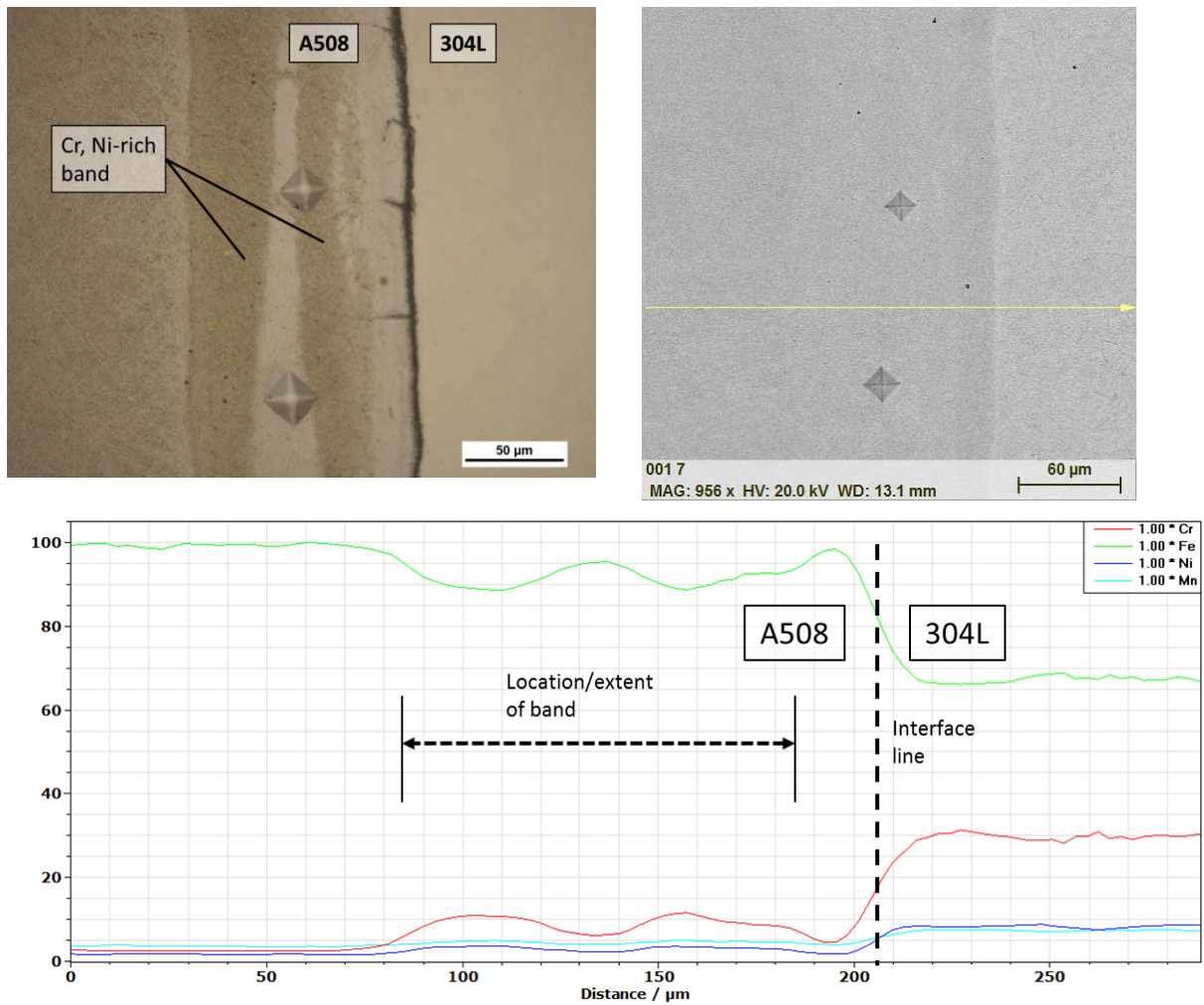


Figure 4.12: Line EDX analysis across the Cr- and Ni-rich band and the joint interface in A508/304L joint in the thin interfacial zone region (note that the y-axis scale is only qualitative).

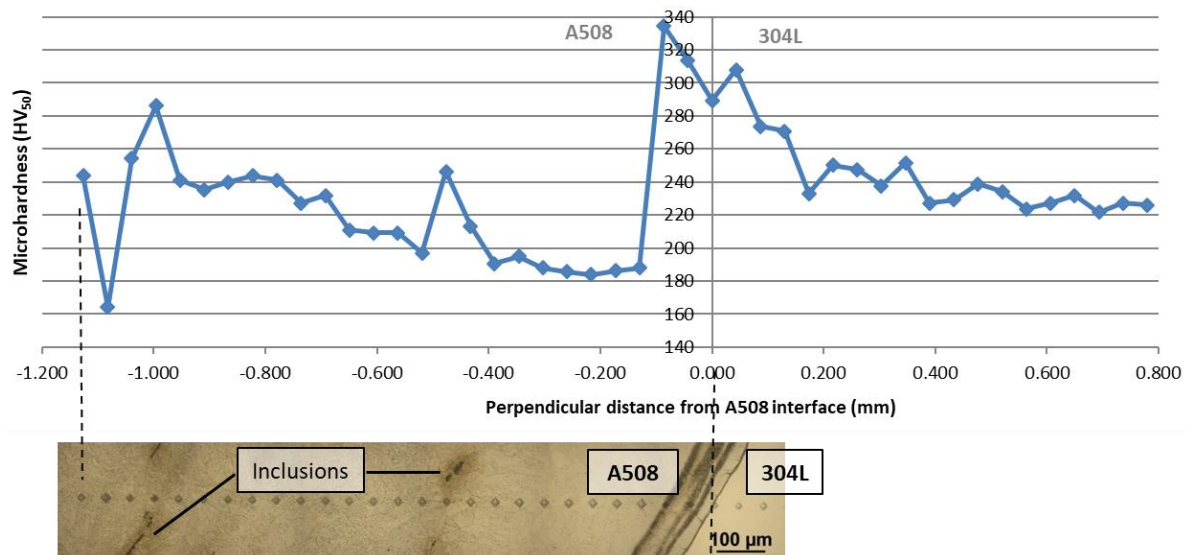


Figure 4.13: Detailed microhardness profile of A508/304L joint in the thin transition zone region, with the corresponding image of A508 side of interface. Note that because the hardness profile was measured at 30° offset from the perpendicular of the interface line, the distance axis in the plot is 'normalised' to the perpendicular distance from the interface line (using $\cos(30^\circ)$), so the axis does not exactly match with the scale bar in the image.

At the interface itself, the boundary between A508 and 304L are rough (indicating martensitic band formation) and precipitate dense, as can be seen in Figure 4.14. This may be due to carbon pileup at the interface as the carbon tries to migrate into the 304L side of the interface, which caused supersaturation of carbon and thus led to formation of precipitates. Figure 4.14 also confirms that there is lack of precipitates in the A508 side of the interface, which is almost featureless even after etching, except for perhaps the grain boundaries (possibly PAGB).

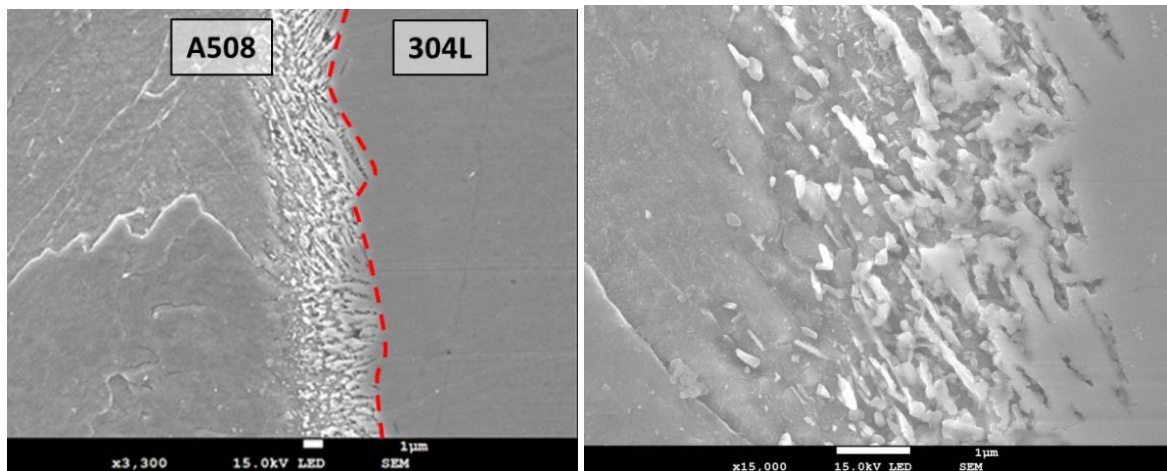


Figure 4.14: Scanning electron micrographs (SE mode) of A508 steel side of the interface of A508/304L joint at the thin transition zone region. The two images represent different magnifications of the same site.

4.4.3 Microstructure of 304L side of interface in A508/304L joint

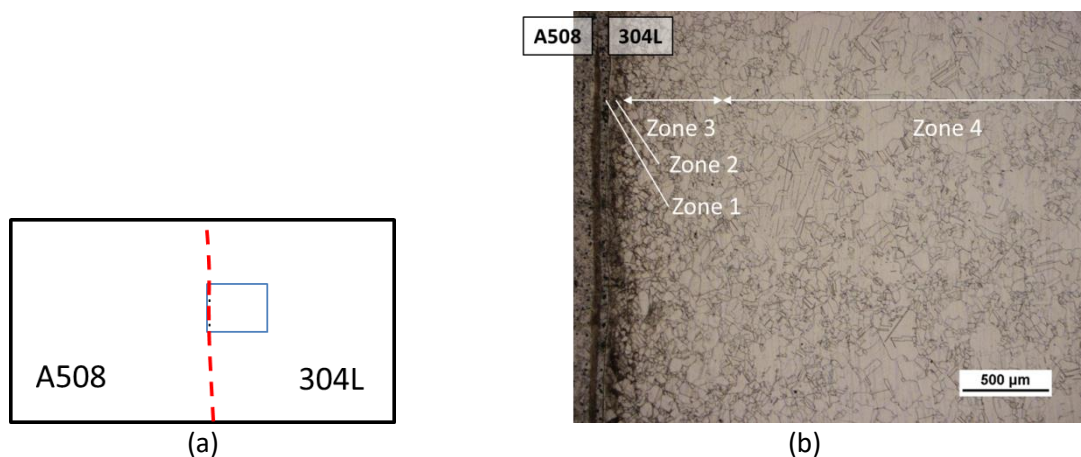


Figure 4.15: (a) Location of 304L side of interface. (b) Overview of etched 304L side of the interface of A508/304L joint in the thin transition zone region.

Figure 4.15 shows the location and an overview of several distinct microstructural zones in the 304L side of the interface. Figure 4.16 shows that, Zone 1, which is about 5 μm wide and is located next to the interface line, is shown to be devoid of precipitates. Zone 2, which is about 80-110 μm wide, has

both intergranular and intragranular precipitates. The intergranular precipitates are most likely some sort of Cr carbide like $M_{23}C_6$, but those inside the grains still need to be identified. Zone 3, which is about 400-450 μm wide, is similar to the Zone 2, but without the intragranular precipitates. Finally, Zone 4 is the bulk 304L.

Higher resolution images of the zones using SEM are presented in Figure 4.17. Zone 1 is shown to have no precipitates, even in grain boundaries. Zone 2 contains spheroid and needle precipitates within grains, and also precipitates in the grain boundaries as well as twinning boundaries. Zone 3 contains precipitates in the grain boundaries as well as twinning boundaries, but no precipitates within grains. The microstructure of Zone 4 is just the bulk 304L microstructure shown in a previous section.

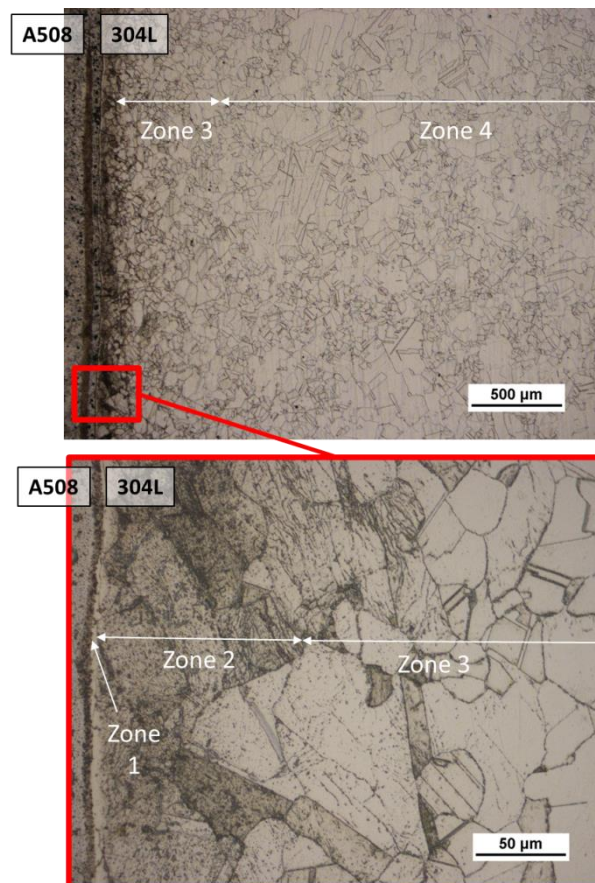


Figure 4.16: Optical micrograph of 304L steel side of the interface of A508/304L joint. Divided into distinct microstructural zones.

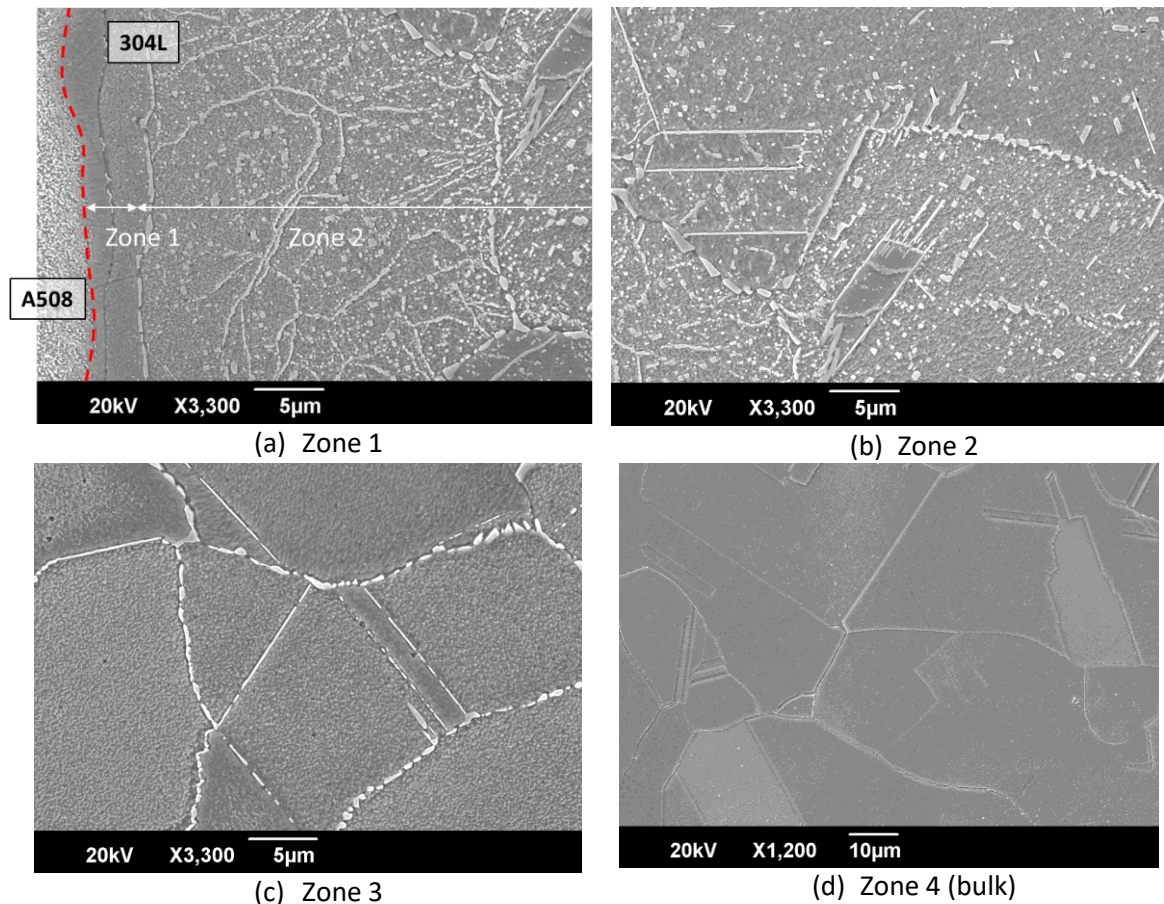


Figure 4.17: Scanning electron micrographs (SE mode) of the zones in the 304L steel side of the interface of A508/304L joint at the thin transition zone region. (a) Zone 1 (b) Zone 2 (c) Zone 3 (d) Zone 4.

4.4.4 Nanoindentation testing

Nanoindentation profile across the interface of the A508/304L joint in the thin transition zone region were measured, with 30 μm spacing between each indents, using a Berkovich indenter. Each indent was made with a controlled depth of 1 μm. Two profiles, with 33 indents each, were made from -480 μm to 480 μm distance from the A508 interface, as schematically shown in Figure 4.18. The nanoindentation profile traverses the entirety of PDZ of the A508 side of the interface, up to the start of Zone 4 (the 304L bulk). So all distinct microstructural zones can be said to be covered by the nanoindentation profile.

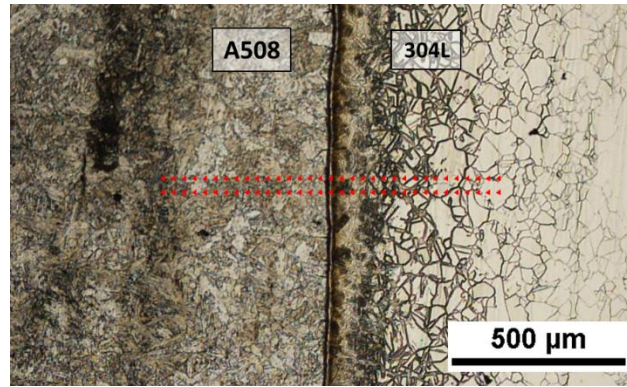


Figure 4.18: Schematic of the nanoindentation profile across the A508/304L joint in the thin transition zone region.

Each of the 66 indents yielded a P - h curve. For clarity, only 33 of these P - h curves, from the indents of the lower line in Figure 4.18, are reported in Figure 4.19. The P - h curves were used to optimise the parameters (elastic modulus E , Poisson's ratio ν , yield stress σ_y , and hardening exponent n) in FE simulation so that the simulated data would best match the measured P - h curves. An example of the optimisation process is shown in Figure 4.20. Figure 4.20(a) shows that the simulated P - h curves (in green) almost converges with the measured P - h curve (in red) for this particular indent. Figure 4.20(b) shows the parameters tested in each iteration of the optimisation process. The final (109th) iteration, where the normalised error is 0.381, yields the following parameters:

- $E = 366.95$ GPa
- $\nu = 0.2856$
- $\sigma_y = 603.49$ MPa
- $n = 0.247$

Profiles of the optimised nanoindentation parameters across the thin transition zone in the A508/304L joint using Limits Sets 1, 2, and 3 are presented in Figure 4.21, Figure 4.22, and Figure 4.23, respectively. Figure 4.21 shows that, when the upper limit of 300 GPa for E was imposed, the optimised E tends to scatter between 299 and 300 GPa. This indicates that the 300 GPa constraint has forced the values of E to be lower than they should at all indents. Figure 4.22 shows that, when the upper limit for E was increased to 400 GPa, many points still tend to go toward the maximum, though the normalised error decreased to 0.836 (compared to average normalised error of 1.153 when the

upper limit of 300 GPa for E was imposed). Normally, the value of E for steels should be around 180 to 220 GPa. The optimised E values in comparison are too high, which indicates that there may be issues with the optimisation method. Figure 4.23 shows the optimised parameters when E was derived from the stiffness S of each indent, by using Equations 3.1 and 3.3, while ν was assumed to be 0.3; only the other two parameters (σ_y and n) were optimised. It shows that on the A508 side of the interface, E is between 190 GPa and 210 GPa, but the stiffness spikes on the 304L side of the interface to 220 GPa, and then gradually decreasing to between 180 GPa and 190 GPa. While Limits Set 3 is the best for determining E , the optimised parameters yielded an average normalised error of 1.998, meaning is the most difficult for the simulated P - h curve to converge with the measured P - h curve. Also the σ_y profile in the Limits Set 3 is slightly elevated compared to those in the Limits Sets 1 and 2. This may be due to the fact that the optimisation process in Limits Set 3 compensates the inability to increase E (because E for each indent is pre-determined) by increasing the σ_y values, resulting in a higher σ_y values than it should be. Still, all three σ_y profiles have the same general trend, i.e. decreases from left side in the A508 bulk, to a trough on the A508 side of the interface, followed by a spike at the interface, and then gradually decreases deeper into 304L side of the interface.

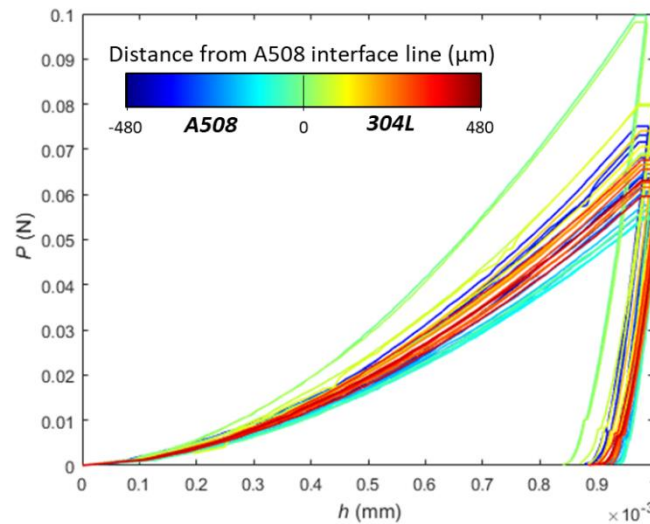


Figure 4.19: P - h curves measured from 33 of the nanoindentations across the A508/304L joint in the thin transition zone region.

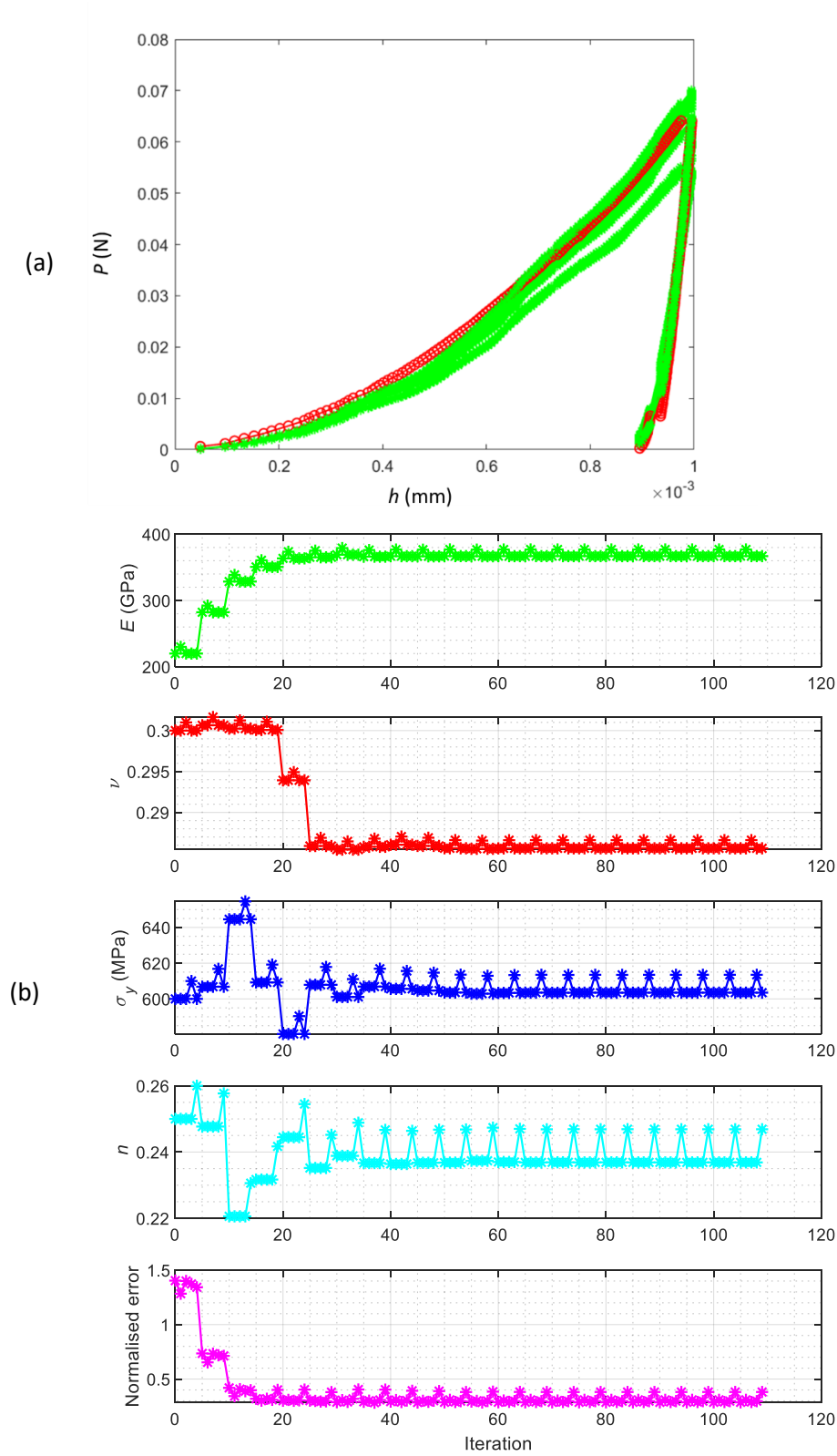


Figure 4.20: An example of a typical nanoindentation optimisation result (Limits set 2, indent #3) (a) Measured P-h curves (red) and simulated P-h curves (green) (b) Iterations of parameters (elastic modulus E , Poisson's ratio ν , yield stress σ_y , and hardening exponent n) tested in FE simulations.

The fact that the results are very different depending on the limits set, particularly for E , suggests that the inverse method used here may not have resulted in well-optimised $P-h$ curves. This is a known issue with single-objective functions, including this inverse method. It is possible that, in each optimisation problem, the solver has found a local minimum in the error function and converged on this, even though this may not be the global minimum. Once the solver did so, it cannot move from this local minimum to seek other local minima or indeed the global minimum.

Note that convergence has *not* been defined by whether the normalised error (i.e. the overall difference between the experimental and simulated $P-h$ curves) reaches a small value threshold. Recall that convergence is reached, terminating the optimisation process, when the solver attempts to take a step norm that is smaller than the defined lower bound on the size of the step ($\text{TolX} = 1\text{e-}8$), or that results in a *change* in the value of the objective function that smaller than the defined lower bound ($\text{TolFun} = 1\text{e-}8$). Perhaps the disadvantage of this optimisation process is that it was designed to make the solver converge to the first local minimum found, which is problematic in single-objective functions that may have many local minima. This is the reason why incorporating the Limits Sets is important, as they would help reduce the range of possible local minima in the optimisation space, but the ones used here seem to have failed to prevent the solver from converging to local minima that are not the global minimum. (The different Limits Sets resulted in different local minima being found by the solver, but none seem to be the global minimum.) An expansion of the inverse method by incorporating additional experimental data into a multi-objective optimisation function may alleviate this issue.

E is the particularly problematic parameter, as the optimised values of E kept reaching values that were higher than the known range of E values for steels. As mentioned, Limits Set 3 is perhaps the best for determining E precisely because Young's Modulus was directly calculated from the experimental $P-h$ curve, instead of being obtained through the optimisation process. Reasonable values of E can only be obtained when E (as well as ν) were excluded from the optimisation process,

suggesting that the inverse method in the current form cannot manage to produce reasonable values of E . Note that because the four parameters being optimised were not independent of each other in the inverse method, the results of the other three parameters presented here may also not be accurate and have to be taken with caution; even though, for example, all three σ_y profiles appear to have the same general trend.

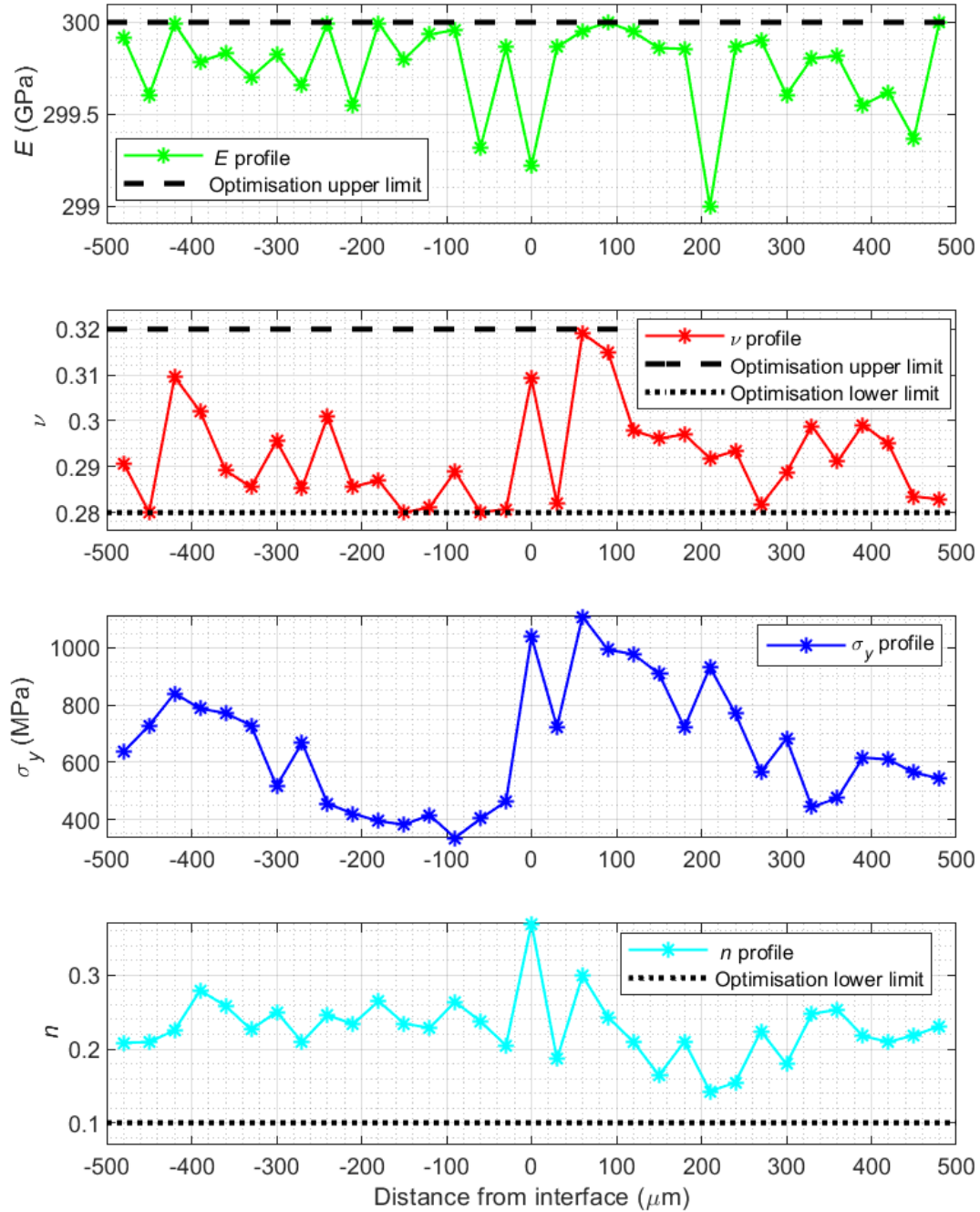


Figure 4.21: Profile of the optimised nanoindentation parameters across the thin transition zone in the A508/304L joint using Limits Set 1. The average normalised error is 1.153.

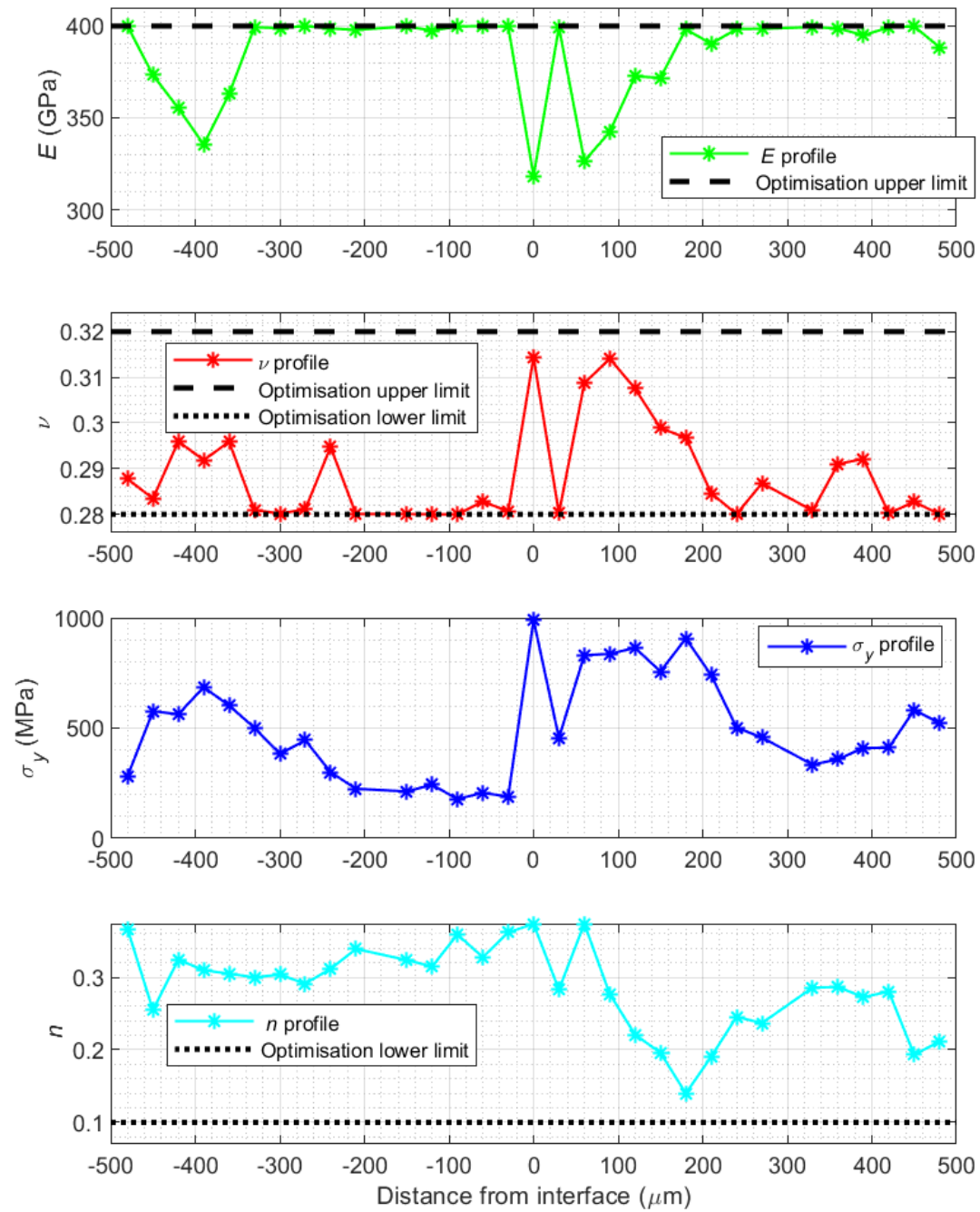


Figure 4.22: Profile of the optimised nanoindentation parameters across the thin transition zone in the A508/304L joint using Limits Set 2. The average normalised error is 0.836.

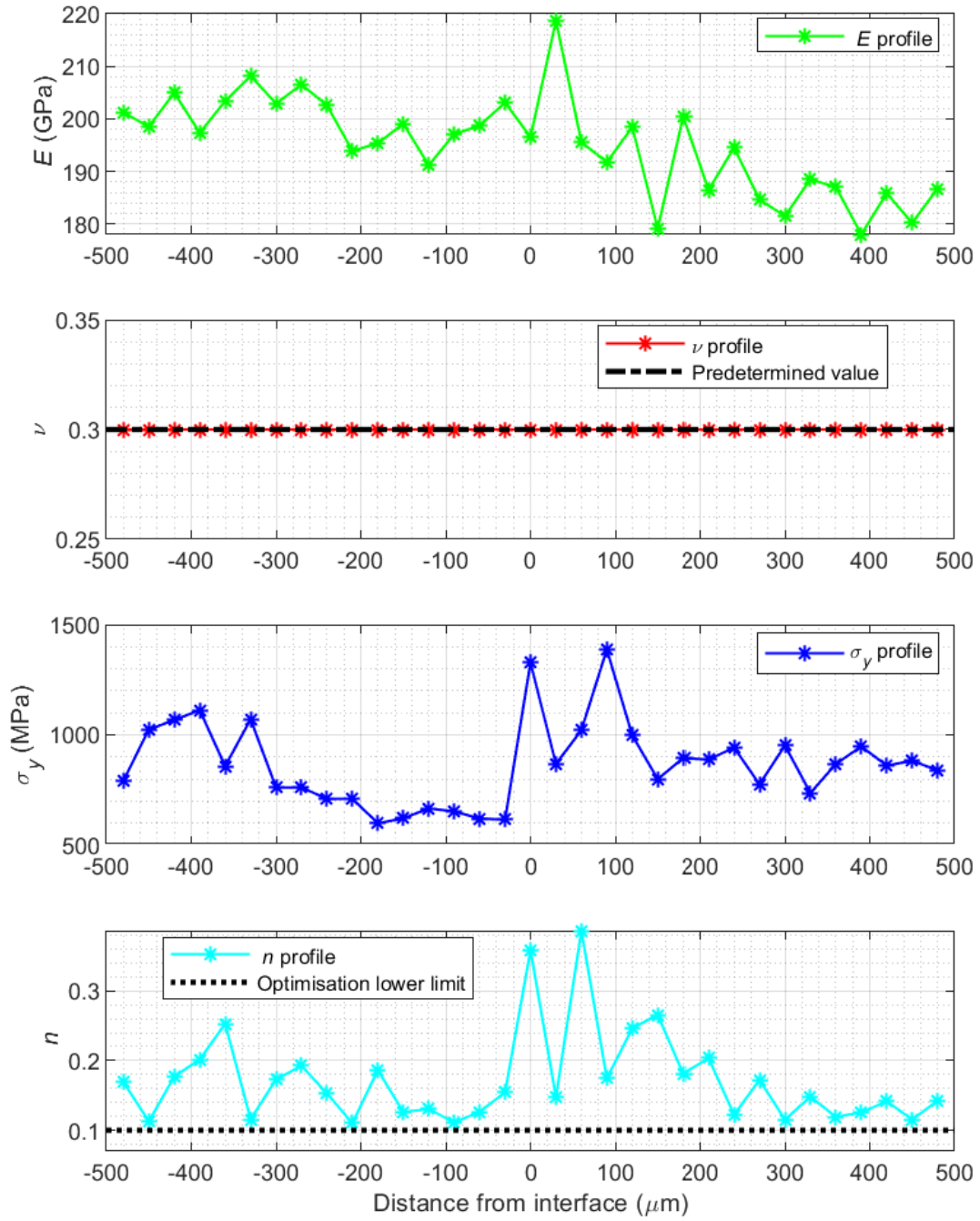


Figure 4.23: Profile of the optimised nanoindentation parameters across the thin transition zone in the A508/304L joint using Limits Set 3. The average normalised error is 1.998.

4.5 Microstructure of medium thickness transition zone region of A508/304L

4.5.1 Cross-weld composition profile

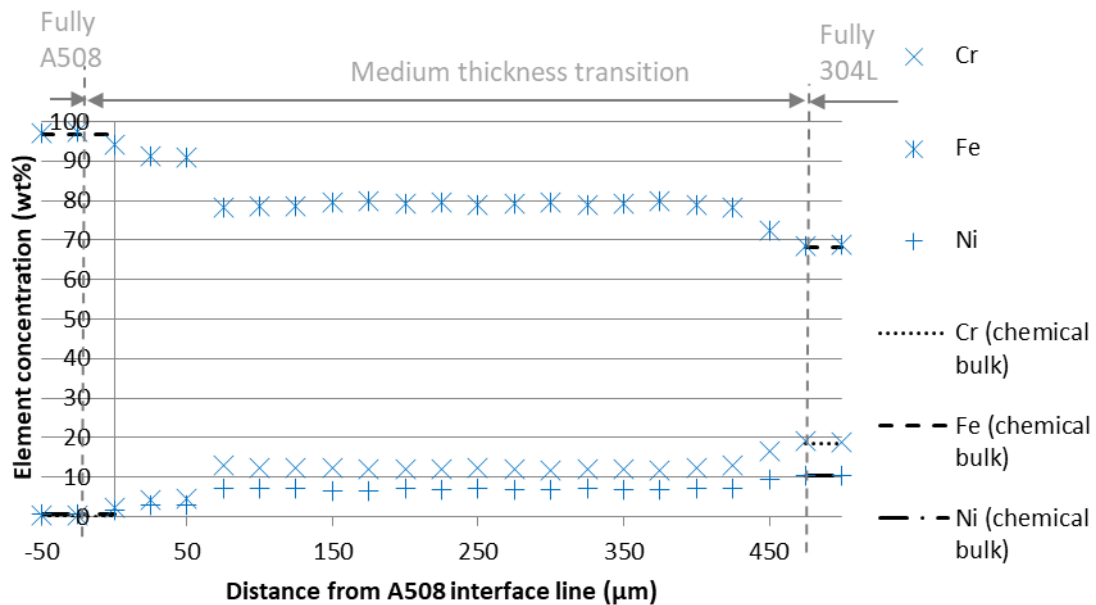


Figure 4.24: Composition profile of major elements (Cr, Fe, Ni) across medium thickness transition zone region of A508/304L joint. The values for the chemical bulk compositions for each alloy originates from the chemical analysis provided by supplier of the bulk alloys.

The composition profile across the interface of the A508/304L joint in the medium thickness transition zone region were measured, with 25 μm spacing between each measurement, using EDX analysis. This is presented in Figure 4.24, showing that there is clearly an EBW effect in the location of EDX measurement as the transition of composition between one alloy to another occur in a wide range. The concentration of Fe in fully A508 is 97 wt%, while it is around 69 wt% in fully 304L, which agrees with the nominal compositions of the alloys provided by the manufacturer. The same is true for Cr and Ni (Cr is 0.2 wt% in A508 and 19 wt% in 304L, while Ni is 0.6 wt% in A508 and 11 wt% in 304L). The change/transition in composition occurs at a range of approximately 500 μm, from -25 μm to 475 μm distance from the A508 interface, meaning that the start of the change in composition from the fully A508 into the transition which does not exactly match the interface line.

The change in composition across the transition zone is also not uniform or linear. The composition of Fe changes gradually from A508 bulk values at -25 μm to about 91 wt% (4.4 wt% for Cr, and 2.8wt% for Ni) at 50 μm. This may indicate the presence of a partially mixed zone (PMZ). The composition

then changes to 79 wt%, 12 wt%, and 7 wt% for Fe, Cr and Ni, respectively, at 75 μm , staying roughly constant to 425 μm . From 425 μm to 475 μm , the composition changes gradually to the bulk 304L values.

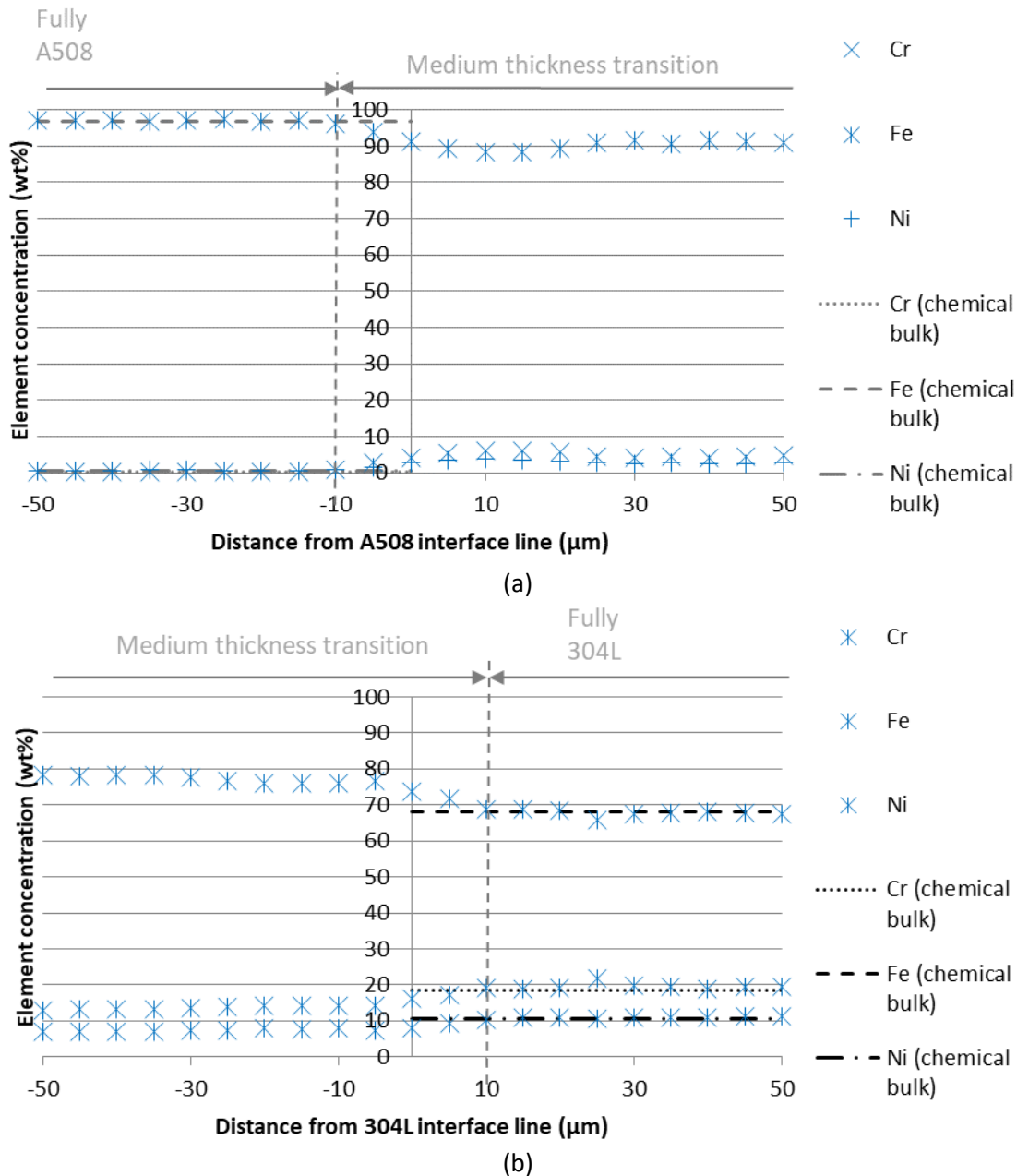


Figure 4.25: EDX profile in the vicinity of the interface lines between (a) A508-medium transition and (b) medium transition-304L.

