Master Thesis:

Hot cracking characterization in dissimilar welds of C-Mn Steel and Ni-base alloy

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CHAPTER 1
1.0 INTRODUCTION

The Organization of Petroleum Exporting Countries (OPEC) World Outlook (WOO) provides projections for the medium-term (to 2018) and long-term (to 2035) on an annual basis for oil demand and supply. It demonstrates that oil will continue to play a major part in satisfying the world’s growing energy needs. As the global energy landscape progressively changing due to increases in the population, the demands of the fossil fuels also increase. Over the projection period 2010-2035, energy demand in the Reference Case\(^1\) increase by 52%. Fossil fuels accounted for 82% of energy supply in 2010 and constitute 80% of the global total by 2035. Table 1.1 shows statistically the prospect of energy demand until 2035. In addition, Figure 1.1 depicts the demands for fossil products in 2012 and 2035. Consequently, these statistics reflect that oil and gas industry and all industries related to the oil and gas, are still in development. This development has two main aspects which are finance, technology.

<table>
<thead>
<tr>
<th>Energy Type</th>
<th>Levels (mboe/d)</th>
<th>Growth % p.a.</th>
<th>Fuel shares %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2010</td>
<td>2020</td>
<td>2035</td>
</tr>
<tr>
<td>Oil</td>
<td>81.2</td>
<td>89.7</td>
<td>100.2</td>
</tr>
<tr>
<td>Coal</td>
<td>69.8</td>
<td>84.9</td>
<td>104.0</td>
</tr>
<tr>
<td>Gas</td>
<td>54.8</td>
<td>69.0</td>
<td>99.8</td>
</tr>
<tr>
<td>Nuclear</td>
<td>14.3</td>
<td>16.0</td>
<td>21.6</td>
</tr>
<tr>
<td>Hydro</td>
<td>5.8</td>
<td>7.4</td>
<td>10.1</td>
</tr>
<tr>
<td>Biomass</td>
<td>24.4</td>
<td>28.0</td>
<td>35.2</td>
</tr>
<tr>
<td>Other renewables</td>
<td>1.8</td>
<td>3.6</td>
<td>10.7</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>251.9</td>
<td>298.6</td>
<td>381.7</td>
</tr>
</tbody>
</table>

Table 1.1 World supply of primary energy in the reference case [OPEC WOO].

![Figure 1.1 Global product demand, 2012 and 2035 [OPEC WOO].](image)

Offshore pipe laying is coupled strongly to oil and gas industry for the extraction and transportation of hydrocarbons from the sea bed. These pipes have two dissimilar layer. The inner layer is manufactured from a corrosion resistance alloy (CRA), however, the outer layer is

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\(^1\) Reference Case: It is primarily constructed using the output from OPEC’s world energy model (OWEM). This is based on the world policies for energy supply and consumption.
normally carbon-manganese steel. The purpose for considering the CRA as the inner layer of the pipe is the corrosivity of the oil and gas (hydrocarbons). These pipes can be manufactured in two different ways. If the inner and outer layer of the pipe bonded metallurgically, they are called clad pipes. If these two layers are bonded mechanically, they are called line pipes. During the pipe laying, the pipe’s joints are first welded and then coated at the vessel. Allseas Company is one of the pioneers in pipe laying industry. The pipes with length of almost 12 metre are welded together while they laid down at the sea bottom. Allseas has developed a semi-automatic welding system (Phoenix) that provides superior welding quality. A “robot” moves along the pipe groove over a belt. The welding material is fed continuously from a reel by a manipulator arm into the weld. The welding in clad or line pipes can be considered to be dissimilar welds since these pipes consist of two dissimilar metal layers. Besides, generally in welding technology, the principle to choose appropriate filler metal is to keep the consumable the same as the base metal in order to have a reasonable chemical and physical compatibility between the filler and base metal. Consequently, the possibility for the formation of microstructure defects is expected to be minimized. In practice, for the clad/line pipes often superalloys or stainless steel filler wires are used for the whole gap since these filler metals showed an remarkable strength, fracture toughness and corrosion resistivity [1,9]. However, these wires are expensive and indeed incompatible with outer layer (carbon-manganese steel) of the pipes. In order to apply and investigate the just mentioned principle for the clad/line pipes two various filler wires should be used for each layer of the pipe, which is a superalloy for inner part and a low carbon steel filler wire for the outer part of the pipe respectively. Figure 1.2 schematically depicts the structure of the two filler metals and associating pipe layers at the groove.

As can be seen from Figure 1.2, two different filler materials I and II are applied. The compatibility of these two materials in terms of chemical and physical properties is the most important factor to avoid or at least minimizing the introduction of microstructural defects in the weld metal with the focus on the filler material II. In order to investigate the compatibility and mixing phenomenon between filler materials I and II, a specific welding test geometry is designed to simplify the complex situation of the real weld. This is represented by Figure 1.3 in which a steel substrate is cladded first with a Ni-base filler wire (Layer 1/ wire I) on which the second layer deposited with low carbon steel wire (Layer 2/ wire II). By depositing these two layers, it is possible to study the various concepts during evolution of the microstructure such as; mixing, composition, solidification behavior and possible microstructure defects mainly cracks.
Figure 1.3 Schematic of filler wires deposition.

An preliminary investigation has been done on dissimilar welds of superalloy Inconel 625 as the filler wire (I) and low carbon steel as the filler wire (II) by depositing these two wires as it is shown in Figure 1.3. Following the deposition of the first layer (Inconel 625) by gas metal arc welding (GMAW) the steel wire was deposited on top of the first layer. This has been done in order to evaluate the microstructure of both layers. The investigation showed that cracking occurred in different regions of the second carbon steel layer. The cracks were characterized by optical and electron microscopy. Optical microscopy revealed the diluted dendritic microstructure of the steel weld bead and the position of cracks relative to the weld bead. The study showed that hot cracks (i.e. solidification and liquation cracks) were found respectively at the center of the fusion zone and heat affected zone regions of the subsequent weld passes. Besides, local chemical analysis of the dendritic microstructure showed segregation of low melting point elements to the interdendritic region and the formation of eutectic phases at this regions. These eutectic phases lead to the formation of the heat affected zone (HAZ) liquation cracks during subsequent welding passes.

In this study, based on the results achieved from the preliminary investigation, a systematic study was conducted to reveal the mechanisms leading to the hot crack formation, in order to avoid cracking or minimizing the crack susceptibility in the microstructure. The welding technique selected is gas tungsten arc welding (GTAW ) instead of GMAW in order to control the process more accurately and minimizing the dilution of steel by the nickel layer. Furthermore, beside using Alloy 625 as one of the filler wires for the first layer, an almost pure nickel consumable is deposited to avoid the formation of any second phase particles at the interdendritic region of the microstructure and secondly to compare the microstructure of the diluted steel layer by these two Ni-base filler wires (Alloy 625 and pure nickel). Characterization and assessment of the cracks is carried out by optical and scanning electron microscope (SEM) equipped with secondary, backscattered electron and energy dispersive spectroscopy.