Because the interface lines (A508-transition boundary and transition-304L boundary) seem to occur in transition compositions, i.e. do not appear to mark the boundary between fully bulk-material composition and the transition composition, a closer look at the composition in the vicinity of the interface lines is taken. An EDX profile with 5 μm spacing between each measurement is presented in

Figure 4.25 in the vicinity of the interface lines (i.e. the boundary between fully bulk-material composition and the transition composition). Figure 4.25(a) shows that the composition starts to shift from being fully A508 to the transition composition, at $-10\ \mu\text{m}$ into the A508 side of the interface. Similarly, Figure 4.25(b) shows that the composition starts to shift from being fully 304L to the transition composition, at $10\ \mu\text{m}$ into the 304L side of the interface. This confirms the fact that the interface lines do not coincide with start of the shift in composition from the bulk values; instead the shift starts to occur about $10\ \mu\text{m}$ within the bulk side of the interfaces.

4.5.2 Microstructure of A508 side of interface

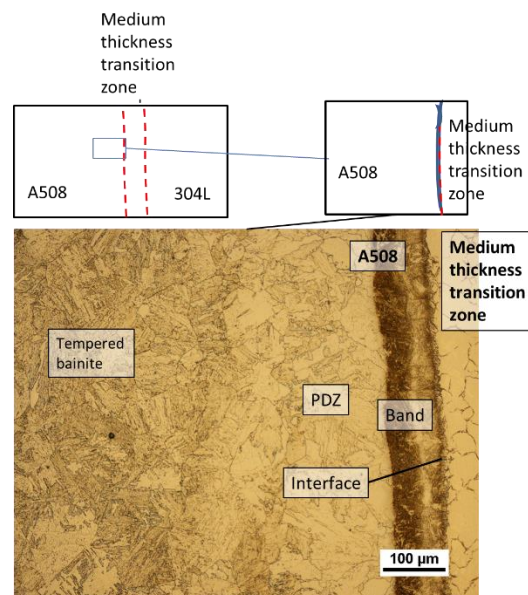


Figure 4.26: A508 side of the interface in the medium thickness transition zone region of A508/304L joint.

The location of examination for microstructure of the A508 in the vicinity of the interface is displayed in Figure 4.26. Similar to the thin transition zone region, the A508 does not etch well near the interface, as can be seen in Figure 4.27. The PDZ is almost featureless even after etching, except for perhaps the grain boundaries. However, the PDZ in this region is thinner, about $200\ \mu\text{m}$ wide.

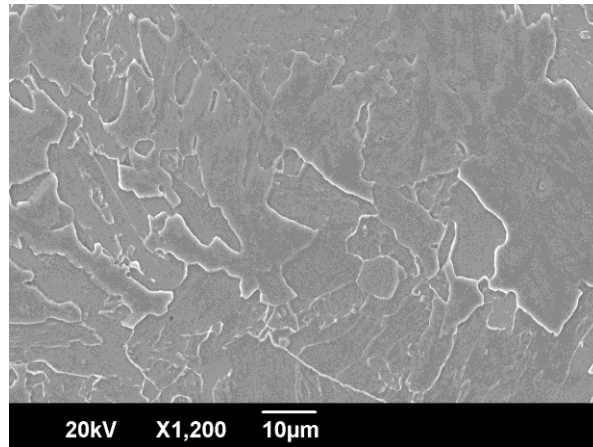


Figure 4.27: Precipitate depleted zone (PDZ) at the A508 side near the interface, in the medium thickness transition zone region.

The semi-continuous band to the left of the interface within the A508 alloy is also present, and is considerably thicker (about 100 µm), though it is adjacent to the interface instead of having a PDZ gap between it and the interface, as shown in Figure 4.28. The interface itself is rough, indicating the formation of martensitic band of about 10 µm wide, and full of precipitates, perhaps due to carbon pileup at the interface as the carbon tries to migrate into the transition side of the interface.

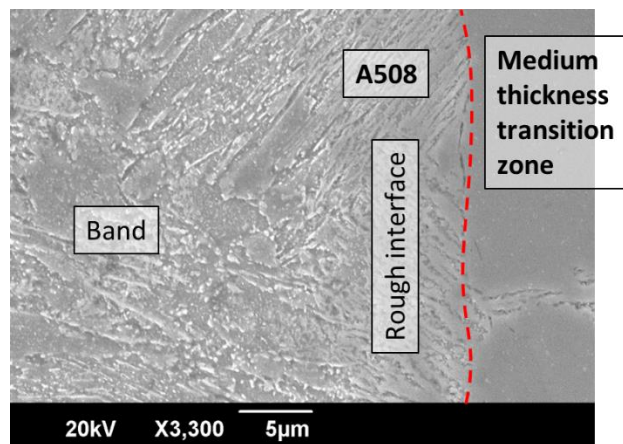


Figure 4.28: The interface A508 steel side of A508/304L joint at the medium thickness transition zone region.

4.5.3 Microstructure of medium thickness transition zone and the 304L side of the interface

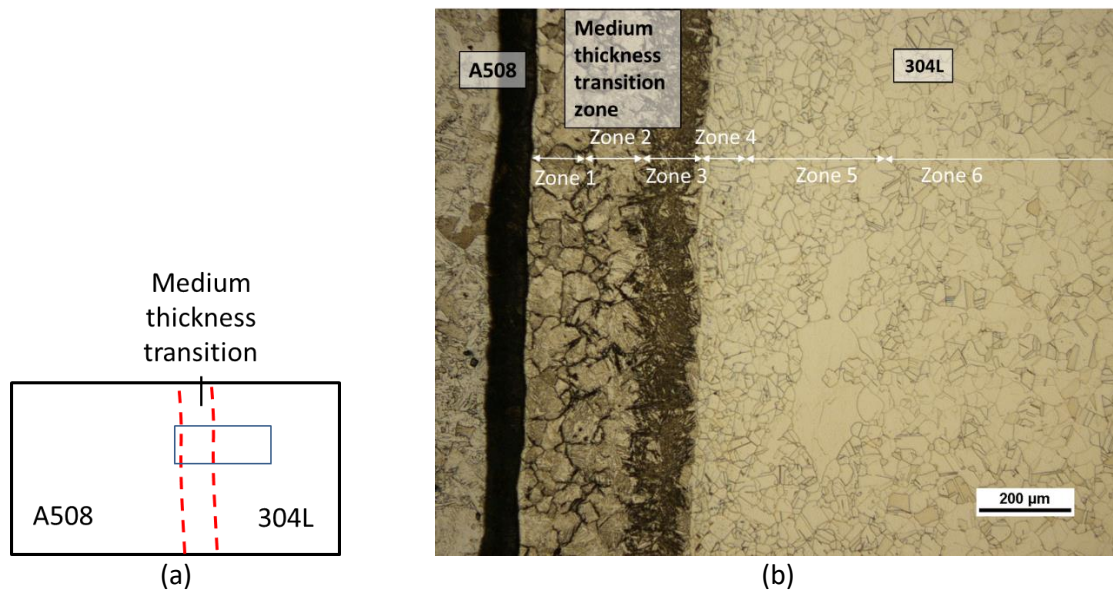


Figure 4.29: (a) Location of medium thickness transition zone. (b) Overview of etched medium thickness transition zone and 304L side of the interface of A508/304L joint in the medium transition zone region. The medium thickness transition zone consists of Zones 1-3, and 304L side consists of Zones 4-6.

Figure 4.29 shows the location and an overview of several distinct microstructural zones in the medium thickness transition zone and 304L side of the interface. The medium thickness transition zone, which is about 360-400 μm wide, is divided into Zones 1-3, and 304L side is divided into Zones 4-6. Figure 4.29(b) shows that Zone 1, which is about 80-100 μm wide, has a grain structure with twins and intergranular precipitates that are arranged in a plate martensite fashion. Zone 2, which is about 100-120 μm wide, has even thicker intergranular plate martensite precipitates, but no twins. Zone 3, which is about 160-180 μm wide, has a very (tempered) martensitic structure with dense precipitates, which is similar to the hard band found in the A508 side of the interface. Zone 4, which is about 100-110 μm wide and lies on the 304L side of the interface, has equiaxed twinned austenitic grain structure with both intergranular and intragranular precipitates. Zone 5, which is about 290-350 μm wide, is similar to Zone 4, but without the intragranular precipitates. Finally, Zone 6 is the bulk 304L.

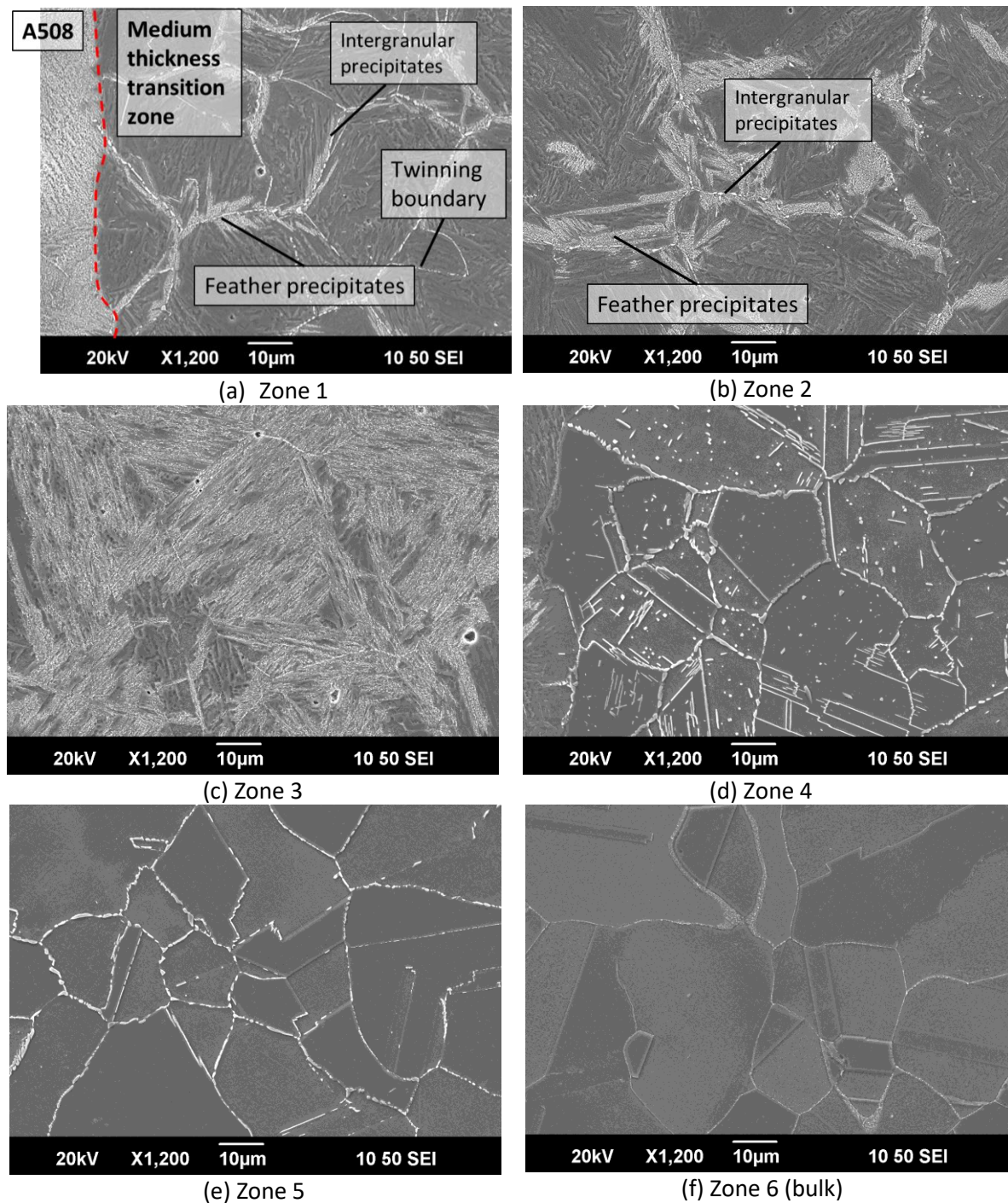


Figure 4.30: Scanning electron micrographs (SE mode) of the zones in the 304L steel side of the interface of A508/304L joint at the medium thickness transition zone region. (a) Zone 1 (b) Zone 2 (c) Zone 3 (d) Zone 4 (e) Zone 5 (f) Zone 6.

Higher resolution images of the zones using SEM are presented in Figure 4.30. Zone 1 has intergranular precipitates that are expected to be $M_{23}C_6$. The fine precipitates of the plate martensite surrounds the grain boundaries. Zone 2 also contains intergranular precipitates and plate martensite precipitates, but the intergranular precipitates are scarcer and the plate martensite precipitate formations are thicker. Zone 3 contains dense plate martensite, but the intergranular precipitates are thin. Zone 4

contains spheroid and needle precipitates within grains, and also precipitates in the grain boundaries as well as twinning boundaries. Zone 5 contains precipitates in the grain boundaries as well as twinning boundaries, but no precipitates within grains. The microstructure of Zone 6 is just the bulk 304L microstructure.

4.5.4 EBSD crystal structure in the vicinity of the medium thickness transition zone

EBSD analysis of the medium thickness transition zone and the surrounding bulk material, presented in Figure 4.31, shows that A508 side and 304L side of the respective interface lines with the transition zone have BCC and the FCC crystal structures, respectively. The hard band in the A508 side of the interface has BCC crystal structure, the same as the rest of A508. The medium thickness transition zone has a more complex set of crystal structures. Much of the area in this transition zone is non-indexed. Interestingly, while most of this zone appears to have BCC crystal structure, there is a band of FCC crystal structure adjacent to the A508 interface and fades into the BCC structure further from the A508 interface. Most of the FCC crystal structure in this region is within 80 μm from the A508 interface, which roughly corresponds to Zone 1. The presence of FCC crystal structure, hence austenitic crystal structure, in Zone 1, may explain the presence of the twinning boundary observed there. EDX mapping, displayed in Figure 4.32, shows that the composition of Fe, Mn, and Ni are more or less constant throughout the medium thickness transition zone.

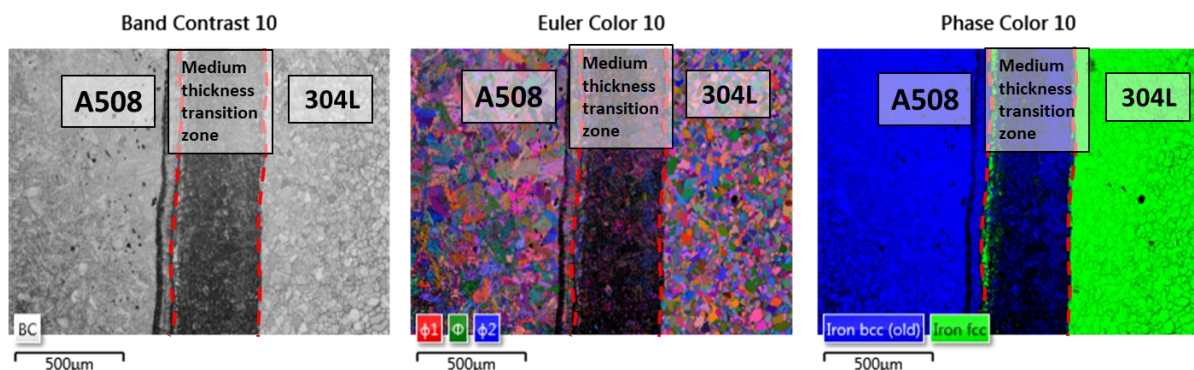


Figure 4.31: EBSD mapping of the vicinity of the medium thickness transition zone in the A508/304L joint. Black indicates non-indexed solutions.

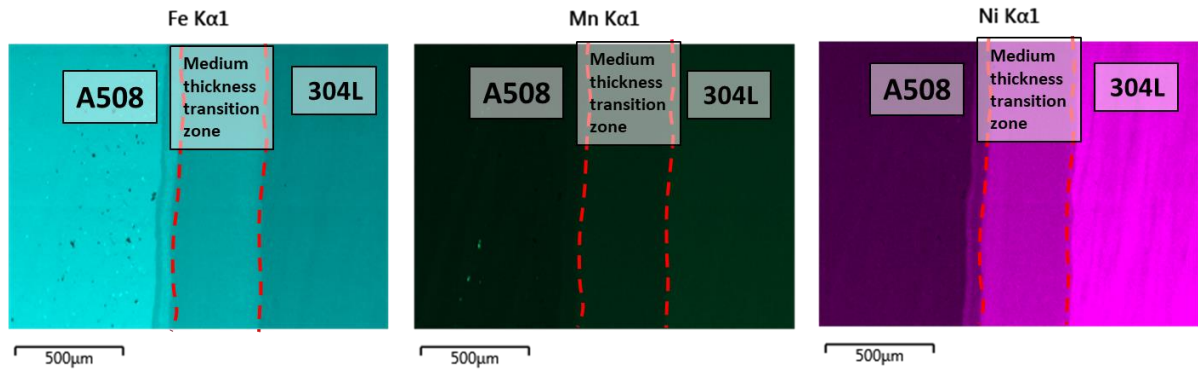


Figure 4.32: EDX mapping of Fe, Mn, and Ni in the vicinity of the medium thickness transition zone in the A508/304L joint.

4.6 Microstructure of thick transition zone region of A508/304L

4.6.1 Cross-weld composition profile

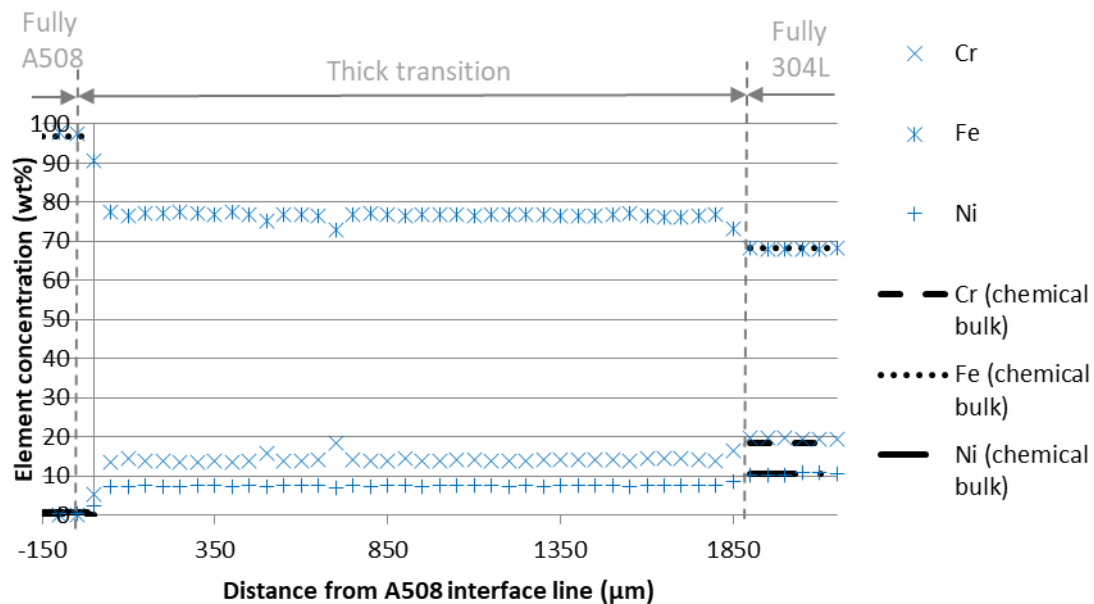


Figure 4.33: Composition profile of major elements (Cr, Fe, Ni) across thick transition zone region of A508/304L joint. The values for the chemical bulk compositions for each alloy originates from the chemical analysis provided by supplier of the bulk alloys.

The composition profile across the interface of the A508/304L joint in the thick transition zone region were measured, with 50 µm spacing between each measurement, using EDX analysis. This is presented in Figure 4.33, showing that there is clearly an EBW effect in the location of EDX measurement as the transition of composition between one alloy to another occur in a wide range (about 1950 µm wide). The concentration of Fe in fully A508 is 97 wt%, while it is around 68 wt% in fully 304L, which agrees with the nominal compositions of the alloys provided by the manufacturer. The same is true for Cr and Ni (Cr is 0.17 wt% in A508 and 19 wt% in 304L, while Ni is 0.4 wt% in A508 and 11 wt% in 304L).

The change/transition in composition occurs from -50 μm to 1900 μm distance from the A508 interface, meaning that the start of the change in composition from the fully A508 into the transition which does not exactly match the interface line. At most of the transition zone, the composition of Fe, Cr, and Ni are more or less constant at 77 wt%, 14 wt%, and, 7.5 wt% respectively from 50 μm to 1800 μm .

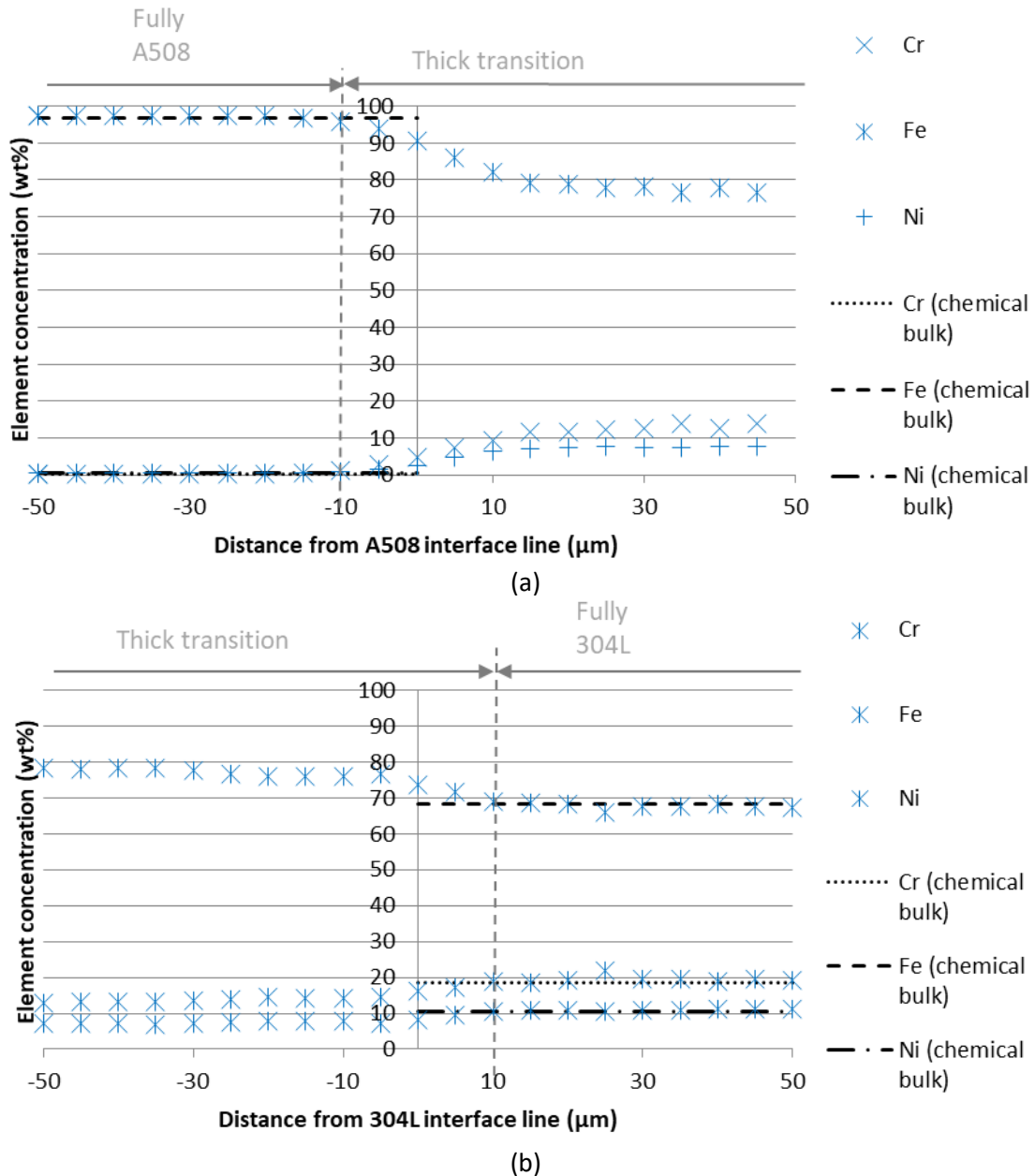


Figure 4.34: EDX profile in the vicinity of the interface lines between (a) A508-thick transition and (b) thick transition-304L.

The interface lines (A508-transition boundary and transition-304L boundary) seem to occur in transition compositions, i.e. do not appear to mark the boundary between fully bulk-material

composition and the transition composition; hence a closer look at the composition in the vicinity of the interface lines is taken. An EDX profile with 5 μm spacing between each measurement is presented in Figure 4.34 in the vicinity of the interface lines (i.e. the boundary between fully bulk-material composition and the transition composition). Figure 4.34(a) shows that the composition starts to shift from being fully A508 to the transition composition, at 10 μm into the A508 side of the interface. Similarly, Figure 4.34(b) shows that the composition starts to shift from being fully 304L to the transition composition, at 10 μm into the 304L side of the interface. This confirms the fact that the interface lines do not coincide with start of the shift in composition from the bulk values; instead the shift starts to occur about 10 μm within the bulk side of the interfaces.

4.6.2 Microstructure of A508 side of interface

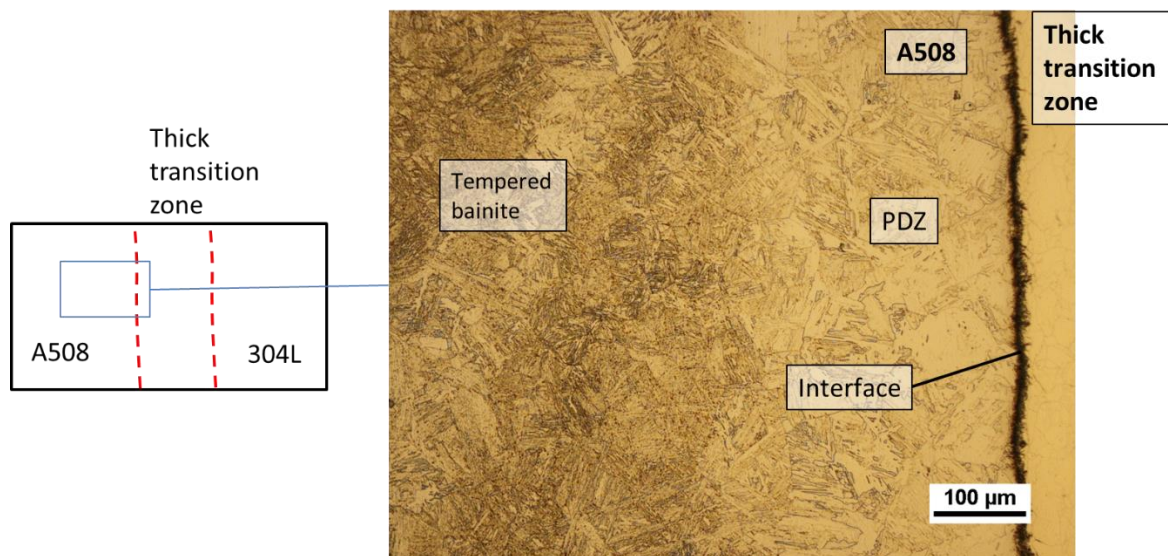


Figure 4.35: A508 side of the interface in the thick transition zone region of A508/304L joint.

The location of examination for microstructure of the A508 in the vicinity of the interface is displayed in Figure 4.35. Similar to the other regions, the A508 does not etch well near the interface, as can be seen in Figure 4.36. The PDZ in this region is also thin, about 200 μm wide.

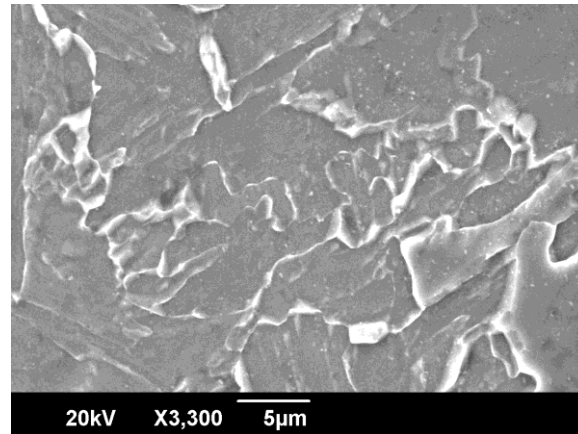


Figure 4.36: Precipitate depleted zone (PDZ) at the A508 side near the interface, in the thick transition zone region.

In this region, there is no semi-continuous band to the left of the interface within the A508 alloy. As in the other regions, the interface itself is rough, indicating the formation of martensitic band, and full of precipitates.

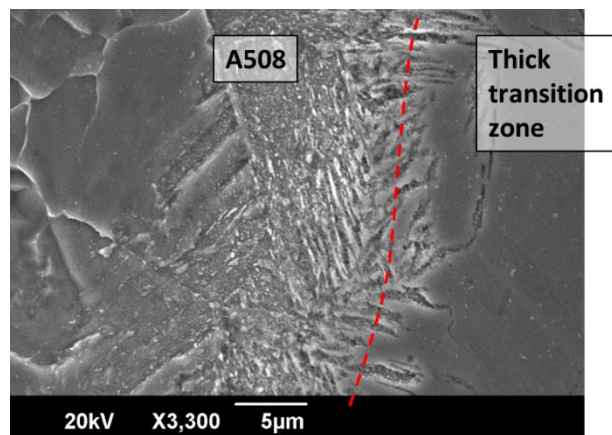


Figure 4.37: The interface A508 steel side of A508/304L joint at the thick transition zone region.

4.6.3 Microstructure of thick transition zone and 304L side of interface

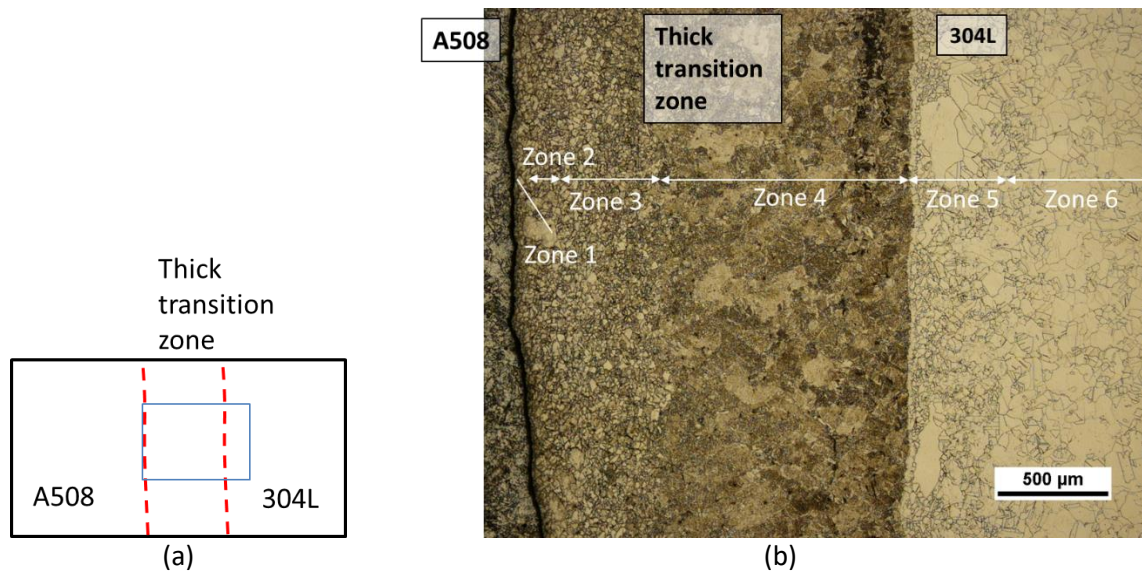


Figure 4.38: (a) Location of thick transition zone. (b) Overview of etched thick transition zone and 304L side of the interface of A508/304L joint in the thick transition zone region. The thick transition zone consists of Zones 1-4, and 304L side consists of Zones 5 and 6.

Figure 4.38 shows the location and an overview of several distinct microstructural zones in the thick transition zone and 304L side of the interface. The thick transition zone, which is about 1700-1800 μm wide, is divided into Zones 1-4, and 304L side is divided into Zones 5 and 6. Figure 4.39(a) shows that Zone 1, which is about 40-60 μm wide, has dense intergranular and intragranular precipitates in an equiaxed twinned austenitic grain structure. Zone 2, which is about 110-140 μm wide, also has intergranular and intragranular precipitates, but the intragranular precipitates are not as dense. Zone 3, which is about 400-500 μm wide, is similar to Zone 2, but has partial plate martensite formation in parts of the grain boundaries. Figure 4.39(b) shows that Zone 4, which is about 1100-1200 μm wide, has a tempered martensitic grain structure. A semi-continuous band is also present in this zone. Zones 5 and 6 are the 304L side of the interface, with Zone 5 having noticeably smaller grain size compared to Zone 6 (bulk 304L).

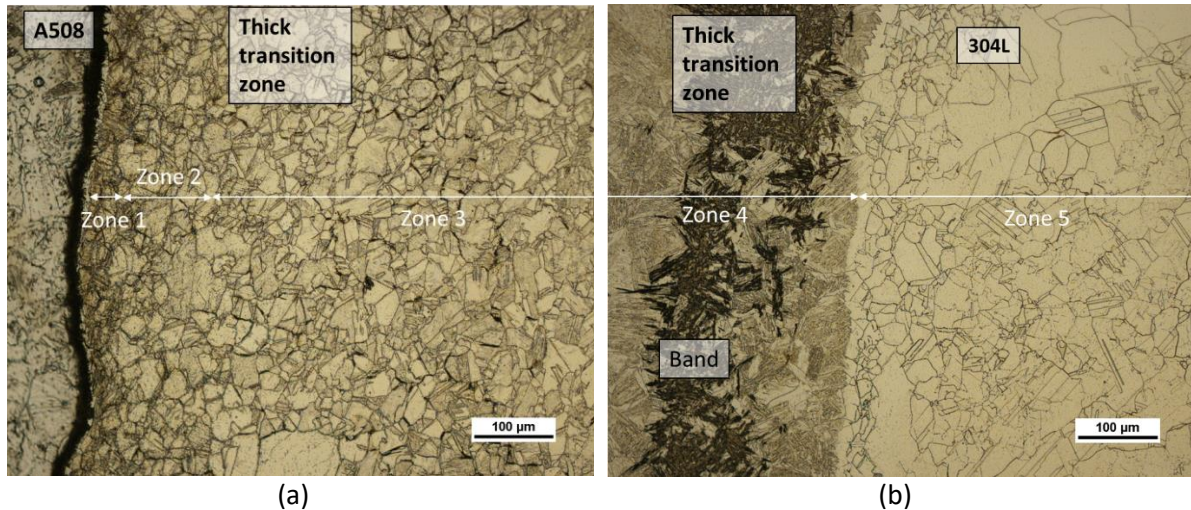


Figure 4.39: (a) Zones 1-3 in the thick transition zone and (b) Zones 4 and 5 in 304L, in the A508/304L joint.

Higher resolution images of the zones using SEM are presented in Figure 4.40. Zone 1 contains spheroid, needle and fine precipitates within grains, and also precipitates in the grain boundaries as well as twinning boundaries. Zone 2 is similar to Zone 1, except that there is no fine intragranular precipitates. Zone 3 is similar to Zone 2, but has fine precipitates of the plate martensite that surrounds the grain boundaries in addition. Zone 4 has a tempered martensitic grain structure. Most of Zone 4 is devoid of the precipitates, except in the semi-continuous band, which is precipitate dense. Zone 5, which lies in the 304L side of the interface, contains precipitates in the grain boundaries as well as twinning boundaries, but no precipitates within grains. The microstructure of Zone 6 is just the bulk 304L microstructure.

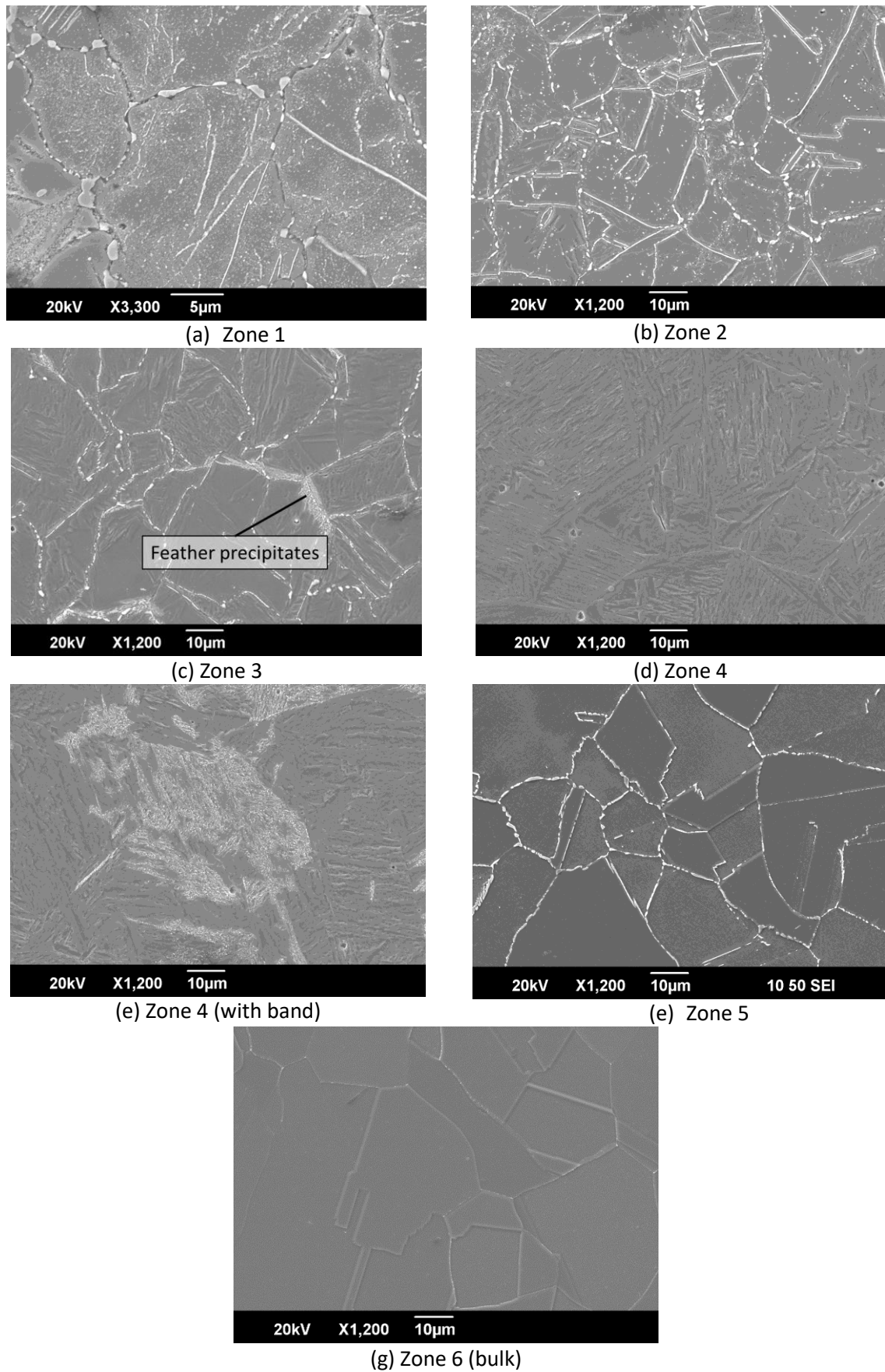


Figure 4.40: Scanning electron micrographs (SE mode) of the zones in the 304L steel side of the interface of A508/304L joint at the thick transition zone region. (a) Zone 1 (b) Zone 2 (c) Zone 3 (d) Zone 4 (e) Zone 4 with band (f) Zone 5 and (g) Zone 6.

4.6.4 EBSD crystal structure in the vicinity of the thick transition zone

EBSD analysis of the thick transition zone and the surrounding bulk material, presented in Figure 4.41, shows that A508 side and 304L side of the respective interface lines with the transition zone have BCC and the FCC crystal structures, respectively. The thick transition zone has a more complex set of crystal structures. Much of the area in this transition zone is non-indexed. While most of this zone appears to have BCC crystal structure, there is a band of FCC crystal structure adjacent to the A508 interface and fades into the BCC structure further from the A508 interface. Most of the FCC crystal structure in this region is within 500-600 μm from the A508 interface, which roughly corresponds to Zones 1, 2 and 3. The presence of FCC crystal structure, hence austenitic crystal structure, in Zones 1, 2 and 3 may explain the presence of the twinning boundary observed there. EDX mapping, displayed in Figure 4.42, shows that the composition of Fe, Mn, and Ni are more or less constant throughout the thick transition zone.

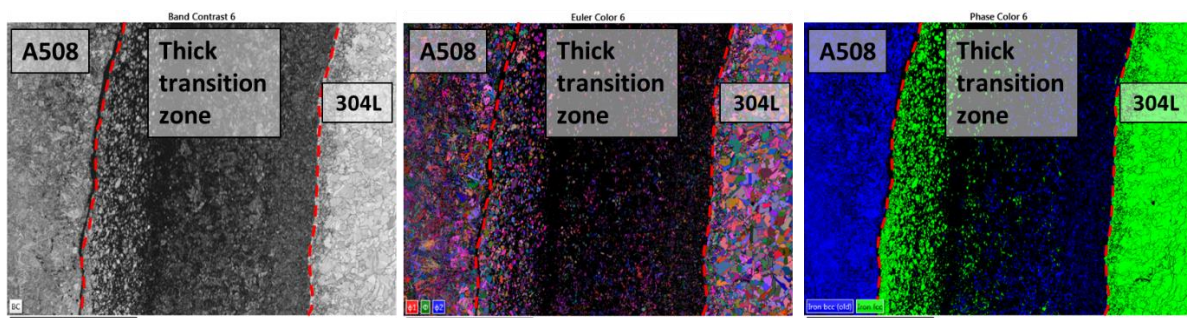


Figure 4.41: EBSD mapping of the vicinity of the thick transition zone in the A508/304L joint. Black indicates non-indexed solutions.

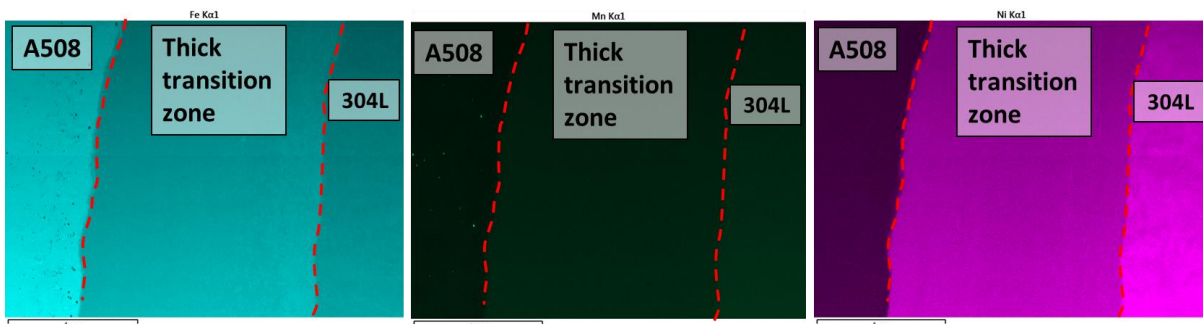


Figure 4.42: EDX mapping of Fe, Mn, and Ni in the vicinity of the thick transition zone in the A508/304L joint

4.7 Thermodynamic simulations of phase equilibria

4.7.1 A508

The phase equilibria for A508 was calculated using the software package ThermoCalc and the TCFe7 database. The composition of A508 G3 used for ThermoCalc calculations is presented in Table 4.2 and some minor elements normally found in A508 have been omitted to simplify the calculation. These minor elements can be introduced at a later stage of the work if their presence leads to significant effects, including sulphur which creates the tendency to form MnS inclusions, which was observed in A508.

Table 4.2: Input composition of A508 Gr 3 for ThermoCalc calculations.

Element	Amount (wt%)
C	0.24
Cr	0.11
Fe	97.04 (Bal)
Mn	1.42
Mo	0.58
Ni	0.61
S	0.01

In the majority of alloys, graphite is suspended as a phase in the simulation because of its slow kinetics for formation and instead various carbide phases are permitted to form. The equilibrium phase fractions predicted as function of temperature (from 500°C to 1200°C) are plotted in Figure 4.43. Note that the minimum temperature of 500°C is chosen due to the fact that the TCFe7 database is not reliable below this temperature.

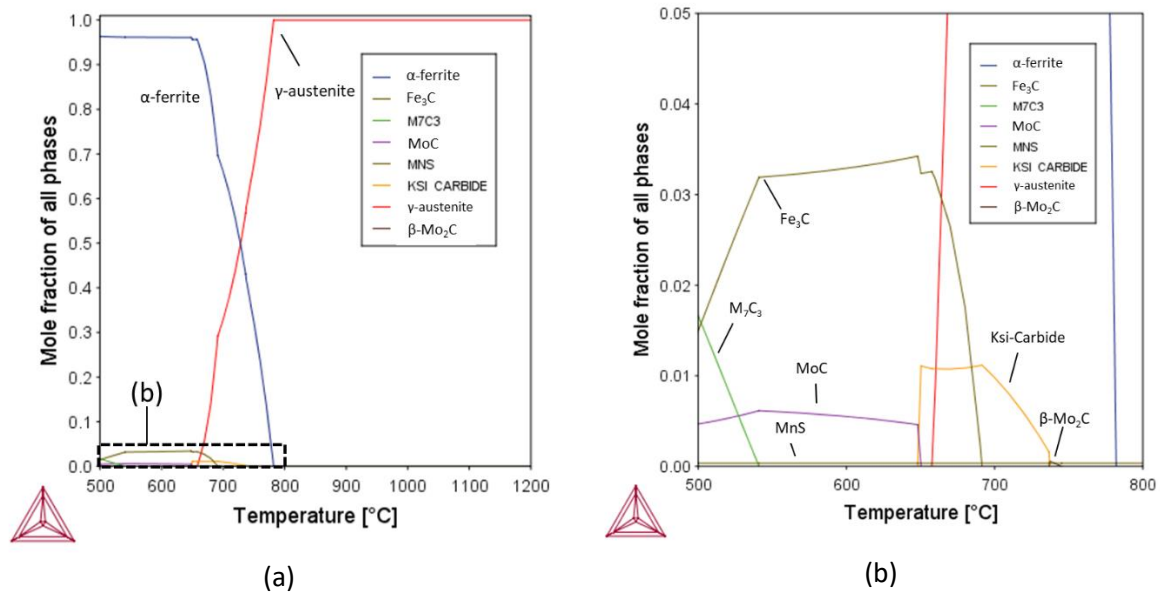


Figure 4.43: Phase fractions as a function of temperature for A508 steel, as calculated by ThermoCalc using the TCFE7 database, showing (a) all phases, and (b) secondary phases. The dashed box in (a) indicates the portion of the plot represented in (b).

The matrix phase is identified as γ -austenite (FCC) and α -ferrite (BCC). The precipitates are identified as follows:

- **HCP_A3:** $\beta\text{-Mo}_2\text{C}$ (molybdenum carbide)
- **KSI_CARBIIDE:** $\text{Fe}_{11}\text{Mo}_6\text{C}_5$ or Fe_2MoC
- **CEMENTITE:** Fe_3C
- **MC_SHP:** MoC
- **M7C3:** M_7C_3 (where M is a combination of Fe, Cr and other elements)
- **MNS:** MnS inclusion

The α -ferrite and cementite phases shown here would in reality be replaced by pearlite, bainite, and/or martensite; exactly which of these three would appear in the A508 depends on the cooling rate of the alloy from austenitisation treatment, but the most likely transformation that would appear

in the alloy used here is from γ -austenite to bainite. According to Figure 4.43(b), after tempering at 680°C, A508 is actually partially austenitised, but this may be inaccurate.

β -Mo₂C and Ksi-carbide are molybdenum-rich nano-size carbides that are shown to form in A508 after exposure to high temperature at relatively short amount of time (homogenization at 910°C for 9 h, a tempering at 650°C for 9 h, an austenitizing at 880°C for 8 h, a quenching and a tempering at 650°C during 1 h.) [28].

As can be seen in Figure 4.43(b), the presence of MnS inclusion, as observed in the microstructure, is mostly constant at mole fraction of 0.00035 from 500°C to 800°C. Different types of molybdenum carbides appear depending on temperature. Below 650°C, MoC (molybdenum monocarbide) appears. From 650°C to about 737°C, MoC disappears and Ksi-carbide appears. Since Ksi-carbide also contains Fe, some Fe are leeched from the matrix; indeed, there is a slight dip in the mole fraction of α -ferrite at 737°C to feed Fe into Ksi-carbide, as can be seen in Figure 4.43(a). At a small temperature range from 737°C to about 745°C, Ksi-carbide mostly dissolves in the matrix, though some molybdenum forms the β -Mo₂C.

4.7.2 304L

The phase equilibrium for 304L is calculated using ThermoCalc and the TCFE7 database. The composition of 304L used for ThermoCalc calculations is presented in Table 4.3. SIGMA phase has been suspended. The equilibrium phase fractions as a function of temperature (from 500°C to 1200°C) are plotted in Figure 4.44.

Table 4.3: Input composition of 304L for ThermoCalc calculations.

Element	Amount (wt%)
C	0.029
Cr	18.57
Fe	69.247 (Bal)
Mn	1.62
Ni	10.45

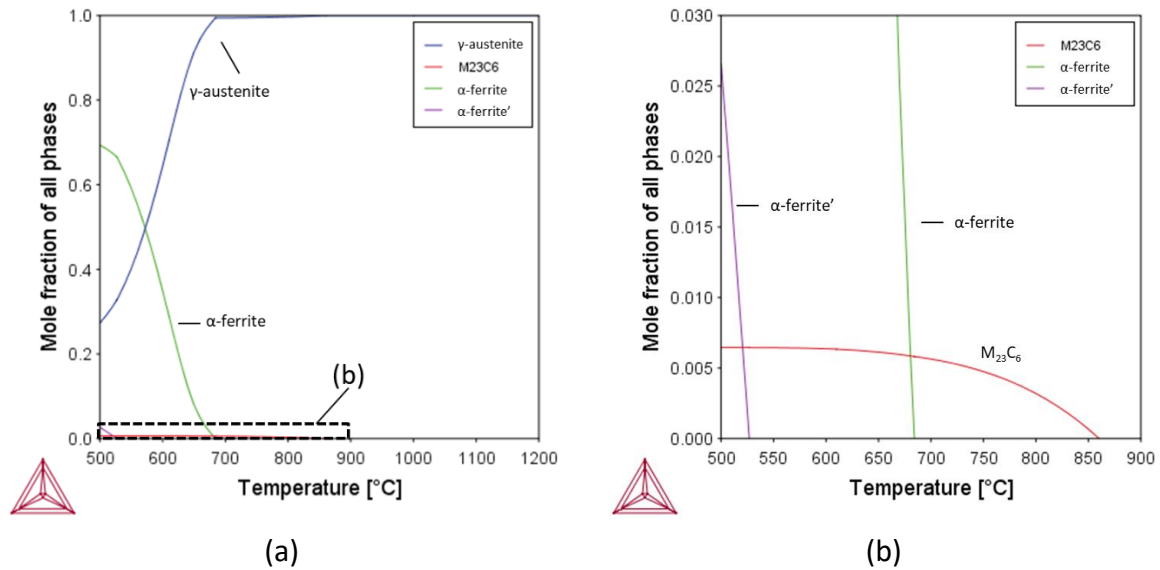


Figure 4.44: Phase fractions as a function of temperature for 304L steel, as calculated by ThermoCalc using the TCFE7 database, showing (a) all phases, and (b) secondary phases. The dashed box in (a) indicates the portion of the plot represented in (b).

The matrix phase is identified as γ -austenite (FCC) and α -ferrite (BCC) below 650 °C. Note that α -ferrite' (alongside α -ferrite) resulted from a spinodal disintegration or decomposition of the BCC phase due to a miscibility gap. α -ferrite is Fe-rich whereas α -ferrite' is Cr-rich, though both have BCC crystal structure. The precipitates are identified as $M_{23}C_6$, which is a Cr-rich carbide. According to Figure 4.44(a), BCC α -ferrite phase should appear in 304L below 680 °C. However, under relatively fast cooling, 304L is not transformed from FCC due to slow diffusion of Cr and Ni. Figure 4.44(b) shows that $M_{23}C_6$ are expected to form in small amounts below about 850 °C, which may lead to stainless steel sensitisation if exposed to high temperature below 850 °C for a long time, despite being a low carbon L-grade. So the tempering operation at 680 °C during the post-forge heat treatment (PFHT) may have led to a small amount of sensitisation, though precipitates were not detected in 304L bulk.

4.7.3 Medium thickness transition zone

From Figure 4.24, the composition across the medium thickness transition zone varies. However, a large portion of the transition zone has constant composition of the main alloying elements (Fe, Ni, and Cr) and to a lesser extent other major elements (Mn and Mo), even though there are many distinct microstructural zones within this constant composition. While the major elements are of similar

composition in this zone, it is apparent that the composition of carbon may vary across this zone (as exhibited by the absence and presence of carbides). Hence it is valuable to know that types of phases would appear as a result of varying amounts of local carbon content in the transition zone during PFHT (austenitisation/solution treatment at 980°C followed by water quenching and then tempering at 680°C). The phase equilibrium for this can be calculated using ThermoCalc. The composition of the medium thickness transition zone used for ThermoCalc calculations is presented in Table 4.4. The TCFE7 database is used because the Fe content is >50 wt%. The equilibrium phase fractions as a function of carbon content (0 to 2 wt% C) on the PFHT temperatures are plotted in Figure 4.45.

Table 4.4: Input composition of the medium thickness transition zone in the A508/304L joint for ThermoCalc calculations.

Element	Amount (wt%)
C	(varies)
Cr	12.19
Mn	1.52
Fe	79.09
Ni	6.91
Mo	0.29

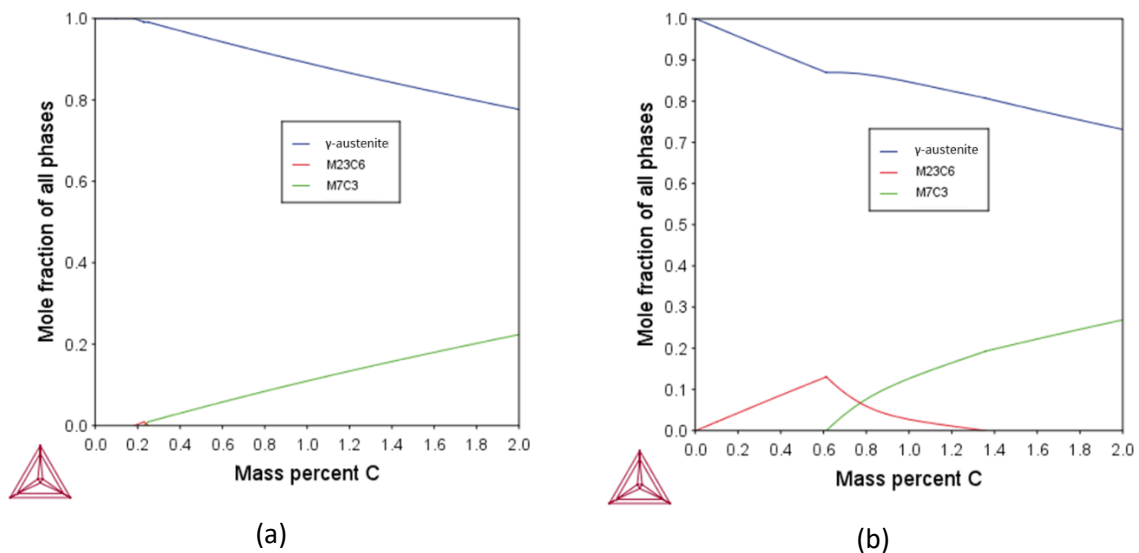


Figure 4.45: Phase fractions as a function of wt% carbon for the medium thickness transition zone in the A508/304L joint, as calculated by ThermoCalc using the TCFE7 database, at the temperature of (a) 980°C during the austenitisation/solution treatment and (b) 680°C during tempering.

Figure 4.45(a) shows that during the austenitisation/solution treatment, in locations where there is less than 0.2 wt% C, there is only one phase (γ-austenite). At locations where there is about 0.2 wt%

C, there is a very small amount of $M_{23}C_6$ carbide. Above 0.2 wt% C, there is an increasing amount of M_7C_3 carbide in locations with greater local carbon content. Figure 4.45(b) shows that during tempering, there is a phase which is still largely γ -austenite, though $M_{23}C_6$ carbide is present below 1.4 wt% C and M_7C_3 is above 0.6 wt% C. Both of these plots show that during PFHT, the matrix phase should only consist of γ -austenite, while some carbides (either $M_{23}C_6$ or M_7C_3) may be present, depending on the local carbon content.

4.7.4 Thick transition zone

The thick transition zone is similar to the medium transition zone in while a large portion of the cross-weld composition is largely constant when it comes to the main alloying elements (Fe, Ni, and Cr) and to a lesser extent other major elements (Mn and Mo), as can be seen from Figure 4.33, there are many distinct microstructural zones within this constant composition. The absence and presence of carbides in different zones also indicate varying local carbon content. The composition of the thick transition zone used for ThermoCalc calculations is presented in Table 4.5. The TCFE7 database is used because the Fe content is >50 wt%. The equilibrium phase fractions as a function of carbon content (0 to 2 wt% C) on the PFHT temperatures are plotted in Figure 4.46.

Table 4.5: Input composition of the thick transition zone in the A508/304L joint for ThermoCalc calculations.

Element	Amount (wt%)
C	(varies)
Cr	14.19
Fe	76.65
Mn	1.36
Mo	0.33
Ni	7.47

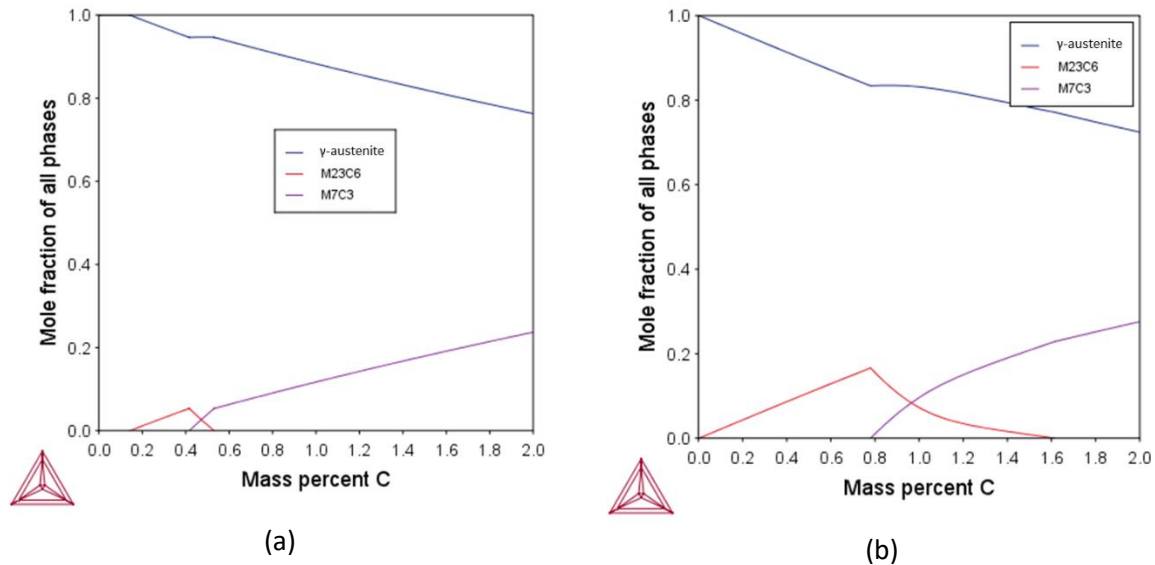


Figure 4.46: Phase fractions as a function of wt% carbon for the thick transition zone in the A508/304L joint, as calculated by ThermoCalc using the TCFE7 database, at the temperature of (a) 980°C during the austenitisation/solution treatment and (b) 680°C during tempering.

Similar to the medium thickness transition zone, Figure 4.46(a) shows that during the austenitisation/solution treatment, in locations within the thick transition where there is less than 0.14 wt% C, there is only one phase (γ-austenite). At locations where there is between 0.2 wt% and 0.53 wt% C, there is some M₂₃C₆ carbide. Above 0.4 wt% C, there is an increasing amount of M₇C₃ carbide in locations with greater local carbon content. Figure 4.46(b) shows that during tempering, there is a phase which is still largely γ-austenite, though M₂₃C₆ carbide is present below 1.6 wt% C and M₇C₃ is above 0.8 wt% C. Again, both of these plots show that during PFHT, the matrix phase should only consist of γ-austenite, while some carbides (either M₂₃C₆ or M₇C₃) may be present, depending on the local carbon content.

4.8 Tensile testing

As seen in Figure 4.1, the transition zone region varies in thickness across the joints. In order to assess whether this variation results in a difference in mechanical behaviour for different thicknesses, two tensile specimens were extracted from both the thick and thin transition zone regions of the joint, perpendicular to the interface as shown in Figure 4.47. In addition, tensile specimens of bulk materials from the joints were also obtained in order to compare the mechanical performance of the cross-weld

properties with those of the bulk material. As material was limited, the bulk tensile specimens were cut parallel to the interface.

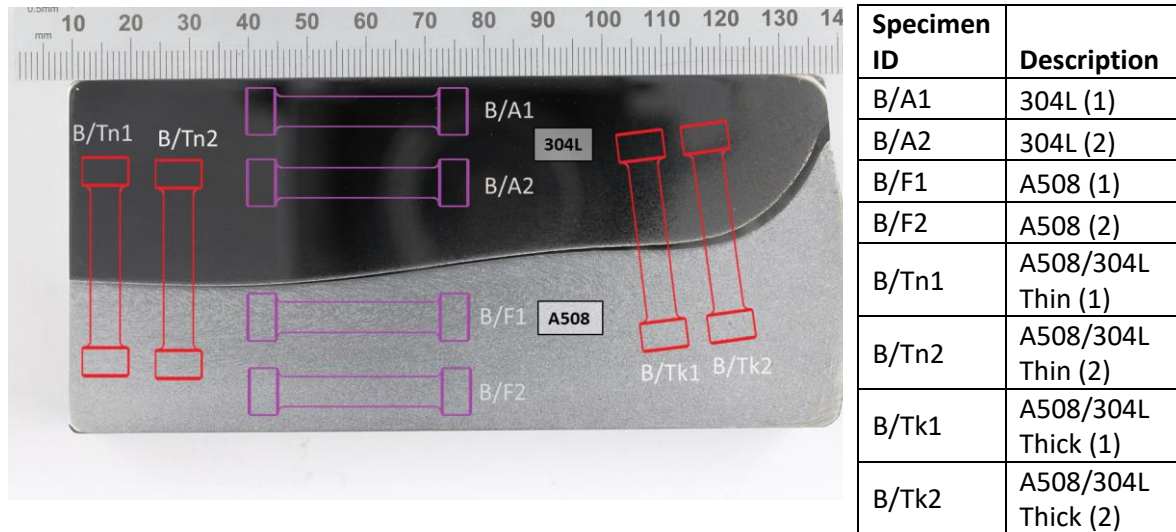


Figure 4.47: Origin of tensile test specimens of A508/304L joint. Two specimens each were obtained from the thinner transition zone region (B/Tn1 and B/Tk1) and thicker transition zone regions (B/Tk1 and B/Tk2) and bulk alloys in the lateral direction (B/A1-B/F2).

4.8.1 Tensile stress-strain curves and material properties

The stress-strain curves from the tensile tests of specimens from A508/304L joints are presented in Figure 4.48, and their extracted tensile properties are presented in Table 4.6. The A508 specimens (B/F1 and B/F2) have the highest UTS and yield strength among all of the specimens, though are not identical to each other. Again, the difference in the A508 curves is perhaps due to the flow that can be seen in Figure 4.1. The specimen closer to the interface (i.e. specimen B/F1) comes from the part of A508 that experience more flow and deformation compared to the other specimen (B/F2). The UTS and yield stress of B/F1 is somewhat lower than that of B/F2, though the elongation is identical. The greatest ductility is exhibited by the 304L specimens (B/A1 and B/A2), even though the elongation somewhat differs. In terms of the cross-weld specimens, the curve and tensile properties for two thin transition zone region specimens (B/Tn1 and B/Tn2) are nearly identical. Similarly, the thick-transition zone region specimens (B/Tk1 and B/Tk2) are also nearly identical. While all four cross-weld specimens have similar yield stress, the thin transition zone region specimens (B/Tn1 and B/Tn2) have slightly

higher ductility but lower UTS. Overall the cross-weld specimens have UTS, yield stress, and elongation intermediate between that of A508 and 304L. This indicates that the tensile properties across the weld are comparably strong and ductile (or more so) compared to the bulk alloys, showing the viability of the fabrication process.

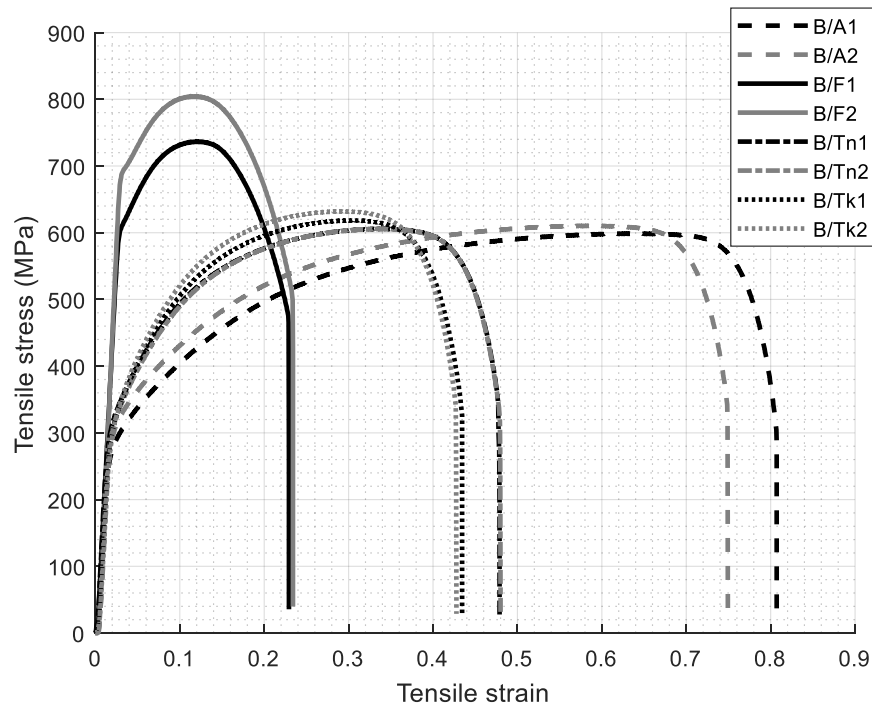


Figure 4.48: Stress-strain curves of tensile specimens from the A508/304L joint.

Table 4.6: Measured tensile properties of the A508/304L joint according to the stress-strain curves.

Type	Specimen	Test results		
		0.2% Proof (MPa)	UTS (MPa)	EL (%)
Bulk alloy	B/A1	253	599	81
	B/A2	279	611	75
	B/F1	602	737	23
	B/F2	685	805	23
Cross-weld	B/Tn1	286	606	48
	B/Tn2	286	606	48
	B/Tk1	286	619	43
	B/Tk2	289	633	43

4.8.2 Fractography

The fracture locations of the tensile test specimens for the cross-weld specimens, presented in Figure 4.49, allows an initial evaluation of whether the interface (i.e. the fusion region) between the two alloys was at least as strong as the bulk material for each of the joints.

Figure 4.49(a) and Figure 4.49(b) show that for both the thin and thick transition zone regions of the A508/304L joint, the fracture occurred well into the 304L bulk, indicating the fusion between the two alloys is at least as strong as 304L. All the necking occurred within the 304L bulk.

The hardness trough near the A508 side of the interface (see Figure 4.2) does not appear to result in a weakness that would cause tensile failure to occur there. This could be due to the constraint effect inherent in tensile specimens that experience non-uniform deformation or stresses—especially those with cross-sections such as the circular one used here—unlike hardness tests where there is no constraint effect. The strength and elongation are affected by the constraint of adjacent materials with differing mechanical properties. Hence, since the hardness troughs occur in a relatively narrow zone, the bulk A508 and the A508 interface line (both of which are high-strength) provided constraint that prevented excessive elongation and weakening in this narrow zone. Hence, severe elongation had to occur within the 304L side, where relatively low hardness occurred in a much wider range, with the fracture occurring far from the A508 interface line (or from the transition zone).

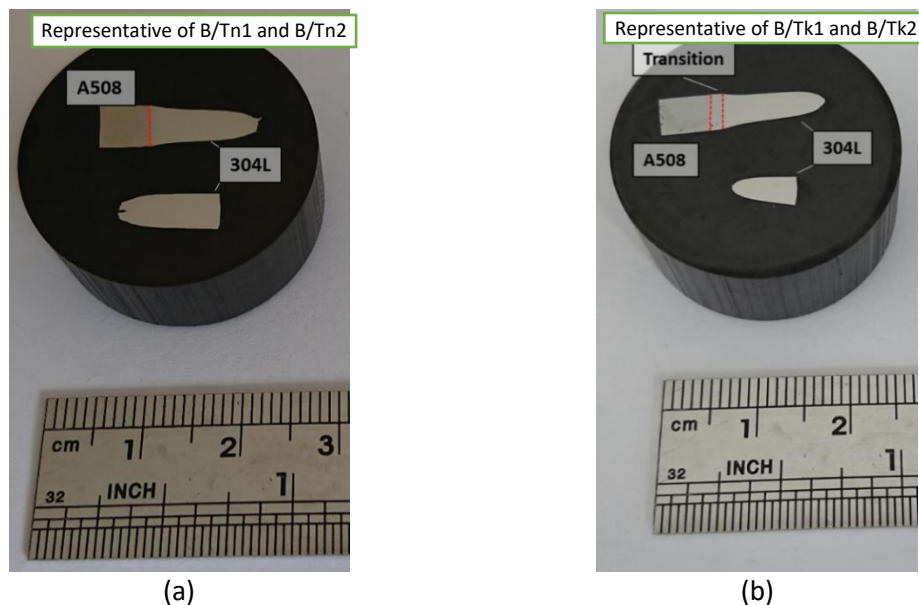


Figure 4.49: Fractured tensile testing specimens from (a) B/Tn1 Thin transition zone region of A508/304L joint (b) B/Tk1 Thick transition zone region of A508/304L joint.

Fracture behaviours of the tensile tested specimens can be further illuminated by examining the fracture surface topography using scanning electron microscopy. The fracture surface of B/Tn1 (thin transition zone region) and B/Tk1 (thick transition zone region) are shown in Figure 4.50(c) and Figure

4.50(d). In both cases, dimples dominate but cleavage and 'rivers' are also common, indicating a combination of brittle and ductile fractures. This most resembles the fracture surface of B/A1 304L bulk (Figure 4.50(a)), which is consistent with the observation in Figure 4.49(a) and Figure 4.49(b), i.e. the fracture occurs in 304L portion of specimen B/A1, so the fracture surface should resemble that of 304L.

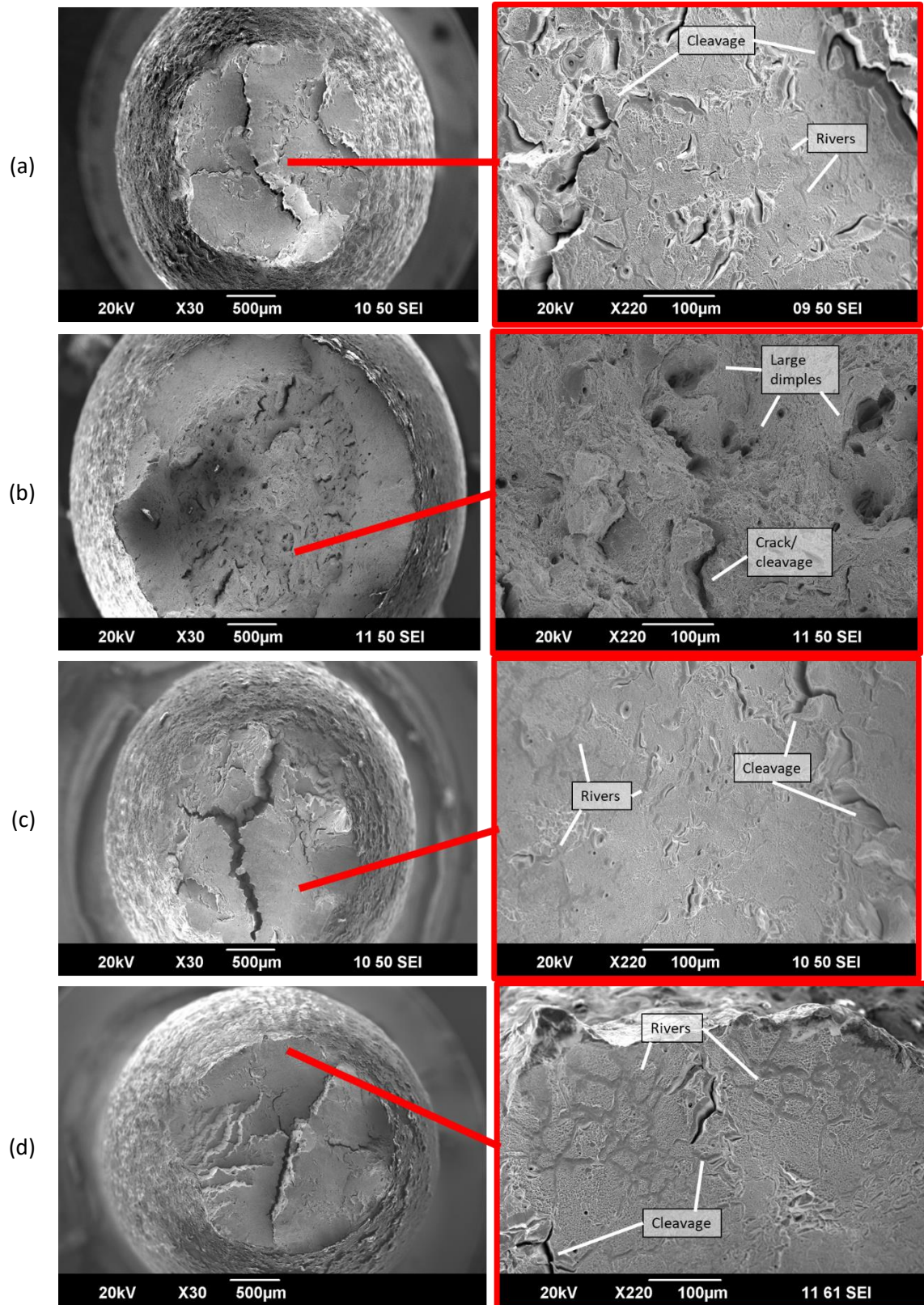


Figure 4.50: SEM (secondary electron mode) images of fracture surface topographies of the tensile specimens from A508/304L joint at (a) B/A1 304L bulk (b) B/F1 A508 bulk (c) B/Tn1 Thin transition zone region (e) B/Tk1 Thick transition zone region.

4.9 Discussion

4.9.1 Semi-continuous hard band

In each of the three regions examined, there is a hard semi-continuous band. This band is shown to be Cr and Ni rich, at the expense of Fe and has high hardness values. While it always occurs in the vicinity of the transition zones, exactly where in relation to the transition zone it occurs varies from region to region. A schematic of where the hard band occurs throughout the A508/304L joint is illustrated in Figure 4.51. In the thin transition zone region, the hard band is about 50 μm into the A508 side of the interface. In the medium thickness transition zone region, the hard band moves to the interface and also splits into the transition zone (as can be seen in Zone 3 of the medium thickness transition zone region). The hard band gradually dissipates in the A508 side of the interface at the thick transition zone region, but is maintained in the transition zone close to the interface with 304L (Zone 4 in the thick transition zone region).

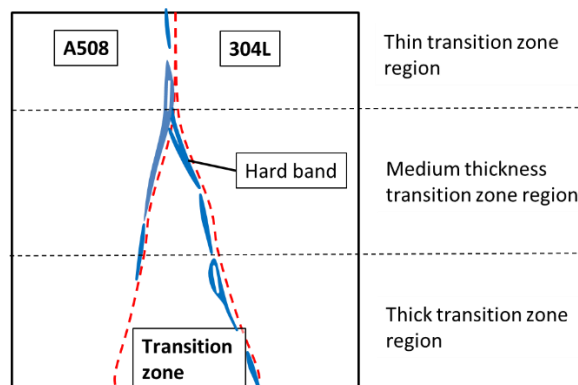


Figure 4.51: Schematic illustration of where the hard band occurs in the A508/304L joint divided into the three regions.

It is unclear how or what stage of the fabrication process this band formed. All EBSD measurements show that the hard band appears in zones with BCC crystal structures (whether in the A508 side or in the transition zone), though it tends to have a smaller block, packet and lath morphologies compared to the surrounding BCC area. The decreased size of these morphologies in the band is likely caused by high local carbon content during the rapid cooling phase (quenching) of the austenitisation/solution treatment at 980°C as part of the PFHT, as increased carbon content promotes the formation of smaller block, packet, and lath [98], and the precipitates then formed during the tempering process

at 680°C. This means that the band location already contains increased carbon content prior to PFHT, so EBW and/or the forging operations were responsible for creating this band. It is possible that during either operation, the Cr was 'expelled' from the surrounding A508, forming PDZ, and then gathered to form this band, and carbon then diffused into this Cr-rich band due to the lowered chemical potential of carbon caused by the elevated Cr content. Or, the semi-continuous and inconsistent nature of this band may have been created due to undesired factors such as impurities.

Whatever the cause, tensile testing shows that it may not have any significant role in limiting or otherwise undermining—through e.g. strain accumulation in adjacent softer regions—the mechanical performance of the joint, at least in room temperature, as the elongation and fracture occurs in 304L bulk, away from any occurrence of hard band.

4.9.2 Behaviour of A508 near the interface

Disregarding the hard band (justified by the fact that it does not occur exclusively in the A508 side of the interface), generally the microstructure can be summarised schematically in Figure 4.52. Because the solution treatment operation was done on the whole joint during the first part of PFHT, the grain size in A508 are more or less uniform throughout. Away from the interface, the bulk A508 material has a typical tempered bainite microstructure. Closer to the interface, however, the precipitates gradually thins out in the PDZ, until none can be detected. Adjacent to the interface is a rough formation of martensitic interfacial band. It is possible that PDZ formed due to carbon migration and subsequent pileup at the interface as the carbon tries to migrate into the 304L or the transition zone side of the interface during PFHT. It is also noticeable that EBSD analyses show that the A508 side of the interface is fully BCC, including the (tempered) martensitic interface.

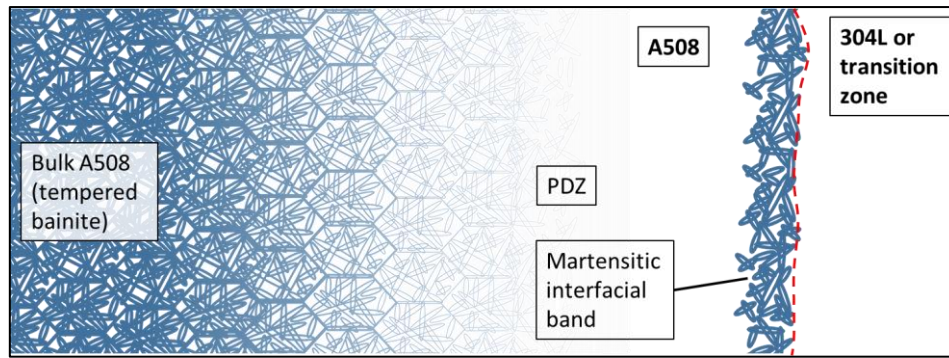


Figure 4.52: Schematic illustration of the microstructure of the A508 side of the interface in the A508/304L joint.

The martensitic interfacial band is approximately 10 μm wide. This correlates with the start of the change in composition on the A508 side of the interface, as can be seen in Figure 4.6, Figure 4.25(a), and Figure 4.34(a). Hence, the formation of the martensitic interfacial band can be attributed to the gradient in composition across the interface, where the local high alloy content leads to high hardenability that promotes the formation of martensite upon cooling during PFHT.

4.9.3 Behaviour of 304L near the interface

The microstructure of the 304L side of the interface is summarised schematically in Figure 4.53. While all zones have equiaxed twinned austenitic grain structure with FCC crystal structure, there are noticeable differences between different regions in terms of precipitates present. In the thin transition zone region, there is a zone with very dense intragranular and intergranular (as well as twin) precipitates, as can be seen in Zone 2 in the region. The precipitates gradually become less dense further away from the interface. This less dense precipitate zone (still containing both intragranular and intergranular as well as twin precipitates) also appears adjacent to the medium thickness transition zone, corresponding to Zone 4 of that region. Still further from the interface, the intragranular precipitates disappear, leaving only the intergranular and twin precipitates. This is Zone 3 in the thin transition zone region and Zone 5 in the medium thickness transition zone region. In the thick transition zone region, this zone is adjacent to the interface with the transition zone, and corresponds to Zone 5. Lastly, the intergranular and twin precipitates gradually thins out to the bulk 304L further away from the interface, where twinning boundaries are visible, but the no precipitates were observed.

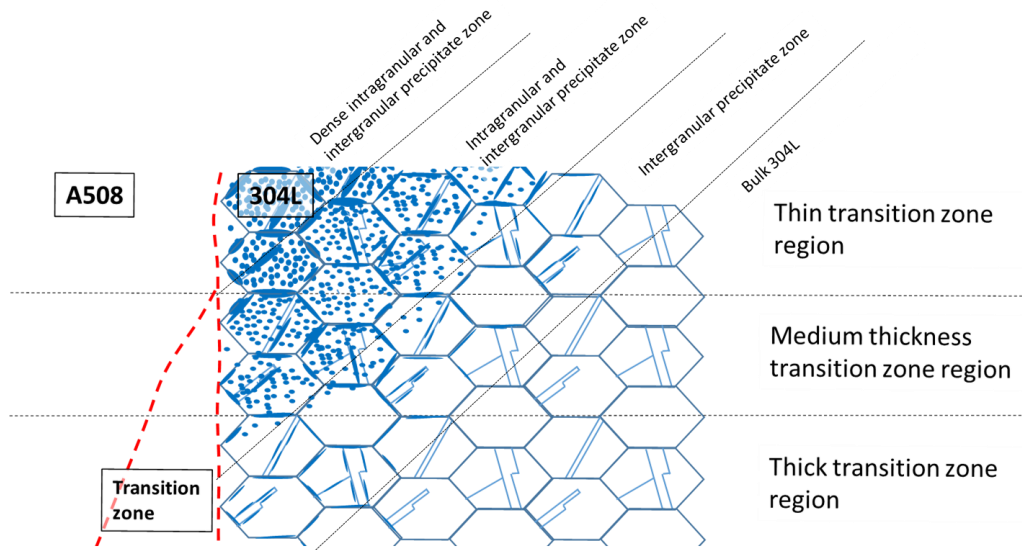


Figure 4.53: Schematic illustration of the microstructure of the 304L side of the interface in the A508/304L joint.

It is possible that the difference in behaviour in the different regions can be attributed to the thickness of the transition zones. The density of precipitates at a given location is correlated with the level of local carbon content; the higher the carbon content, the denser precipitates. In the thin transition zone region, where the 304L was joined almost directly with A508, there is high carbon in the 304L side of the interface, gradually decreasing further from the interface. In the medium thickness transition zone region, there is less carbon in the 304L side, and still less in the thick transition zone region. The difference in carbon content adjacent to the interface may be due to the difference in successful carbon migration into the 304L side. In the thin transition zone, carbon can migrate directly from the A508 side in to the 304L side, thus a relatively large amount of carbon can cross into the 304L side. In the transition zone, as a result of the mixing of A508 and 304L during EBW operation, the carbon content is less than A508, thus could supply less carbon to migrate into the 304L, resulting in the less carbon migration in the 304L side. Also, the carbon that may traverse from the A508 side, through the transition zone, into the 304L side, is less likely to arrive in the 304L when the transition zone is thick, i.e. the thick transition zone represents a barrier to carbon migration into 304L side.

4.9.4 Transition zone

Formation of the transition zone is likely caused by the EBW operation, which resulted in the transition zone being thicker at the edges due to the width of the EBW fusion zone itself being thicker at the edges prior to forging, which created a wider mixed zone there.

In terms of microstructure, the most prominent feature of the transition zone is the presence of both austenitic (FCC) and ferritic/tempered martensitic (BCC) structures, as can be seen in Figure 4.54. Where these structures take place within the transition zone is also counterintuitive; the FCC zone (corresponding to Zones 1 & 2 in the thick transition zone) is located adjacent to the A508 alloy, which is exclusively BCC, while the BCC zone (corresponding to Zone 4 in the thick transition zone, and Zone 3 in the medium thickness transition zone) is located adjacent to the 304L alloy, which is exclusively FCC. At the two interfaces, the boundaries between BCC and FCC crystal structures are sharp. Within the transition zone, the shift from FCC on one side to BCC on the other side is gradual. This shift is characterised by a mixture of FCC and BCC crystal structures, where martensitic plates start to form in the grain boundaries of the austenitic matrix (these plates were then transformed to BCC crystal structure during PFHT). The plates appear as the 'feathers' found in Zone 3 in the thick transition zone, and Zones 1 and 2 in the medium thickness transition zone. However, the high hardness found within the medium thickness transition zone (see Figure 4.2(b)), exceeding 400 HV₂₀₀, suggests that the martensite there has not been fully tempered.

It is unclear why a portion of the transition zone is retained austenite, while the other portion is martensitic. There is no observed spike in the austenite stabiliser Ni (nor a spike of another weaker austenite stabiliser, Mn) where the FCC crystal structure occurs. According to the ThermoCalc simulations plotted in Figure 4.45 and Figure 4.46, the portions of the transition zones with the constant major element compositions (i.e. most of the transition zone, traversing all three FCC, mixture, and BCC zones) should contain only γ -austenite matrix during both the solution treatment at 980°C and the tempering treatment at 680°C, even with varying carbon content. But parts of the

transition zone retains austenitic structure at room temperature after air-cooling following tempering, while the rest converts to BCC. Since composition alone could not explain the difference in behaviour, it is possible that the formation of martensite region (that then transformed to BCC) was mechanically induced, possibly by residual strain caused by difference in the coefficients of thermal expansion between A508 and 304L. This strain may have accumulated when the joint was cooled down from the tempering process to room temperature, as 304L (which has higher coefficient of thermal expansion) shrinks to a greater degree compared to A508.

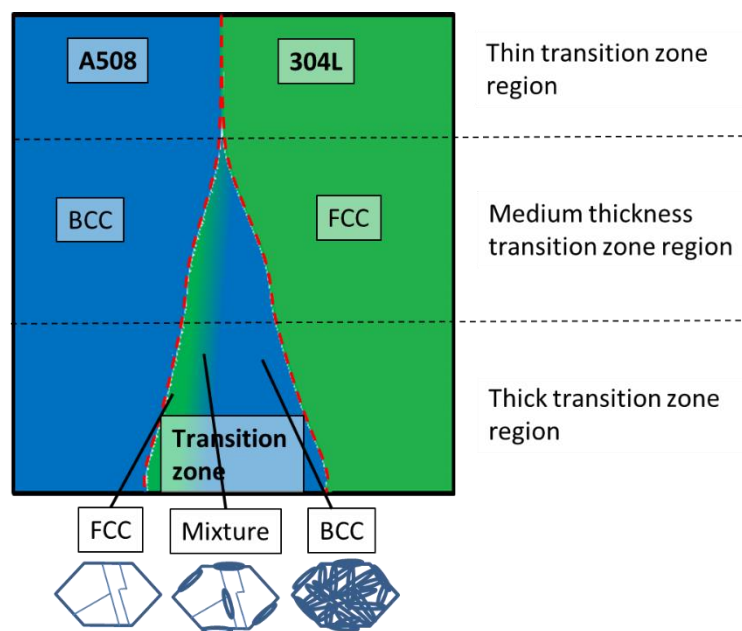


Figure 4.54: Schematic diagram of the crystal structure distribution in the A508/304L joint, with illustrations of the resulting microstructural features in the transition zone.

Despite having a complex microstructure, the presence of thick transition zone may not always be detrimental, as tensile testing shows that while the tensile samples from the thin transition zone region (i.e. B/Tn1 and B/Tn2) has slightly higher ductility, the tensile samples from the thick transition zone (B/Tk1 and B/Tk2) has similar 0.2% proof yield stress and slightly higher UTS.

4.9.5 Nanoindentation profile and material properties

The most important parameter from the nanoindentation simulation optimisation is σ_y , as the location within the weld with the lowest yield stress may be the weakest, and thus failure under load may occur there. Figure 4.55 shows the profiles of the optimised nanoindentation σ_y across the interface

in the A508/304L joint using Limits Sets 1, 2 and 3. Each profile is superimposed with microhardness profile from Figure 4.13. The profile of the optimised nanoindentation σ_y correlates well with the microhardness measurement, with the exception between -100 μm and 0 μm from the interface. This can be explained by the fact that the microhardness profile deliberately traversed and picked up the hard band, whereas the nanoindentation profile missed this semi-continuous band, thus no spike in hardness near the interface.

While the nanoindentation profile traverses across all distinct microstructural zones in the 304L, it does not go far enough into the 304L bulk to capture the low hardness of the 304L bulk far away from the interface. Figure 4.2(a) shows that the microhardness only reached bulk 304L values about 700 μm from the interface, even though bulk 304L microstructure appears less than 600 μm from the interface. This may be due to the residual strain near the interface that resulted from the difference in the coefficients of thermal expansion between A508 and 304L. It can be inferred that if the nanoindentation profile extends further into the 304L bulk, the resulting σ_y values would decrease to a value similar to, or less than, that of the PDZ region of A508. Indeed, tensile testing at all cross-weld locations yielded necking and fracture deep in the 304L bulk, suggesting that 304L bulk is the weakest part of the joint and thus have the lowest σ_y .

The difference in hardness between the PDZ and the hard band (or the interface, in locations where there is no hard band) does not appear to introduce strain incompatibility that would cause a large enough strain accumulation in the PDZ that failure would occur there.

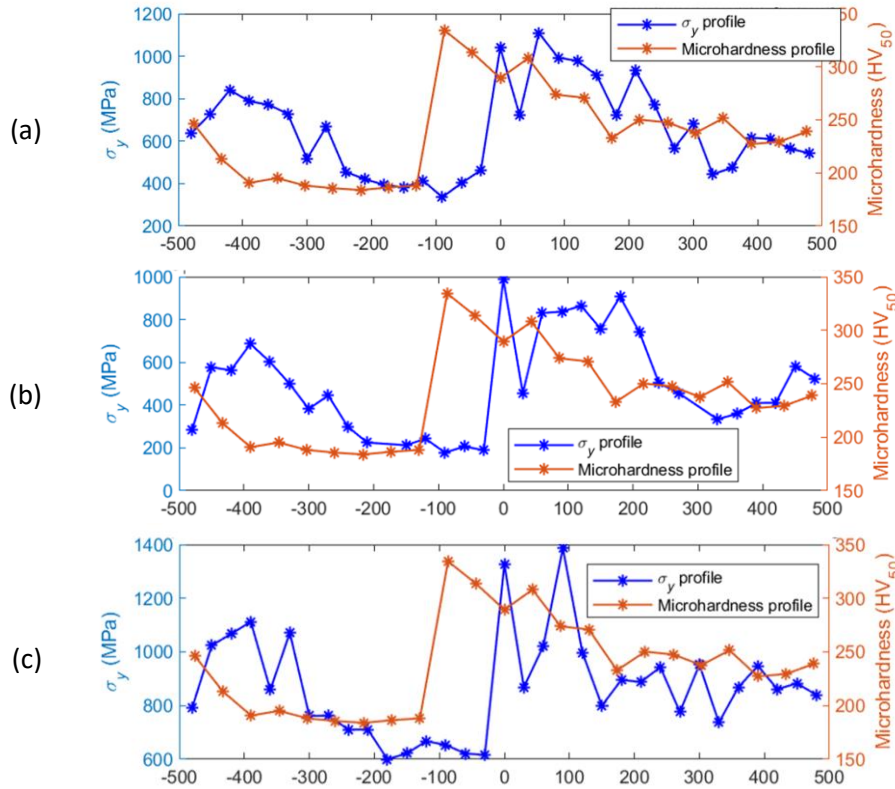


Figure 4.55: Profile of the optimised nanoindentation yield strength σ_y across the interface in the A508/304L joint using (a) Limits Set 1 (b) Limits Set 2 and (c) Limits Set 3. Each profile is superimposed with microhardness profile from Figure 4.13.

4.10 Conclusions

The following observations can be made about the TransForge A508/304L joint:

- A508 and 304L steels—which previously have only been joined together with the use of electrodes/fillers such as 308L [99] in fusion DMW—have been directly joined successfully, demonstrating good weldability of this pair of dissimilar alloys when utilising the TransForge fabrication technique.
- The interfacial regions varies in different parts of the joint; the centre of the joint contain the thin transition zone region, whereas the transition zone gets thicker nearer to the edge of the joints.
- Overall the cross-weld specimens (from both the thin and the thick transition zone region) have UTS, yield stress, and elongation intermediate between that of A508 and 304L. Necking and fracture occurred deep in the 304L bulk in all cases. This indicates that the tensile

properties across the weld are comparably strong and ductile (or more so) compared to the bulk alloys, showing the viability of the fabrication process.