The structure of this thesis is as follows. An overview on the background information of existing knowledge regarding to the dissimilar welds of nickel base alloys with carbon steel. Secondly, finding the optimum welding parameters in order to minimize the heat input while a good deposition still be feasible. The thermal cycle analysis would help to have better understanding of the heat dissipation role in the microstructure evolution by the means of experimental analysis and modeling. Besides, mapping the corresponding phase diagrams based on the microstructure composition by the associating thermodynamic software (Thermo-calc). This can help to predict the phase transformation sequences and subsequently the stable phases at the room temperature. Moreover, characterization of the microstructure features by optical and scanning electron microscopy in order to understand the possible mechanisms in the formation of the microstructural cracks.
2.0 BACKGROUND

2.1 Fusion arc welding
During arc welding a pool of molten metal is formed directly below the heat source. The shape of this molten pool is influenced by the flow of both heat and liquid metal, associated with energy (heat) transport, with melting occurring ahead of the heat source and solidification when the heat source has passed. The heat input determines the volume of molten metal and in a case of welding process with filler wire addition, dilution and weld metal composition, as well as the thermal conditions under which solidification take place. Beside solidification, the crystal growth rate which is geometrically related to welding travel speed and weld pool shape is affected by the heat input. Hence, weld pool shape, weld metal composition, cooling rate and growth rate are all factors interrelated to heat input which will affect the solidification microstructure. Here in the following section, specifically the heat input of the gas tungsten arc welding GTAW is explained.

2.2 Heat input
One of the most important characteristics of the arc is its non-linear voltage distribution. This causes a disproportionality large fraction of the total arc energy generated in the arc to be transferred to the cathode and the anode. Consequently, this phenomenon makes the arc an effective heat source in welding. In gas tungsten arc welding polarity can be in three modes; Direct-Current Electrode Negative (DCEN), Direct-Current Electrode positive (DCEP) and Alternating Current (AC). In this study, the most common polarity in GTAW which is DCEN, is described. In this process the electrode (tungsten) is connected to the negative terminal of the power supply. Electrons are emitted from the tungsten electrode and accelerated while travelling through the arc. A significant amount of energy, called the work function, is required for an electron to be emitted from the electrode. When the electron enters the work piece, an amount of energy equivalent to the work function is released. This is why in GTAW with DCEN more power (about two-thirds) is located at the work end of the arc and less (about one-third) at the electrode end. Consequently, a relatively narrow and deep weld is produced.

The amount of heat transferred per unit time to the anode \( Q_a \) can be calculated using the energy balance. For the anode and its immediate surroundings (anode fall zone), this leads to following equation \([2]\):\
\[
Q_a = V_a I + C_a V_{col} I - \phi_a I - \frac{3k(T_{col} - T_a)}{2e} I,\quad (2.1)
\]

The term \( V_a I \) represents the energy produced in the anode fall zone; \( C_a V_{col} I \) is the fraction of the energy produced in the arc column that is transferred to the anode by heat conduction, convection and radiation; \( \phi_a I \) is the energy required to emit the electrons from the anode; The term \( 3k(T_{col} - T_a)/2e \) is the energy required to raise the temperature of the electrons from the anode temperature \( T_a \) to the arc column temperature \( T_{col} \).

An important quantity in arc welding is the process efficiency \( \eta_p \). This is the fraction of the total arc energy that is transferred to the work piece (either directly or via the consumable electrode). For the different variants of the arc welding process different efficiencies can be defined. However, particularly for GTAW with DCEN polarity the process efficiency can be defined:

\[
\eta_p = \frac{\alpha}{V_I} X 100\%\quad ,\quad (2.2)
\]
Table 2.1 shows typical values of $\eta_p$ for different variants of the arc welding process.

<table>
<thead>
<tr>
<th>Arc welding process</th>
<th>$\eta_p$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GTA welding (electrode negative)</td>
<td>50-80</td>
</tr>
<tr>
<td>GTA welding (electrode positive)</td>
<td>40-70</td>
</tr>
<tr>
<td>Plasma arc welding</td>
<td>50-80</td>
</tr>
<tr>
<td>SMA welding</td>
<td>70-90</td>
</tr>
<tr>
<td>GMA welding</td>
<td>60-90</td>
</tr>
<tr>
<td>SA welding</td>
<td>90-100</td>
</tr>
</tbody>
</table>

The melting efficiency $\eta_m$ is the fraction of the total arc energy, which is required to heat and melt the metal (base and filler metal). The melting efficiency can be estimated using the following equation:

$$\eta_m = \frac{qAv}{V} \quad , \quad (2.3)$$

where $q$ represents the amount of energy necessary to heat and melt a unit volume of the metal, $A$ is the surface area of the transverse cross-section of the weld, and $v$ is the travel speed (the speed of the welding arc relative to the work piece). The melting efficiency increases with the energy density of the arc, because the higher the energy density, the faster the metal is heated and the less heat will be lost by heat conduction. Table 2.2 shows melting efficiencies and energy densities of some welding processes.

Another important quantity in arc welding is the heat input. The heat input determines the peak temperature and the cooling rate in and around the weld, and in this way influences the structure and properties of the welded joints that will be discussed in section 2.6 and 2.7. The heat input $W$ is defined as the amount of heat transferred to the work piece per unit length of the weld, and can be written as:

$$W = \frac{\eta_p V I}{v} \quad , \quad (2.4)$$

The melting efficiency $\eta_m$ and energy density of some welding processes are shown in Table 2.2.

<table>
<thead>
<tr>
<th>Welding process</th>
<th>$\eta_m$ (%)</th>
<th>Energy density (MW/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxyfuel gas welding</td>
<td>2-10</td>
<td>10 - 10²</td>
</tr>
<tr>
<td>GTA welding</td>
<td>20-40</td>
<td>10² - 10³</td>
</tr>
<tr>
<td>Plasma arc welding</td>
<td>40-60</td>
<td>10³ - 10⁴</td>
</tr>
<tr>
<td>Laser welding</td>
<td>90-100</td>
<td>10⁴ - 10⁵</td>
</tr>
<tr>
<td>Electron beam welding</td>
<td>90-100</td>
<td>10⁶ - 10⁷</td>
</tr>
</tbody>
</table>
2.3 Power Density Distribution of Heat Source

In gas tungsten arc welding process (GTAW) with direct current electrode negative (DCEN), the shape of the electrode tip affects both the shape and power density distribution of the arc. As the electrode tip becomes blunter, the diameter of the arc decreases and the power density distribution increases as illustrated in Figure 2.1. Glickstein showed [4], in Figure 2.2, that the arc becomes more constricted as the conical tip angle of the tungsten electrode increases. Moreover, Savage et al. [5] observed that, under the same welding parameters such as current, speed and arc gap, the weld bead depth-width ratio increases with increasing tip angle of the conical tip of the tungsten electrode. Key [6] also illustrated a similar effect of the tip angle, at least with argon shielding, as shown in Figure 2.3.