- Sensitisation is not evident in 304L bulk, despite the PFHT operation.
- A semi-continuous martensitic hard band (with dense precipitates) formed in A508 side of the interface and in the transition zone side of the interface with 304L. It is unclear how this band formed. Nevertheless, it does not appear to affect the mechanical performance of the joint.
- Near the interface, the A508 side experienced PDZ, resulting in a low hardness there. At the interface, a martensite band formed on the A508 side due to composition gradient.
- Varying degrees of precipitate formation on the 304L side of the interface, in different transition zone regions, are caused by the difference in local carbon content, suggesting that there were different degrees of carbon migration into the 304L side of the interface, depending on the thickness of the transition zone. 304L adjacent to thicker transition zone experienced less carbon migration into it.
- The different types of crystal structures that formed within the transition zone cannot be explained by composition alone, suggesting mechanically induced martensite formation. High hardness values ($>400 \text{ HV}_{200}$) observed in the medium thickness transition zone suggests that some of the martensite in the transition zone has not been fully tempered despite the PFHT.
- The elevated hardness at the 304L side of the interface, including part of the 304L bulk, suggests that there is residual strain near the interface at room temperature.
- Optimised parameter values derived from nanoindentation data using the inverse method resulted in a cross-weld yield stress profiles that correlates well with microhardness profile. However, the fact that the results are very different depending the on the limits set, particularly for E , suggests that inverse method used here may not have resulted in well-optimised P - h curves. Since the four parameters being optimised in the inverse method were not independent of each other, the results of the other three parameters, including yield stress may also not be accurate and should to be taken with caution.

5 CHARACTERISATION OF A508/IN600 TRANSFORGE JOINT

5.1 Introduction

A macroscopic investigation of the A508/IN600 joint is presented in Figure 5.1 and shows some evidence of flow experienced by the lower strength A508 material during the forging process at the centre of the joint, not just at the edge where the lip is formed. Similar to the A508/304L joint, it can also be seen that there are differences in the thickness of the transition zone between the centre of the specimen and the outer edge of the specimen. The centre of the TransForge joint contains the thin transition zone region, whereas the transition zone gets thicker nearer to the edge of the joints.

As the interfacial regions are distinctive in different parts of the joint, more than one location of the transition zone has to be studied in order to fully evaluate the interface between the two alloys. Therefore, the distinct interfacial regions are divided and studied as follows:

- Thin transition zone region, which originates from the centre of the forged A508/IN600 joint (the region around 'a' in Figure 5.1).
- Medium thickness transition zone region, which has the transition zone intermediate in thickness between the thin and thick transition zones, originating from a region between the thin and thick transition zone regions (the region around 'b' in Figure 5.1)
- Thick transition zone region, which originates from a region close to the outer edge of the forged A508/IN600 joint (the region around 'c' Figure 5.1).

Characterisation methods employed for this joint is similar to that carried out for the A508/304L joint:

- Cross-weld microhardness profiles in the different regions above to provide an indication of cross-weld microstructural variability.
- The microstructural studies of the bulk materials, followed by the microstructure in the vicinity of the interface between the two alloys, divided into the different regions above.

- Nano indentation testing to determine the cross-weld elastic-plastic properties of the thin transition zone region, by using the inverse method.
- Thermodynamic simulations of phase equilibria, using the ThermoCalc software, to assist the identification of phases present, such as possible precipitate phases and whether the matrix phases should be predominantly austenitic (FCC) or ferritic (BCC).
- Tensile testing to evaluate whether the microstructural features have any impact on the mechanical performance of the joint.

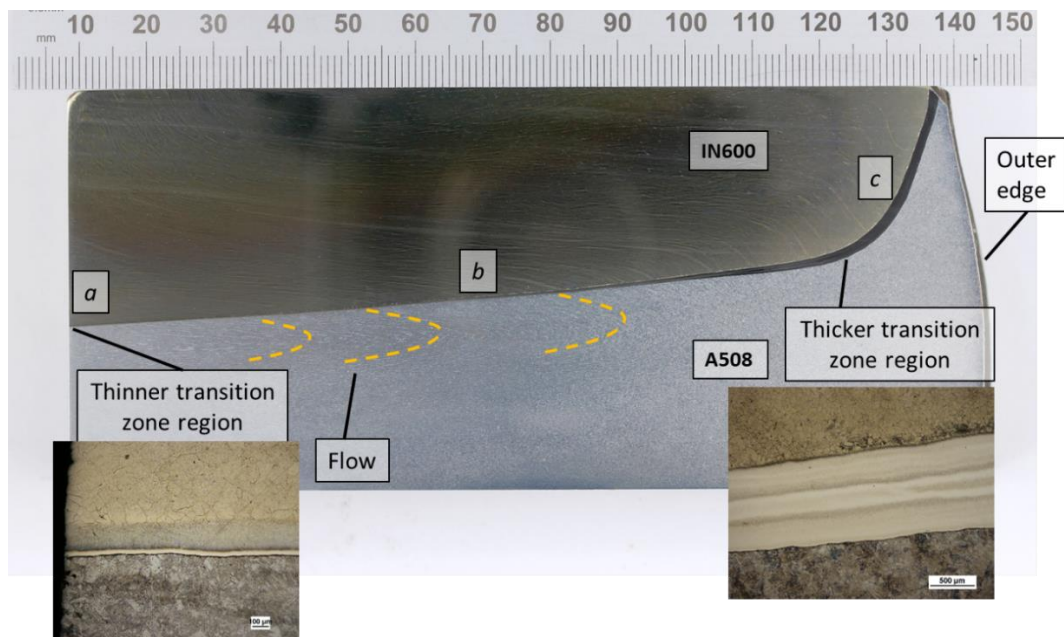


Figure 5.1: TransForge A508/IN600 joint after forging and post-forge heat treatment. Etched in 2% Nital followed by an electrolytic etch in sulphuric acid. (a) indicates the thin transition zone region, (b) indicates the medium thickness transition zone region, and (c) indicates the thick transition zone region,

5.2 Cross-weld microhardness profiles of A508/IN600 joint

Hardness values were taken across the interface of the A508/IN600 joint in three locations (thin, medium, and thick transition zone regions) with 100 µm spacing between indents, these hardness profiles are presented in Figure 5.2. In addition, bulk alloy microhardness values were measured on 10 points far (>10mm) from the interface in each alloy, which are presented in Table 5.1. As with the 304L joint, there is a scatter of hardness values throughout the A508 material

For the thin-transition zone region (Figure 5.2(a)), the microhardness profile shows that in the A508 bulk, the hardness has an average value of approximately 240 HV₂₀₀. This is comparable to the bulk

measurement shown in Table 5.1. The hardness is fairly constant throughout the A508, up to the interface. The transition zone is thinner than the 100 μm spacing, so the profile does not show a transition zone. At the interface, the hardness reaches a local peak of about 268 HV_{200} . On the other side of the interface, in the IN600, the hardness drops sharply to an average of 176 HV_{200} , which is roughly consistent with the hardness value of IN600 bulk in Table 5.1.

For the medium thickness transition zone region (Figure 5.2(b)), the hardness in the A508 bulk has an average of approximately 238 HV_{200} , which again is comparable to the bulk measurement shown in Table 5.1. At the interface, the hardness reaches a local peak of about 264 HV_{200} . On the other side of the interface, the hardness drops sharply to an average of 173 HV_{200} in the transition zone (up to 0.35 mm from the A508 interface). The hardness drops to a minimum of 159 HV_{200} at 0.5 mm. The IN600 bulk material has a similar hardness value (176 HV_{200} average).

For the thick-transition zone region (Figure 5.2(c)), in the A508 bulk the hardness has an average value of 256 HV_{200} . This is still comparable to the bulk measurement shown in Table 5.1 but is slightly higher than in the other two regions and the scatter is less pronounced. At the interface, the hardness reaches a local peak of about 268 HV_{200} . On the other side of the interface, the hardness drops sharply to an average of 178 HV_{200} in the transition zone (up to 1.5 mm from the A508 interface), followed by a dip in hardness to 160 HV_{200} at 1.6 mm. The IN600 bulk material has an average hardness of 185 HV_{200} , which is roughly similar to the transition zone average.

Table 5.1: Microhardness measurements of the bulk material in the A508/IN600 joint. 10 measurements were taken at points far (>10mm) from the interface in each case.

	Microhardness measurements (HV_{200})	
	A508	IN600
Mean	249.2	180.5
Range	60.0	27.6
Std Dev	23.2	9.4
Std Err	7.3	3.0

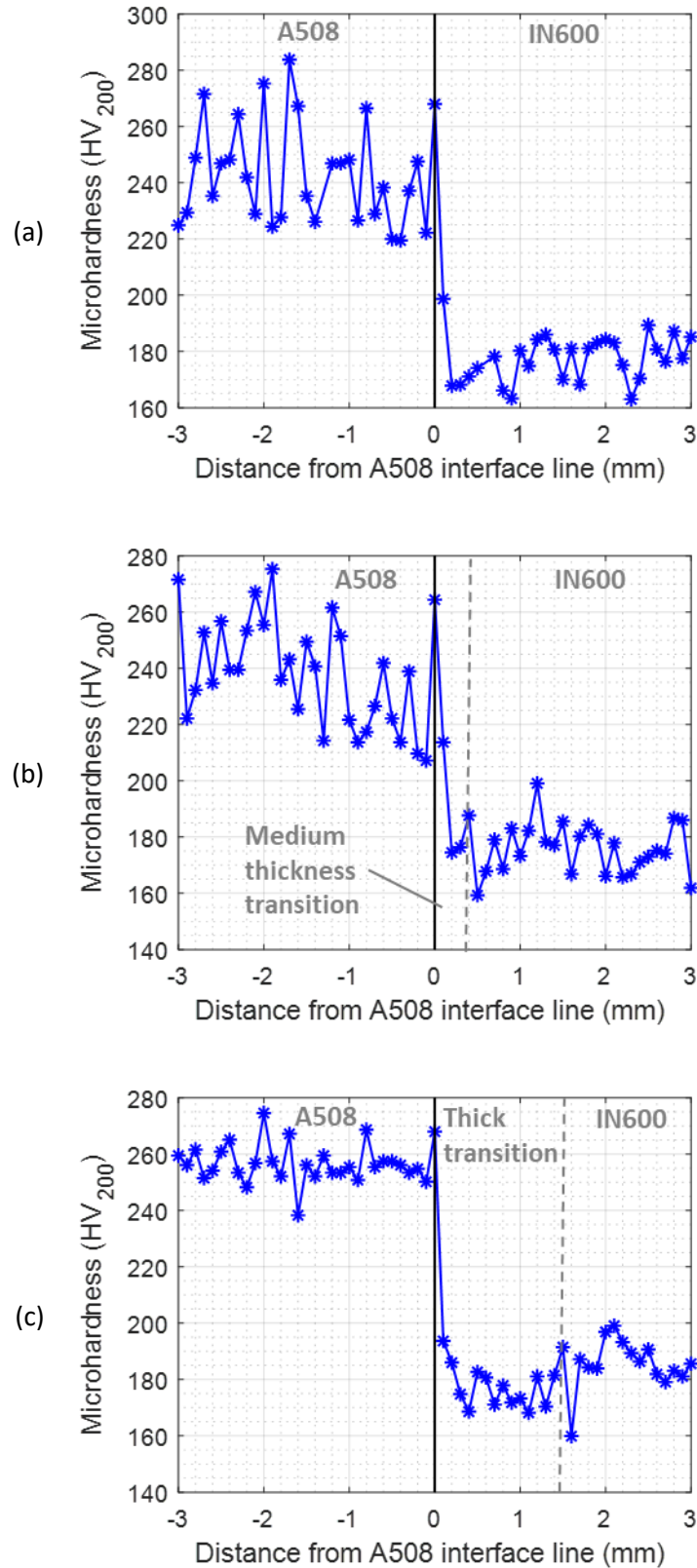


Figure 5.2: Microhardness profiles across interface of the A508/IN600 joint at (a) thin transition zone region (b) medium transition zone region and (c) thick transition zone region. The dotted line indicates the boundary between transition zone and IN600 viewed under optical microscope.

5.3 Microstructure of bulk materials

The microstructure of the bulk materials in the joint, away from the interface between the alloys, were analysed in order to provide the starting point for the microstructural study of the joint. This can then be compared with the microstructure in the vicinity of the interface between the alloys in order to assess the weld integrity.

The microstructure of etched A508 bulk is presented in Figure 5.3. This shows typical tempered bainitic structure with some MnS inclusions visible.

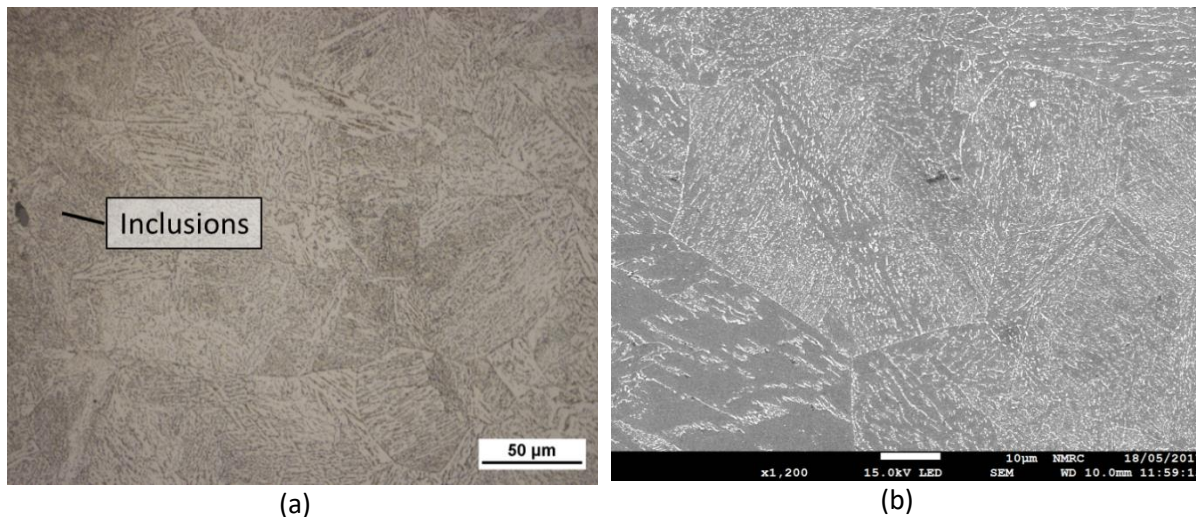


Figure 5.3: (a) Optical micrograph and (b) scanning electron micrograph of A508 bulk from A508/IN600 joint.

The microstructure of etched IN600 bulk is presented in Figure 5.4. Figure 5.4(a) shows that there are grain and twin boundaries in this alloy. A closer look in Figure 5.4(b) shows there are intergranular as well as intragranular precipitates, and these can be a mixture of $M_{23}C_6$ and M_7C_3 carbides [36]. The grain boundaries are serrated, which is thought to improve creep life and creep ductility [100], thus suggesting that the post-forge heat treatment temperature is suitable for this alloy specifically.

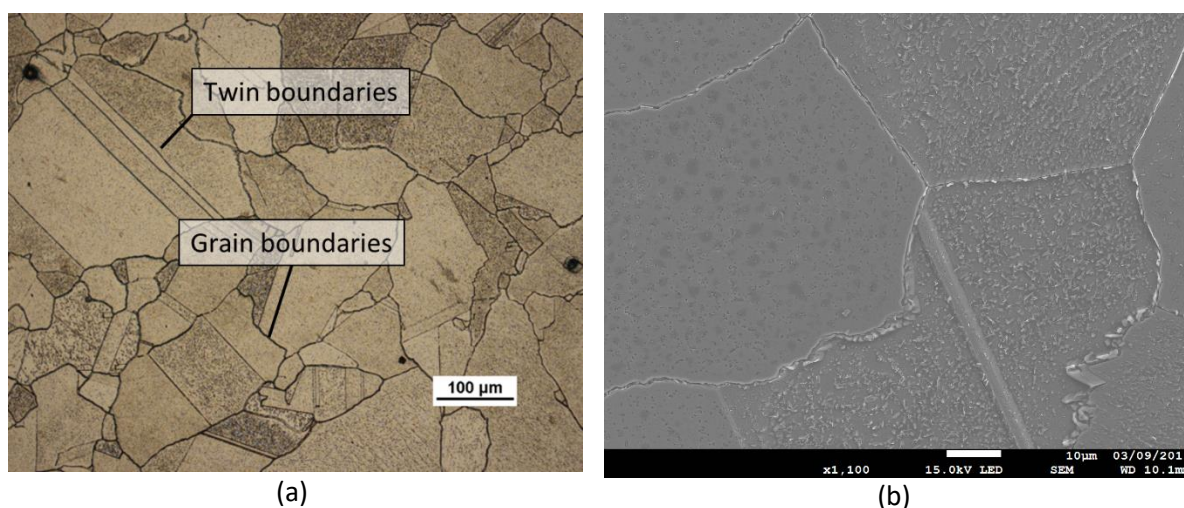


Figure 5.4: (a) Optical micrograph and (b) scanning electron micrograph of IN600 bulk from A508/IN600 joint.

5.4 Microstructure of thin transition zone region of A508/IN600

5.4.1 Cross-weld composition profile

The composition profile across the interface of the A508/IN600 joint in the thin transition zone region was measured, with 5 μm spacing between each measurement, using EDX analysis. This is presented in Figure 5.5, which shows that the concentration of Fe in A508 is 97 wt% and begins to transition, less than 7 μm from interface, smoothly to around 18 wt% in the IN600 side of the interface. The latter is well above the nominal amount of Fe in IN600 (8.74 wt%) mentioned in the materials specification. The opposite is true for Ni, which is 0.7 wt% in A508 and 67 wt% in IN600 (nominal Ni content in IN600 is 74.37 wt%). In another EDX measurement taken across a greater distance, as shown in Figure 5.6, it is shown that there is a region of about 200 μm where the composition is intermediate between that of A508 and IN600. The change/transition in composition occurs at a longer range; all these indicate the presence of a relatively wide mixed zone at the interfacial area even in the thin transition zone region. The thin transition zone composition then shifts to IN600 in about 40 μm range.

However, measurements taken away from interface (>3mm from interface) shows that the concentration of major elements (Fe: 8.9 ± 0.1 wt%, Ni: 75.6 ± 0.3 wt%, Cr: 15.5 ± 0.3 wt%) do coincide with the specification compositions.

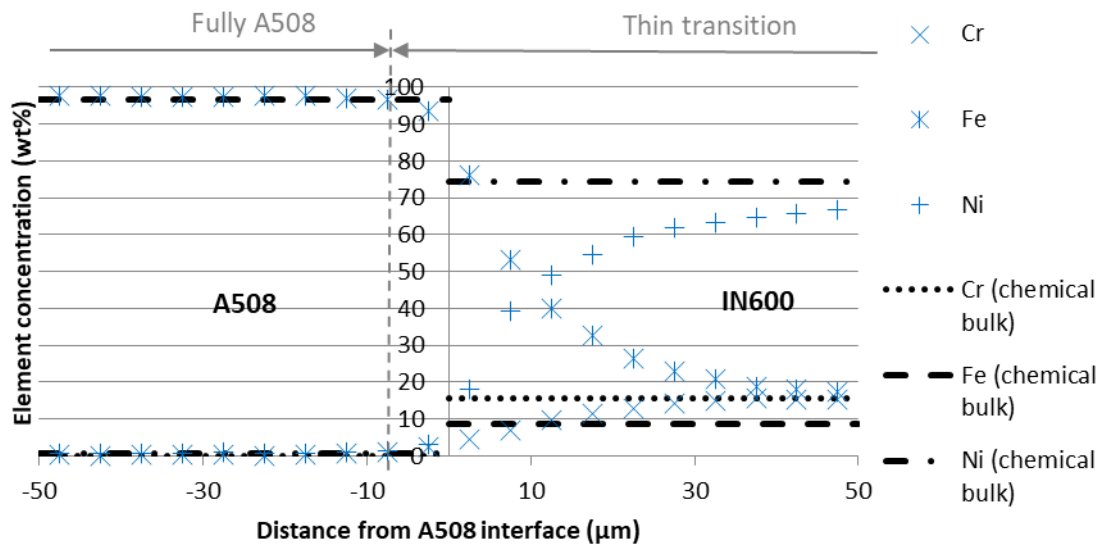


Figure 5.5: Composition profile of major elements (Cr, Fe, Ni) across the thin transition zone in A508/IN600 joint.

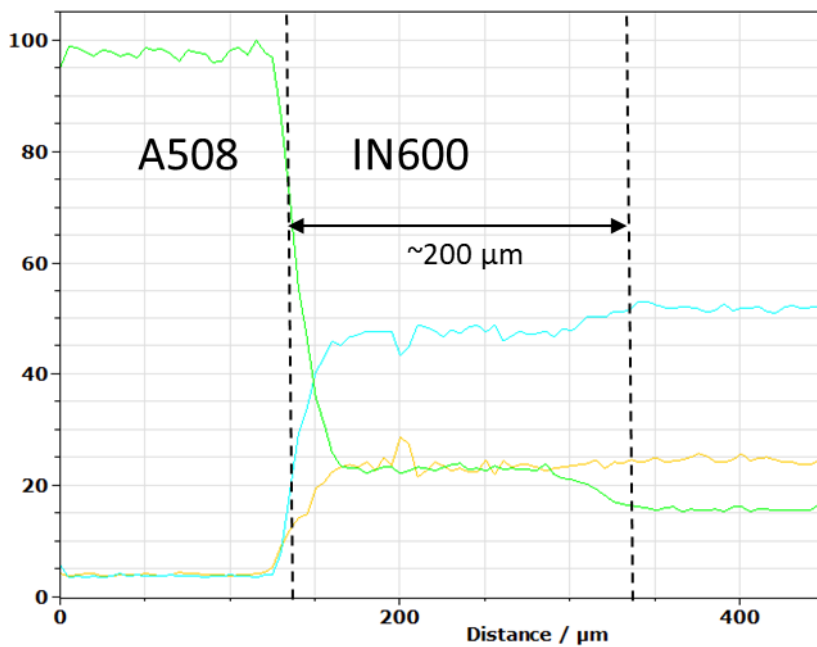


Figure 5.6: Qualitative composition profile of major elements (Cr, Fe, Ni) across the thin transition zone in A508/IN600 joint, taken at greater distance. The dotted line is the A508 interface.

5.4.2 Microstructural evolution of A508 side of interface

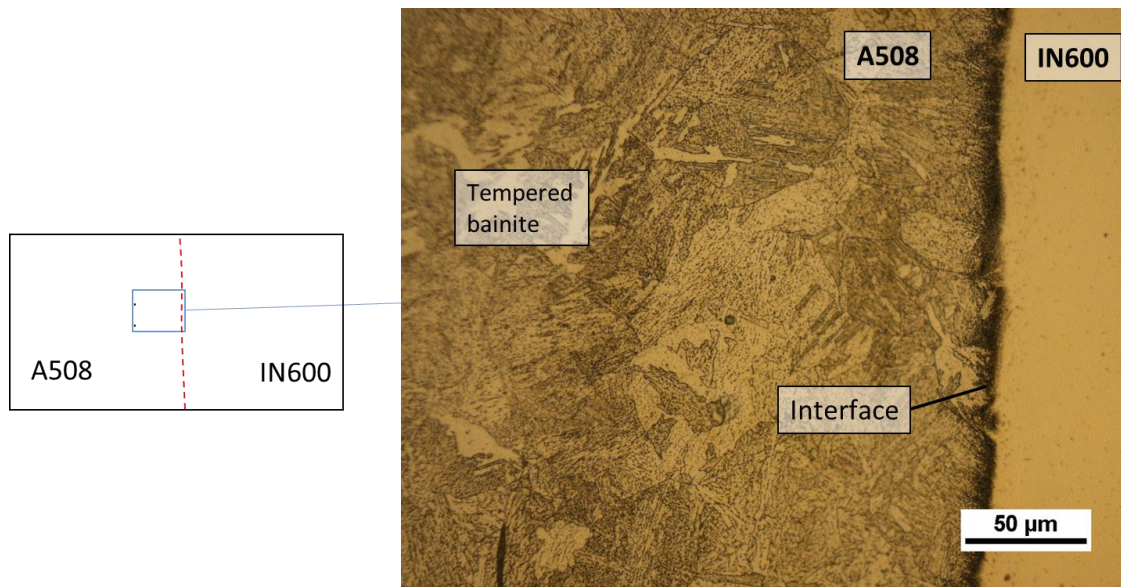


Figure 5.7: A508 side of the interface in the thin transition zone region of A508/IN600 joint.

The location of examination for microstructure of the A508 in the vicinity of the interface is displayed in Figure 5.7. The figure also illustrates the etched A508 side of the interface of A508/IN600 joint, showing that there appears to be little microstructural variation in A508 as a function of distance from the interface. This could explain the flat hardness trend observed in the microhardness profile in Figure 5.2(a), unlike for the A508/304L joint, which contains a trough in hardness corresponding to the precipitate-depleted zone (PDZ) adjacent to the interface. There is a rough interface with plenty of precipitates in the vicinity that may indicate a martensitic band, as can be seen in Figure 5.8.

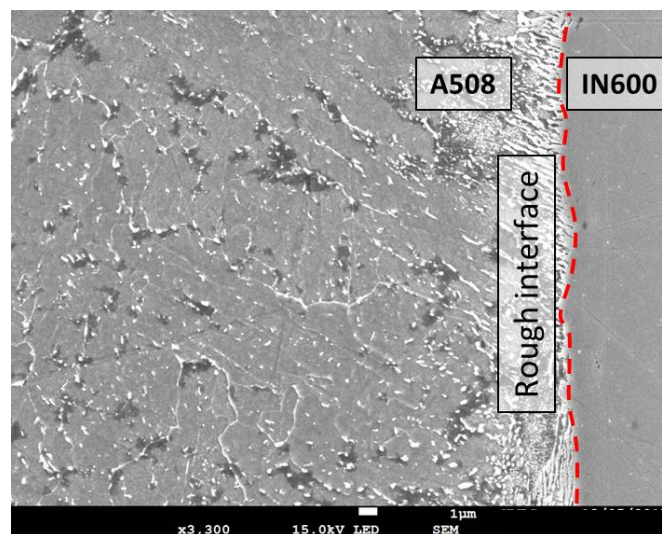


Figure 5.8: Scanning electron micrograph (SE mode) of A508 side of the interface in the thin transition zone region of A508/IN600 joint.

5.4.3 Microstructural evolution of IN600 side of interface (including the thin transition zone)

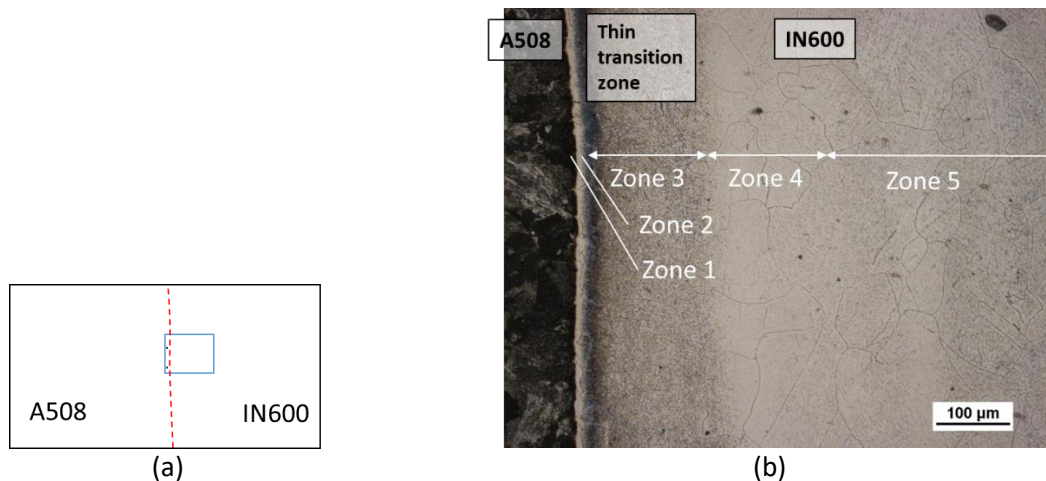


Figure 5.9: (a) Location of IN600 side of interface. (b) Overview of etched IN600 side of the interface of A508/IN600 joint in the thin transition zone region.

Figure 5.9 shows the location and an overview of several distinct microstructural zones in the IN600 side of the interface. Figure 5.10 shows that Zone 1, which is about 10 μm wide and located next to the interface line, is shown to be devoid of precipitates. Zone 2, which is about 20 μm wide, is very precipitate dense. Zone 3 is about 200 μm wide, containing slightly less precipitates than Zone 2. Zone 4, which is 100 μm wide, has precipitates mostly in the grain boundaries. Finally, Zone 5 is the bulk IN600. When comparing zone widths with the composition profile in Figure 5.6, it appears that the thin transition region comprises Zones 1-3 in the IN600 side of the interface.



Figure 5.10: Optical micrograph of IN600 alloy side of the interface of A508/IN600 joint in the thin transition zone region.

Higher resolution images of the zones using SEM are presented in Figure 5.11. Zone 1 is shown to have no precipitates, even at the grain boundaries. The precipitates in Zone 2 are both intergranular and intragranular. The intragranular precipitates are densely packed and have different morphologies, with both needle and spheroid shapes. Zone 3 also has these precipitates, but they are more spaced apart. Zone 4 contain mostly intergranular precipitates, though intragranular precipitates are also present. The microstructure of Zone 5 is just the bulk IN600 microstructure shown in a previous section.

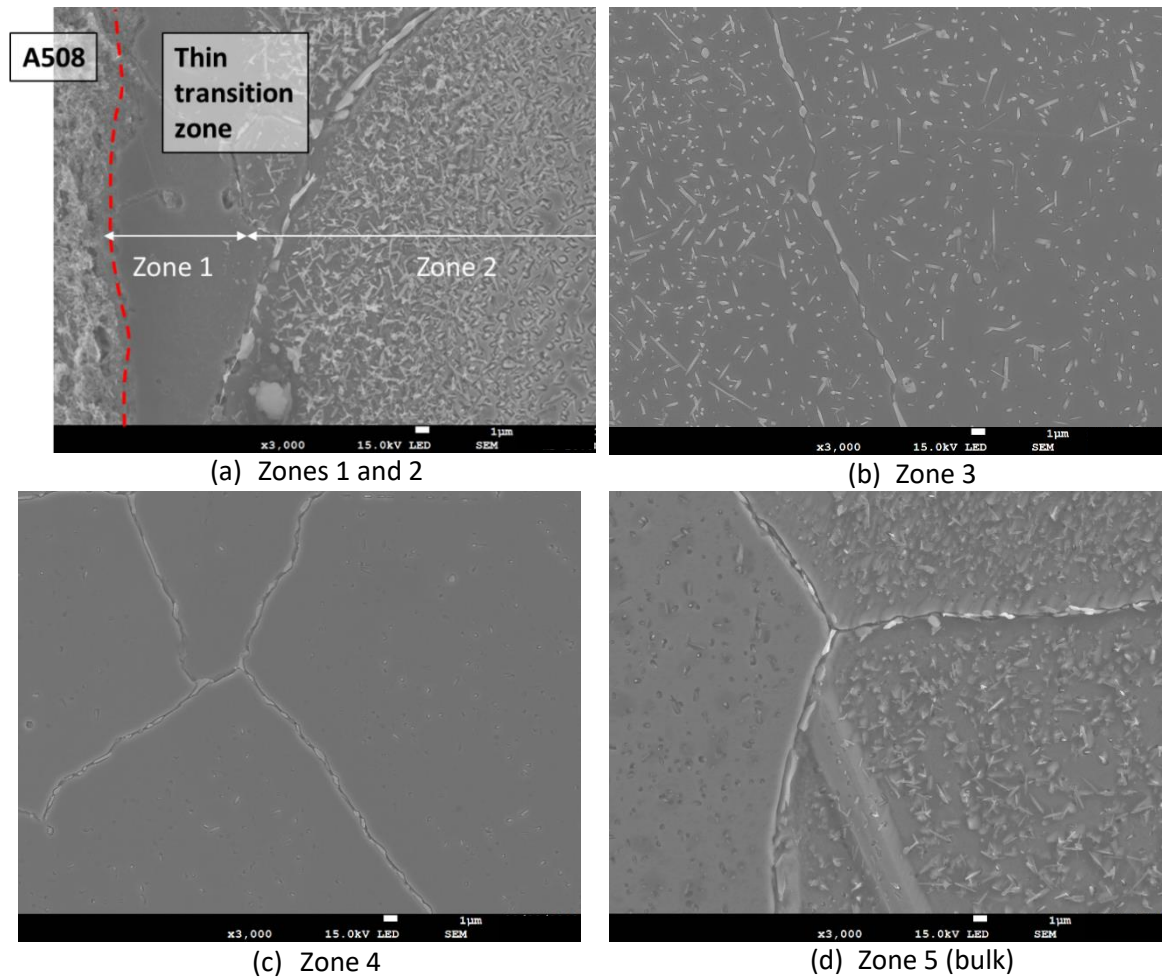


Figure 5.11: Scanning electron micrographs of the zones in the IN600 side of the interface of A508/IN600 joint in the thin transition zone region. (a) Zones 1 & 2, (b) Zone 3, (c) Zone 4 and (d) Zone 5.

5.4.4 EBSD crystal structure in the vicinity of the thin transition zone

EBSD analysis of the thin transition zone and the surrounding bulk material, presented in Figure 5.12, shows that A508 side and thin transition zone (originally thought of as the full IN600) side of the interface line have BCC and the FCC crystal structures, respectively. The boundary between the BCC and FCC crystal structures coincides with the observed boundary between A508 and the thin interfacial zone. On the interface at the A508 side, the grains are relatively fine, indicating a distinct zone/band of microstructure, possibly martensite band formation. EDX mapping, displayed in Figure 5.13, shows that the BCC/FCC boundary occurs when Fe is still mostly A508 in composition and while Ni is still low.

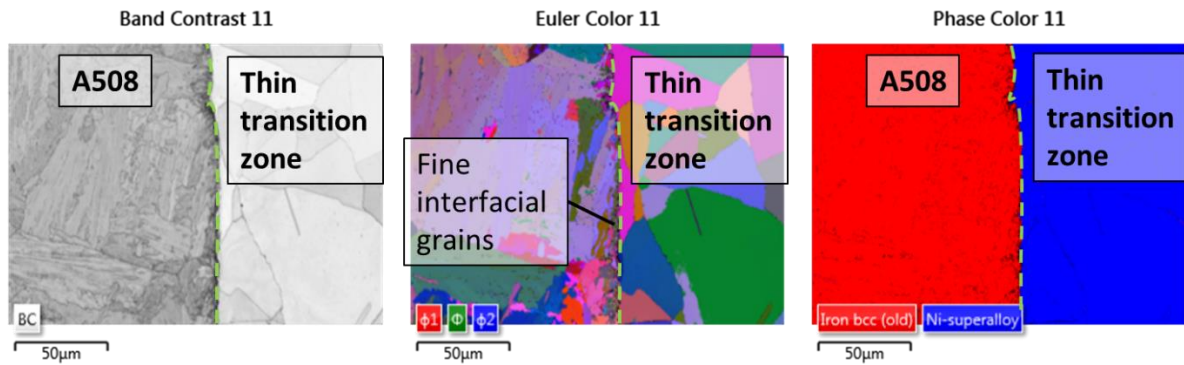


Figure 5.12: EBSD mapping of the vicinity of the thin transition zone in the A508/IN600 joint. Black indicates non-indexed solutions.

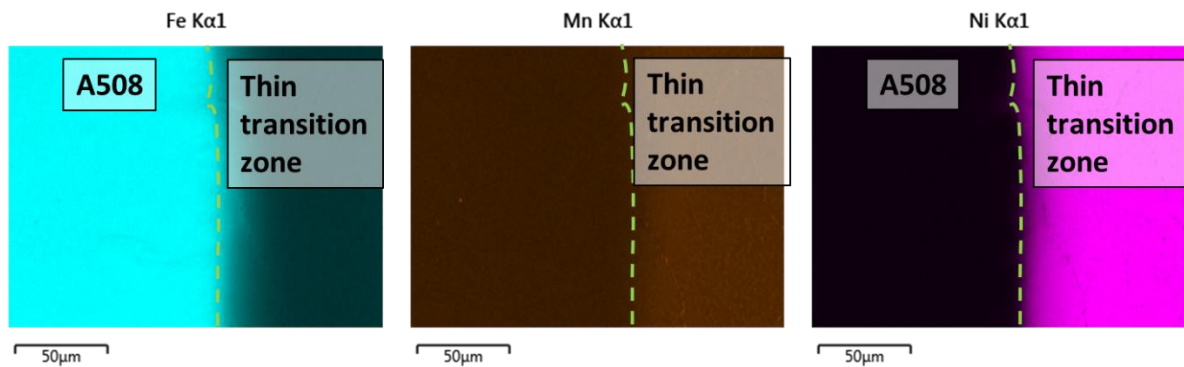


Figure 5.13: EDX mapping of Fe, Mn, and Ni in the vicinity of the thin transition zone in the A508/IN600 joint.

5.4.5 Nanoindentation testing

Similar to that carried out for the A508/304L joint, nanoindentation profile across the interface of the A508/IN600 joint in the thin transition zone region were measured, with 30 μm spacing between each indent, using a Berkovich indenter. Each indent was made with a controlled depth of 1 μm . Two profiles, with 33 indents each, were made from -480 μm to 480 μm distance from the A508 interface, as schematically shown in Figure 5.14. The nanoindentation profile traverses deep in the A508 bulk, up to the start of Zone 5 (the IN600 bulk). So all distinct microstructural zones can be said to be covered by the nanoindentation profile. For clarity, only 33 of these P - h curves, from the indents of the lower line in Figure 5.14, are reported in Figure 5.15.

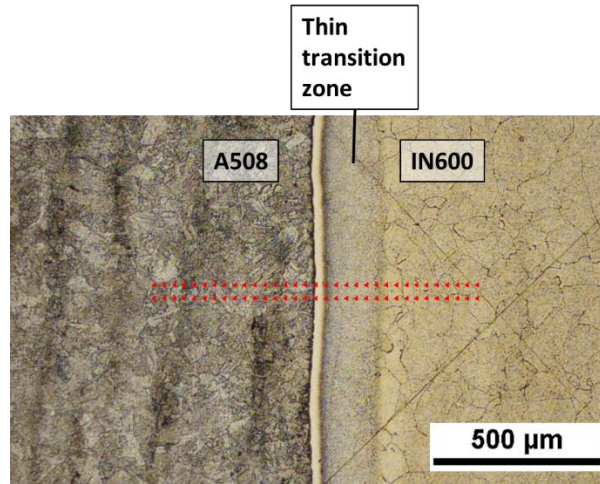


Figure 5.14: Schematic of the nanoindentation profile across the A508/IN600 joint in the thin transition zone region.

Profiles of the optimised nanoindentation parameters across the thin transition zone in the A508/IN600 joint using Limits Sets 1, 2, and 3 are presented in Figure 5.16, Figure 5.17, and Figure 5.18, respectively. Figure 5.16 shows that, when the upper limit of 300 GPa for E was imposed, the optimised E tends to mostly scatter close 300 GPa. This indicates that the 300 GPa constraint has forced the values of E to be lower than they should at all indents. Figure 5.17 shows that, when the upper limit for E was increased to 400 GPa, most of the points increase to more than 300 GPa but does not hug the maximum value of 400 GPa, and the average normalised error decreased slightly to 0.419 (compared to average normalised error of 0.611 when the upper limit of 300 GPa for E was imposed). Normally, the value of E for both the A508 and IN600 should be from 200-210 GPa. The optimised E values are far higher than this. The σ_y profile in the Limits Set 1 is slightly higher than that of Limits Set 2, even though they follow a general trend, indicating that the optimisation process compensated the constraint provided by the 300 GPa limit in Limits Set 1 by increasing σ_y values. Figure 5.18 shows the optimised parameters when E was derived from the stiffness S of each indent, by using Equations 3.1 and 3.3, while ν was assumed to be 0.3; only the other two parameters (σ_y and n) were optimised. It shows that on the A508 side of the interface, E is between 190 GPa and 210 GPa, but the stiffness dips in the thin transition zone to less than 180 GPa, and then increasing to between 180 GPa and 200 GPa in the rest of the thin transition zone; and in the IN600, E is between 185 and 210 GPa. While Limits Set 3 is the best for determining E , the optimised parameters yielded an average normalised error of

1.404, meaning is the most difficult for the simulated P - h curve to converge with the measured P - h curve. Also the σ_y profile in the Limits Set 3 is slightly elevated compared to those in the Limits Sets 1 and 2. This may be due to the fact that the optimisation process in Limits Set 3 compensates the inability to increase E (because E for each indent is pre-determined) by increasing the σ_y values, resulting in a higher σ_y values than it should be. Still, all three σ_y profiles have the same general trend, i.e. relatively flat in the A508 bulk, gradually increasing to a peak around the interface, and then gradually decreases within the thin transition zone, and flat again in IN600. As with the previous chapter, the fact that the results are very different depending the on the limits set, particularly for E , suggests that inverse method used here may not have resulted in well-optimised P - h curves.

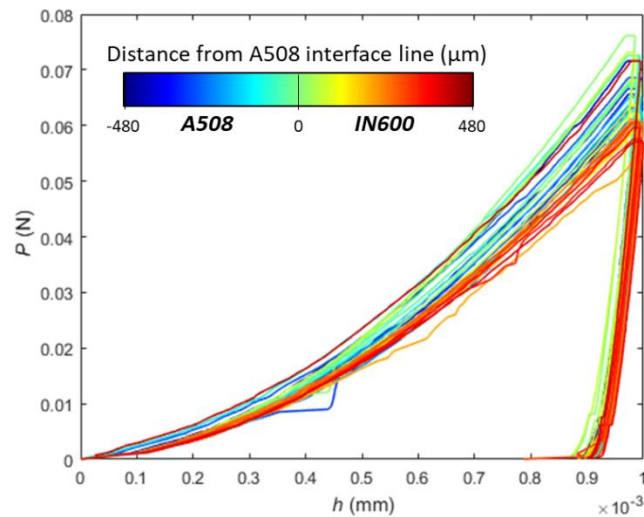


Figure 5.15: P - h curves measured from 33 of the nanoindentations across the A508/IN600 joint in the thin transition zone region.

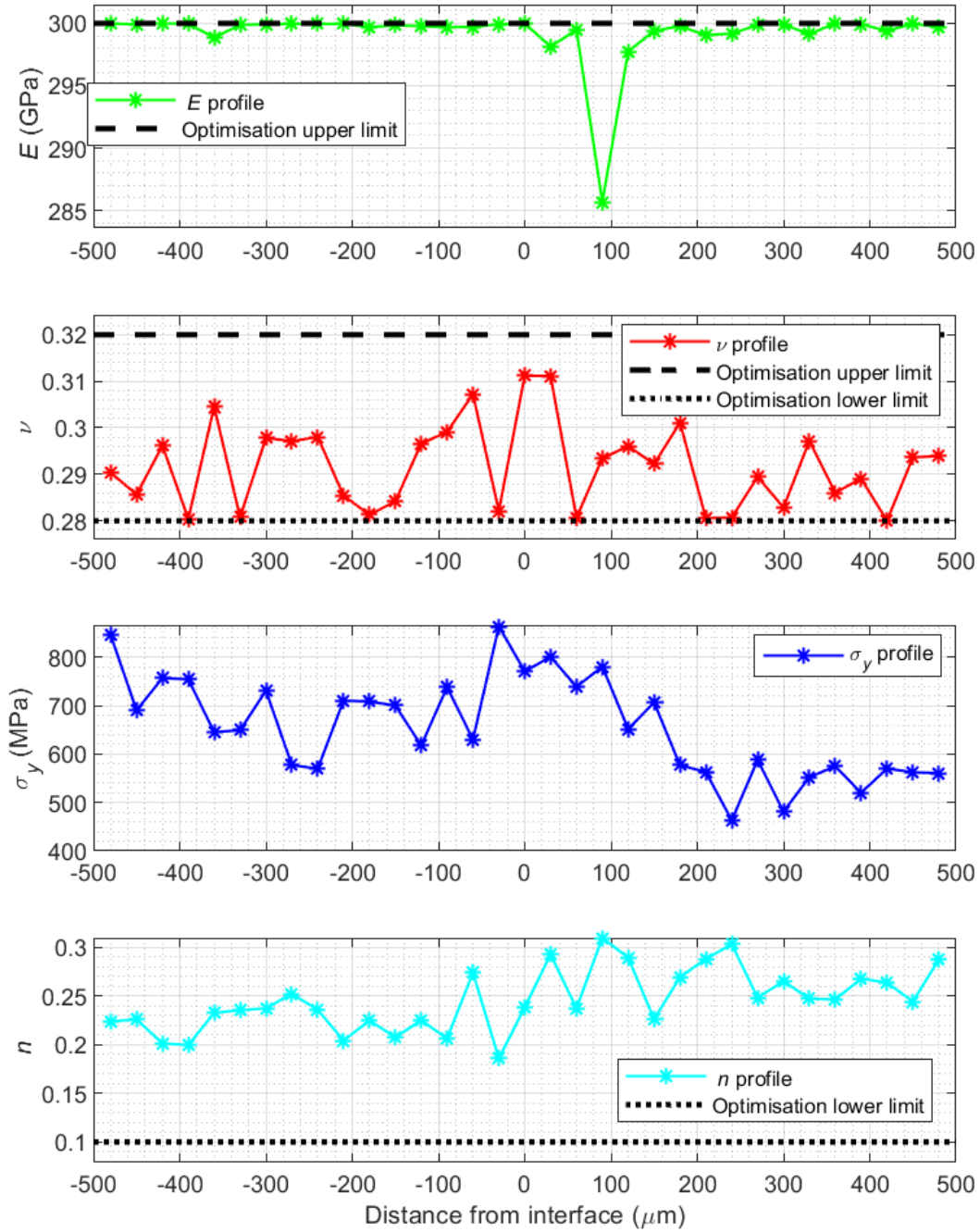


Figure 5.16: Profile of the optimised nanoindentation parameters across the thin transition zone in the A508/IN600 joint using Limits Set 1. The average normalised error is 0.611.

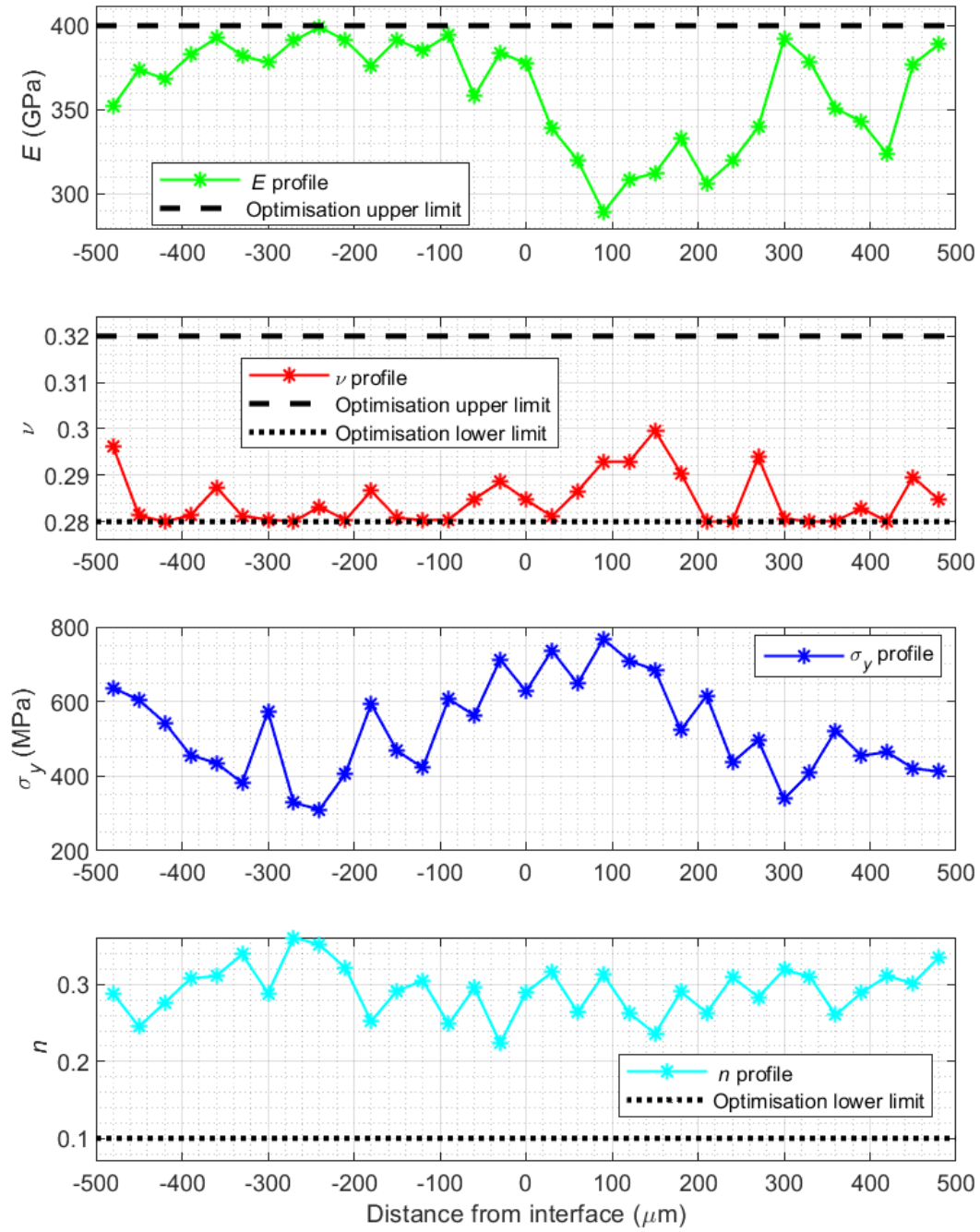


Figure 5.17: Profile of the optimised nanoindentation parameters across the thin transition zone in the A508/IN600 joint using Limits Set 2. The average normalised error is 0.419.

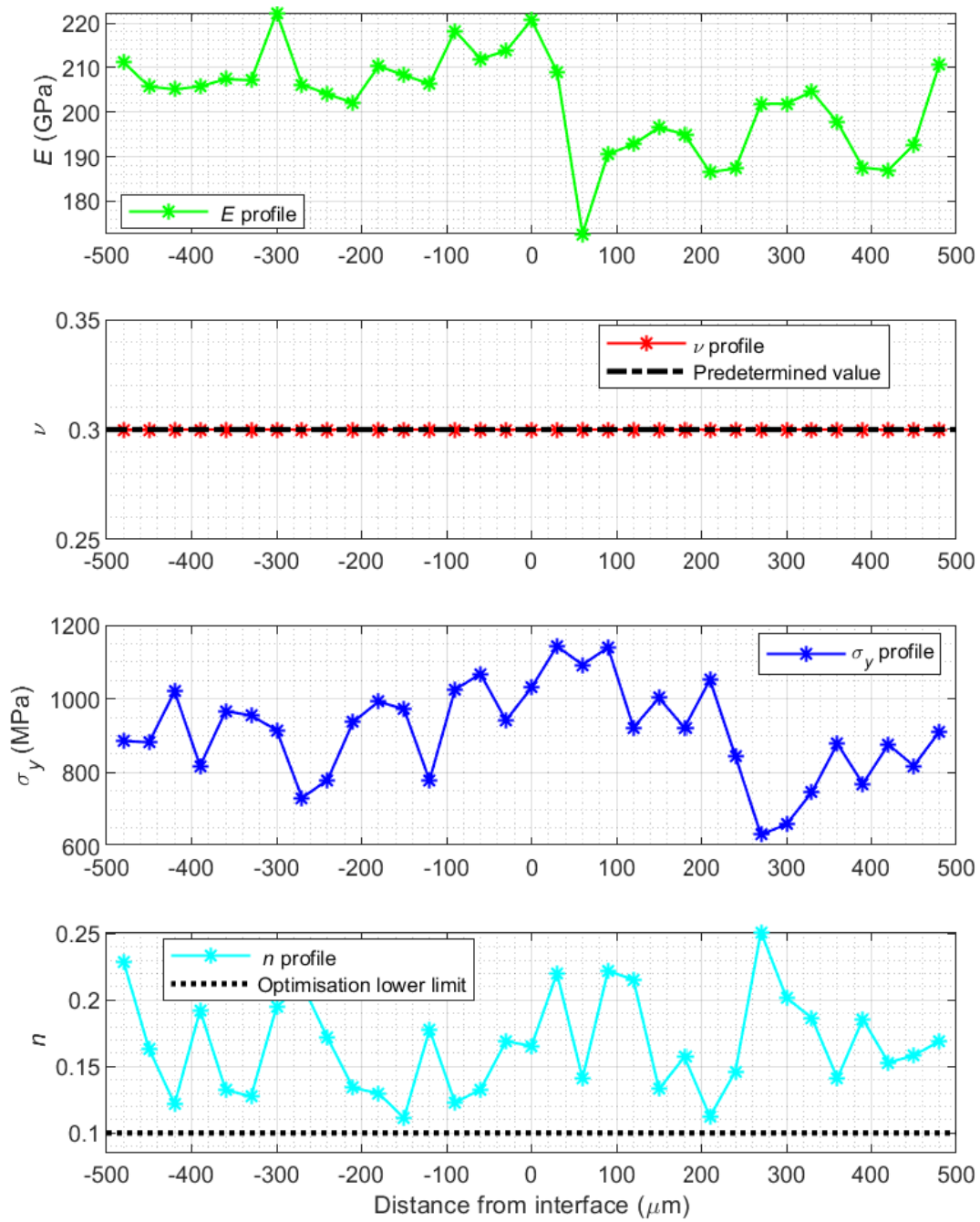


Figure 5.18: Profile of the optimised nanoindentation parameters across the thin transition zone in the A508/IN600 joint using Limits Set 3. The average normalised error is 1.404.

5.5 Microstructure of medium thickness transition zone region of A508/IN600

5.5.1 Cross-weld composition profile

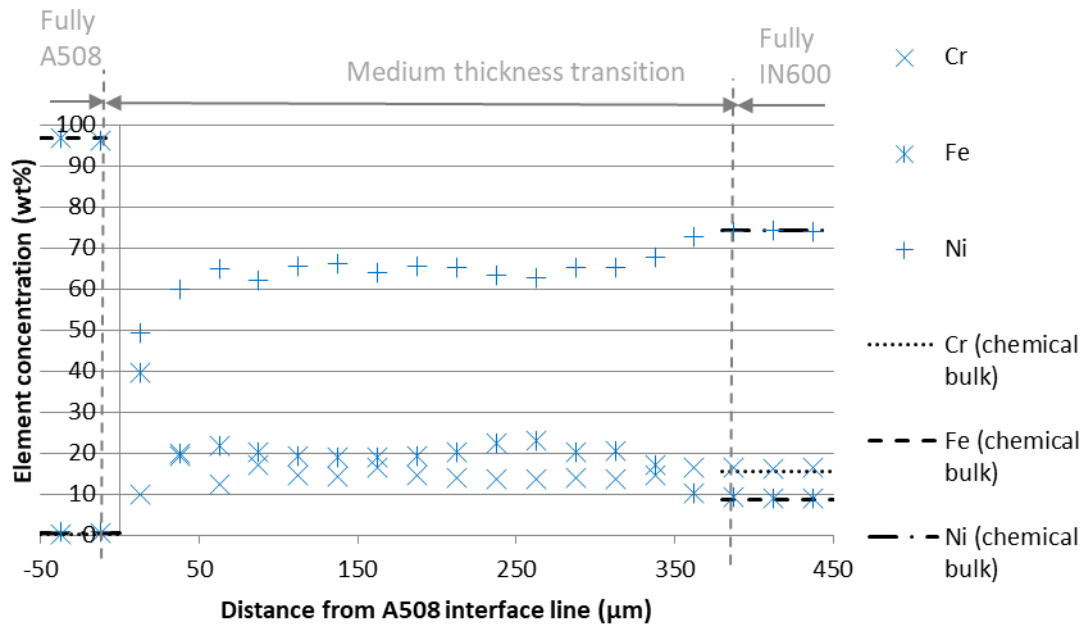


Figure 5.19: Composition profile of major elements (Cr, Fe, Ni) across medium thickness transition zone region of A508/IN600 joint. The values for the chemical bulk compositions for each alloy originates from the chemical analysis provided by supplier of the bulk alloys.

The composition profile across the interface of the A508/IN600 joint in the medium thickness transition zone region were measured, with 25 μm spacing between each measurement, using EDX analysis. This is presented in Figure 5.19, showing that there is clearly an EBW effect in the location of EDX measurement as the transition of composition between one alloy to another occur in a wide range. The concentration of Fe in fully A508 is 97 wt%, and it is around 9 wt% in fully IN600, which agrees with the nominal compositions of the alloys provided by the manufacturer. The same is true for Cr and Ni (Cr is 0.2 wt% in A508 and 16 wt% in IN600, while Ni is 0.6 wt% in A508 and 74 wt% in IN600). The change/transition in composition occurs at a range of approximately 400 μm, from -12.5 μm to 387.5 μm distance from the A508 interface, meaning that the start of the change in composition from the fully A508 into the transition which does not exactly match the interface line. At most of the transition zone, the composition of Fe, Cr, and Ni are scatter between 19-23 wt%, 12-17 wt%, and, 62-66 wt% respectively from 62.5 μm to 312.5 μm.

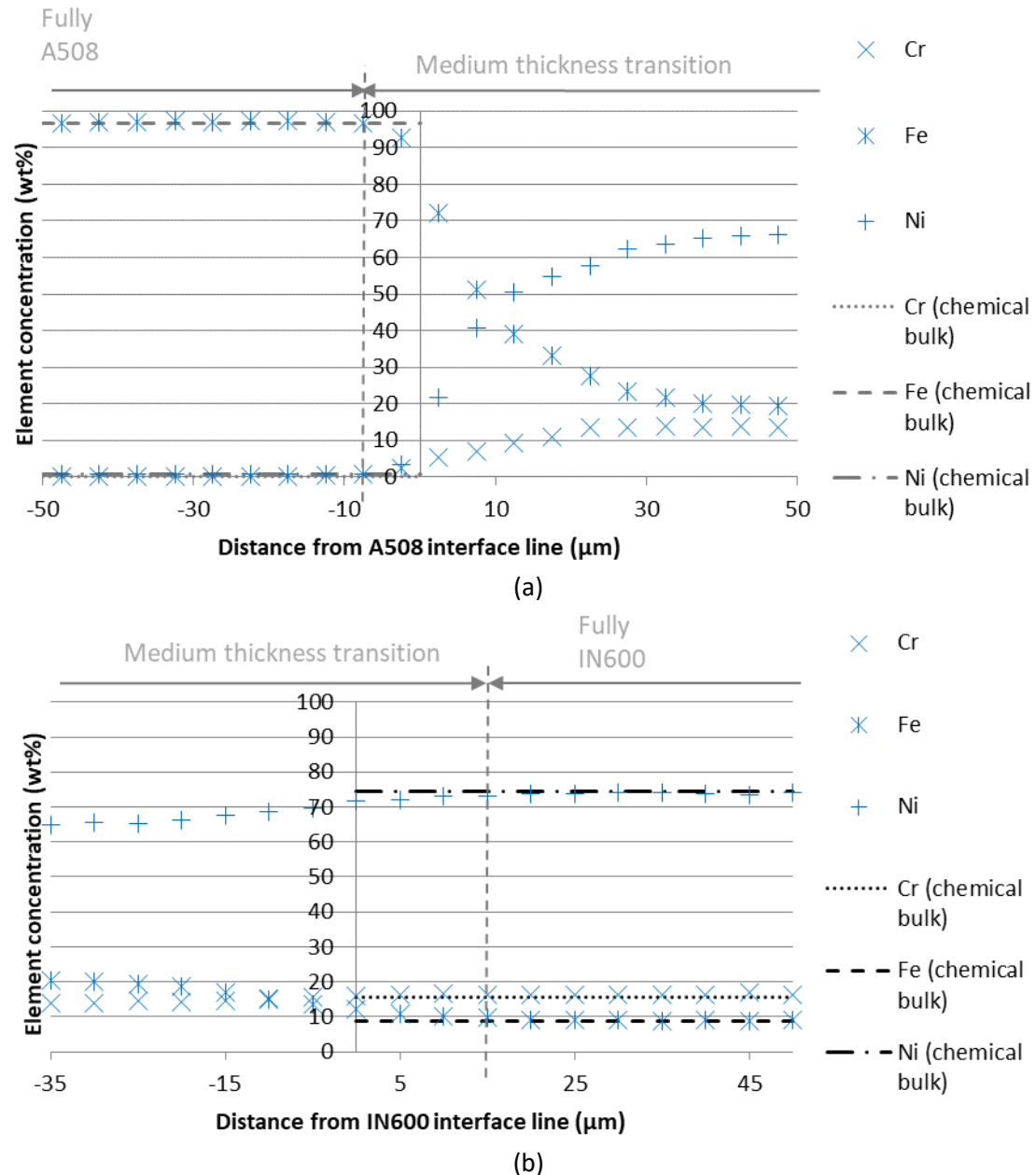


Figure 5.20: EDX profile in the vicinity of the interface lines between (a) A508-medium transition and (b) medium transition-IN600.

Because the interface lines (A508-transition boundary and transition-IN600 boundary) seem to occur in transition compositions, i.e. do not appear to mark the boundary between fully bulk-material composition and the transition composition, a closer look at the composition in the vicinity of the interface lines is taken. An EDX profile with 5 μm spacing between each measurement is presented in Figure 5.20 in the vicinity of the interface lines (i.e. the boundary between fully bulk-material composition and the transition composition). Figure 5.20(a), which is very similar to Figure 5.5, shows that the composition starts to shift from being fully A508 to the transition composition, at less than

10 μm into the A508 side of the interface. Similarly, Figure 5.20(b) shows that the composition starts to shift from being fully IN600 to the transition composition, at 15 μm into the IN600 side of the interface. This confirms the fact that the interface lines do not coincide with start of the shift in composition from the bulk values, though the offset magnitudes are different.

5.5.2 Microstructure of A508 side of interface

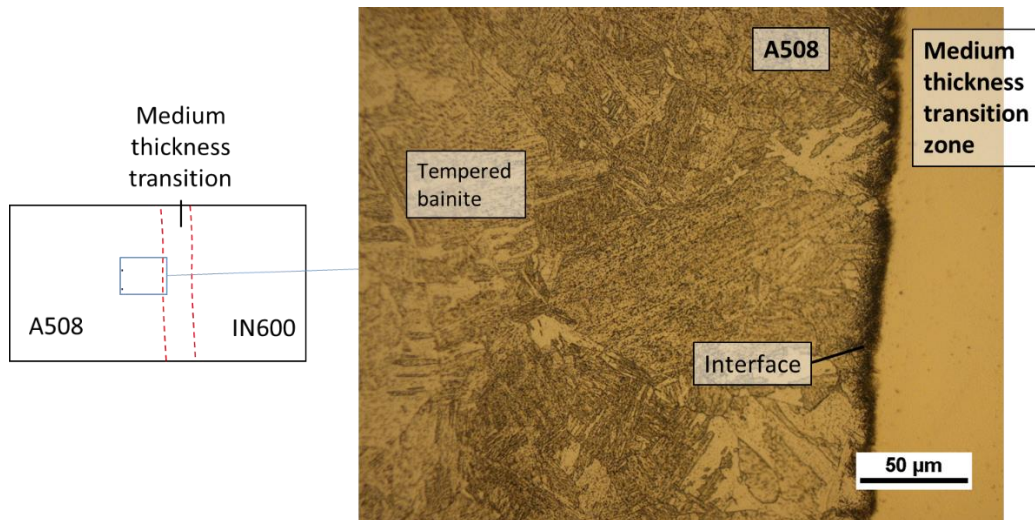


Figure 5.21: A508 side of the interface in the medium thickness transition zone region of A508/IN600 joint.

The location of examination for microstructure of the A508 in the vicinity of the interface is displayed in Figure 5.21. Similar to the thin transition zone region, there appears to be little microstructural variation in A508 as a function of distance from the interface at the A508 side of the interface. There is a rough interface with a large number of precipitates in the vicinity that may indicate a martensitic band, as can be seen in Figure 5.22.

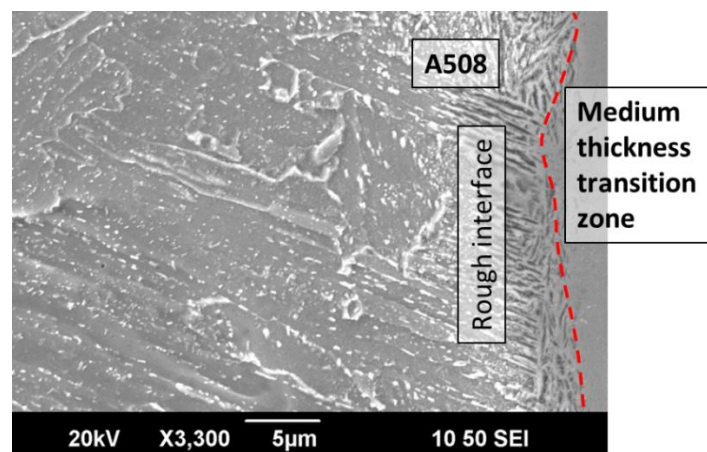


Figure 5.22: The interface A508 steel side of A508/IN600 joint at the medium thickness transition zone region.

5.5.3 Microstructure of medium thickness transition zone and the IN600 side of the interface

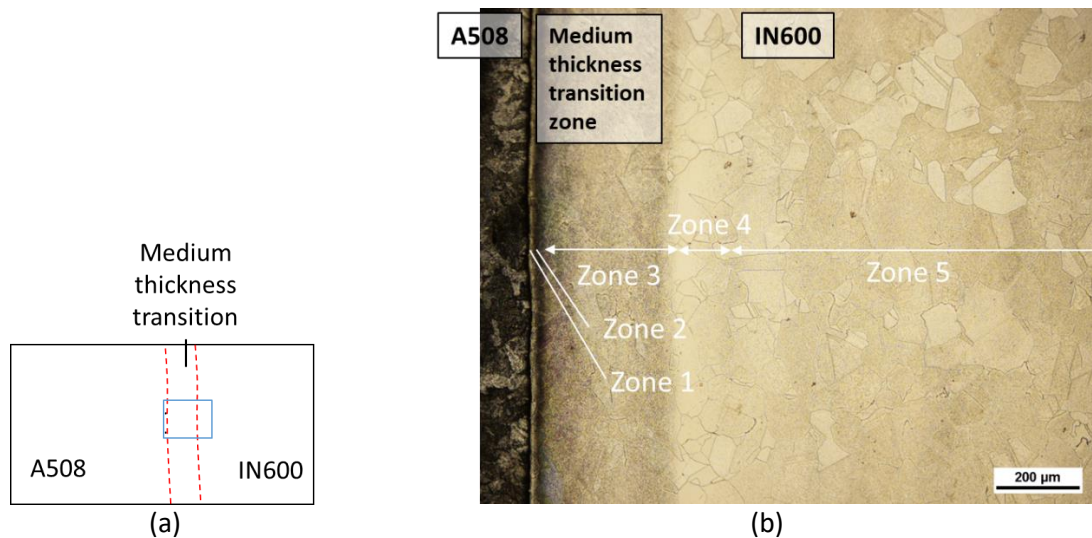


Figure 5.23: (a) Location of medium thickness transition zone. (b) Overview of etched medium thickness transition zone and IN600 side of the interface of A508/IN600 joint in the medium transition zone region. The medium thickness transition zone consists of Zones 1-3, and IN600 side consists of Zones 4 and 5.

Figure 5.23 shows the location and an overview of several distinct microstructural zones in the medium thickness transition zone and IN600 side of the interface. The medium thickness transition zone, which is about 350 μm wide, is divided into Zones 1-3, and IN600 side is divided into Zones 4 and 5. Figure 5.24 shows that Zone 1, which is about 10 μm wide and located next to the interface line, is shown to be devoid of precipitates. Zone 2, which is about 30 μm wide, is very precipitate dense. Zone 3 is about 300 μm wide, containing slightly less precipitates than Zone 2. Zone 4, which is 100 μm wide, has precipitates mostly in the grain boundaries. Finally, Zone 5 is the bulk IN600. These zones are very similar to those in the thin transition zone region; only the widths of Zones 2 and 3 differ. Higher resolution images of the zones using SEM are presented in Figure 5.25. These are similar to those in the thin transition zone region presented in Figure 5.11.

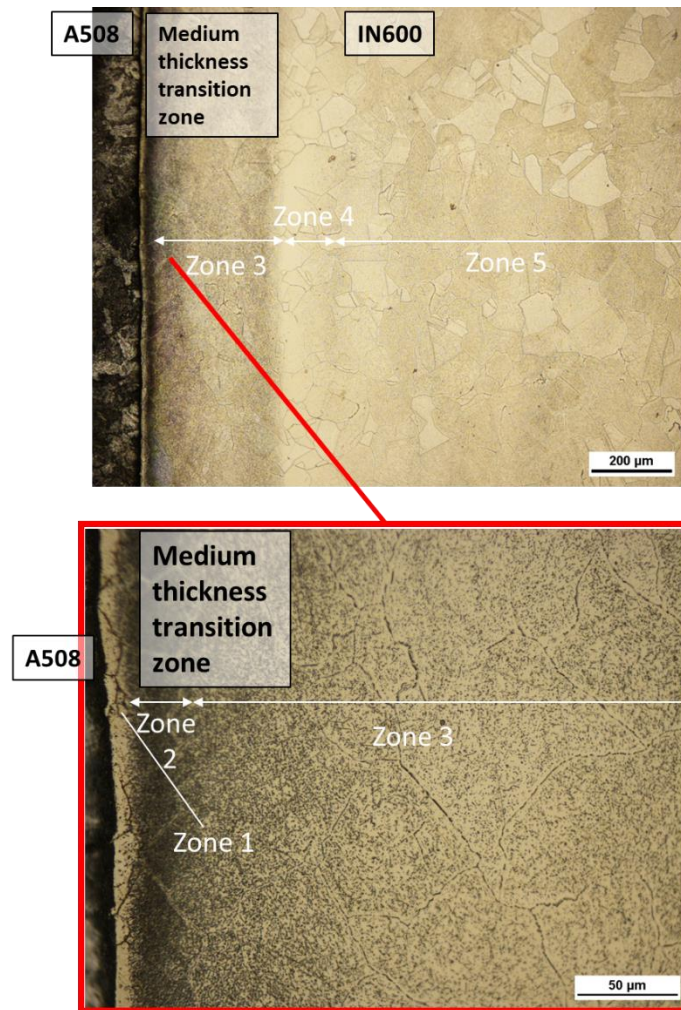


Figure 5.24: Optical micrograph of IN600 alloy side of the interface of A508/IN600 joint in the medium thickness transition zone region.

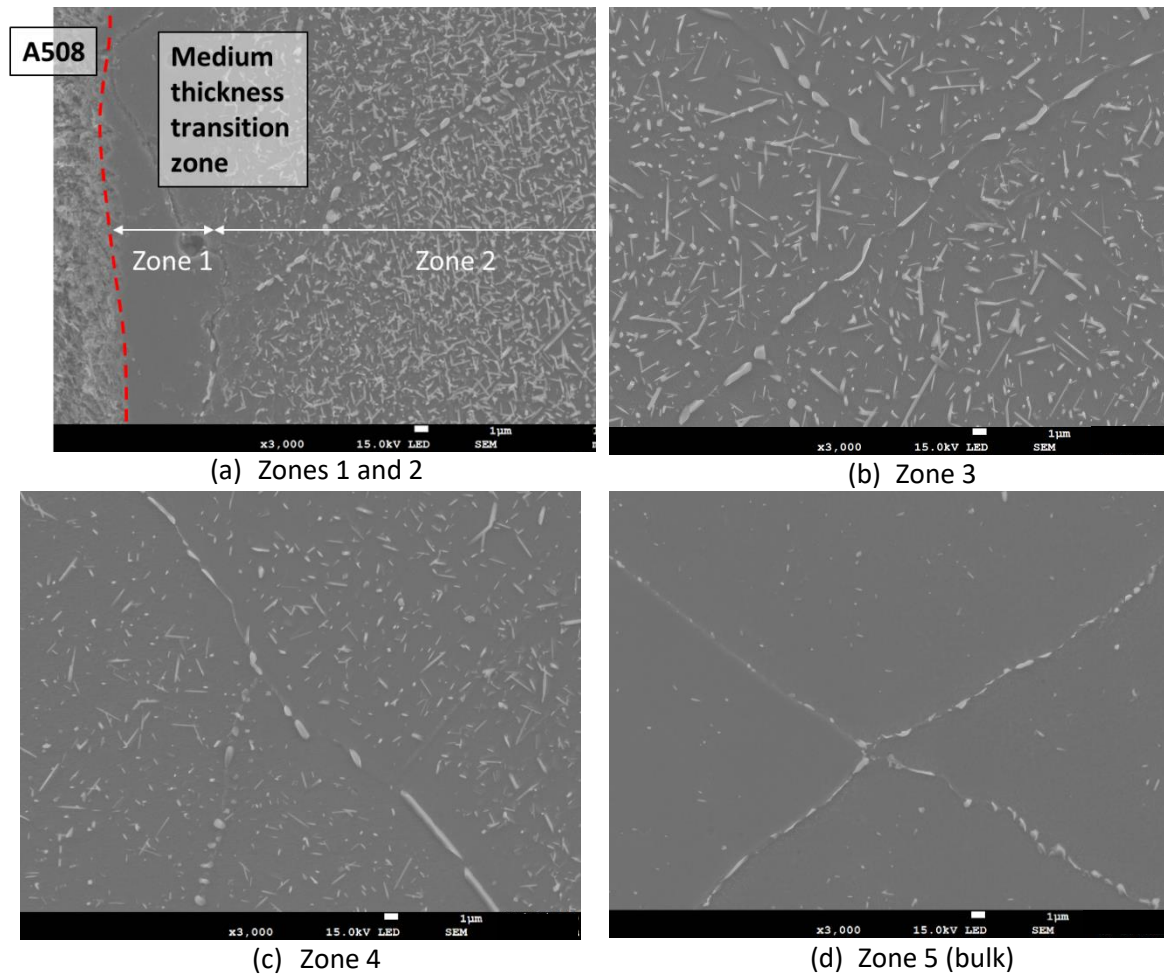


Figure 5.25: Scanning electron micrographs of the zones in the IN600 side of the interface of A508/IN600 joint in the medium thickness transition zone region. (a) Zones 1 & 2, (b) Zone 3, (c) Zone 4 and (d) Zone 5.

5.5.4 EBSD crystal structure in the vicinity of the medium thickness transition zone

EBSD analysis of the thin transition zone and the surrounding bulk material, presented in Figure 5.26, shows that A508 side has a BCC crystal structure, whereas both the medium transition zone and IN600 have a FCC crystal structure. In terms of crystal structure, grain morphology, and grain size, the medium transition zone and IN600 side are virtually indistinguishable and uniform. EDX mapping, displayed in Figure 5.27, shows while there are zones of slightly lower Fe concentrations (coinciding with slightly higher Ni concentrations) that run parallel to the interfaces, the composition within the medium transition zone is mostly uniform.

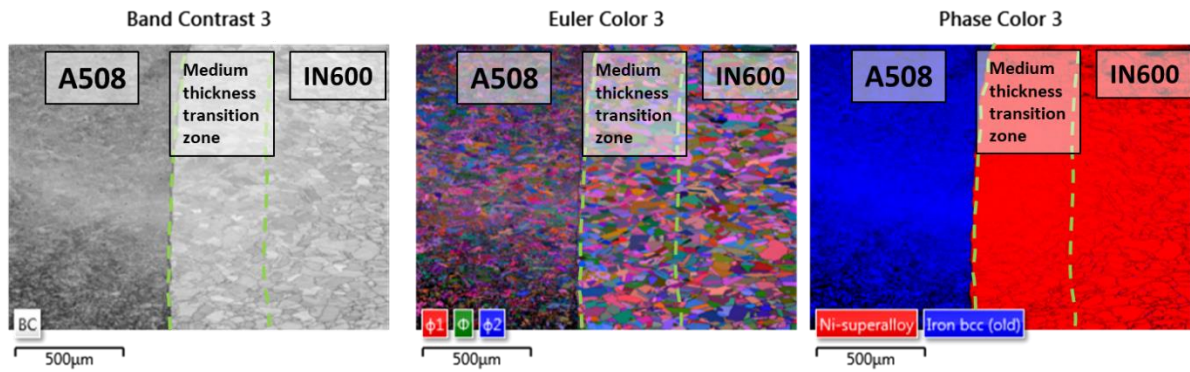


Figure 5.26: EBSD mapping of the vicinity of the medium thickness transition zone in the A508/IN600 joint. Black indicates non-indexed solutions.

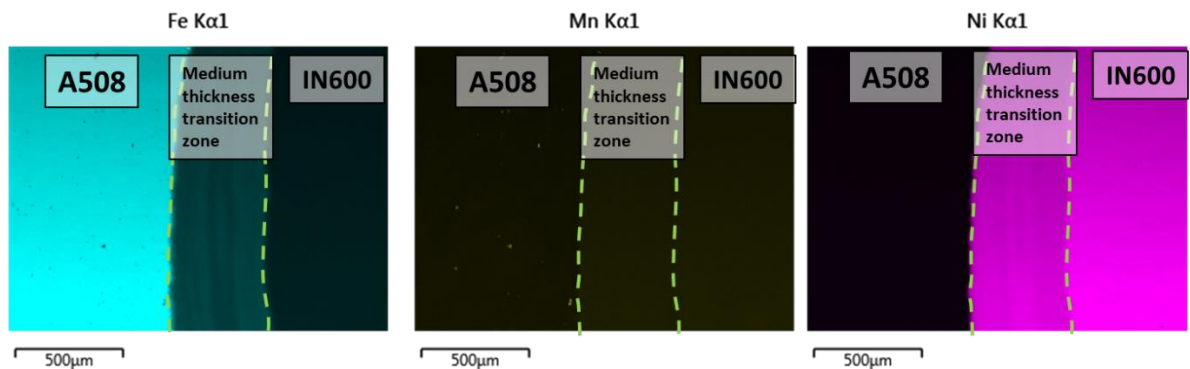


Figure 5.27: EDX mapping of Fe, Mn, and Ni in the vicinity of the medium thickness transition zone in the A508/IN600 joint.

5.6 Microstructure of thick transition zone region of A508/IN600

5.6.1 Cross-weld composition profile

The composition profile across the interface of the A508/IN600 joint in the thick transition zone region were measured, with 50 µm spacing between each measurement, using EDX analysis. This is presented in Figure 5.28, showing that there is clearly an EBW effect in the location of EDX measurement as the transition of composition between one alloy to another occur in a wide range (about 1650 µm wide). The concentration of Fe in fully A508 is 97 wt%, while it is around 9 wt% in fully IN600, which agrees with the nominal compositions of the alloys provided by the manufacturer. The same is true for Cr and Ni (Cr is 0.2 wt% in A508 and 16 wt% in IN600, while Ni is 0.6 wt% in A508 and 74 wt% in IN600). The change/transition in composition occurs from -25 µm to 1625 µm distance from the A508 interface, meaning that the start of the change in composition from the fully A508 into the transition which does not exactly match the interface line. At most of the transition zone, the composition of Fe,

Cr, and Ni are scatter between 35-41 wt%, 10-15 wt%, and, 46-53 wt% respectively from 75 μm to 1525 μm .

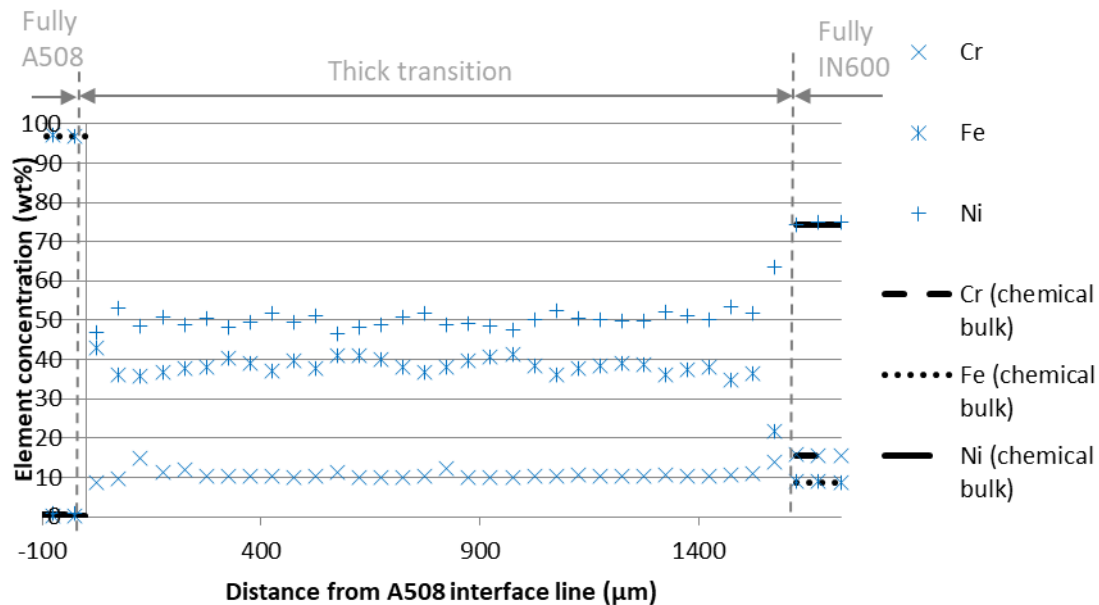


Figure 5.28: Composition profile of major elements (Cr, Fe, Ni) across thick transition zone region of A508/IN600 joint. The values for the chemical bulk compositions for each alloy originates from the chemical analysis provided by supplier of the bulk alloys.

The interface lines (A508-transition boundary and transition-IN600 boundary) seem to occur in transition compositions, i.e. do not appear to mark the boundary between fully bulk-material composition and the transition composition; hence a closer look at the composition in the vicinity of the interface lines is taken. An EDX profile with 5 μm spacing between each measurement is presented in Figure 5.29 in the vicinity of the interface lines (i.e. the boundary between fully bulk-material composition and the transition composition). Figure 5.29(a) shows that the composition starts to shift from being fully A508 to the transition composition, at 7.5 μm into the A508 side of the interface. Similarly, Figure 5.29(b) shows that the composition starts to shift from being fully IN600 to the transition composition, at 10 μm into the IN600 side of the interface. This confirms the fact that the interface lines do not coincide with start of the shift in composition from the bulk values.

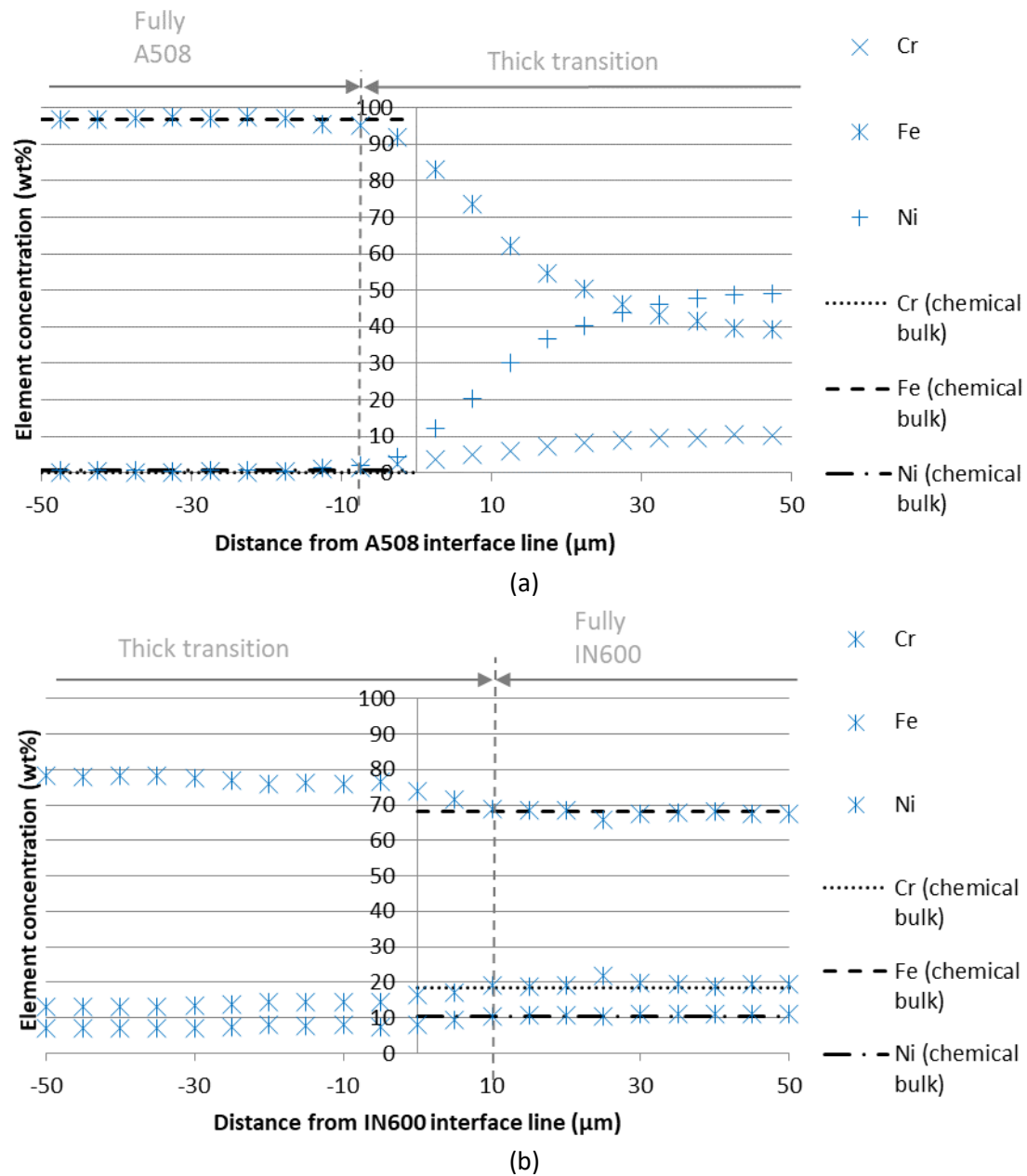


Figure 5.29: EDX profile in the vicinity of the interface lines between (a) A508-thick transition and (b) thick transition-IN600.

5.6.2 Microstructure of A508 side of interface

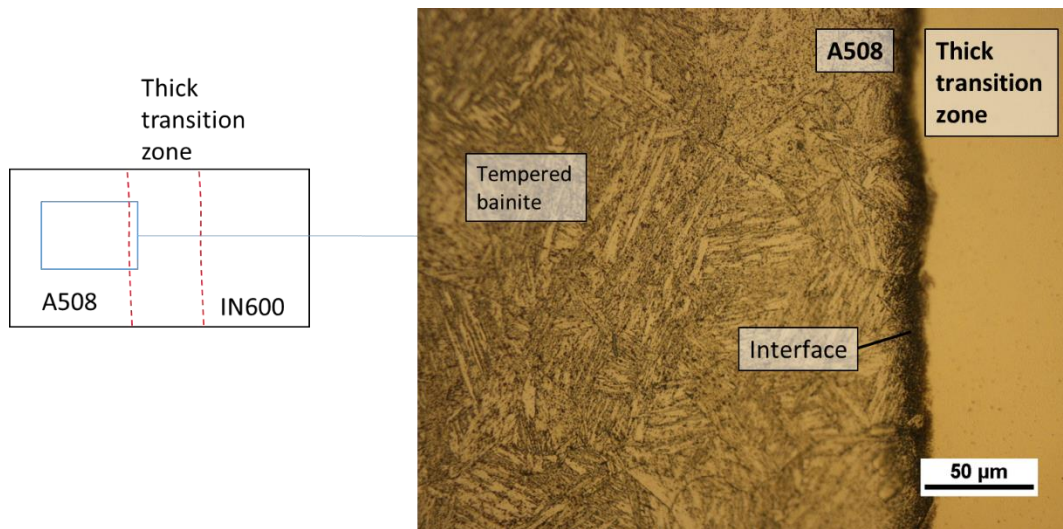


Figure 5.30: A508 side of the interface in the thick transition zone region of A508/IN600 joint.

The location of examination for microstructure of the A508 in the vicinity of the interface is displayed in Figure 5.30. Similar to the thin and medium thickness transition zone regions, there appears to be little microstructural variation in A508 as a function of distance from the interface at the A508 side of the interface. There is also a rough interface with plenty of precipitates in the vicinity that may indicate a martensitic band, as can be seen in Figure 5.31.

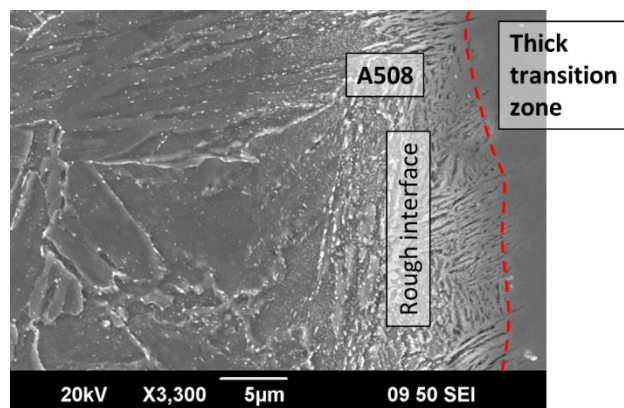


Figure 5.31: The interface A508 steel side of A508/IN600 joint at the thick transition zone region.

5.6.3 Microstructure of thick transition zone and the IN600 side of the interface

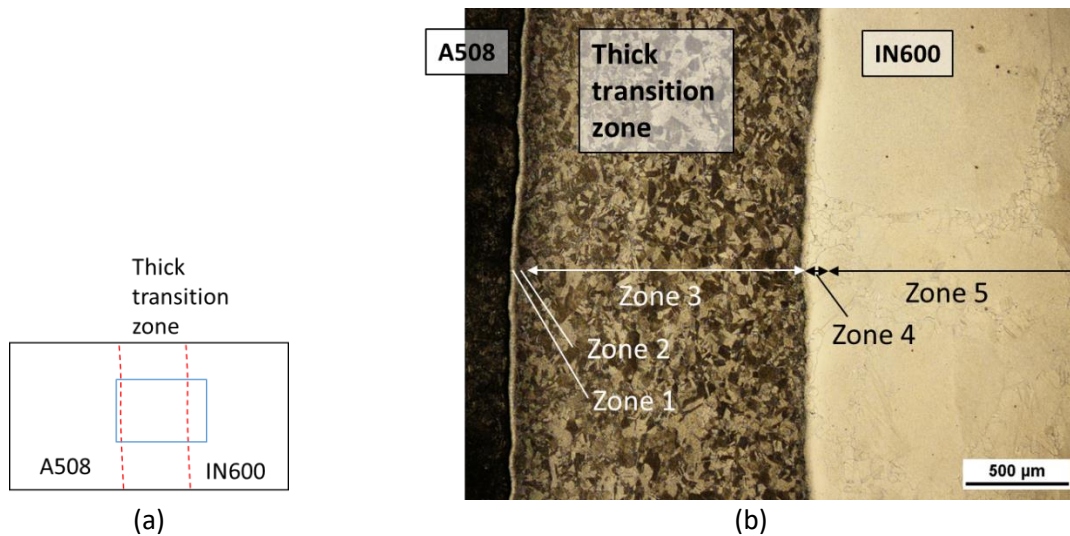


Figure 5.32: (a) Location of thick transition zone. (b) Overview of etched thick transition zone and IN600 side of the interface of A508/IN600 joint in the thick transition zone region. The thick transition zone consists of Zones 1-3, and IN600 side consists of Zones 4 and 5.

Figure 5.32 shows the location and an overview of several distinct microstructural zones in the thick transition zone and IN600 side of the interface. The thick transition zone, which is about 1420-1450 μm wide, is divided into Zones 1-3, and IN600 side is divided into Zones 4 and 5. Figure 5.33 shows that Zone 1, which is about 20 μm wide and located next to the interface line, is devoid of precipitates. Zone 2, which is about 60 μm wide, is very precipitate dense. Zone 3 is about 1320-1350 μm wide, containing slightly less precipitates than Zone 2, and the amount of precipitation varies from grain to grain. Zone 4, which is 60-70 μm wide, has precipitates mostly in the grain boundaries. Finally, Zone 5 is the bulk IN600. While Zone 5 still has the grain and twin boundaries, and both intergranular and intragranular precipitates, the size of the grains vary greatly, as can be seen in Figure 5.34. The bulk IN600 in this region are made of large grains surrounded by small grains in between.

Higher resolution images of the zones using SEM are presented in Figure 5.35. Zone 1 is shown to have no precipitates, even at the grain boundaries. The precipitates in Zone 2 are both intergranular and intragranular. The intragranular precipitates are densely packed and have different morphologies, with both needle and spheroid shapes. Zone 3 also has these precipitates, but they are more spaced apart, and precipitate densities are different in different grains. Zone 4 mostly intergranular

precipitates, though intragranular precipitates are also present. The microstructure of Zone 5 is consistent with the bulk IN600 microstructure.

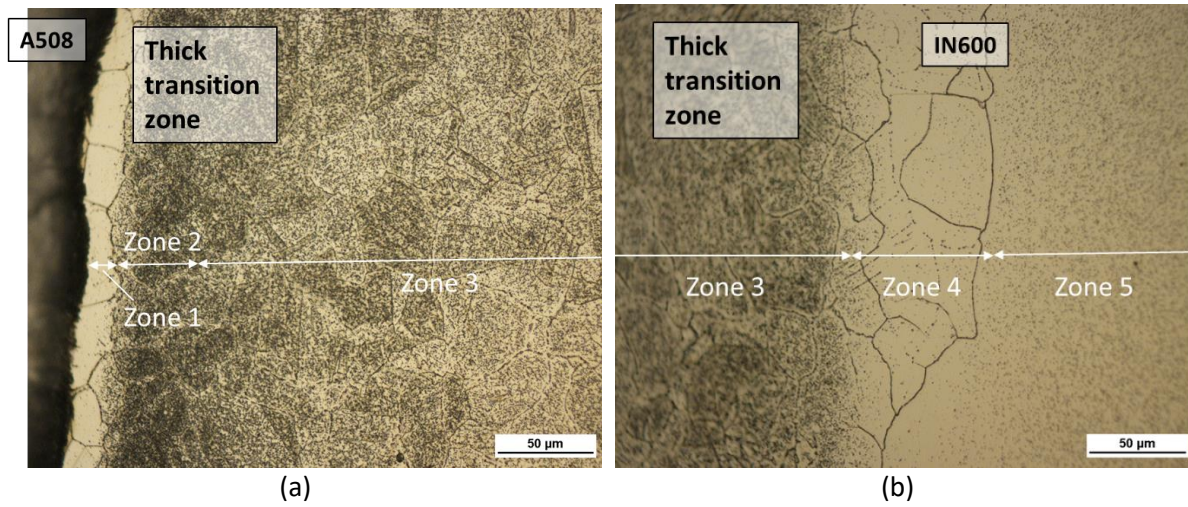


Figure 5.33: (a) Zones 1-3 in the thick transition zone and (b) Zones 4 and 5 in IN600, in the A508/IN600 joint.