**Figure 2.1** Effect of electrode tip angle on shape and power density distribution of gas-Tungsten arc [3].

**Figure 2.2** Effect of electrode tip angle on shape of gas Tungsten arc. Reprinted from Glickstein [4].
2.4 Fluid flow in the weld pool

In fusion welding processes, part of the base material is melted. In welding processes that no consumables (filler) are used, the weld pool consists of molten base metal. However, in welding processes with consumables, the weld pool consists of a mixture of molten base material and molten consumable. The composition of this mixture is determined by the mixing ratio $M$, which is defined as:

$$M = \frac{\text{weight of molten base material}}{\text{total weight of molten material}} \times 100 \quad (2.5)$$

The value of $M$ depends on the welding process and on the welding parameters used.

The shape and size of the weld pool have an important influence on the solidification microstructure, due to the fact that during solidification the primary grains of the weld metal grow perpendicular to the fusion boundary. Both shape and size of the weld pool depend on the material physical properties (thermal conductivity, specific heat) and the welding processes with their associating welding parameters.

In particular the heating power and the travel speed play a significant role. It is widely accepted, that by increasing the heating power the width and depth of the weld pool increase, whereas with increasing travel speed, the weld pool becomes more elongated: the shape changes from a circle (zero travel speed) via an ellipse to a tear shape.

Generally, in the weld pool several physical and chemical phenomena occur. Since during fusion welding processes high heating and cooling rates take place, equilibrium situations are usually not reached.

One of the most important mechanisms is fluid flow in the weld pool due to convection process that affects the shape and dimension of the weld pool.

Usually, two different flow patterns are distinguished, see Figure 2.4:

- Radial outward directed flow, which leads to a relatively wide and shallow weld pool;

- Downward directed flow, which results in a relatively narrow and deep weld pool;
2.4.1 Driving forces for fluid flow

The driving forces for fluid flow in the weld pool are the buoyancy force, the Lorentz force, the shear stress induced by the surface tension gradient at the weld pool surface and the shear stress acting on the pool surface by the arc plasma. Moreover, the arc pressure can also apply force on the pool surface, but its effect on fluid flow is small, especially below 200(A) [7, 8], which is usually the case for GTAW process. The driving forces causing fluid flow in the weld pool, shown in Figure 2.5, are described next [3].

Figure 2.5 Driving forces for weld pool convection: (a, b) buoyancy force; (c, d) Lorentz force; (e, f) shear stress caused by surface tension gradient; (g, h) shear stress caused by arc plasma [3].
• **Buoyancy Force**

The density of liquid metal ($\rho$) decreases with increasing temperature ($T$). As the heat source is located above the centre of the pool surface, the liquid metal is warmer at point a and cooler at point b. Point b is near the pool boundary, where the temperature is lowest at the melting point. As shown in Figure 2.5a, gravity causes the heavier liquid metal at point b to sink. Consequently, the liquid metal falls along the pool boundary and rises along the pool axis, as shown in Figure 2.5b.

• **Lorentz Forces**

In the case of arc welding for instance GTAW process with DC electrode negative (Cathode), the electric current in the work piece converges toward the tungsten electrode and hence near the centre of the pool surface. This converging current field, together with the magnetic field it induces, causes a downward and inward electromagnetic (Lorentz-) force, as shown in Figure 2.5c. As such, the liquid metal is pushed downward along the pool axis and rises along the pool boundary, as shown in Figure 2.5d. The area on the pool surface where the electric current passes through is called the anode spot ($\Pi b^2$, where $b$ is the effective radius of the current density distribution).

The smaller the anode spot, the more the current field converges from the work piece (through the weld pool) to the anode spot, and hence the greater the Lorentz force becomes to push the liquid metal downward, which leads to the higher depth to width ratio [3].

• **Shear Stress induced by Surface Tension Gradient**

In the absence of a surface-active agent, the surface tension ($\gamma$) of the liquid metal decreases with increasing temperature ($T$), namely, $\frac{\partial \gamma}{\partial T} < 0$. As shown in Figure 2.5e, the warmer liquid metal with a lower surface tension at point $a$ is pulled outward by the cooler liquid metal with a higher surface tension at point $b$. In other words, an outward shear stress is induced at the pool surface by the surface tension gradient along the pool surface. This causes the liquid metal to flow from the centre of the pool surface to the edge and return below the pool surface, as shown in Figure 2.5f. Surface-tension-driven convection is also called thermocapillary convection or Marangoni flow [3].

• **Shear Stress Induced by Plasma Jet**

In the case of arc welding, the column of the arc is composed of neutral particles, such as atoms and molecules (both in the excited and in the non-excited state), and electrically charged particles, such as electrons and ions. The negative particles (electron and negative ion) always move towards the anode under the influence of the electric field, while the positive particles move towards the cathode. As it has already mentioned above, an important property of the arc column is its electric neutrality. This condition of the arc is always referred to as plasma. The plasma forms a flow in the arc, which is directed from the electrode towards the weld pool. When the arc reaches the surface of the weld pool, this plasma spreads out in radial direction along the surface of the weld pool. Therefore, it forms a drag force, which causes outward directed flow, as shown in Figure 2.5g. This drag force causes the liquid metal to flow from the centre of the weld pool surface to the pool edge and returns below the pool surface, as shown in Figure 2.5h [2].

In all of the driving forces that are briefly mentioned above for fluid flow in the weld pool, it appears that in most cases the plasma and buoyancy forces are negligibly small and only the Maragoni-flow and Lorentz-flow should be considered. In addition, which of these two driving forces are dominant in a specific situation depends on the welding process, the welding parameters and the chemical composition of the weld metal. Generally, in most situations
Maragoni-flow plays the dominant role. However, in the case of arc welding Lorentz-flow usually dominants the flow pattern.
2.5 Dissimilar welding

Ni based filler metals are often used to weld dissimilar alloys, particularly steels, stainless steels and Ni base alloys. Generally, these alloys provide good corrosion resistance, strength and fracture toughness relative to steel fillers or even austenitic stainless steels [9]. The proper selection of filler metals for these dissimilar welding combinations is essential. Understanding of the metallurgical behaviour of dissimilar welds can guide us to prevent problems during its fabrication or service life time. This section describes the welding mixture phenomenon and weldability associated with dissimilar welds that involve Ni-base alloys as filler metals [10].

2.5.1 Application of dissimilar welds

There are several engineering advantages that Ni-base alloys provide for dissimilar welding combinations (e.g. steel to austenitic stainless steel and/or austenitic stainless steel to Ni-base alloys). In general, these applications take advantage of several inherent properties of Ni-base alloy welding materials that make them ideal choices for specific types of applications. Each application in which the Ni-base welding material is used to weld other base metals usually takes advantage of unique characteristics of the as-cast or as-deposited weld metal that allows it to provide equal to or better properties than that of the wrought base metal. In the first case, Ni-base filler metals (normally the solid-solution strengthened alloys) are used since these kind of strengthened alloys provide quite the same strength relative to base metals compare to precipitation strengthened alloys. They are used to weld overlay structural materials, such as Cr-Mo alloy steel applied in water wall tubing in power boilers. In another case, Ni-base filler metals are used to join low alloy steels to austenitic stainless steels in power plant applications requiring elevated temperature creep and thermal-fatigue resistance. When austenitic stainless steels are welded directly to carbon steels either by using autogenous “pressure welding” or by using an austenitic stainless steel filler metal, the difference in coefficient of thermal expansion (CTE) can lead to fatigue and creep failures in the HAZ of the carbon steel after extended exposure at elevated temperature. The use of a Ni-base filler metals provides a gradation of CTE across the weld joint that better distributes stresses resulting from CTE differences between the carbon steel and stainless steel at elevated temperatures. There are also metallurgical advantages realized by the use of Ni-base filler metals for this dissimilar combination. In addition, it has to be considered that Ni-base alloys have good corrosion resistivity against corrosive environment. Therefore, the Ni-base weld metal can overcome the well-known pitting and crevice corrosion when it used to weld lower corrosion resistant base metal [10].

2.5.2 The relation between Fusion Zone composition and process parameters

During fusion welding of dissimilar alloys an intermediate chemical composition will be formed in the weld metal. The chemical composition will have a significant influence on microstructure evolution of the weld pool, and its associating corrosion resistant, mechanical properties, and weldability. Thus, it is useful to understand how variations in processing parameters affect the weld metal composition for dissimilar weld applications. The final fusion zone chemical composition will depend on the individual compositions of the base metal and filler metals and the degree of dilution of the filler metal by the base metal(s). It should be mentioned that dilution is defined based on the change of filler metal composition by mixing with the base metal. Figure 2.6 express the simplest schematic form of dilution equation, where a single pass weld is deposited onto a base metal of a different composition. The degree of mixing for the simple case of Figure 2.6 is equated below by the dilution equation, D

\[ D \text{ (Dilution)} = \frac{A_{bm}}{A_{bm} + A_{fm}}, \]  

(2.6)
where \( A_{bm} \) is the melted cross-sectional area of the base metal and \( A_{fm} \) is the cross-sectional area of the deposited filler metal. The concentration of any element \( i \) in the fusion zone \( (C_{ifz}) \) can be determined through the knowledge of dilution and concentration of the element in the base metal \( (C_{ibm}) \) and filler metal \( (C_{ifm}) \) via:

\[
C_{ifz}^i = DC_{ibm}^i + (1-D)C_{ifm}^i \tag{2.7}
\]

This assumes that sufficient mixing occurs in the liquid state so the macroscopic composition gradients are not present in the weld deposit. In previous works by [11,12] it was shown that concentration gradients in welding of dissimilar metals only exist near the fusion boundary over distances on the order of a few hundred microns.

In the equation 2.7 the fusion zone composition will vary linearly with dilution from the filler metal composition (for \( D=0 \)) to the base metal composition (for \( D=1 \)). The welding process and its associating welding parameters strongly influence the dilution level [13,14]. In simple single pass welds, energy and mass balance considerations can be used to show that the dilution depends on the heat source power \( (VI) \) and the volumetric filler metal feed rate \( (V_{fm}) \) as:

\[
D=\frac{1}{1+\eta_p \eta_m VI-E_{bm}E_{fm}} \tag{2.8}
\]

where \( \eta_p \) and \( \eta_m \) are the energy transfer and melting efficiencies and \( E_{bm} \) and \( E_{fm} \) are the melting enthalpies of the base metal and filler metal. Values of \( \eta_p \) for different welding processes have been measured by calorimetric means and are mentioned in Table 2.1. However, the values for \( \eta_m \) that were already mentioned in the Table 2.2, can be estimated based on the welding parameters (e.g. current, travel speed) and intrinsic thermal properties of the materials such as heat capacity and melting enthalpy. The quantity \( \eta_p \eta_m VI \) represents the melting power, which is the fraction of heat source power that is actually used for melting the base metal and the filler metal.
Figure 2.7 Effect of filler metal feed rate and power on dilution for stainless steel [11].

Figure 2.7 depicts a graphical plot of Equation 2.8. This plot can guide us through the influence of process parameters on dilution. The filler metal feed rate ($V_{fm}$) is plotted as a function of the melting power ($\eta_p \eta_m V I$) and the slopes which correspond to various calculated dilution levels are plotted in 10% increments. In the Figure, Type 308 stainless steel is deposited onto carbon steel. Besides, a boundary between an “inoperable range” and “operable range” is also plotted. The line with 0% dilution represents the condition that no mixing occurs between base metal and filler metal. Measured data for deposition of Type 308 stainless steel onto carbon steel by the submerged arc welding process are also plotted for validation of the calculations. In this case, the extra melting power cannot be absorbed by the filler metal if the filler metal feed rate is fixed, so the base metal absorbs the extra melting power which leads to an increase in the melting rate of the base metal with an accompanying increase in dilution. It can be seen from Figure 2.7 that with a feeding rate of zero, 100% dilution in weld pool is achieved autogenously. However, for constant melting power, by increasing the feeding rate the dilution in weld pool decrease. In this case, dilution decreases because the cross sectional area of deposited filler metal increases. It should be mentioned that the filler metal consumes a larger portion of the total power and less energy is available to melt the base metal. As a result, the base metal volumetric melting rate decreases and dilution is reduced. The trend shown in Figure 2.7 would be similar for Ni-base filler metals. This is of particular importance in processes such as gas tungsten arc welding (GTAW) and plasma arc welding (PAW) where wire feed rate is independent of welding current. Power ratio and energy density equations have been developed by Gandy et al. to describe this behaviour [15]. Although the heat source travel speed does not appear in Equation 2.8, it affects the dilution indirectly by affecting the melting efficiency. Melting efficiency increases with increasing travel speed. [16, 17] This increase in melting efficiency produces a concomitant increase in dilution via Equation 2.8. This trend has been observed experimentally. [11, 18] The consumable electrode arc processes generally do not permit independent control over the filler metal feed rate and power. For these processes, the filler metal feed rate is set and the electrical characteristics of the power source control the required current (power) level. In the non-consumable electrode processes, the filler metal feed rate and power can be controlled independently, and dilution is generally easier to control with these processes such as gas tungsten arc cold wire feed process. Each welding process affects the dilution differently since it controls the energy transfer and melting efficiency values ($\eta_p$ and $\eta_m$). Therefore, welds made with different welding processes under identical welding parameters will generally produce
different dilution levels. Moreover, weld beads with different filler and base metals under identical processing conditions will also produce different dilution levels due to changes in the melting enthalpy values ($E_{bm}$ and $E_{fm}$). There are other factors that affect the dilution level in multipass welds. For example, an increase in the degree of overlap between adjacent passes will decrease the dilution because fewer base metal is melted. An increase in the preheat or interpass temperature will increase the dilution because it reduces the amount of energy required for the melting the base metal, so increasing the base metal melting rate. Finally, choosing the optimum welding parameters for process is one of the most significant factors to reach the desired microstructure.