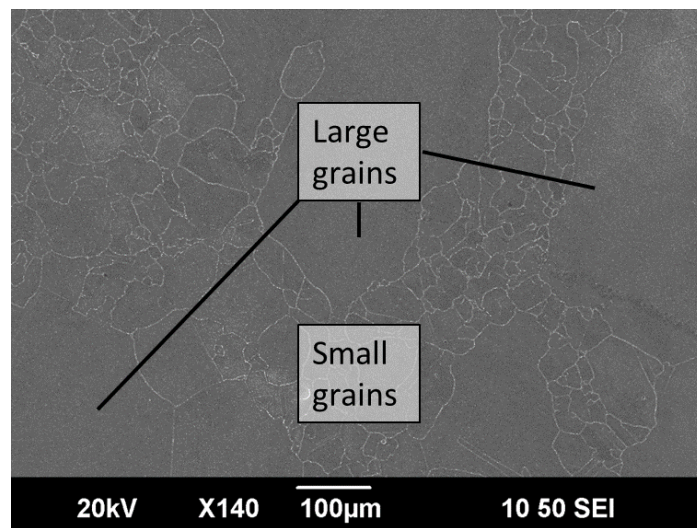


Figure 5.34: Scanning electron micrograph of the IN600 bulk in the thick transition zone region of A508/IN600 joint.

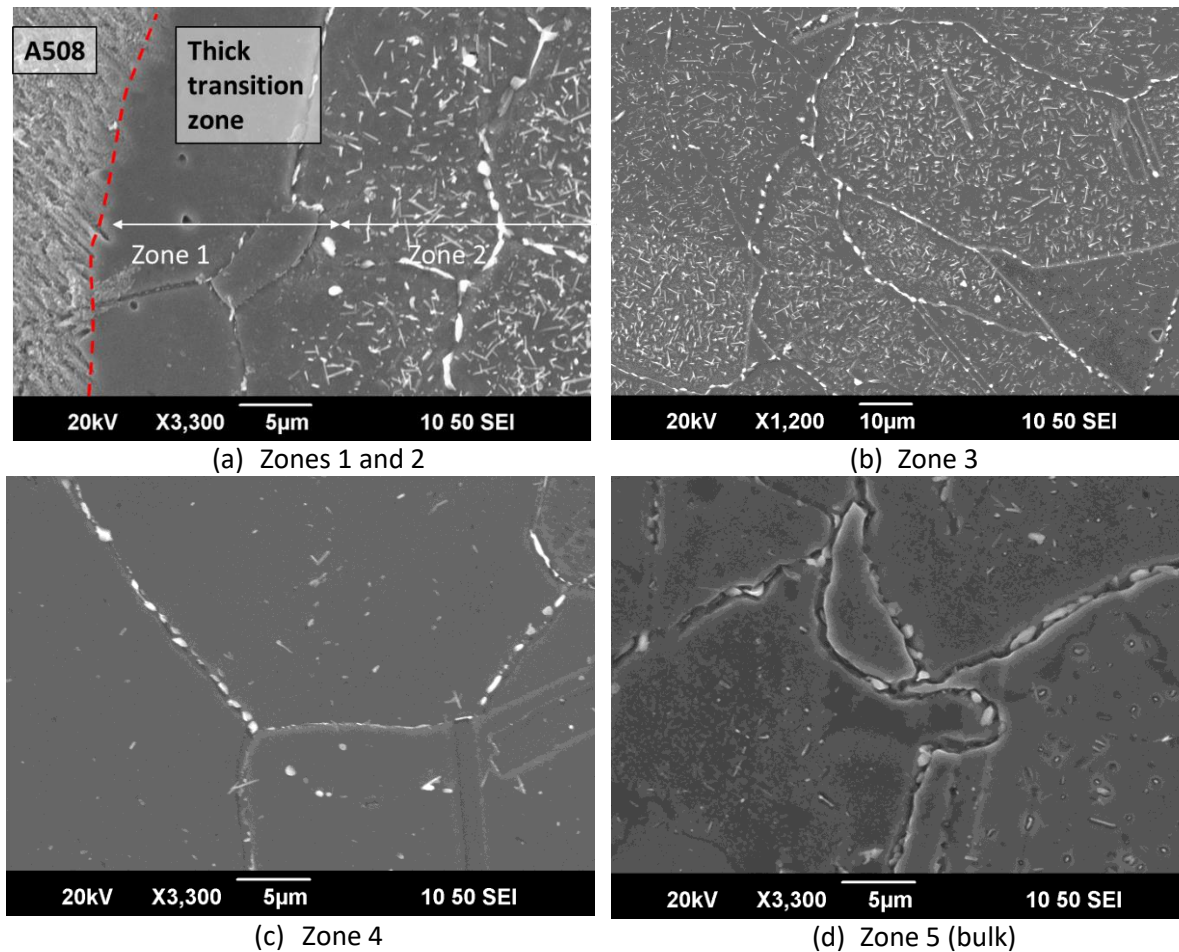


Figure 5.35: Scanning electron micrographs of the zones in the IN600 side of the interface of A508/IN600 joint in the thick transition zone region. (a) Zones 1 & 2, (b) Zone 3, (c) Zone 4 and (d) Zone 5.

5.7 Thermodynamic simulations of phase equilibria

5.7.1 IN600

The phase equilibrium for IN600 was calculated using ThermoCalc and the TCNI8 database. The composition of IN600 used for ThermoCalc calculations is presented in Table 5.2. γ' (represented by FCC_L12 phase in ThermoCalc), an intermetallic which is considered to be the 'ordered' version of γ (represented by FCC_A1 phase in ThermoCalc), is suspended because it is not found in IN600 in practice, though its existence is possible under certain circumstances [101]. The equilibrium phase fractions as a function of temperature (from 500°C to 1200°C) are plotted in Figure 5.36.

Table 5.2: Input composition of IN600 for ThermoCalc calculations.

Element	Amount (wt%)
C	0.07
Cr	15.6
Fe	8.74
Mn	0.53
Ni	75.06 (Bal)

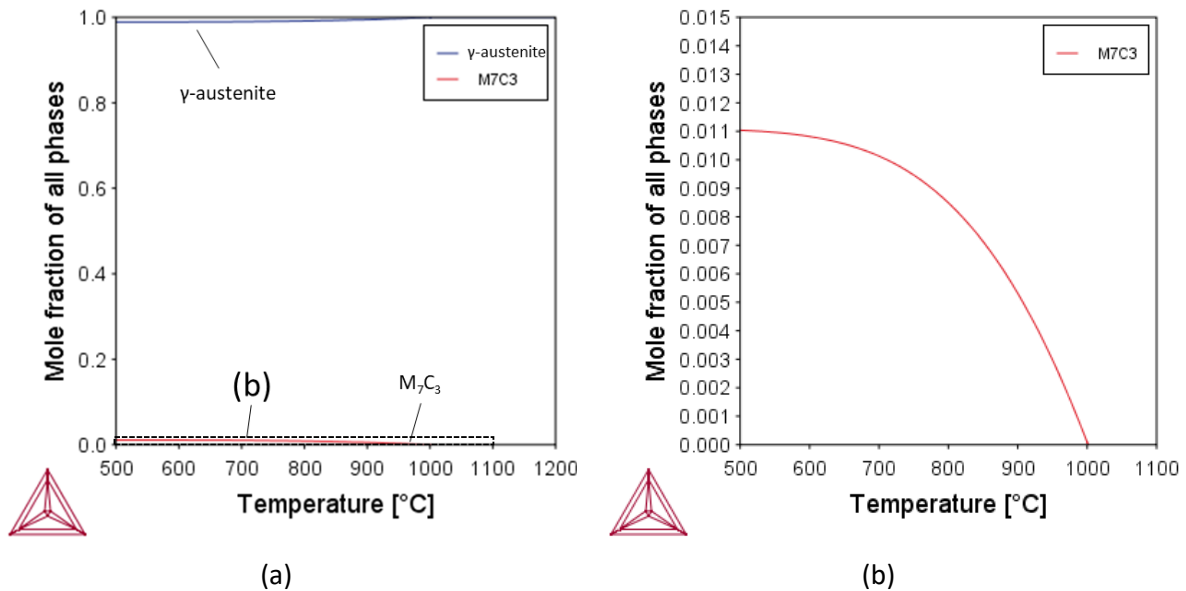


Figure 5.36: Phase fractions as a function of temperature for IN600 alloy, as calculated by ThermoCalc using the TCNI8 database, showing (a) all phases, and (b) secondary phase. The dashed box in (a) indicates the portion of the plot represented in (b).

The matrix phase is identified as γ -austenite (FCC) in the whole temperature range. The precipitates are identified as only M_7C_3 , which form at high temperatures below 1000°C. In practice, in many cases a fraction of M_7C_3 transforms to $M_{23}C_6$ in the 600°C -700°C temperature range [102]. So despite the prediction here of the formation of only M_7C_3 , the IN600 alloy likely has $M_{23}C_6$ as well due to the 680°C tempering.

5.7.2 Medium thickness transition zone

From Figure 5.19, the composition across the medium thickness transition zone varies. However, a large portion of the transition zone has mostly constant—albeit scattering in a narrow range—compositions of the main alloying elements (Fe, Ni, and Cr) and to a lesser extent other major elements (Mn and Mo). The composition of the medium thickness transition zone used for ThermoCalc calculations is presented in Table 5.3, taken from the mean composition from 62.5 μm to 312.5 μm

range in Figure 5.19 for each element. The carbon content is assumed to be halfway between that of A508 and that of IN600, i.e. 0.16 wt%, to reflect the mixing that occurs in the transition zone. The TCNI8 database is used for the calculation, with ordered γ' phase (represented by FCC_L12 phase in ThermoCalc) suspended. The equilibrium phase fractions as a function of temperature (from 500°C to 1200°C) are plotted in Figure 5.37. The matrix phase is identified as γ -austenite (FCC) in the whole temperature range. There are two types precipitates that form in this temperature range: $M_{23}C_6$ carbide and M_7C_3 carbide. The $M_{23}C_6$ carbide forms below 710°C, whereas the M_7C_3 carbide forms between 560°C and 1200°C.

Table 5.3: Input composition of the medium thickness transition zone in in the A508/IN600 joint for ThermoCalc calculations.

Element	Amount (wt%)
C	0.16
Cr	14.30
Mn	0.69
Fe	20.39
Ni	64.49 (Bal)
Mo	0.14

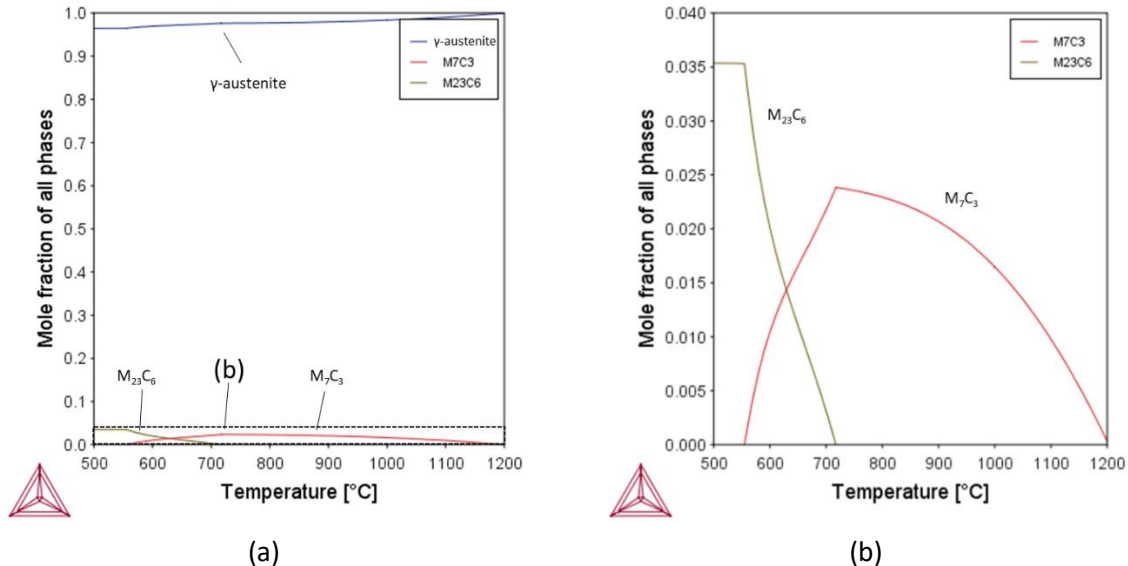


Figure 5.37: Phase fractions as a function of temperature for medium thickness transition zone in the A508/IN600 joint, as calculated by ThermoCalc using the TCNI8 database, showing (a) all phases, and (b) secondary phase. The dashed box in (a) indicates the portion of the plot represented in (b).

5.7.3 Thick transition zone

The composition of the thick transition zone used for ThermoCalc calculations is presented in Table 5.4, taken from the mean composition from 75 μm to 1525 μm range in Figure 5.28 for each element. The carbon content is again assumed to be halfway between that of A508 and that of IN600, i.e. 0.16 wt%. The TCNI8 database is used for the calculation, with ordered γ' phase (represented by FCC_L12 phase in ThermoCalc) suspended. The equilibrium phase fractions as a function of temperature (from 500°C to 1200°C) are plotted in Figure 5.38. The matrix phase is identified as γ -austenite (FCC) in the whole temperature range. There are two types precipitates that form in this temperature range: M_{23}C_6 carbide and M_7C_3 carbide. The M_{23}C_6 carbide forms below 800°C, whereas the M_7C_3 carbide forms between 560°C and 1020°C.

Table 5.4: Input composition of the thick transition zone in the A508/IN600 joint for ThermoCalc calculations.

Element	Amount (wt%)
C	0.16
Cr	10.61
Mn	0.85
Fe	38.20
Ni	50.10 (Bal)
Mo	0.24

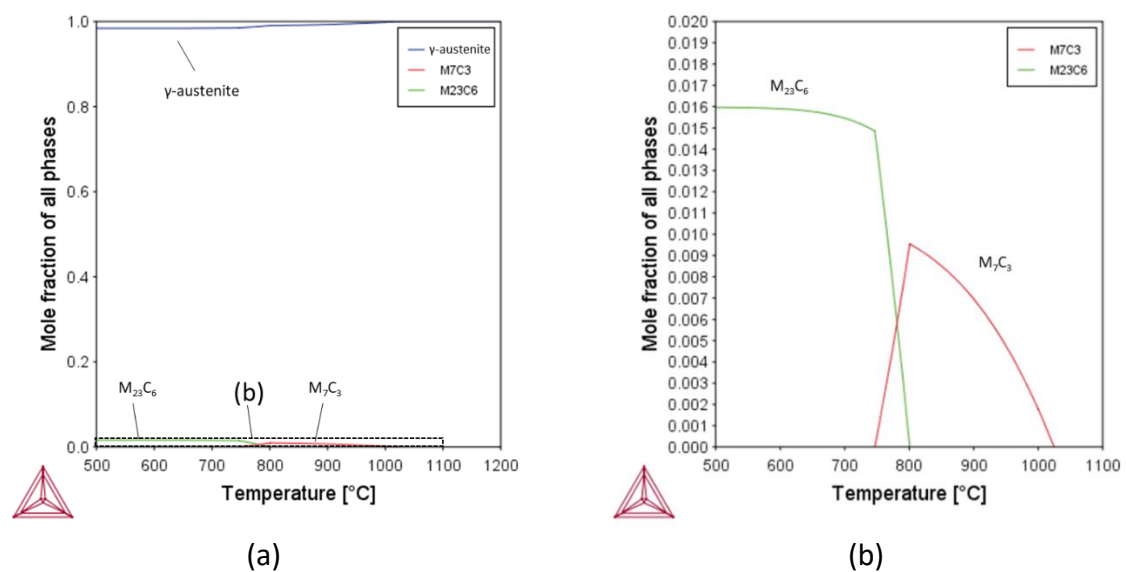


Figure 5.38: Phase fractions as a function of temperature for thick transition zone in the A508/IN600 joint, as calculated by ThermoCalc using the TCNI8 database, showing (a) all phases, and (b) secondary phase.

5.8 Tensile testing

As seen in Figure 5.1, the transition zone region varies in thickness across the joints. In order to assess whether this variation results in a difference in mechanical behaviour for different thicknesses, two tensile specimens were extracted from both the thick and thin transition zone regions of the joint, perpendicular to the interface as shown in Figure 5.39. In addition, tensile specimens of bulk materials from the joints were also obtained in order to compare the mechanical performance of the cross-weld properties with those of the bulk material. As material was limited, the bulk tensile specimens were cut parallel to the interface.

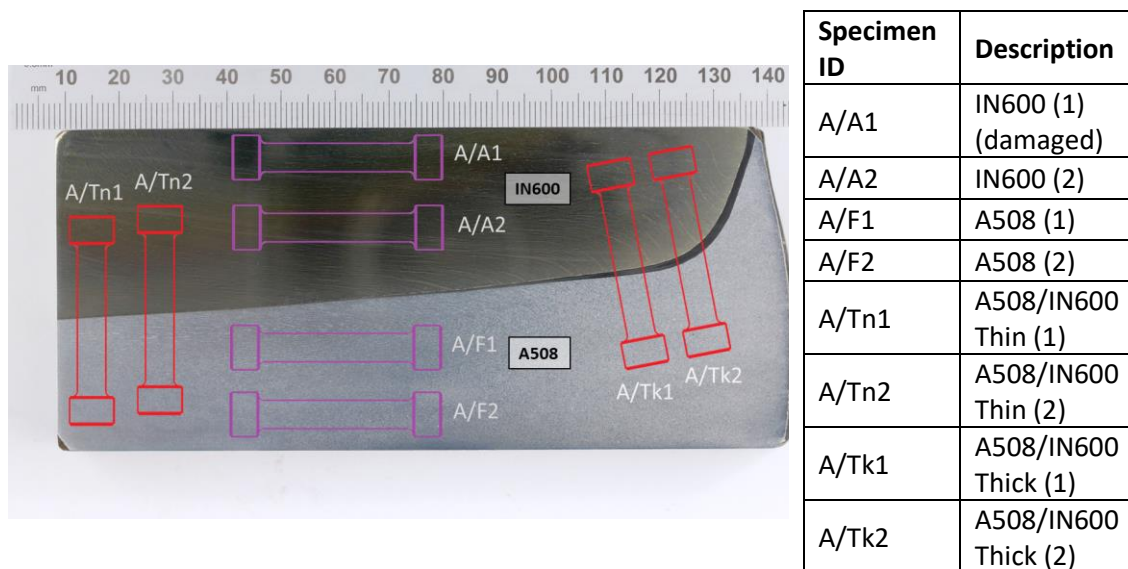


Figure 5.39: Origin of tensile test specimens of A508/IN600 joint. Two specimens each were obtained from the thinner transition zone region (A/Tn1 and A/Tn2) and thicker transition zone regions (A/Tk1 and A/Tk2) and bulk alloys in the lateral direction (A/A1- A/F2). Note that one of the IN600 bulk specimen (specimen A/A1) was damaged and thus could not be considered here.

5.8.1 Tensile stress-strain curves and material properties

The stress-strain curves from the tensile tests of specimens from A508/IN600 joints are presented in Figure 5.40. Tensile properties extracted from the curves are presented in Table 5.5. As with the other joint, the A508 specimens (A/F1 and A/F2) have the highest UTS and yield strength among all of the specimens, though are not identical to each other. The difference in the A508 curves is perhaps due to the flow that can be seen in Figure 5.1. The specimen closer to the interface (i.e. specimen A/F1) came from the part of A508 that experienced more flow and deformation compared to the other

specimen (A/F2). The UTS, yield stress, and elongation of A/F1 is somewhat lower than that of A/F2, thus indicating that the increased flow seems to have an effect of decreasing the UTS, yield stress, and ductility. The IN600 sample exhibited greatest ductility. In terms of the cross-weld specimens, the curve and tensile properties for two thin transition zone region specimens (A/Tn1 and A/Tn2) are nearly identical. On the other hand, the thick-transition zone region specimens (A/Tk1 and A/Tk2) have completely different curves. While the two have similar UTS, A/Tk1 has lower yield stress and but higher elongation. Nevertheless, overall the cross-weld specimens have UTS, yield stress, and elongation intermediate between that of A508 and IN600. This can be considered as a success for the fabrication process, at least for this particular joint, as the tensile properties across the weld are comparably strong and ductile (or more so) compared to the bulk alloys.

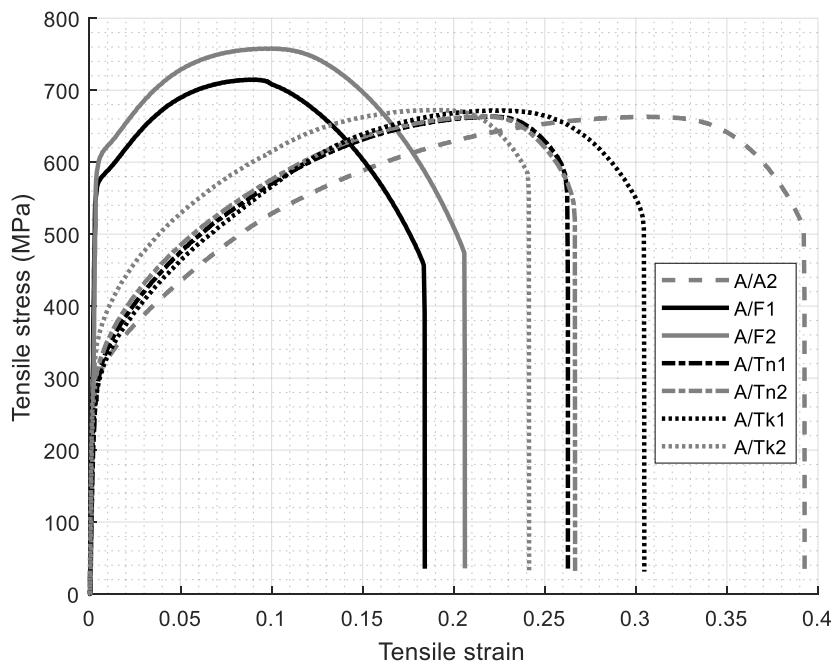


Figure 5.40: Stress-strain curves of tensile specimens from the A508/IN600 joint.

Table 5.5: Measured tensile properties of the A508/IN600 joint according to the stress-strain curves.

Type	Specimen	Test results		
		0.2% Proof (MPa)	UTS (MPa)	EL (%)
Bulk alloy	A/A2	291	663	39
	A/F1	572	715	18
	A/F2	606	758	21
Cross-weld	A/Tn1	297	663	26
	A/Tn2	304	664	27
	A/Tk1	277	672	30
	A/Tk2	340	673	24

5.8.2 Fractography

The fracture locations of the tensile test specimens for the cross-weld specimens, presented in Figure 5.41, allow an initial evaluation of whether the interface (i.e. the fusion region) between the two alloys was at least as strong as the bulk material for each of the joints.

Figure 5.41(a) shows that for the thin transition zone region of the A508/IN600 joint, the fracture ran through the A508 bulk, close to the interface with the IN600 alloy, despite A508 being both harder throughout (see Figure 5.2(a)) and slightly stronger in terms of UTS. This may have been caused by the constraint effect near the interface between the two alloys. While the IN600 alloy initially yielded first, due to its low proof stress, it is far more ductile; as the stressed was increased, the ductility of IN600 absorbed the additional stress, until the stress reached the proof stress of the A508 alloy, which then began to deform plastically. The plastic deformation of A508 near the interface was affected by reduction of area in the nearby IN600 due to the constraint effect. When the stress reached a certain value, the failure location was largely stochastic, i.e. could have occurred anywhere in IN600 or in nearby bulk A508. The result was the same for both specimens A/Tn1 and A/Tn2, although only A/Tn1 is presented here. This indicates that the fusion is sufficiently strong, i.e. stronger than either bulk material and that the strain accumulated in the A508 side of the interface and caused the fracture to occur there.

The fracture for the thick transition zone region of the A508/IN600 joint, the two tests yielded somewhat different results. This might be partly due to the fact that the pair of tensile specimens (A/Tk1 and A/Tk2) were not exactly identical, as one specimen (i.e. A/Tk2) may have had slightly thicker transition zone region than the other, as can be seen in Figure 5.39. Figure 5.41(b) shows that specimen A/Tk1 failed deep in the IN600 bulk, with necking within the IN600 portion extended all the way to the boundary between the thick transition zone and the IN600 bulk. The fracture in specimen A/Tk2, shown in Figure 5.41(c), however, cuts through the boundary between the transition zone region and the IN600 bulk, with the cracking also detoured into the IN600 bulk. The different fracture behaviours as seen in Figure 5.41(b) and (c) were caused by the weakening in the IN600 portion in A/Tk1, which lowered the yield stress, even though the UTS are similar with that of A/Tk1; but the long necking ensured high elongation at failure. This could indicate that the boundary between the transition zone region and the IN600 bulk may have comparable strength to the IN600, in the part of the joint where the transition zone region is particularly thick.

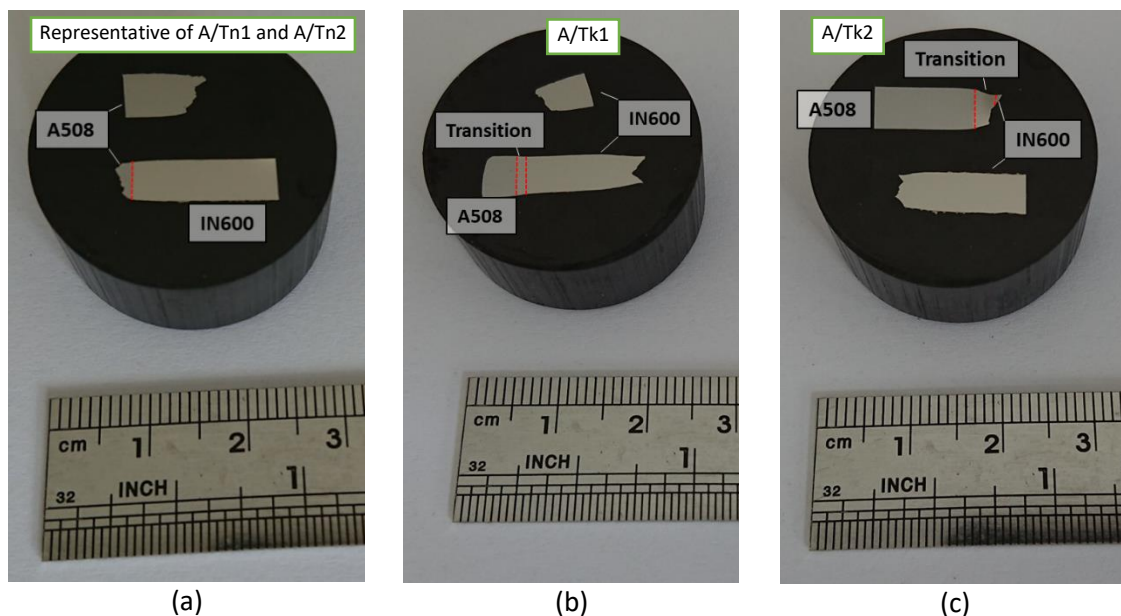
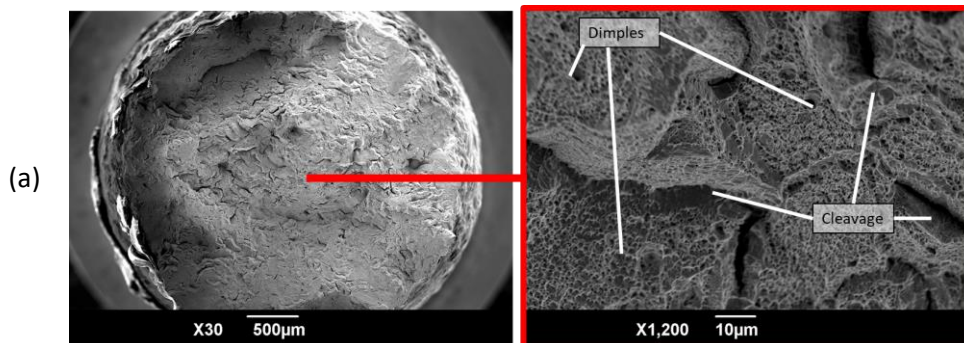


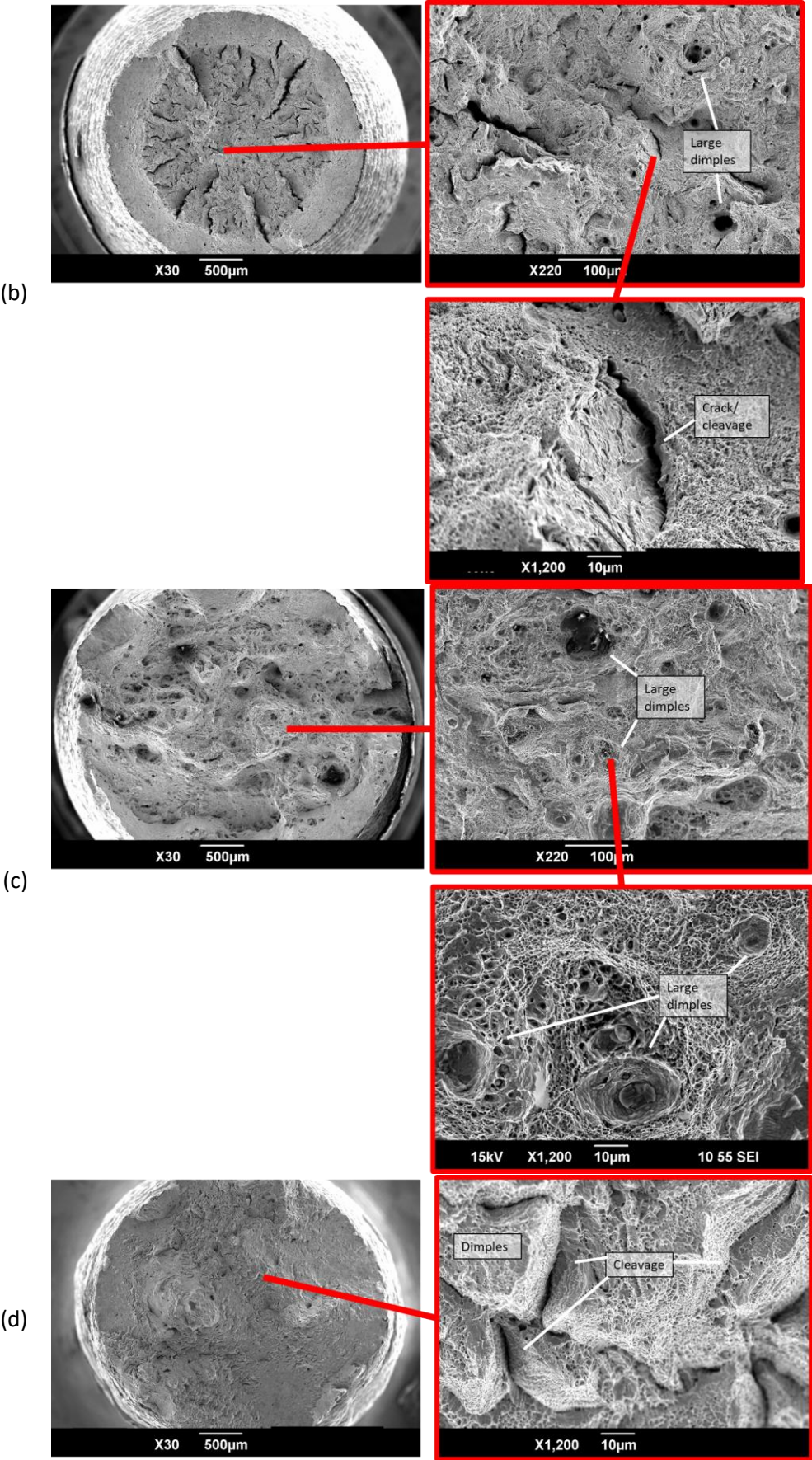
Figure 5.41: Fractured tensile testing specimens from (a) A/Tn1 Thin transition zone region of A508/IN600 joint (b) A/Tk1 Thick transition zone region of A508/IN600 joint (c) A/Tk2 Thick transition zone region of A508/IN600 joint

Fracture behaviours of the tensile tested specimens can be further illuminated by examining the fracture surface topography using scanning electron microscopy. The fracture surface of A/Tn1 (thin transition zone region of A508/IN600 joint) is shown in Figure 5.42(c). It can be seen that there are

mostly dimples, indicating ductile failure, though there are also some cracks/cleavage, and the large dimples are likely due to the presence of MnS inclusions. This most resembles the fracture surface of A/F1 A508 bulk (Figure 5.42(b)), which is consistent with the observation in Figure 5.41(a), i.e. the fracture occurs in A508 portion of specimen A/Tn1, so the fracture surface should resemble that of A508. Similarly, the fracture surface of A/Tk1 (one of the thick transition zone region specimen of A508/IN600 joint), shown in Figure 5.42(d), most resembles the fracture surface of A/F1 IN600 bulk (Figure 5.42(a)), because the fracture in A/Tk1 occurs in IN600 portion as shown in Figure 5.41(b). The fracture surface in IN600 indicates a combination of ductile and brittle fracture due to the presence of both dimples and intergranular cleavages.

The fracture surface of A/Tk2 (the other thick transition zone region specimen of A508/IN600 joint), shown in Figure 5.42(d), is more complex due to the fact that the fracture went through more than one zone. A portion of the fracture surface resembles the IN600 bulk (with the cleavage and dimples), whereas the other has only dimples. The fracture behaviour of this specimen may be summarized in Figure 5.43. Crack first appears at boundary between the IN600 bulk and the transition zone, then propagates along this boundary, and then detours from the boundary into the IN600 bulk.





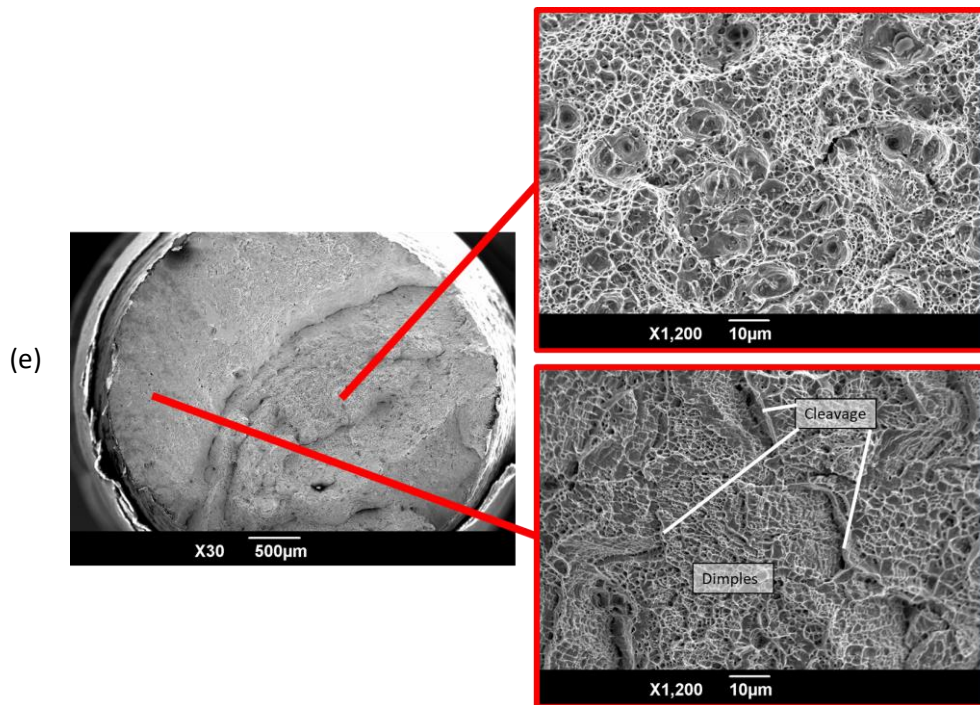


Figure 5.42: SEM (secondary electron mode) images of fracture surface topographies of the tensile specimens from A508/IN600 joint at (a) A/A2 IN600 bulk (b) A/F1 A508 bulk (c) A/Tn1 Thin transition zone region (d) A/Tk1 Thick transition zone region (e) A/Tk2 Thick transition zone region

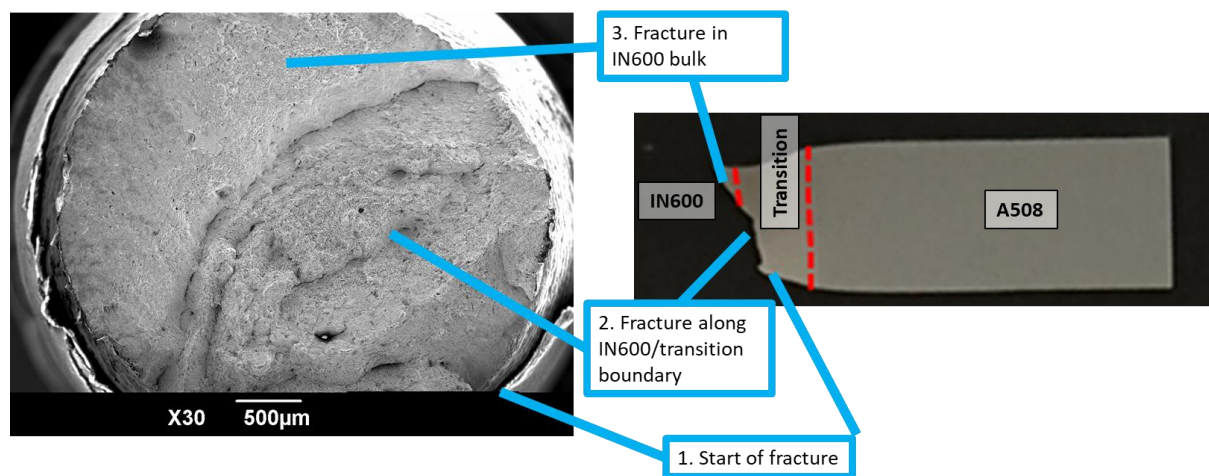


Figure 5.43: Fracture behaviour of A/Tk2 Thick transition zone region of the A508/IN600 joint

5.9 Discussion

5.9.1 Behaviour of A508 near the interface

The microstructure of the A508 side of the interface is summarised schematically in Figure 5.44. Because the solution treatment operation was carried out on the whole joint during the first part of post forge heat treatment (PFHT), the grain size in A508 are more or less uniform throughout. A508 side of the interface exhibit a typical tempered bainite microstructure. There is no noticeable PDZ,

which confirms the general observation that the decarburized layer in the ferritic side of the joint is far less severe when a ferritic steel (in this case the A508 steel) is joined to a nickel-based metal, compared to an austenitic stainless steel metal [53, 55, 56], when heated to a high temperature (in this case, the PFHT). This means that there was relatively little carbon migration from the A508 into the transition zone with the IN600. The martensitic interfacial band that formed adjacent to the interface correlates with the start of the change in composition on the A508 side of the interface, as can be seen in Figure 5.5, Figure 5.20(a), and Figure 5.29(a). Hence, the formation of the martensitic interfacial band can be attributed to the gradient in composition across the interface.

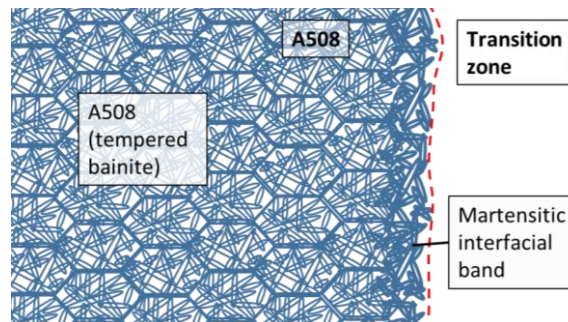


Figure 5.44: Schematic illustration of the microstructure of the A508 side of the interface in the A508/IN600 joint.

5.9.2 Behaviour of the transition zone and IN600

Observations made in the thin transition zone region suggest that there is a transition zone of at least 200 μm wide even towards the centre, i.e. far away from the outer edge, of the joint illustrated in Figure 5.1. Since the EBW fusion zone is not shown to penetrate that far towards the centre, it is possible that the thin transition zone (as well as possibly the medium thickness transition zone) was created as a result of interdiffusion during the forging, and to a lesser extent the PFHT, operations.

Despite having differing compositions, microstructurally the transition zone and the IN600 are quite similar. The grain morphology, grain sizes, and crystal structure of both are similar (both have FCC crystal structure), according to the EBSD image in Figure 5.26 and confirmed by the ThermoCalc calculations. There is no sharp boundary between IN600 and the transition zone, even in the thick transition zone region. The main difference between the IN600 bulk and the transition zone (and its subzones) is the amount of precipitates that appear, shown schematically in Figure 5.45. From

ThermoCalc calculation results presented in Figure 5.36, Figure 5.37, and Figure 5.38, the precipitates in the transition zone as well as IN600 are likely to be composed of a combination of $M_{23}C_6$ and M_7C_3 carbides. The precipitate density variation seems to be only a function of distance from the A508 interface, and is not delineated by grain boundaries. Adjacent to the A508 interface, there is a narrow PDZ. About 10 μm from the interface, the precipitates get particularly dense, before tapering to a lower density in deeper into the transition zone. On the IN600 side adjacent to the boundary between the transition zone and IN600, the precipitate formation had decreased to so low that it is mostly present in on the grain boundaries. The precipitates begin to gradually appear again in the IN600 bulk.

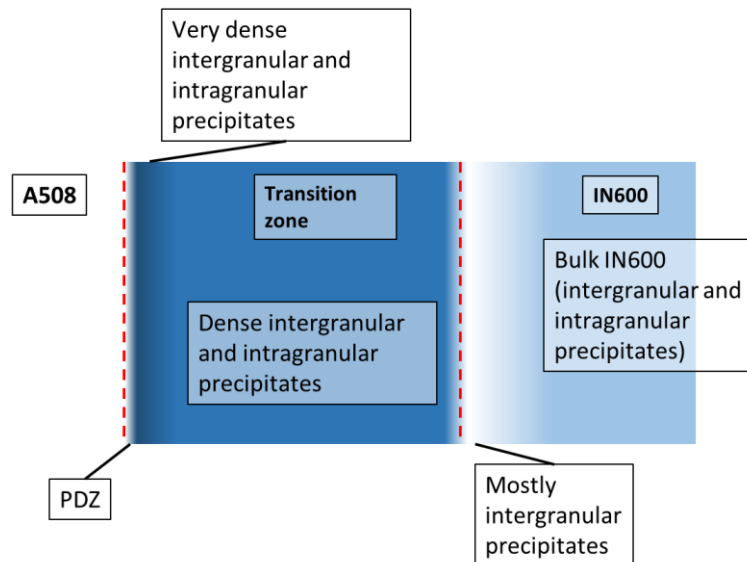


Figure 5.45: Schematic illustration of the microstructure of the transition zone and the IN600 side in the A508/IN600 joint, showing variations in precipitate density.

The presence of PDZ shows that there may be some degree of carbon migration into the very dense precipitate zone. However, the formation of higher density of precipitates in the transition zone may have also been caused by decreased in carbon solubility in the matrix phase of the transition zone. A ThermoCalc calculation of the phases in the thick transition zone, medium thickness transition zone, and IN600, at the tempering temperature of 680°C, is shown in Table 5.6. Indeed even when the carbon content of the transition zone regions is assumed to be as low as that of IN600 (0.07 wt%), the mole and volume fractions of the carbides in the transition zone regions are significantly greater than that of the IN600. This indicates that during PFHT, the greater amount of carbides would form in the

transition zone compared to IN600, even if they contain the same amount of carbon. The high density of precipitates in the transition zone also did not increase the hardness of the transition zone above that of the IN600 bulk, as can be seen in the microhardness profiles in Figure 5.2. This suggests that the strengthening effect of extra precipitates is compensated by the decrease in solid solution strengthening of carbon in matrix.

Table 5.6: The mole and volume fractions of the phases in the thick transition zone, medium thickness transition zone, and IN600, at the tempering temperature of 680°C, as calculated by ThermoCalc using TCNI8 database. C is assumed to be 0.07 wt% in the transition zones, same as IN600. The rows of interest, showing the total carbides in each location, are coloured in yellow.

Location	Phases present	Mole fraction	Volume fraction
Thick transition zone	γ -austenite	0.984	0.986
	$M_{23}C_6$	0.016	0.014
Medium thickness transition zone	γ -austenite	0.986	0.987
	Carbides total	0.014	0.013
	$M_{23}C_6$	0.011	0.010
	M_7C_3	0.003	0.003
IN600	γ -austenite	0.990	0.991
	M_7C_3	0.010	0.009

5.9.3 Mechanical performance and material properties

It is unclear why the failure would occur on the A508 side of the interface in the thin transition zone cross-weld tensile specimens A/Tn1 and A/Tn2. There is no PDZ in the A508 side near the interface to provide a weak zone where strain could accumulate. There is no grain size variation within A508 side to provide weakness, as the varying grain sizes in HAZ of fusion welding would. Both the microhardness profile (Figure 5.2(a)) and the σ_y profiles from nanoindentation testing (Figure 5.16, Figure 5.17, and Figure 5.18) carried out across thin transition zone region indicates that the weakest point should be the IN600 bulk. In short, there is no known factor that would cause the fracture to have occurred on the A508 side instead of elsewhere.

The fracture in the thick transition zone cross-weld tensile specimens A/Tk1 and A/Tk2 occur in the IN600 bulk or near the transition-IN600 boundary, respectively. While the actual fracture locations are

different, in both specimens there are extensive necking in the IN600 bulk. The fact that there are large grains in the IN600 bulk in the thick transition zone region (i.e. near the outer edge of the joint), as observed in Figure 5.34, may have caused necking and fracture to preferentially occur there. There is also necking in the transition zone in both specimens, so the high density of precipitates in the transition zone provided limited extra strength to the matrix there, at least in room temperature where the tensile testing was held.

Nonetheless, despite the differing fracture locations, the cross-weld specimens have UTS, yield stress, and elongation intermediate between that of A508 and IN600, showing that the tensile properties across the weld are comparably strong and ductile (or more so) compared to the bulk alloys.

5.10 Conclusions

The following observations can be made about the TransForge A508/IN600 joint:

- The interfacial regions varies in different parts of the joint; the centre of the joint contain the thin transition zone region, whereas the transition zone gets thicker nearer to the edge of the joints.
- Overall the cross-weld specimens (from both the thin and the thick transition zone region) have UTS, yield stress, and elongation intermediate between that of A508 and IN600. The fracture occurred in A508 side of the interface in the thin transition zone cross-weld tensile specimens, while for the thick transition zone cross-weld tensile specimens the fracture occurred in the IN600 bulk or near the transition-IN600 boundary. Despite the differing fracture locations, tensile properties across the weld are comparably strong and ductile (or more so) compared to the bulk alloys, showing the viability of the fabrication process.
- There is no PDZ in the A508 side of the interface, consistent with other observations that the decarburized layer in the ferritic side of the joint is far less severe when it is joined to a nickel-based metal. At the interface, a martensite band formed on the A508 side due to composition gradient.