The large composition differences between the Ni-base alloys and steels can result in a wide range of weld metal microstructure and properties. The presence of high amounts alloying elements with different solubility in the solid phase and thus different segregation behaviour can lead to formation of second phase constituents. This topic will be discussed in more detail in section 2.8. Often these phases cause embrittlement of the microstructure and generally decrease the weldability of the parent metal. In addition, dissimilar welds between Ni-base alloys and steels form a compositional transition region between the diluted weld metal and the base metal. In many cases, this transition region may contain a layer of martensite as a result of carbon diffusion from the steel into the nickel weld pool particularly at the fusion line [10].
2.5.3 Weld metal microstructure in dissimilar welds

In order to determine the composition of the weld metal particularly in dissimilar welds between steel and Ni-base alloys, the dilution Equation 2.6 can be used. In the case that the filler metal is a Ni-base alloy and dilution is not extremely high, the weld metal will be fully austenitic as a result of the high concentration of nickel with a fcc structure. Meanwhile, in the presence of alloying elements the possibility of the formation of a second phase particle is high because of segregation during the solidification process. For the determination of the microstructure and the approximate chemical composition of the weld metal, the Schaeffler diagram is widely used (see Figure 2.8). This diagram was developed primarily to predict the weld metal constitution (phase balance) of stainless steels, but can be applied to dissimilar welds with carbon steels and stainless steels.

In this figure the vertical axis shows the Nickel equivalent but cannot show the chemical composition of the filler material because of the limitations in the size of Figure. However, the virtual tie lines can be used to determine the effect of the base metal on dilution and provide an insight into the microstructure that may form in the composition transition zone adjacent to the fusion boundary. For instance, as it is hypothetically depicted in Figure 2.8, a nickel base filler material has been used to weld a austenitic stainless steel and carbon steel. Based on the level of dilution the structure of the weld metal can be altered. For dissimilar welding of carbon steel with Ni-base filler wire, if the dilution be higher than almost 75% some martensite will form in the weld metal and above 85% the microstructure will be fully martensitic. However, in the case of austenitic stainless steel no matter what the level of dilution is, the weld metal will be fully austenitic. Generally, dilution levels above 50-60% are difficult to achieve in practice. It should be considered that a composition transition region at the fusion boundary exist that confines the composition range represented by means of Schaeffler diagram and predicts that some martensite must be present at the fusion boundary. The Schaeffler diagram only predicts formation of three major phases austenite (fcc), ferrite (bcc) and martensite (bct) [10]. Other constituents such as carbides, Laves phase, Sigma phase and etc. that can potentially be formed in these dissimilar welds are not predictable by the Schaeffler diagram. The formation of these second phase constituents can be of particular interest, especially on dissimilar combinations with carbon steels where the composition of the diluted filler metal can introduce high levels of carbon. These second phase constituents can cause microstructure defects during solidification that will be discussed in more details in the section 2.8.

![Figure 2.8 The Schaeffler diagram with tie lines to a Ni-base alloy superimposed for a carbon steel and austenitic stainless steel. The percentages along the tie lines represent the dilution level [10].](image-url)

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2.6 Solidification behaviour of Ni-base alloys

Generally, in order to describe the solidification behaviour and microstructure evolution in the fusion zone of any alloy, there are some concepts that necessarily should be considered. These basic concepts are solute redistribution, solidification modes, constitutional supercooling, microsegregation and banding, the dendrite-arm or cell-spacing and finally the solidification path. As it has been already mentioned, the microstructure assessment of dissimilar weld between the Ni-base alloy and C-Mn is the main goal of this project. Particularly, the microstructure evolution and weldability of C-Mn steel layer after deposition on top of a Ni-base alloy is a point of interest. In addition, cracking susceptibility and characterization of the cracks that are formed during the deposition of C-Mn steel on Ni-base alloys should be addressed in order to find the solution to avoid such defects. In the dissimilar welding involving Ni-base alloys there are several investigations by Lippold and DuPont regarding the characterization of their solidification behaviour and cracks in Ni-base alloys. However, hardly any literature is available regarding the solidification and microstructure evolution in C-Mn steel layers after deposition on top of the Ni-base alloys.

It should be mentioned that as the C-Mn steel layer is highly diluted by the Ni-base layer. The steel layer solidifies primarily austenitic. Therefore, the similar solidification behaviour as in the Ni base alloys is seen in the diluted steel layer. Consequently, in this section based on the existing literatures, the solidification behaviour of Ni-base weld metals is explained since the similar behaviour has been observed in the diluted C-Mn steel weld.

2.6.1 Solidification modes

During solidification of a pure metal the S/L interface is usually planar, unless severe thermal undercooling is imposed. During the solidification of an alloy, however, the S/L interface and hence the mode of solidification can be planar, cellular, or dendritic depending on the solidification condition and the material system involved. The four basic types of the S/L interface morphology observed during the solidification are shown in Figure 2.9 [3].

Figure 2.9 Basic solidification modes (magnification 67x): (a) planar solidification of carbon tetrabromide: (5); (b) cellular solidification of carbon tetrabromide with a small amount of impurity (5); (c) columnar dendritic solidification of carbon tetrabromide with several percent impurity (5); (d) equiaxed dendritic solidification of cyclohexanol with impurity [19]. Reprented from [20].
2.6.2 Constitutional supercooling

During welding of alloys, the solidification front will move with a certain uniform speed, as indicated in Figure 2.10. Because the solubility of the alloying element is smaller in the solid metal than in the liquid metal, solute atoms will be pushed from the solid to the liquid, which will result in an enrichment of the alloying element in the liquid in front of the solid/liquid interface. This enrichment will be opposed by diffusion of the alloying element in the liquid metal. A stationary situation will be established, when the amount of alloying element pushed from the solid into the liquid metal per unit time, is equal to the amount transferred per unit time by diffusion, assuming that flow of the liquid metal along the solidification front can be neglected. The concentration of the alloying element in the liquid metal as a function of the distance to the solid/liquid interface is shown schematically in Figure 2.10 (b).

For equilibrium, expressed by the hypothetical phase diagram depicted in Figure 2.10(a), it is assumed that diffusion in the solid is not limited. In the practice of welding, however, the cooling rate is high and diffusion in the solid plays a minor role, which will result in segregation. By pushing the alloying elements in front of S/L interface, the concentration of alloying elements increase which leads to a decrease in liquidus temperature $T_L$. The value of $T_L$ as function of the distance to solid/liquid interface can be obtained by combining Figure 2.10(b) with the phase diagram which is shown in Figure 2.10(c). The liquid phase is stable in the boundary layer when the temperature is above the liquidus temperature, while if the temperature be below the liquidus temperature, solid and liquid both should coexist. Therefore, the planar S/L interface should breakdown to a cellular or dendritic nature so that solid cells or dendrite cores can coexist with the intercellular or interdendritic liquid (Figure 2.11a and b). The shaded area below the liquidus line in Figure 2.10(a), which depends on $D_L$ and R, indicates the corresponding region of constitutional supercooling in the phase diagram. It is apparent that this region lies in the solid and liquid region of the phase diagram, where the solid and liquid phases should coexist.

Figure 2.10 Constitutional supercooling: (a) phase diagram; (b) composition profile in liquid; (c) liquidus temperature profile in liquid [3].
As is shown in Figure 2.10(b) the thickness of the boundary layer at the steady state is $D_l/R$. Consequently, the slope of the tangent to the liquidus line at the S/L interface is $\Delta T/(D_l/R)$ or $R\Delta T/D_l$. The criteria to ensure that planar interface is formed during solidification should be that the actual temperature gradient $G$ at the S/L interface must be at least equal to $R\Delta T/D_l$ or:

$$G/R \geq \Delta T/ D_l$$

This is the steady-state form of the criterion for planar growth. Based on this criterion, the higher the temperature gradient $G$ and the lower the growth rate, the easier for a planar S/L interfaces to be stable. However, the higher the solidification temperature range ($\Delta T$) and the lower the diffusion coefficient, the planar S/L interface becomes more instable.

Figure 2.11 Nonplanar solidification structure in alloys. (a) Columnar dendrites in a Ni alloy. From New Trends in Materials Processing [21]. (b) Three-dimensional view of dendrites in a Ni-base superalloy. Reprinted from International Trends in Welding Science and Technology [22].
Figure 2.12 shows that the solidification mode changes from planar to cellular, to columnar dendritic, and finally to equiaxed dendritic as the degree of constitutional supercooling continues to increase. The region where dendrites (columnar or equiaxed) and the liquid phase coexist is often called the mushy zone. It is interesting that at a very high degree of constitutional supercooling (Figure 2.12 (d)) the mushy zone can become so wide that it is easier for equiaxed dendrites to nucleate than for the columnar dendrites to stretch all the way across the mushy zone.

Figure 2.12 Effect of constitutional supercooling on solidification mode: (a) planar; (b) cellular; (c) columnar dendritic; (d) equiaxed dendritic. (S, L and M denote solid, liquid and mushy zone respectively [3].
2.6.3 Phase Transformation Sequence

The phase transformation sequence in steel that is deposited on top of the Ni-base substrate is basically dependent on the amount of dilution and mixing from substrate into the top weld. The high amount of nickel that has diluted the steel causes stabilization of the austenite phase in the steel layer. As it has been already mentioned in section 2.4 various parameters can affect the amount of the dilution. In this situation the most helpful solution to understand the phase transformation sequence is the use of phase diagrams. Although many issues and challenges arise in the calculation of ternary phase diagrams, several thermodynamic modelling and experiments have been done by different groups [23-26] for ternary phase diagrams by using CALPHAD methods and mass spectrometric measurements. These investigations could help predicting the sequence of the phase transformation from liquidus temperature to room temperature. The phase transformation plays a significant role for characterizing cracks in the microstructure and also can act as a solution to prevent the formation of such cracks (see section 2.8). In this project, for the plate one (deposited with Alloy 625) the layers that are deposited by welding have three dominant elements being Fe, Ni and Cr. The high concentration of iron comes from the low carbon steel wire that is deposited on the Ni-base alloy substrate. Besides, nickel and chromium dilute the top steel layer during welding the substrate. However, for the plate two (deposited with pure Ni), the dominant elements are Fe and Ni. Consequently, for the plate one the ternary phase diagram that is to be considered is the Fe-Ni-Cr phase diagram and for the plate two the binary Fe-Ni phase diagram. Specifically, for the ternary phase diagram of Fe-Ni-Cr several articles have been published to calculate this ternary phase diagram system [27]. It has been shown that the present thermodynamic models available cannot well represent the low temperature (T<1073 K) phase equilibria. However, above this temperature, it is found that, in the existing thermodynamic assessments, the phase equilibria were well described. It should be mentioned that the most technical issues in steel processing are taking place above this temperature. Tomiska [23] used substantially more accurate thermodynamic data from experimental investigation to enhance the compatibility of the modelling and experimental results for calculation of the phase diagram. His new thermodynamic descriptions reduced the magnetic contribution terms to the expressions of the pure species only. This new approach could yield a distinct improved overall-agreement with experimental data than the results of previous assessments.

In the present study, the aim is to reduce dilution to avoid cracks in the microstructure. Therefore, it is expected that the two most dominant elements of the ternary Fe-Cr-Ni would be first iron and secondly nickel. In this case, based on the existing ternary phase diagram with a dilution of 30% from the Ni-base alloy layer into the carbon steel weld the sequence of the phase transformation which is dependent on the nominal chemical composition is expected to be (see Table 2.3):

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Sequence of the phase transformation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe - Ni\textsubscript{eq} (24%) - Cr\textsubscript{eq} (11%)</td>
<td>L $\rightarrow$ L + $\delta$ (FCC) $\rightarrow$ $\delta$ $\rightarrow$ $\alpha$ (BCC, Fe-rich) + $\delta$ (2)</td>
</tr>
</tbody>
</table>

In Fe-Cr-Ni ternary phase diagram, there is a possibility for the formation of $\sigma$ phase that above 12% wt Ni this phase is suppressed [10].
2.7 Microstructural Evolution in the Fusion zone

As it has been already mentioned in section 2.5.1, the solid-solution strengthened Ni-base alloys are generally used for welding of steel to Ni-base alloys or to austenitic stainless steels. These alloys solidify as austenite even until the terminal stages of solidification. These Ni-base alloys remain austenitic at room temperature. Segregation during solidification of these alloys results in local variation in composition at the solidification subgrain level. In many alloys, the segregation of alloy and impurity elements can lead to the formation of a second phase (or phases) at the end of solidification, i.e. low melting point phases.

2.7.1 Substructure characteristics

- **Solidification Subgrain Boundaries (SSGBs)**

During solidification within a single grain, the finest (sub-grain) structure that can be resolved in a light optical microscope is an array of parallel hexagonal cells or dendrites with distorted cubic structure which are separated by ‘low-angle’ grain boundaries (less than five degrees). These are low angle boundaries since there is virtually no crystallographic misorientation across these boundaries and therefore also known as solidification subgrain boundary (SSGB). These boundaries are evident in the microstructure since their composition is different from that of the bulk microstructure. This difference is a result of microscopic solute redistribution, also referred to as Scheil partitioning. Dendrites typically grow along the three <100> easy growth directions in bcc and fcc structures. Figure 2.12 shows the schematic of dendritic easy growth directions in cubic structures [3, 28].

![Figure 2.13 Schematic of dendrites arm along the three easy growth direction for fcc and bcc.](image)

- **Solidification Grain Boundaries (SGBs)**

The solidification grain boundary (SGB) results from the intersection of packets, or groups of subgrains. Thus, SGBs are the direct result of competitive growth that occurs along the trailing edge of the weld pool. Each of these packets consisting of dendrites has their own crystallographic direction as a result of different growth direction. Therefore, as these packets intersect, they form a boundary with a high angular misorientation. These are referred to ‘high angle’ grain boundaries. This misorientation results in the development of a dislocation network along the SGB.

The SGB also has a compositional component besides its crystallographic component. This composition component is a result of the solute redistribution during solidification. This distribution can be modelled by macroscopic solidification and often leads to a high concentration of solute and impurity elements at the SGBs. These compositions (eutectic composition) are formed at the terminal stages of solidification along the SGBs that are low melting point liquid films. Subsequently, these low melting liquid films promote weld
solidification cracking. In Ni-base alloys the solidification cracking almost always occurs along the SGBs [10].