- Despite having differing compositions, microstructurally the transition zone and the IN600 are quite similar. The grain morphology, grain sizes, and crystal structure of both are similar (both have FCC crystal structure), and both have similar hardness values. The main difference between the IN600 bulk and the transition zone (and its subzones) is the amount of precipitates ($M_{23}C_6$ and M_7C_3 carbides) that appear; this may have been caused by carbon migration during EBW, forging and PFHT, but the elevated amount of carbides that appear in the transition zone may also have been caused by the decreased carbon solubility in the γ -austenite matrix.

6 MICROSTRUCTURAL EVOLUTION OF DISSIMILAR METAL WELD BETWEEN P92 AND IN82/IN182 UNDER CREEP AND THERMAL AGEING

6.1 Introduction

In this chapter, the microstructural evolution of DMW between P92 and IN82/IN182 under thermal ageing and creep (all at 630°C), is characterised. Even though the weld metal consist of both IN82 (root weld) and IN182 (multipass weld), P92 is assumed to interface with IN182 weld only, not with IN82. Characterisation of non-crept samples are presented first to provide understanding of the microstructural behaviour of the joint at high temperature. The results for the crept samples are then discussed to explain weld behaviour under mechanical loading at high temperature. The focus is largely on P92 side of the interface as this is the most likely location of failure in laboratory creep tests as well as in service.

6.2 Characterisation of post weld heat treated and thermally aged welds

The samples in this section are labelled as the following:

- Sample **T** – post weld heat treated (PWHT) sample, which underwent tempering treatment at 770°C for 8 hours after weld fabrication, as received from Doosan Babcock.
- Samples **HT-109h**, **HT-500h**, **HT-1000h**, **HT-1453h**, **HT-3000h**, and **HT-5862h** – samples that underwent thermal ageing at 630°C for 109, 500, 1000, 1453, 3000, and 5863 hours, respectively. Samples HT-109h, HT-1453h, and HT-5862h were created to provide thermally aged samples with the same high temperature exposure durations as the failed creep tests, while samples HT-500h, HT-1000h, and HT-3000h were created to provide additional data for thermal ageing durations in between.

6.2.1 Microhardness profile across interface line

Hardness values were taken across the interface of the P92/IN182 weld in samples T, HT-109h, HT-1453h, and HT-5862h with 100 μm spacing between indents; these hardness profiles are presented in Figure 6.1. For sample T, from the left, the bulk P92 hardness is around 217 HV_{200} , until about 3mm from the interface. The hardness then gradually decreases until a minimum of around 195 HV_{200} at about 2mm from the interface, corresponding to the intercritical HAZ (ICHAZ) between the bulk and fine-grained HAZ (FGHAZ). The microhardness then increases to a peak around 245 HV_{200} at 0.2 mm, slightly decreasing at the interface itself (about 240 HV_{200}). The hardness then decreases to around 205 HV_{200} in the IN182 side, around 0.4 mm from the interface.

For sample HT-109h, the average hardness of bulk P92 is marginally higher at about 220 HV_{200} . The hardness in the left side of HAZ (from -3mm to -1.5 mm) is also slightly higher, with the minimum being 204 HV_{200} . This indicates the hardening of ICHAZ. At the right side of the HAZ (from -1.5 mm to the weld line), however, the hardness decreased from sample T, and the hardness peak is at the interface itself, where the hardness is similar to that of sample T. This indicates the softening of FGHAZ and CGHAZ. The behaviour in the IN182 side is mostly similar to that of sample T.

For sample HT-1453h, the average hardness of bulk P92 is similar to that of HT-109h. The hardness in the left side of HAZ (from -3mm to -1 mm) is also similar. At the right side of the HAZ (from -1 mm to the weld line), however, the hardness increased from sample HT-109h, and the hardness peak is now at 250 HV_{200} at about 0.2 mm from the interface, though the hardness decreases at the interface to a value similar to that of sample HT-109h. This indicates the hardening of FGHAZ and CGHAZ. The high hardness values in the IN182 side due to the presence of two overlapping weld beads at the location where the hardness profile is taken.

For sample HT-5862h, the average hardness of bulk P92 and the hardness in the left side of the HAZ (from -3mm to -1 mm) are similar to that of HT-1453h. At the right side of the HAZ (from -1 mm to the weld line), however, the hardness decreased from sample HT-1453h. The peak was now 240 HV_{200} at

about 0.2 mm from the interface. The microhardness at the interface also decreased, to about 225 HV₂₀₀. On the IN182 side of the weld line, the profile is mostly similar to that of other samples except HT-1453h.

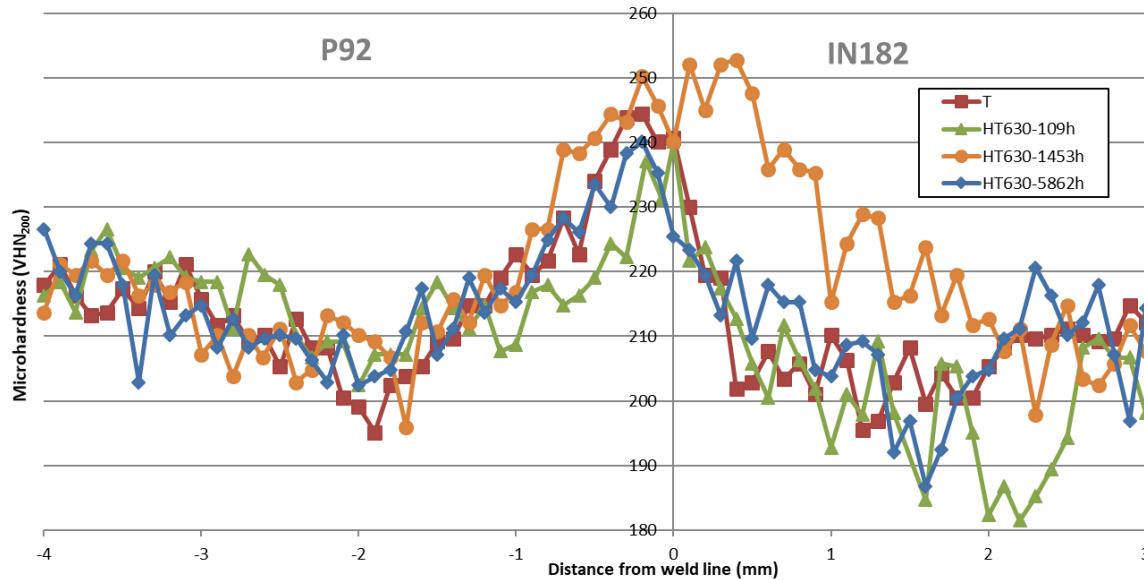


Figure 6.1: Microhardness profile across the P92/IN182 weld interface in samples T, HT-109h, HT-1453h, and HT-5862h.

6.2.2 Composition profile across interface line

The composition profile across the weld line of the P92/IN182 joint in sample T and HT-109h was measured using EDX analysis, with 5 μm spacing between each measurement. This is presented in Figure 6.2, which shows that the composition of major elements in the P92 side of the interface in sample T are close to the manufacturer specification of P92 alloy (88 wt% Fe, 9 wt % Cr, 1.5 wt% W, etc.). In the IN182 side of the weld line, there is a partially mixed zone (PMZ) where the composition transitions from the P92 alloy to the IN182 alloy, between the weld line and 10 μm into the IN182 side. Even deep in the IN182 side, the composition does not reach the specification IN182 electrode composition (e.g. the nickel content is only about 60 wt% far from the interface, in contrast to the 69 wt% Ni in the electrode specification), though this is expected due to the effect of dilution of the weld metal during fusion DMW.

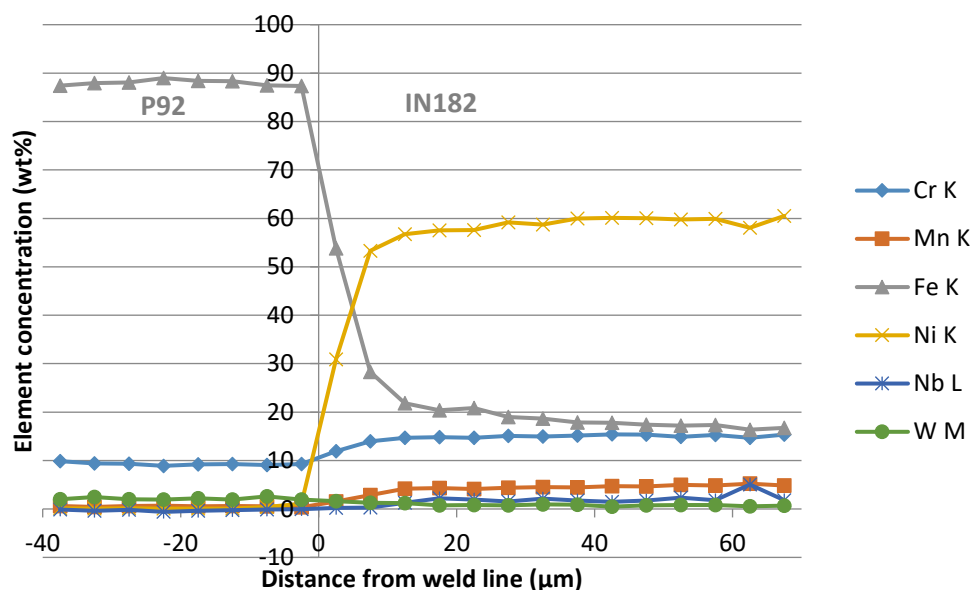


Figure 6.2: Composition profile of six largest elements across weld interface in T sample.

6.2.3 Microstructural evolution of P92 side of interface

Microstructural evolution of the P92 bulk is presented in Figure 6.3. Figure 6.3(a) shows the typical tempered martensite structure in sample T, with precipitates, mainly $M_{23}C_6$ carbides, already present on the prior austenite grain boundaries (PAGB) as well as packet boundaries, and finer MX-type carbonitrides dispersed throughout. Figure 6.3(c), (e), and (g) show that the precipitates in PAGB and packet boundaries grow and become more numerous with increasing thermal ageing duration. A clear prior austenite grain (PAG) is highlighted in Figure 6.3(c). Figure 6.3(b) shows that, in sample T, only $M_{23}C_6$ carbides are visible, shown by a slightly brighter spots in the image. Starting from sample HT-109h (Figure 6.3(d)), bright spots in the BSE mode start to appear, indicating the formation of Laves phase. EDX measurements on these bright spots, an example of which is shown in Figure 6.4, shows that it is W-rich, indicating the presence of Laves phase. The brightness of the Laves phase in BSE mode is caused by the presence of concentrated amount of W, which increased the local average atomic mass, which in turn causes that part of the BSE image to appear brighter than the surrounding area.. The number of and size of these bright spots, and thus the Laves particles, continues to grow and coarsen with increasing thermal ageing duration, as can be seen in Figure 6.3(f) and Figure 6.3(h).

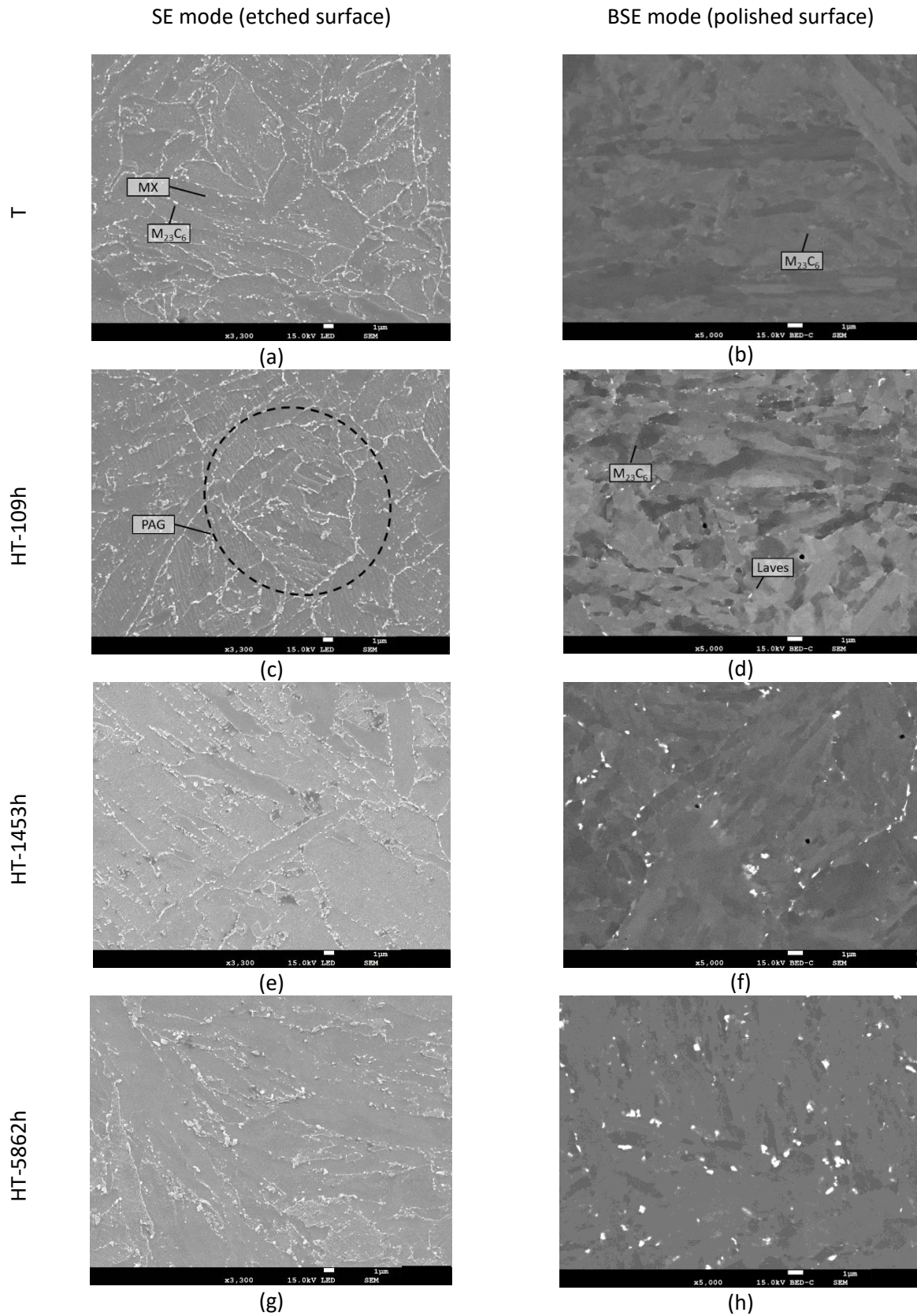


Figure 6.3: Scanning electron micrographs of P92 bulk in samples T (a and b), HT-109h (c and d), HT-1453h (e and f), and HT-5862h (g and h). Note that the SE mode images were taken from etched surfaces, whereas the BSE mode images were taken from polished surfaces.

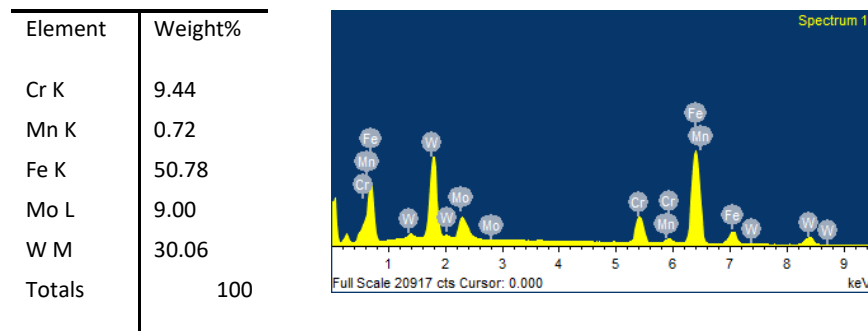


Figure 6.4: EDX analysis of the bright phase present in P92 bulk under BSE mode in SEM, indicating the presence of Laves phase.

Adjacent to the weld, there is a HAZ visible on the P92 side, as shown in Figure 6.5. The CGHAZ is about 800-1000 μm wide, depending on location. The FGHAZ is also about 800-1000 μm wide. The boundary between the ICHAZ and FGHAZ lies about 1800 μm to 2000 μm from the weld line. This corresponds to location where the minimum hardness values were measured in Figure 6.1. SEM images of these zones are presented in Figure 6.6, showing a typical large PAG in CGHAZ and typical small grains in FGHAZ, in comparison with bulk P92 PAG shown in Figure 6.3(c). For the sake of brevity, only the images from sample T are shown here, as the HAZ does not experience further development in terms of grain size and distribution during thermal ageing; only the precipitates develop further (e.g. the formation and coarsening of Laves), and these were already discussed for bulk P92.

Figure 6.7 shows the SEM image of the P92 side of the interface in samples T, HT-109h, HT-1453h, and HT-5862h. The weld interface is a smooth line that divides P92 from IN182. Starting from sample HT-109h, there seems to be a formation of precipitates that roughly lines up along the interface in the HT630-109h sample, but not in the T sample. This may indicate the formation of Type I interfacial carbide, which may have a role in crack nucleation in creep failures that failed in Type II mode. These carbides grow more prominent the longer the thermal ageing duration.

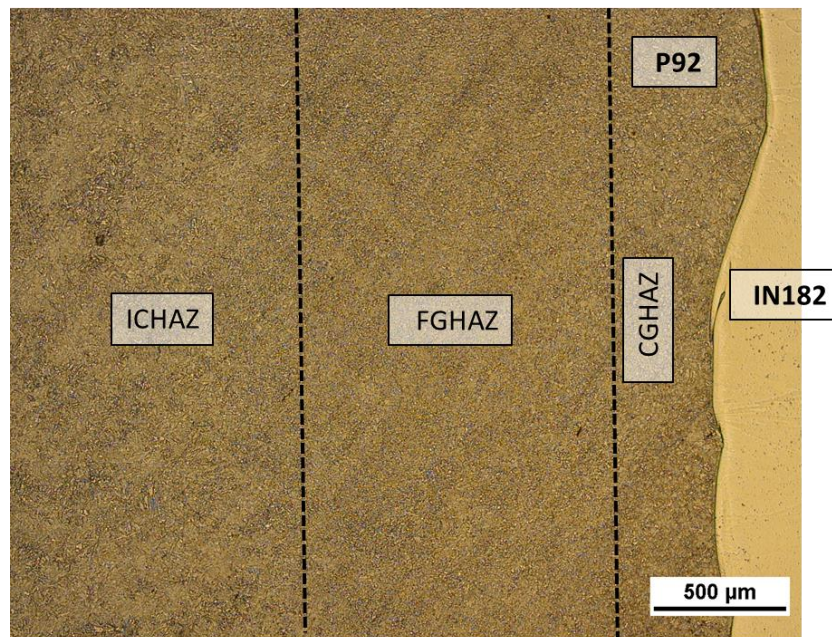
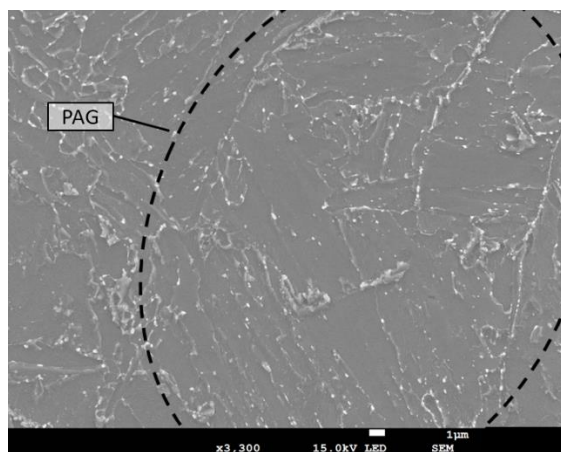
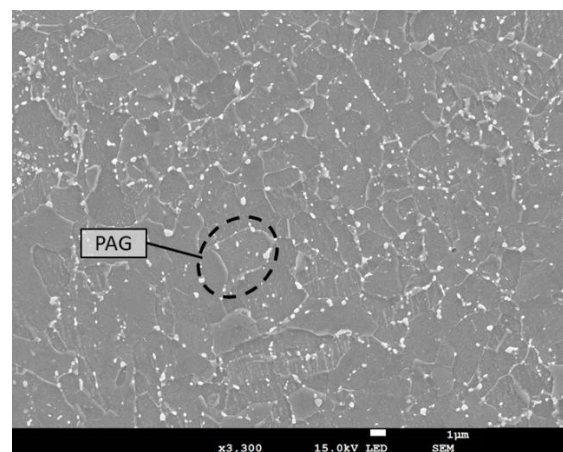


Figure 6.5: HAZ of P92 side of the weld in sample T, showing the intercritical HAZ (ICHAZ), fine-grained HAZ (FGHAZ), and coarse-grained HAZ (CGHAZ).



(a)



(b)

Figure 6.6: Scanning electron micrographs of sample T at (a) CGHAZ and (b) FGHAZ.

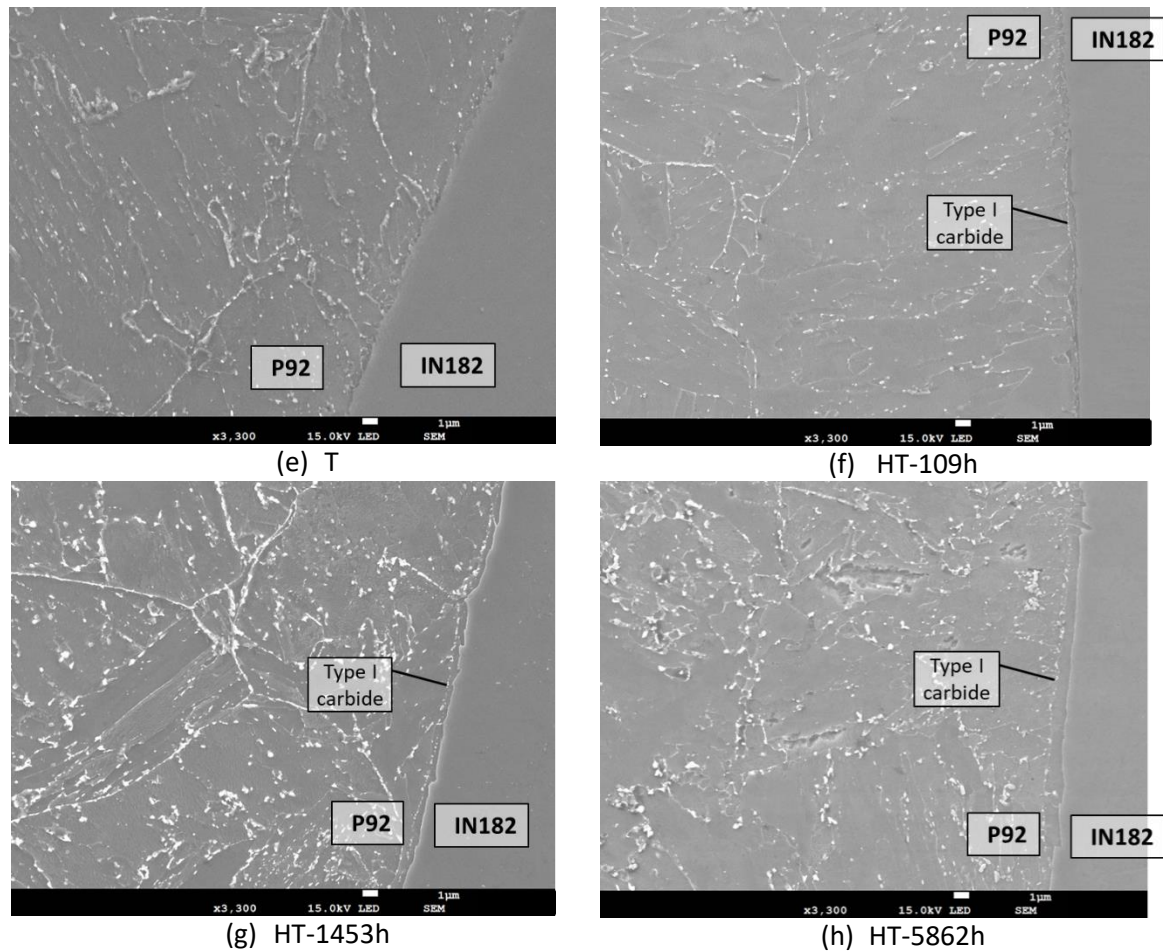


Figure 6.7: P92 side of the weld line in sample (a) T (b) HT-109h (c) HT-1453h and (d) HT-5862h.

6.2.4 Microstructural evolution of IN182 side of interface

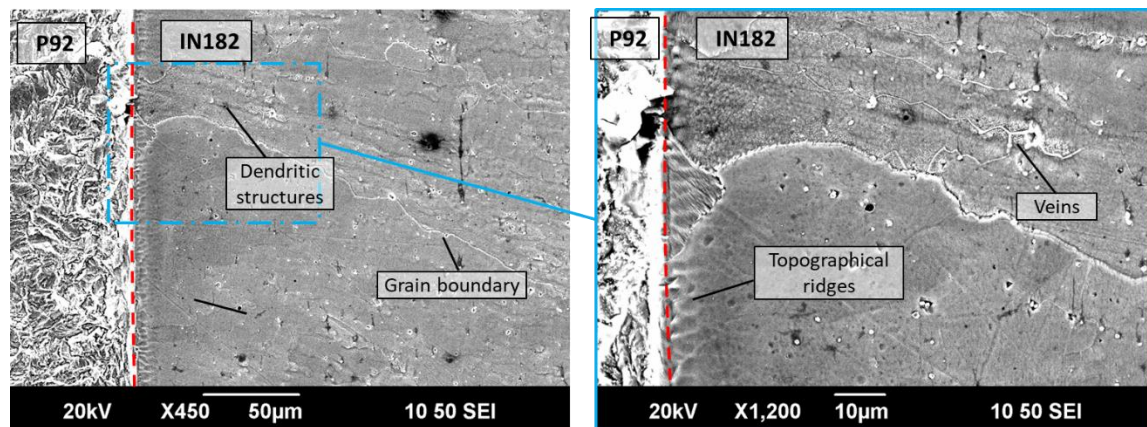


Figure 6.8: Scanning electron micrographs (SE mode) of the IN182 side of the weld line in sample T.

Figure 6.8 shows that the microstructure of IN182 in sample T consists of dendritic substructure with some veins running roughly perpendicular to the weld line. The veins likely contain precipitates such as Nb-rich MX that would appear bright under BSE mode of SEM, as shown in Figure 6.9. This is confirmed by EDX analysis presented in Figure 6.10. Next to the interface, there is a series of

topographical ridges that extends along the interface, about 10µm wide, which may correspond to the presence of PMZ. Figure 6.11 shows that the precipitates that form along these grain boundaries became coarser the longer the thermal ageing duration.

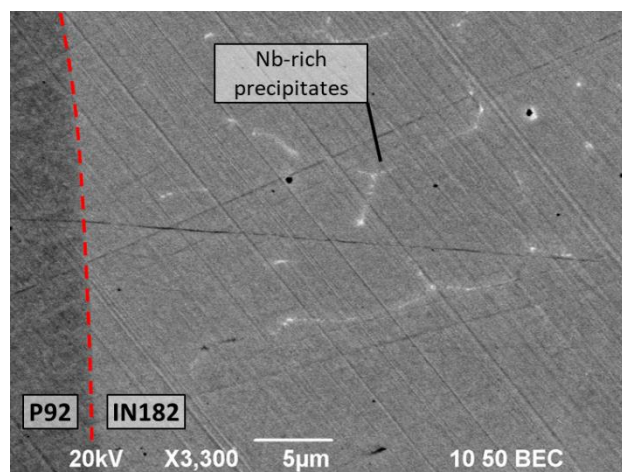


Figure 6.9: Scanning electron micrographs (BSE mode) of the IN182 side of the weld line in T sample. Polished surface.

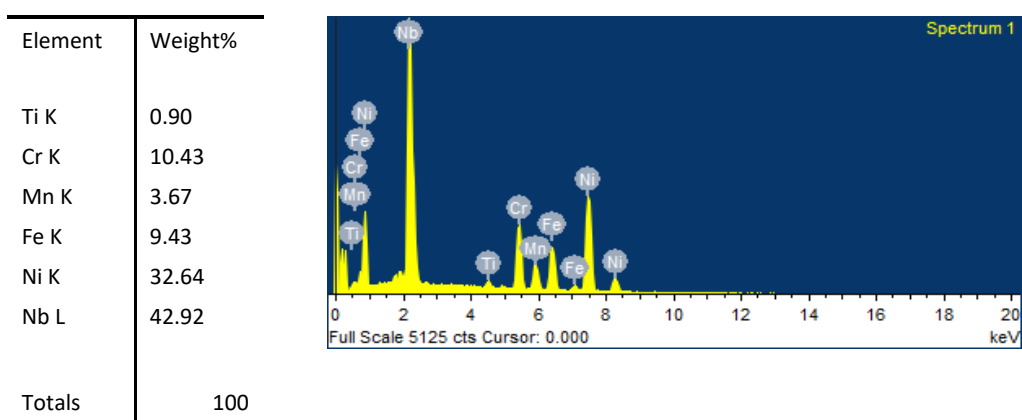


Figure 6.10: EDX analysis of particles in IN182 side that appeared bright in BSE mode in SEM.

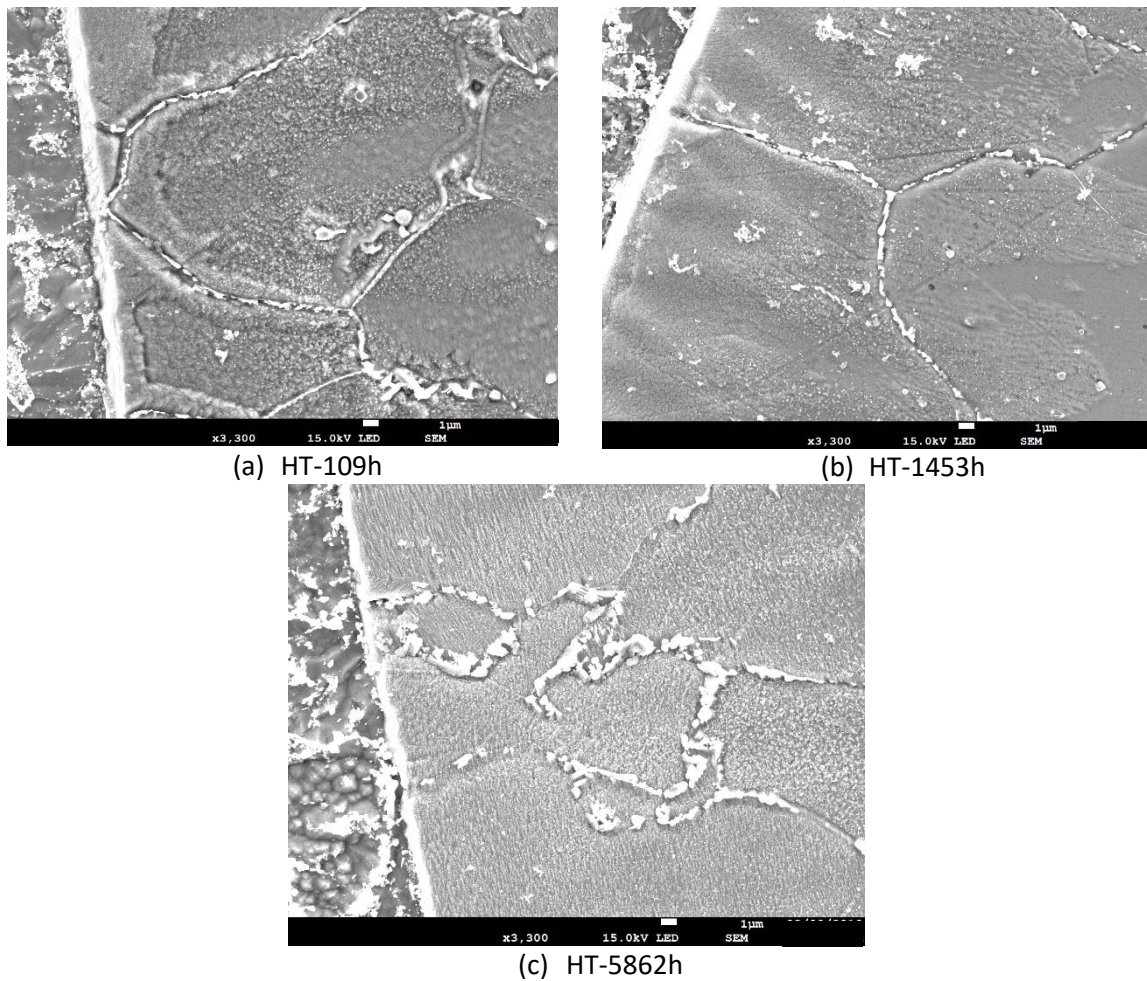


Figure 6.11: IN182 side of the interface in samples (a) HT-109h (b) HT-1453h and (c) HT-5862h.

6.3 Characterisation of crept welds

6.3.1 Creep fracture characteristics

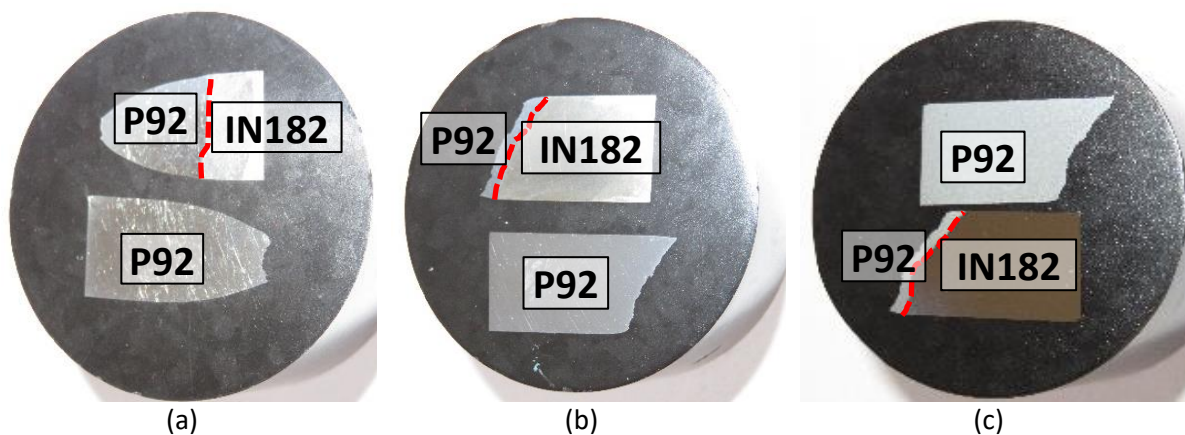


Figure 6.12: Crept P92/IN182 samples, resulted in failure time of (a) 109h (b) 1453h and (c) 5862h.

The results of the creep tests on P92/IN182 weld performed by Doosan Babcock are shown in Figure 6.12. The specimens were subjected to 154, 129, and 116 MPa of stress, at temperature of 630°C, which resulted in time-to-fracture of 109, 1453, and 5862 hours, respectively. These samples are labelled as **Creep-109h** in Figure 6.12(a), **Creep-1453h** in Figure 6.12(b), and **Creep-5862h** in Figure 6.12(c), respectively. These accelerated creep failures did not reproduce the DMW failures seen in power plants, which tend to occur in the P92 side within a few microns of interface, often called Type II failures [39, 70]. Instead, the failure occurred in the bulk P92 for the short duration creep (109h), and Type IV failure—failure in the HAZ—for the longer duration creep (1453h and 5862h).

While the creep curves are not available for analysis, Table 6.1 shows that Creep-1453h and Creep-5862h samples failed at far lower total strain, indicated by the low elongation values. They also have lower strain in the tertiary stage, as shown by the limited amount of necking, which can be seen as seen in Figure 6.12(b) and Figure 6.12(c) and confirmed by the low reductions of area, indicating a more brittle fracture. This can also be seen in Figure 6.13, where the grains and the voids in Creep-109h appear to be more elongated, in a direction perpendicular to the fracture surface, compared to those in Creep-1453h and Creep-5862h.

Table 6.1: Ductility measurements of creep failure of P92/IN182 weld specimens.

Sample	Elongation (%)	Reduction of area (%)
Creep-109h	10.69	80.88
Creep-1453h	1.67	11.56
Creep-5862h	1.09	7.36

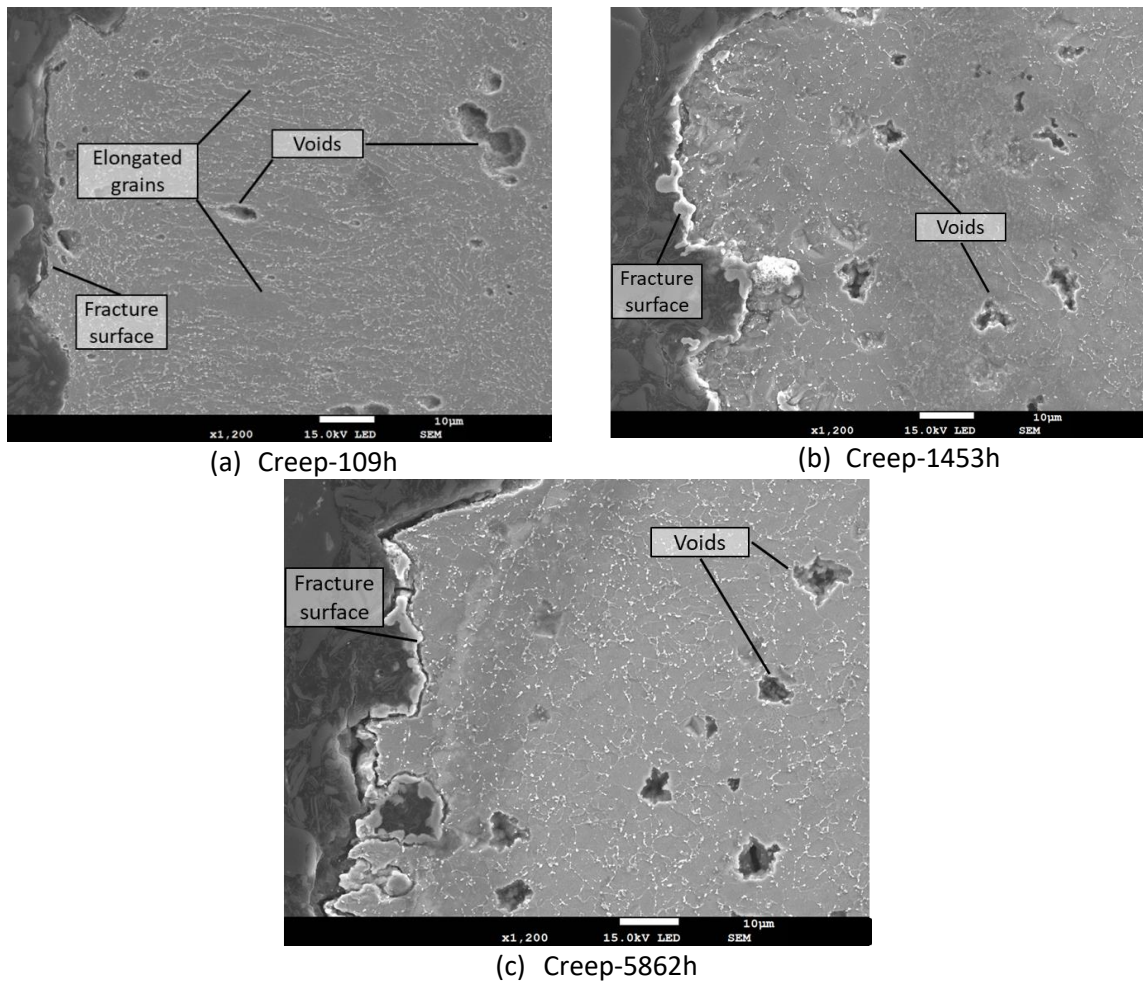


Figure 6.13: P92 microstructure adjacent to the fracture in samples (a) Creep-109h (b) Creep -1453h and (c) Creep -5862h.

Figure 6.14 shows the Larson-Miller parameter ($T(\ln t_r + 20)$, where T is temperature in K and t_r is the stress-rupture time in hours) plot for these P92/IN182 crept welds. The Larson-Miller parameter plots for the P92 bulk and the IN182 weld, whose creep testing data provided by Doosan Babcock are available in the Appendix (Table 0.2 and Table 0.3), are also shown for comparison. The Larson-Miller parameter plot of the crept welds are comparable to that of the P92 bulk and is far below that of IN182 weld, which indicates that the creep failure in the P92/IN182 crept welds can be largely determined by what happens in the P92 side of the weld.

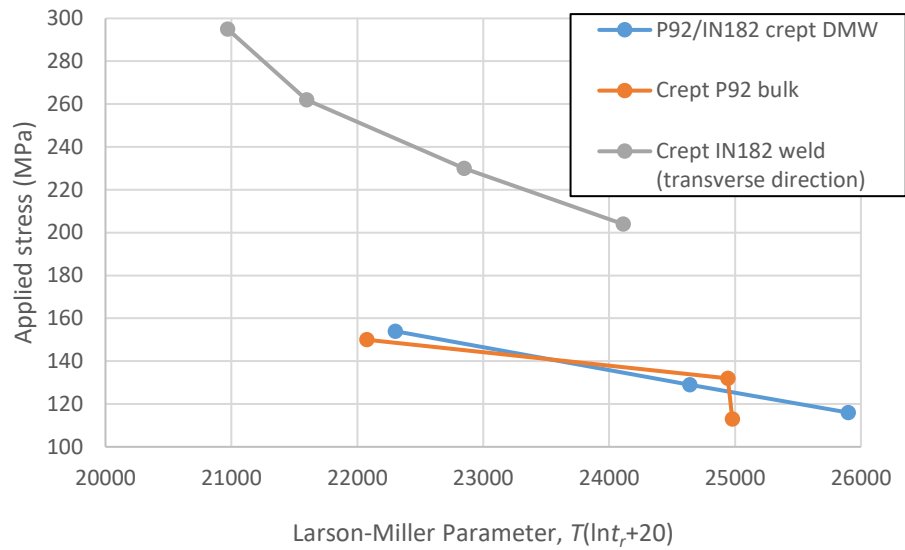


Figure 6.14: Larson-Miller parameter plot for the current P92/IN182 crept welds, in comparison with those of crept P92 bulk and crept IN182 weld. The data for the latter two plots are available in the Appendix.

6.3.2 Microhardness profile across interface line

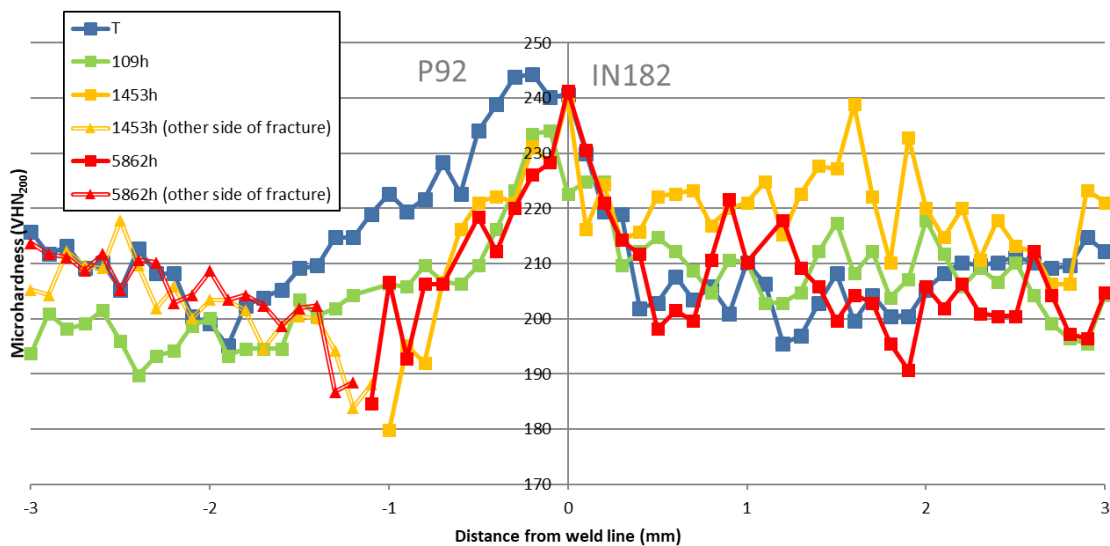


Figure 6.15: Microhardness profile across the weld line in samples T, Creep-109h, Creep-1453h and Creep-5862h.

The hardness profiles across the weld line are plotted for each of the creep samples in Figure 6.15. Microhardness profile for sample T was also added for comparison. In samples Creep-1453h and Creep-5862h samples, the fracture occurs within the HAZ of P92, close to the weld line, so meaningful microhardness profile would have to include the region on the other side of the fracture.

The HAZ in sample T has the highest hardness. The HAZ hardness in the creep samples decreased somewhat during creep, but does not change much after long time. This decrease in hardness is due to the additional tempering that occurs during creep. The Creep-109h sample has lower hardness

away from weld line, likely due to sample elongation, as can be seen in Figure 6.12(a). Creep- 1453h and Creep-5862h has lower hardness (around 190 VHN₂₀₀) in the vicinity of fracture due to cavities and voids (but also due to weak FGHAZ), but otherwise converges to hardness of sample T far from weld line. The cavities extend about 150-200µm from fracture surface.

The minimum hardnesses, where the fracture occurred, of Creep- 1453h and Creep-5862h occur about -1.1 mm to -1 mm from the weld line, corresponding to the FGHAZ. This is different from those shown of the thermally aged welds shown in Figure 6.1, where the minimum hardness occurred somewhere around -1.8 to -2 mm from the interface, corresponding to the boundary between FGHAZ and ICHAZ. This means the fracture under creep would occur within FGHAZ, not at the boundary between FGHAZ and ICHAZ where the minimum hardness in thermally aged samples would occur. The low hardness within FGHAZ measured in the failed Creep- 1453h and Creep-5862h samples was caused by the local elongation that preceded the fracture there, not by a low hardness intrinsic to FGHAZ.

6.4 Comparing P92 precipitate development between creep and thermally aged samples

Comparison of microstructures of P92 side of the interface between crept and thermally aged samples can be made in order to infer the effect of mechanical loading and strain on the microstructural development of P92 under high temperature (630°C), particularly the precipitation development.