- **Migrated Grain Boundaries (MGBs)**

Migrated grain boundaries are mostly seen in fully austenitic weld metal. Basically, the MGB boundary is formed from solidification grain boundaries. As it has been already described above the SGB that forms at the end stages of solidification has both a crystallographic and compositional component. In some cases, when the crystallographic component of the SGB migrates away from the compositional component, a boundary with high angle misorientation (>30 degrees) is formed that is called a migrated grain boundary (MGB). The driving force for such a phenomenon is the same as for simple grain growth in base metals, a lowering of the boundary surface energy. The composition of the boundary varies locally, depending on the composition of the microstructure where it has migrated. It is possible that some segregation can occur along MGBs in the solid state, possibly by a “sweeping” mechanism. The most likely elements are interstitial fast diffusing elements (S, P, B, O, etc.). These elements are swept into the boundary. The formation of second phase particles at the end of solidification along SGBs or SSGBs, can lead to pinning the crystallographic component of the SGB to migrate away, thus preventing the formation of MGBs. The formation of MGBs and the pinning effect is an important part of the mechanisms for Ductility-dip cracking that will be discussed in more details in section 2.8.3. Figure 2.14 shows a schematic of migrated grain boundary [10].

![Figure 2.14](image-url) Schematic of boundaries observed in the weld metals that solidify as austenite [29].
2.7.2 Microsegregation during solidification

The weldability, mechanical properties and corrosion behaviour of Ni-base alloys are largely affected by the solidification behaviour and resultant microstructure of the fusion zone. The fusion zone solidification is controlled by the solute redistribution behaviour. This partitioning occurs across cells and dendrite arms. Solute redistribution of alloying elements in Ni-base alloys can most effectively be evaluated with the aid of the Brody-Flemings equation [30], which is given by:

\[
C_s = KC_0 \left[1 - \frac{f_s}{1 + aK}\right]^{k-1}, \quad (2.9)
\]

where \(C_s\) is the solid composition at the solid/liquid interface, \(C_0\) is the nominal alloy composition, \(f_s\) is the solid fraction, and \(K\) is the equilibrium distribution coefficient. The \(\alpha\) parameter in Equation 2.9 is the dimensionless diffusion parameter defined as:

\[
\alpha = \frac{D_s t_f}{L^2}, \quad (2.10)
\]

Where \(D_s\) is the diffusivity of solute in the solid, \(t_f\) is the solidification time (cooling time between the liquidus and terminal solidus), and \(L\) is half the dendrite arm spacing. The \(D_s t_f\) term shows the distance a solute atom can diffuse in the solid during solidification, while the half dendrite arm spacing, \(L\), represents the length of the concentration gradient [10]. Therefore, when \(D_s t_f \ll L^2\), the solute is able to diffuse only a small fraction of the total gradient length and solid state diffusion will be insignificant. This represents the case that \(\alpha = 0\) and equation 2.9 reduces to the well-known Scheil equation.

\[
C_s = KC_0 \left[1 - f_s\right]^{K-1} (2.11)
\]

The Scheil equation assumes equilibrium at the solid/liquid interface, complete diffusion in liquid, negligible diffusion in the solid and negligible dendrite tip undercooling. Several studies [31-37] have used this concept for describing the solidification behaviour of Ni-base alloys. In all of these studies the solid redistribution behaviour and resultant weld metal microstructure are primarily controlled by the relevant values of \(K\) and \(D_s\) for the alloying elements of interests. The value of \(D_s\) will control the potential for back-diffusion in the solid during solidification. The value of \(K\), which is defined by \(K = C_s/C_l\) (\(C_s\) is the solid composition, \(C_l\) is the liquid composition at a particular temperature), describes how strongly an alloying element partitions to the liquid and solid phases during solidification. For instance elements with \(K < 1\) segregate to the liquid during solidification. Consequently, elements with very low value of \(K\) can produce steep concentration gradient across the cellular or dendritic substructure of the weld. However, elements with sufficient solid-state back diffusion can eliminate the concentration gradient. Therefore, the two parameters \(K\) and \(D_s\) play a significant role in the microstructure development of the fusion zone. Besides, by estimation the \(\alpha\) parameter it is possible to assess the back diffusion potential of alloying elements in nickel. Joo and Takeuchi have calculated the \(\alpha\) value as a function of the cooling rate. By knowing the diffusivity of the interested alloying elements in nickel [10], cooling rate \((\varepsilon)\), solidification temperature range and dendrite spacing-cooling rate relationship, the \(\alpha\) parameter can be directly estimated that can show the potential influence of solid-state diffusion.

\[
t_f = \frac{\Delta T}{\varepsilon}, \quad (2.12)
\]

\[
L = \frac{\lambda}{2} = \frac{Ae^{-n}}{2}, \quad (2.13)
\]
where $\Delta T$ is the solidification temperature range, $\lambda$ is the dendrite spacing, and $A$ and $n$ are material constants. It should be mentioned that Equation 2.12 assumes a linear cooling rate through the solidification temperature range.

![Figure 2.15](image)

**Figure 2.15** Dimensionless $\alpha$ parameter as a function of cooling rate for a wide range of alloying elements typically present in Ni base alloys (From Joo and Takeuchi [38]).

Figure 2.15 shows the $\alpha$ as a function of cooling rate for substitutional alloying elements. This figure represents the $\alpha$ value for the specific elements in an Alloy 713. The values of $A$ and $n$ are almost the same for all Ni-base alloys. Therefore, the same trend is seen in Ni-base alloys, that by increasing the cooling rate, the $\alpha$ value decreases since the solidification time decreases ($t_f$). It is important to note that in all cooling rate conditions the $\alpha$ value is much lower than one, which means solid-state diffusion of substitutional alloying elements in nickel alloys is insignificant during solidification of fusion welds. Besides, experimental results validate these calculations for a variety of elements, including Fr, Cr, Nb, Mo and Si [39, 40]. However, different models have been used such as Lever law and Clyne-Kurz model to illustrate the diffusion behaviour of interstitial alloying elements [39, 41]. Particularly, for carbon and nitrogen complete solid-state diffusion in nickel can be expected as these elements lower activation energy relative to substitutional alloying elements.

Figure 2.16 depicts the directional solidification with limited liquid diffusion and no solid state diffusion. Consequently, neither the solid nor the liquid is uniform in composition during solidification. Because of limited liquid diffusion, the solute rejected from the solid piles up and forms a solute-rich boundary layer ahead of the growth front, as shown in 12.16 (c). Figure 12.16 (b) and (c) represent the initial transient, final transient and solute boundary layer region respectively. When $C_L$, $C_S$ reaches the $C_0/k$ and $C_0$ respectively, a steady-state period is formed, within which $C_L$, $C_S$ and boundary layer remain unchanged. As the boundary layer moves forward, it takes a liquid with composition of $C_0$ and it leaves behind a solid of the same composition $C_0$. Since the input equals the output, the boundary layer remains unchanged. This steady-state condition continues until the boundary layer touches the end of the liquid, that is, when the thickness of the remaining liquid equals that of the steady-state boundary layer. Here, the volume of remaining liquid is already rather small and any further decrease in volume represents a very significant percentage drop in volume. Therefore, the remaining liquid quickly becomes saturated as solidification continues and $C_L$, $C_S$ rise sharply. This final period is called final transient [3].
The analysis of solid redistribution during the solidification of an intercellular or interdendritic liquid during welding is similar to the directional solidification concept with negligible solid diffusion and limited or complete liquid diffusion. Within a volume element of Figure 2.17 the S/L interface still remains planar even though the overall structure is cellular or dendritic. The volume element starts from the centreline of the cell or dendrite arm to the boundary between cells or dendrite arms. Solidification starts in the volume element when the tip of the cell or dendrite arm reaches the volume element. For instance, considering the situation in which the partitioning coefficient is lower than one \( (K<1) \), as is shown in Figure 2.18. No segregation occurs when the diffusion is complete in both the solid and liquid. This requires that \( D_{lt}f_s \gg \ell^2 \) and \( D_{lt}f_s \gg \ell^2 \). Segregation is worst when the diffusion in liquid is complete while no diffusion occurs in solid. In addition to these two situations, the segregation is intermediate when solid phase diffusion is negligible and limited diffusion happens in the liquid phase. In all of these three situations, the concentration at the centreline of the cell is minimum, while it is maximum at the interdendritic regions. Figure 2.18 (b) shows the behaviour of microsegregation for the case with \( K>1 \).