6.4.1 Type I particles

As Type I carbides may play an important role in crack nucleation close to the weld line in service welds, the effect of mechanical stress on Type I carbide was investigated. Note that the precipitates identified as Type I may include the Laves phase or other coarse precipitates. So for the purposes of this analysis, "Type I carbides" are called "Type I particles" henceforth. In Figure 6.16, the small precipitates located parallel to the weld line in sample T were assumed to be Type I particles. Figure

6.17 shows the Type I particles in the crept and thermally aged samples. Parker and Stratford [60] suggested that it is important to measure Type I particles in terms of elliptical major and minor axes, instead of assuming circular shapes, due to the particles sometimes developing into lenticular shapes. So here the measurements are done in terms of the mean area, mean major axis dimension, and mean minor axis dimension, shown in Figure 6.18, Figure 6.19, and Figure 6.20, respectively. Measurements from HT-500h, HT-1000h, HT-3000h were added in order to provide additional data in between the original thermally aged samples. These figures confirm that Type I particles grow continuously in both crept and thermally aged samples. The mean major axis dimensions of Type I carbide particles are similar for both types of samples (though slightly higher in thermally aged samples), as seen in Figure 6.19. However, mean minor axis dimensions of Type I carbide particles are initially similar between the two types of samples, those of the crept samples increase faster in longer durations, as can be seen in Figure 6.20, which in turn coarsens Type I particles slightly (though not significantly) faster in the crept samples, as can be seen in Figure 6.18. It can be deduced that the presence of high stress in Creep-109h samples does not lead to enhanced coarsening of Type I carbide, perhaps due to its short duration. But in longer durations, the presence of (lowered) stress does somewhat impact the coarsening of Type I particles. This is in contrast with the observations made by Parker and Stratford [60], where Type I particles in both crept and thermally aged samples have similar the major and minor axes dimensions at a given duration of high temperature exposure; however, in that particular study, only a small stress was applied (30–80 MPa) which would have led to minimal strain/deformation in the interfacial region, thus the precipitate behaviour in that vicinity would not have been affected significantly by the applied stress.

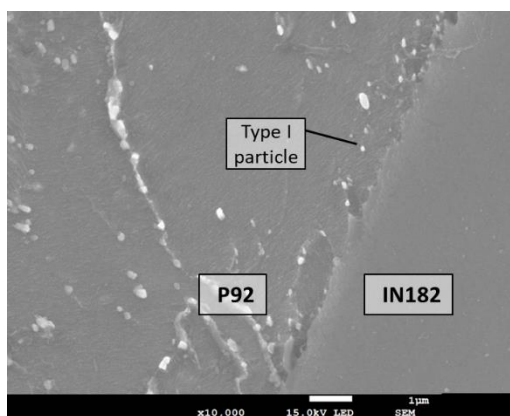


Figure 6.16: P92 near the weld line, showing Type I particles, in sample T.

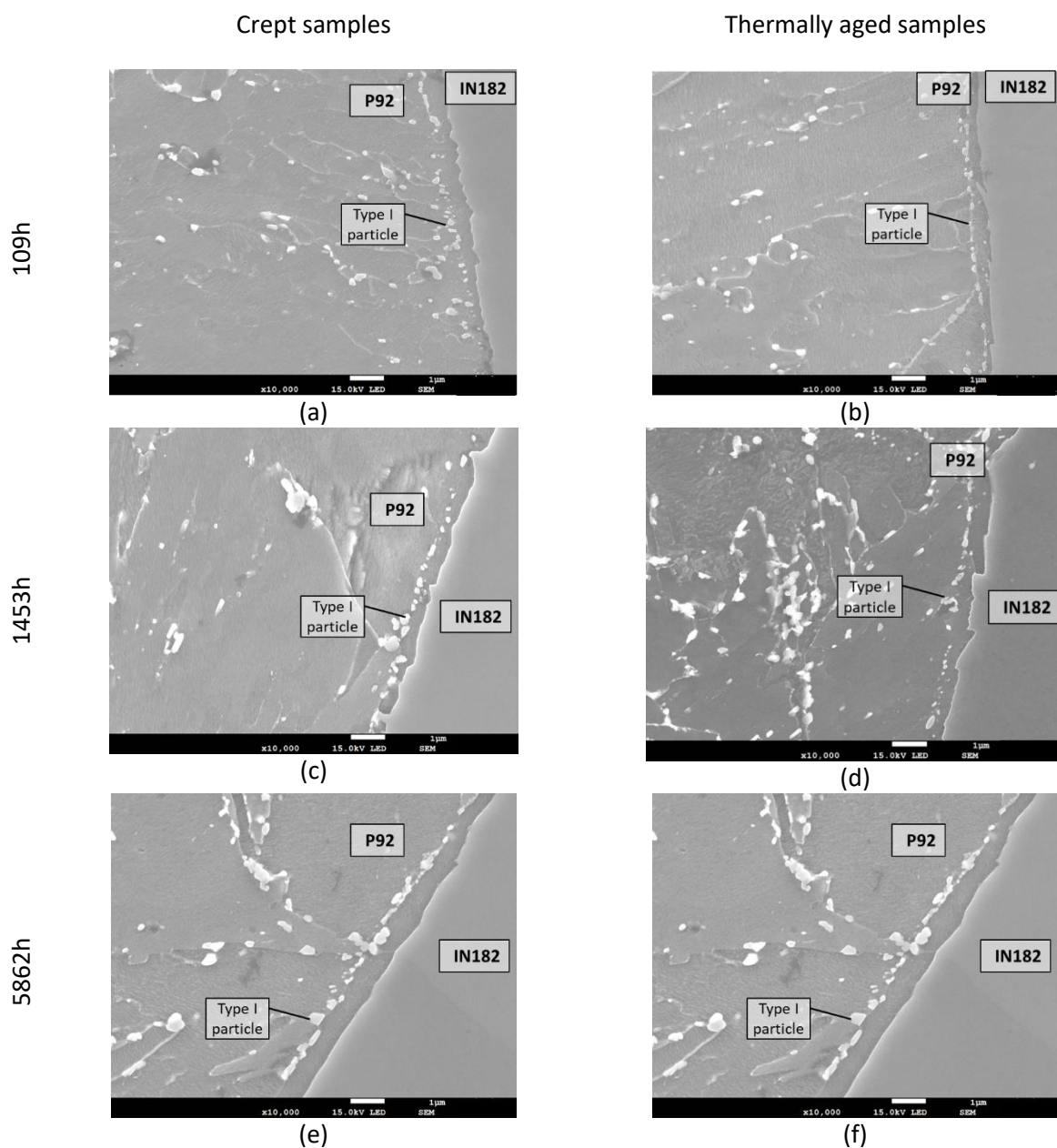


Figure 6.17: P92 near the weld line, showing Type I particles, in samples (a) Creep-109h (b) HT-109h (c) Creep -1453h (d) HT-1453h (e) Creep-5862 and (f) HT-5862h.

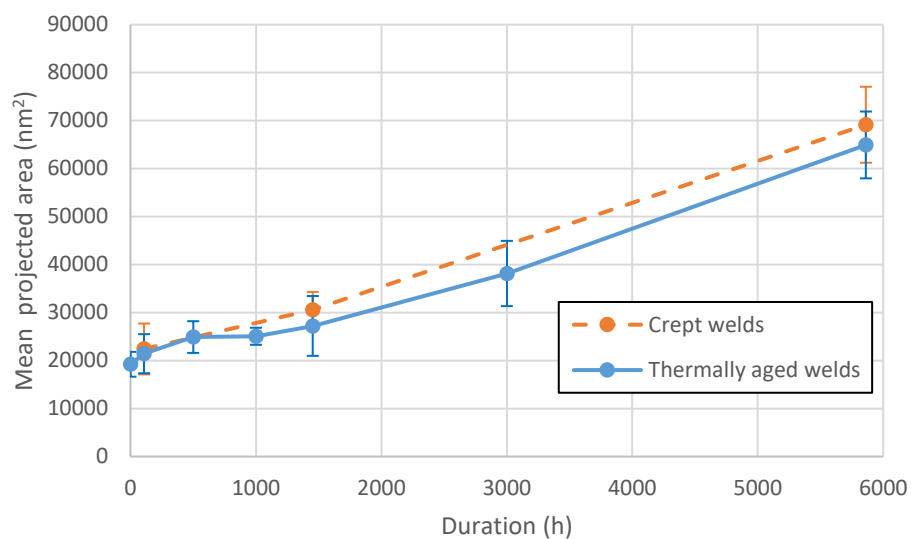


Figure 6.18: Mean projected size of Type I carbide particles in sample T (0h), crept samples and thermally aged specimens. The error bars indicate standard error.

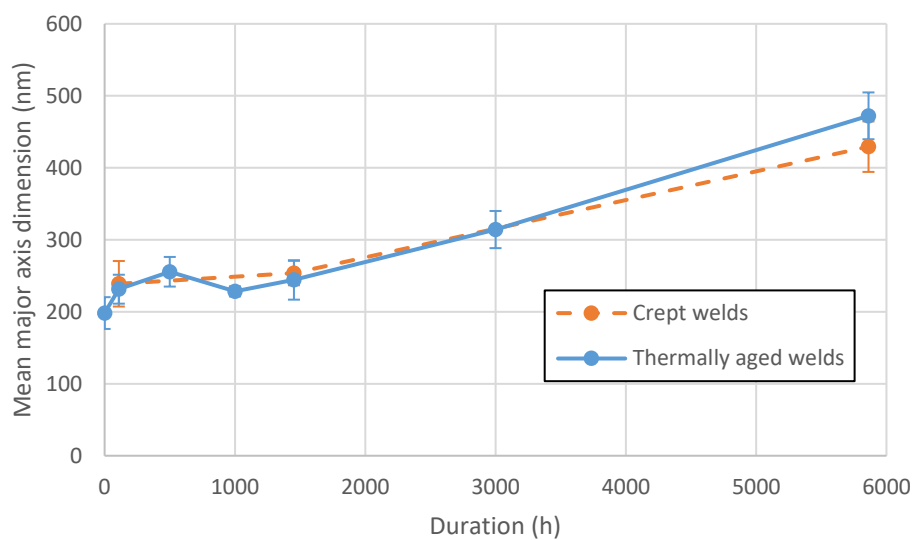


Figure 6.19: Mean major axis dimension of Type I carbide particles in sample T (0h), crept samples and thermally aged specimens. The error bars indicate standard error.

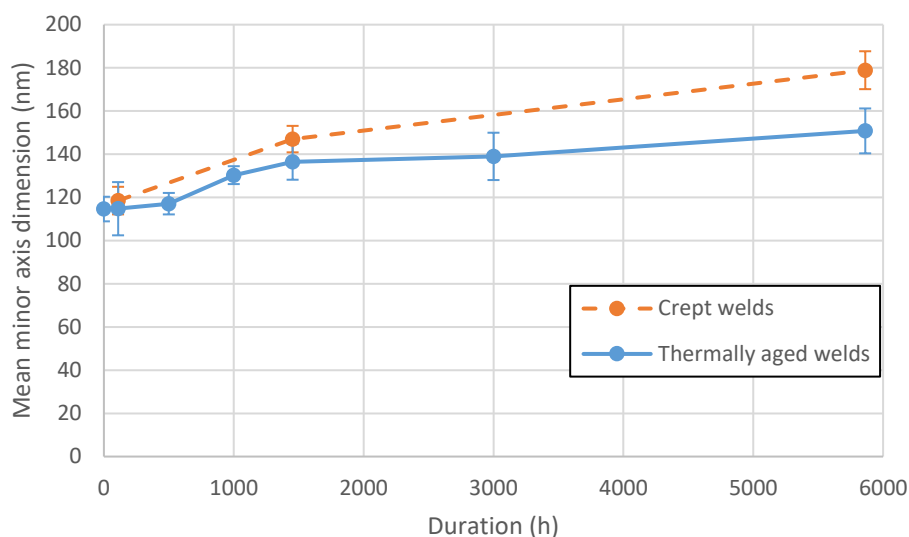


Figure 6.20: Mean minor axis dimension of Type I carbide particles in sample T (0h), crept samples and thermally aged specimens. The error bars indicate standard error.

6.4.2 Laves particles

The development of Laves phase was examined because this type of precipitates can be readily distinguished in the BSE mode of SEM, and may be relevant to Type IV creep failures. Laves may behave differently in different parts of P92 side of the weld due to the differences in grain size, so Laves behaviour in the P92 bulk, CGHAZ, and FGHAZ were investigated. As can be seen Figure 6.3(b), there is no Laves present in sample T, so it is not included in the Laves quantification here. The BSE images of P92 bulk from crept samples and their thermally aged counterparts are presented in Figure 6.21. In the first row (Figure 6.21(a) and Figure 6.21(b)), Creep-109h is shown to have more Laves particles compared to HT-109h, even though each underwent the high temperature of 630°C for the same amount of time (109h). In the longer duration samples, the number density and size of Laves increased in both types of samples. However, the thermally aged samples (HT-1453h in Figure 6.21(d) and HT-5862h in Figure 6.21(f)) developed coarser Laves particles, whereas the crept samples (Creep-1453h in Figure 6.21(c) and Creep-5862h in Figure 6.21(e)) had more numerous Laves particles.

Similar BSE images from crept samples and their thermally aged counterparts from CGHAZ and FGHAZ are presented in Figure 6.22 and Figure 6.23, respectively.

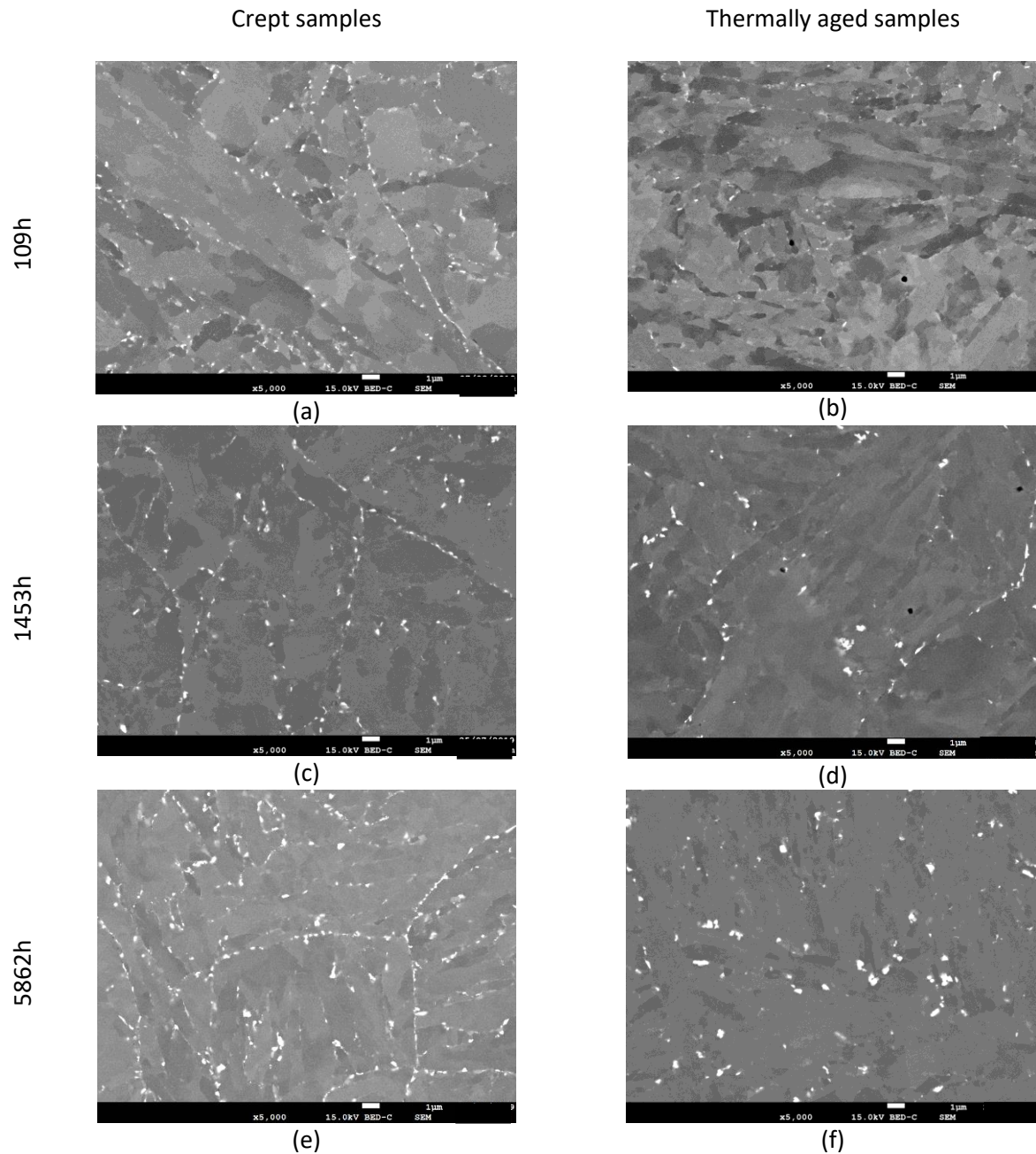


Figure 6.21: Scanning electron micrographs (BSE mode) of P92 bulk in (a) Creep-109h (b) HT-109h (c) Creep -1453h (d) HT-1453h (e) Creep-5862 and (f) HT-5862h.

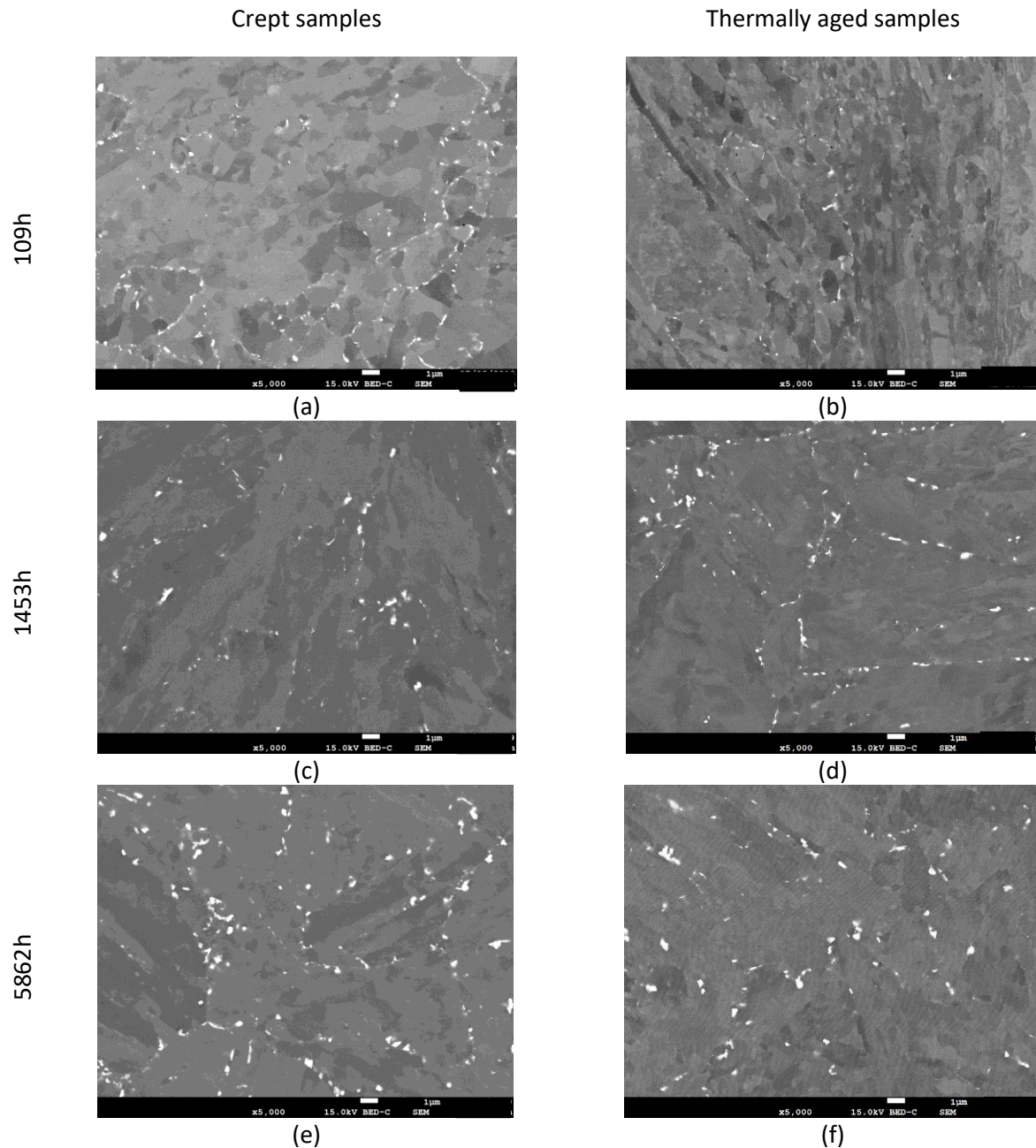


Figure 6.22: Scanning electron micrographs (BSE mode) of P92 CGHAZ in (a) Creep-109h (b) HT-109h (c) Creep -1453h (d) HT-1453h (e) Creep-5862 and (f) HT-5862h.

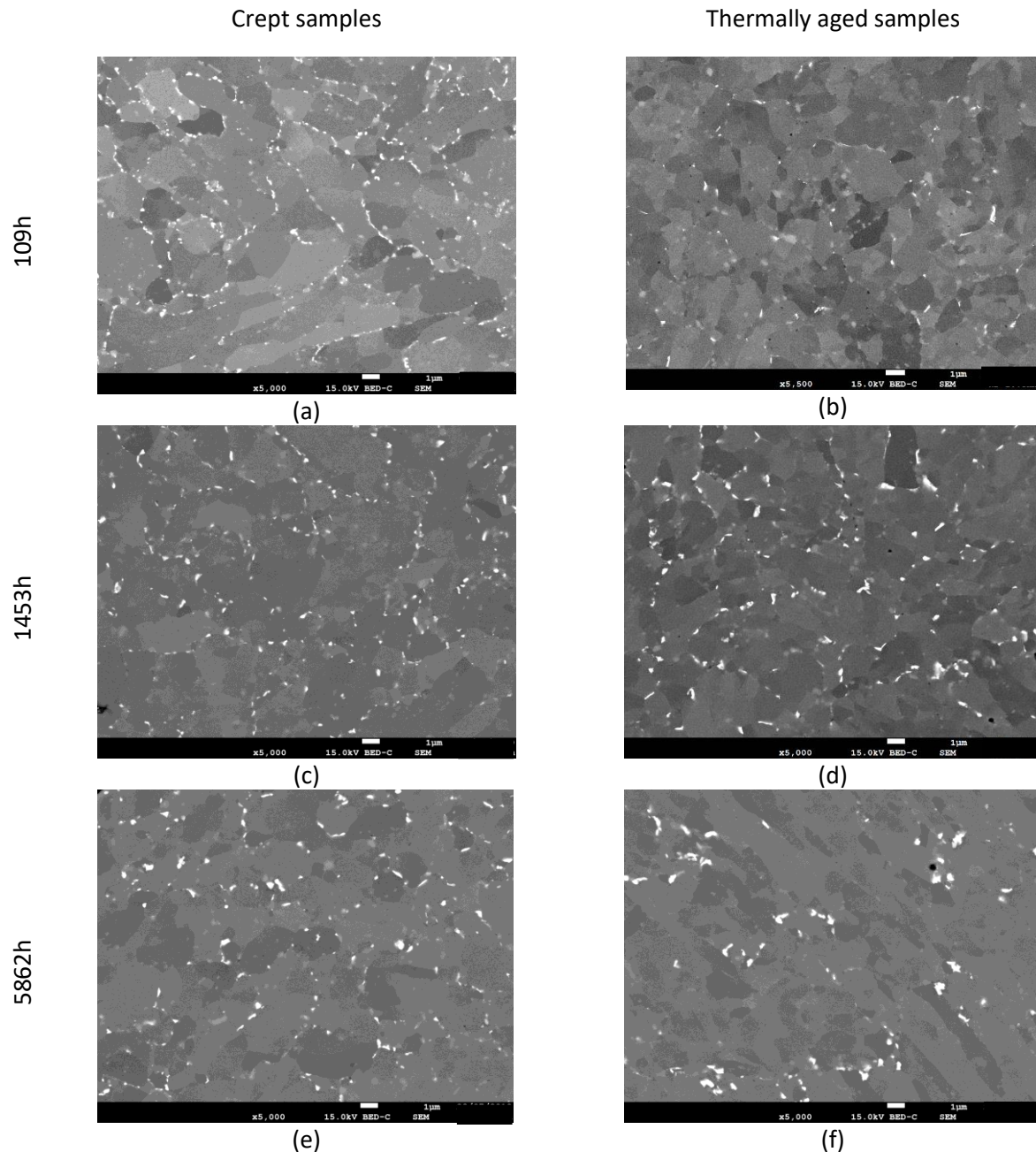


Figure 6.23: Scanning electron micrographs (BSE mode) of P92 FGHAZ in (a) Creep-109h (b) HT-109h (c) Creep -1453h (d) HT-1453h (e) Creep-5862 and (f) HT-5862h.

Quantitative analysis can be employed to better compare the Laves behaviour differences between the two samples. A quantitative analysis of Laves behaviour in P92 bulk, CGHAZ, and FGHAZ, in terms of mean diameter, area fraction, and the number density presented in Figure 6.24, Figure 6.25, and Figure 6.26, respectively. Measurements from HT-500h, HT-1000h, HT-3000h were added in order to provide additional data in between the original thermally aged samples.

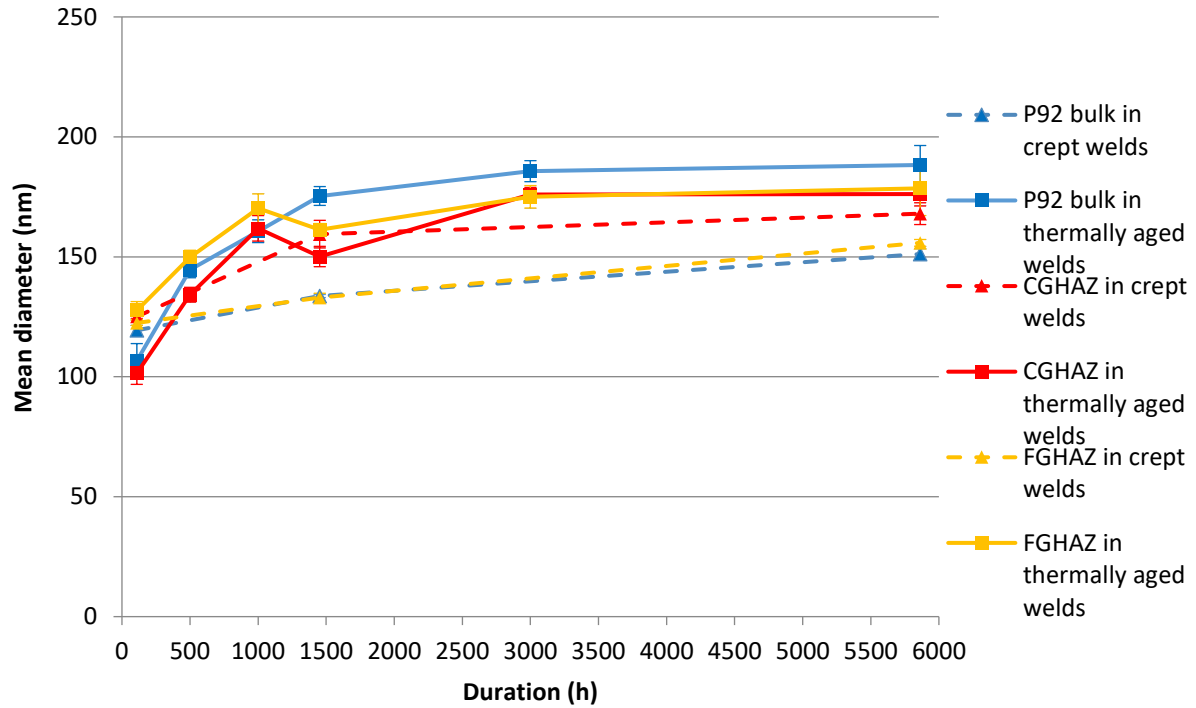


Figure 6.24: Mean diameter of Laves phase in crept samples and thermally aged samples in the P92 bulk, CGHAZ, and FGHAZ. The line colours indicate the locations, while the dashed lines indicate the crept samples and the solid lines indicate the thermally aged samples. The error bars indicate standard error.

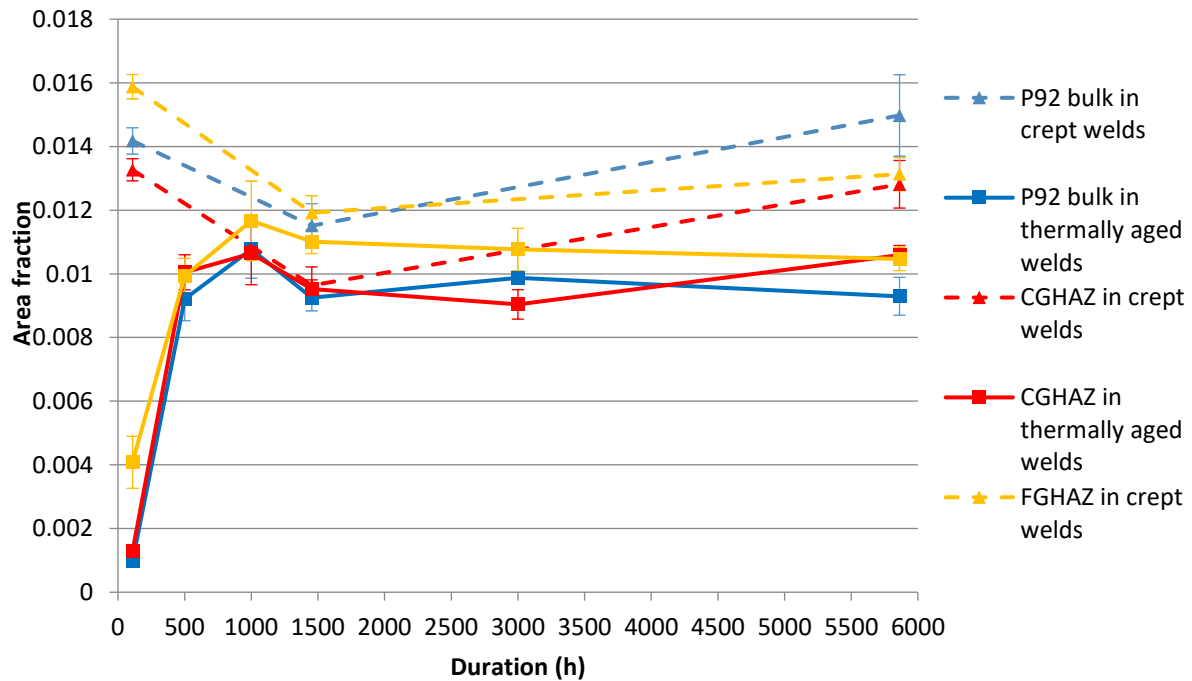


Figure 6.25: Area fraction of Laves phase in crept samples and thermally aged samples in the P92 bulk, CGHAZ, and FGHAZ. The line colours indicate the locations, while the dashed lines indicate the crept samples and the solid lines indicate the thermally aged samples. The error bars indicate standard error.

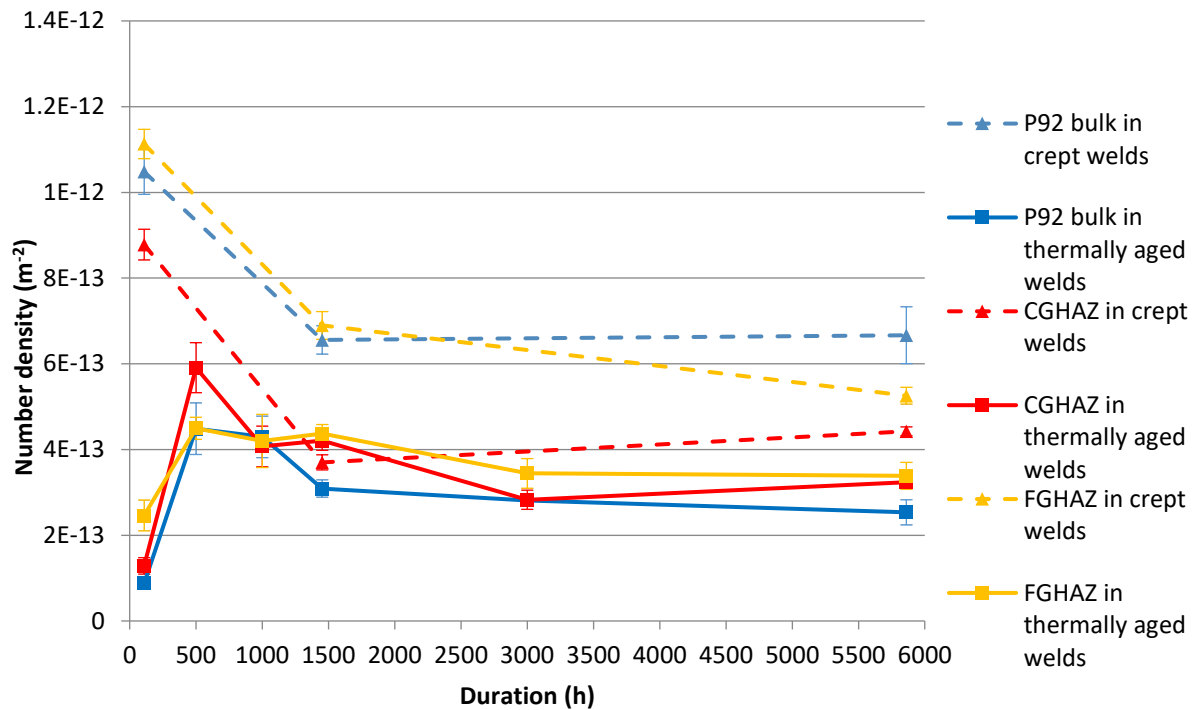


Figure 6.26: Number density of Laves phase in crept samples and thermally aged samples in the P92 bulk, CGHAZ, and FGHAZ. The line colours indicate the locations, while the dashed lines indicate the crept samples and the solid lines indicate the thermally aged samples. The error bars indicate standard error.

Figure 6.24 shows that while both types of samples developed coarser Laves in longer durations, those in the thermally aged samples tended to coarsen faster in all three zones. In terms of area fraction, the Laves in crept samples initially covered a greater percentage of the surface, as can be seen in Figure 6.25, due to a greater number of Laves nucleation reflected by the high initial number density in Figure 6.26, even though the mean diameter is similar to those of thermally aged samples. This indicates that the high stress experienced by sample Creep-109h provided the large strain that would in turn encourage the nucleation of Laves particles, but had little effect on coarsening. The initial number density of Laves phase is particularly high in the FGHAZ, which is understandable given that the small grains in FGHAZ provide larger grain boundary surface area for Laves nucleation to occur. In the intermediate duration of 1453h, the area fractions and number densities of Laves in thermally aged samples increased to mostly match those of the crept samples, though crept FGHAZ and P92 bulk maintained significantly number densities. At 5862h, the area fraction of Laves in the CGHAZ and the bulk of the crept samples increased far higher, though in the FGHAZ of crept samples the Laves have comparable area fraction to those of the thermally aged samples.

It is also interesting to note that for the thermally aged welds, where more data are available, the number density of Laves initially increased to a peak at around 1000h, but then decreased and plateaued from 1453h onwards, even as the mean diameter continues to increase slowly. This indicates that initially there was a great amount of nucleation of Laves but subsequently these particles consolidated to form larger particles, in a process known as Ostwald ripening.

It can be surmised that in higher stress and lower duration crept specimens (such as Creep-109h), the failure occurred at the bulk metal and not at the FGHAZ because the FGHAZ is strong due to its small grains. After short term ageing, the FGHAZ is further strengthened by the formation of Laves phase, which forms in the grain boundaries and adds to the creep strength by precipitation hardening. The small grains provided larger surface area of PAGB per unit volume of material, which means that it is favourable to the formation of Laves phase. This is confirmed by the high number density of Laves in FGHAZ. After long term ageing, however, the Laves phase in the PAGB coarsened. The Laves phase also excessively leached the W and other elements from the matrix, which weakened the grains due to reduced solid-solution strengthening. It also creates greater interfacial energy between the grains, which weakens it. This caused the failure to occur at the FGHAZ in the low stress long duration creep (1453h and 5862h).

6.5 Conclusions

The microstructural evolution of DMW between P92 and IN82/IN182 under thermal ageing and creep (all at 630°C) have been characterised. The following observations can be made:

- In the tempered and thermally aged samples, there are ICHAZ, FGHAZ, and CGHAZ on the P92 side of the interface. The lowest hardness occur on the boundary between FGHAZ and ICHAZ.
- There are MX and $M_{23}C_6$ precipitates in the tempered sample, but no Laves. Subsequent thermal ageing allowed the formation and then coarsening of the Laves phase.
- On the IN182 side of the interface, precipitates formed and coarsened on veins running roughly perpendicular to the weld line and on grain boundaries during thermal ageing.

- For the crept samples, the failure occurred in the bulk P92 for the short duration creep (109h), and Type IV failure—failure in the HAZ—for the longer duration creep (1453h and 5862h). The short duration creep failure is characterised by high ductility and necking, while the longer duration creep failures is characterised by low ductility and limited necking.
- The Larson-Miller parameter plots show that the creep failure in the P92/IN182 crept welds can be largely determined by what happens in the P92 side of the weld.
- The Type IV failure observed in the longer duration creep is shown to have occurred within the FGHAZ. The minimum hardness values were observed near and around the fracture surface. However, this location does not coincide with where the minimum hardness values were observed in tempered and thermally aged samples, i.e. in the boundary between FGHAZ and ICHAZ.

The following conclusions can be made when comparing P92 precipitate development between creep and thermally aged samples:

- Mechanical stresses in crept samples likely accelerated the growth of Type I particles in the minor axis direction, while the major axis dimensions of the Type I particles appear to not be affected.
- High mechanical stresses in creep (as observed in the short duration creep sample) increased the number density of Laves particles, indicating greater Laves nucleation and formation, but may have decreased the coarsening rate.
- Type IV failure in the longer duration creep may be explained by this Laves behaviour in FGHAZ.

7 CONCLUSIONS AND FUTURE WORK

7.1 Conclusions

The main objectives of this research were to characterise materials from the TransForge project examining the feasibility of the innovative fabrication of dissimilar weld joints, as well as to characterise the microstructural evolution of DMW between P92 and IN82/IN182 under creep and thermal ageing. The former was carried out to determine whether the TransForge fabrication technique yields high integrity joints, by investigating the resulting microstructure and performance under tensile testing. The latter was carried out to investigate the microstructural evolution of a fusion weld between P92 and IN82/IN182 under high temperature and to investigate the effect of applied mechanical stress on microstructural evolution of DMW.

The TransForge was demonstrated by joining A508 steel with nickel-based IN600, as well as joining A508 steel with 304L stainless steel using electron beam welding (EBW) and forging to produce a dissimilar metal weld. From results presented and discussed of both TransForge joints, the following conclusions can be made:

- There are differences in the thickness of the transition zone between the centre of the joint specimens and the outer edge of the joint specimens in both types of joints, due to the incomplete penetration of the electron beam welding (EBW).
- Tensile testing shows that, overall, the cross-weld specimens have ultimate tensile stress (UTS), yield stress, and elongation intermediate between the bulk alloys of each joint. This indicates that the tensile properties across the weld are comparably strong and ductile (or more so) compared to the bulk alloys, demonstrating the viability of the fabrication process.
- There is greater variation in microhardness in cross-weld profile in the A508/304L joint, compared to that of A508/IN600 joint, indicating that the A508/304L joint has the more diverse microstructurally distinct zones. In the A508/304L joint, the hardness decreases from

the bulk to the interface in the A508 side of the joint. In the 304L side of the joint the hardness decreases from the interface towards the bulk. This may be explained by the lack of carbides in the A508 side of the interface due to carbon migration to the 304L where there is significant additional carbide precipitation near the interface.

- There appears to be distinct microstructural zones in the austenitic side (either IN600 or 304L) of both joints, as well as in the transition zones formed between the two alloys of each joint.
- The A508/IN600 joint is microstructurally simpler compared to the A508/304L joint. Despite having differing compositions, microstructurally the transition zone (between A508 and IN600) and the IN600 are quite similar. The grain morphology, grain sizes, and crystal structure of both are similar (both have FCC crystal structure), and both have similar hardness values. The main difference between the IN600 bulk and the transition zone (and its subzones) is the amount of precipitates ($M_{23}C_6$ and M_7C_3 carbides) that appear; this may have been caused by carbon migration during EBW, forging and PFHT, but the elevated amount of carbides that appear in the transition zone may also have been caused by the decreased carbon solubility in the γ -austenite matrix, as confirmed by the thermodynamic simulation of phase equilibria using ThermoCalc.
- The A508/304L joint has a transition zone (between A508 and 304L) that has a complex set of subzones. The different types of crystal structures that formed within the transition zone cannot be explained by composition alone, suggesting mechanically induced martensite formation. High hardness values ($>400 \text{ HV}_{200}$) observed in the medium thickness transition zone suggests that some of the martensite in the transition zone has not been fully tempered despite the PFHT. Despite the formation of these complex microstructural features, A508 and 304L have been directly joined successfully, demonstrating good weldability of this pair of dissimilar alloys when utilising the TransForge fabrication technique.
- Experimental P - h curves from nanoindentation tests were used to optimise elastic-plastic parameter values (Young's modulus, Poisson's ratio, yield stress, and strain-hardening

exponent) using the inverse method. Optimising parameters on indents carried out across the weld interface resulted in a cross-weld yield stress profiles that correlates well with microhardness profile. However, the fact that the results are very different depending on the limits set, particularly for E , suggests that inverse method used here may not have resulted in well-optimised P - h curves. Since the four parameters being optimised in the inverse method were not independent of each other, the results of the other three parameters, including yield stress may also not be accurate and should to be taken with caution.

This work has demonstrated the success and viability of the TransForge technique. With the degree of automation possible, the manufacturing process will be faster and more cost-effective using this technique. Thus, TransForge has the potential to be a revolutionary method to join dissimilar metals.

The following conclusions can be drawn from the characterisation of microstructural evolution of DMW between P92 and IN82/IN182 under thermal ageing and creep:

- The Larson-Miller parameter plots show that the creep failure in the P92/IN182 crept welds can be largely determined by what happens in the P92 side of the weld. the failure occurred in the bulk P92 for the short duration creep, and Type IV failure for the longer duration creep. The short duration creep failure is characterised by high ductility and necking, while the longer duration creep failures is characterised by low ductility and limited necking.
- In the tempered and thermally aged samples, there are ICHAZ, FGHAZ, and CGHAZ on the P92 side of the interface. The lowest hardness occur on the boundary between FGHAZ and ICHAZ. In the long duration crept samples, the lowest hardness values occur within the FGHAZ, where the Type IV failure occurred. This indicated that the low hardness within FGHAZ measured long duration crept samples was caused by the local elongation that preceded the fracture there, not by a low hardness intrinsic to FGHAZ.
- There are MX and $M_{23}C_6$ precipitates in the tempered sample, but no Laves. Subsequent thermal ageing allowed the formation and then coarsening of the Laves phase. High

mechanical stresses in creep (as observed in the short duration creep sample) increased the number density of Laves particles, indicating greater Laves nucleation and formation, but may have decreased the coarsening rate. Type IV failure in the longer duration creep may be explained by this Laves behaviour in FGHAZ.

- Mechanical stresses in crept samples likely accelerated the growth of Type I particles in the minor axis direction, while the major axis dimensions of the Type I particles appear to not be affected.

This work indicates that there are differences in the development of both the Type I particles and the Laves phase between crept and thermally aged samples. Hence, in order to simulate microstructural evolution of DMW in service, creep testing needs to be considered. Merely using thermally aged samples, without mechanical stress, to simulate microstructural evolution would likely result in an inaccurate reflection of the microstructural behaviour in service, as the application of stress in service may alter the microstructural behaviour of DMW, at least for some precipitate development including the Type I particles and the Laves phase.

7.2 Future work

For TransForge, the following future work is suggested in order to further understand the fabrication technique and offer the chance for process optimisation:

- Transmission electron microscopy (TEM) characterisation to identify or confirm phases/precipitates that form on the joints, and also to identify any band/zone too thin to be identified by SEM.
- Creep tests and/or thermal ageing of TransForge joints to investigate the microstructural evolution of the joints under prolonged high temperature exposure. Creep tests would be ideal as they would reveal mechanical performance under high temperatures. According to ASME Boiler and Pressure Vessel Code (BPVC), A508 steel is to be utilised in for nuclear

reactors up to 371°C, so any creep tests and/or thermal ageing should be done slightly higher than this temperature, e.g. 450°C.

- DICTRA modelling of the joints in order to simulate the cross-weld diffusion behaviour that occurs under prolonged high temperature exposure. This may reveal any formation of potentially detrimental microstructure due to e.g. carbon migration that would occur if the joint were in service.
- Process modelling of TransForge process.

Regarding the characterisation of microstructural evolution of fusion DMW between P92 and IN82/IN182, the following future work is suggested:

- Interrupted creep tests of these joints, providing crept samples at regular high-temperature intervals with consistent stress applied.
- Another important precipitate that affects the mechanical performance of P92 is the $M_{23}C_6$ carbide. Hence in future work the effect of mechanical stress on $M_{23}C_6$ carbide should be examined.

Furthermore, more work could be done to further refine the optimisation method using the inverse approach to extract elastic-plastic material properties from experimental nanoindentation data, so that the results would provide more accurate material properties values. An expansion of the inverse method by incorporating additional experimental data into a multi-objective optimisation function, such as that presented by Iracheta et al. [103] may alleviate the issue posed by the local minima that may be found by a single-objective function.

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APPENDIX

A number of creep testing results were sent by Doosan Babcock. In this project, the focus is on crept samples originating from a pipe mockup of P92 welded to 740H plate using IN82/IN182 filler. Only P92/IN182 portion of the crept tests are discussed in this project. The test material and history of these are given in Table 0.1. Unfortunately creep curve data are not available.

Table 0.1: Creep testing material and history of pipe mockup of P92 welded to 740H plate using IN82/IN182 filler. PWHT 770°C 8 hours. Courtesy of Doosan Babcock.

Sample	Temperature (°C)	Stress (MPa)	Estimated duration (h)	Final duration (h)	Elongation (%)	Reduction of area (%)
Creep-109h	630	154.0	300	109	10.69	80.88
Creep-1453h	630	129.0	1000	1453	1.67	11.56
Creep-5862h	630	116.0	3000	5862	1.09	7.36

Data of creep testing of P92 bulk material and creep testing of IN182 padding weld (in transverse direction) were also given by Doosan Babcock, presented in Table 0.2 and Table 0.3, respectively. The data from these tests were used to calculate the Larson-Miller parameters for the bulk P92 and IN182, against which the Larson-Miller parameters for the P92/IN182 weld can be compared.

Table 0.2: Creep testing material and history of pipe mockup of P92 bulk material from pipe mockup. Courtesy of Doosan Babcock.

Test Ref.	Temperature (°C)	Stress (MPa)	Estimated duration (h)	Final duration (h)	Elongation (%)	Reduction of area (%)
14/039	630	150.0	1000	85	20.26	76.85
14/040	630	132.0	3000	2033	16.50	52.20
14/041	630	113.0	10000	2116	10.14	28.47

Table 0.3: Creep testing material and history of pipe mockup of IN182 weld from a padding weld, in transverse direction. Courtesy of Doosan Babcock.

Test Ref.	Temperature (°C)	Stress (MPa)	Estimated duration (h)	Final duration (h)	Elongation (%)	Reduction of area (%)
14/053	630	295.0	1000	25	16.26	41.05
14/054	630	262.0	3000	50	21.10	51.40
14/055	630	230.0	10000	200	16.88	35.50
14/056	630	204.0	30000	809	20.66	37.27