Figure 2.16 Solute redistribution during solidification with limited liquid diffusion and no solid diffusion: (a) phase diagram; (b) \( C_L(f_s) \) and \( C_s(f_s) \); (c) composition profiles in solid and liquid [3].

Figure 2.17 volume element for microsegregation analysis: (a) cellular solidification; (b) dendritic solidification [3].
2.7.3 Banding

In addition to the microsegregation phenomena, another mechanism for segregation of solute elements exist that is called Banding. Basically, banding occurs as a result of growth rate fluctuations due to thermal fluctuations. For instance, during solidification when growth rate decrease, the solute elements will segregate less, relative to previous higher growth rate situation which leads to formation of the more solute-rich solidified region and vice versa. Banding can occur by unstable fluid flow in the weld pool [3].

Figure 2.18 Microsegregation profile across cells or dendrite arms: (a) K<1; (b) K>1 [3].
2.8 Weldability and Crack characterization

The term weldability is mainly used to describe the susceptibility to cracking during fabrication. It is widely known that austenitic alloys are always susceptible to hot cracking. Besides, as has already been mentioned in the section 2.4, dilution in the low carbon steel weld by the Ni-base alloy, resulting in the formation of Ni-rich fcc microstructure. Strong segregation of alloying and impurity elements is often seen in such an austenitic microstructure. Basically, the solubility of impurities such as phosphorus and sulphur in the austenite phase is low, which leads to segregation into interdendritic regions. This segregation can act as a mechanism for the crack formation [10].

Generally, cracks are classified in two main types: hot cracks and cold cracks. There are several terminologies that are used in the literature for the formation of hot cracks such as hot cracking, hot tearing, hot fissuring, reheat cracking, microfissuring and ‘polygonization’ cracking in the weld. These terms are indeed confusing and inconsistent since many of these terms refer to different types of “hot” cracking and elevated temperature solid-state cracking (Ductility-Dip Cracking). In an effort to clarify the terms used to describe weld cracking, Hemsworth et al. [42] divided and defined high temperature cracking as two separate types, segregation cracking and ductility-dip cracking. The term “segregation cracking” refers to the type of cracking where intergranular liquid films are present. These forms of cracking, namely weld solidification cracking, HAZ liquation cracking and weld metal liquidation cracking, are now generally referred to “hot” cracking. Thus, by definition, hot cracking is characterized by the presence of a liquid film and occurs at higher temperature, as shown in Figure 2.19. Besides, according to Hemsworth et al. [43], ductility-dip cracking (DDC) can also have three forms that can occur in the weld metal, HAZ or reheated weld metal. The mechanism of DDC is explained in more details in section 2.8.3.

![Figure 2.19 Schematic illustration of ductility as a function of temperature. The dotted line represents a solid-state ductility-dip. (From Nissley [44]).](image)

In the current study, the austenitic microstructure is expected in the top layer weld (C-Mn steel). The austenitic stainless-steel or Ni-base alloys have their own associating mechanisms for the formation of cracks. These include solidification cracking and clad “disbonding” along type two grain boundaries in the weld metal. The most probable mechanisms in the formation of cracks in the microstructure are described based on the existing knowledge for dissimilar weld microstructures and specifically for austenitic (Ni-rich fcc) microstructures.
2.8.1 Fusion zone solidification cracking

Solidification cracking in dissimilar welds joining carbon and stainless steels with Ni-base filler metals, or during cladding of these steels, can potentially be a problem since the weld metals of these combinations will invariably be fully austenitic (fcc-Ni), even under conditions where Ni-base filler metals are heavily diluted by the steel. However, as mentioned previously, in this study the opposite case is tested in which the low carbon steel is overlaid on the Ni-base layer. In this case, the weld metal substructure is indeed dendritic and cellular which is an evidence of austenitic structure. It has been well documented that weld metal primarily solidifies as austenite and is ferritic and austenitic at the room temperature, are inherently susceptible to weld solidification cracking due to the segregation that occurs during solidification and the tendency for liquid films to wet the austenitic grain boundaries [10].

Susceptibility to solidification cracking in dissimilar combinations between Ni-base alloys and steel is a strong function of weld metal dilution by the substrate. The dilution of the steel filler metal by the Ni-base substrate tends to expand the solidification temperature range. Since the width of the solidification temperature range is roughly proportional to cracking susceptibility, dilution by the Ni-base substrate tends to increase the solidification cracking susceptibility of dissimilar welds. Generally speaking, the greater the solidification temperature range (STR), the larger the (S + L) region in the weld metal or the mushy zone and thus the larger the area that is weak and susceptible to solidification cracking. The STR of an alloy increases as a result of either the presence of undesirable impurities such as sulphur (S) or phosphorus (P) in steels and Ni-base alloys or by intentionally added alloying elements.

- Effect of stray elements

Impurities such as S and P are known to cause severe solidification cracking in carbon steel and low-alloy steels even at relatively low concentrations. They have a strong tendency to segregate aggressively (low K values) to the liquid (grain boundaries) during solidification and form low-melting-point compounds (FeS in the case of S), thus widening the solidification temperature range [45]. Figure 2.20 and 2.21 shows the effect of such tramp elements on STR and schematically the effect of impurities on the grain boundaries respectively. In addition, S and P can cause severe solidification cracking in nickel-base alloys [46-48] and ferritic stainless steels [50]. In the case of austenitic stainless steel, their detrimental effect can be significantly affected by the primary solidification phase. It is known that the solubility of sulphur and phosphor in austenite is lower than ferrite, thus, sulphur in austenite will be pushed to the grain boundaries, creating a low melting phase that increase the risk of solidification cracking. Consequently, the primary phase that is formed during solidification can play a significant role in the susceptibility of solidification cracking. For instance, nickel is an austenite stabilizer that promotes the formation of austenite as the primary phase and in this way increase the risk of solidification cracking. As the ratio of the Cr to Ni increases, the primary solidification phase changes from austenite to δ-ferrite, and cracking is reduced. It has been shown by Takalo et al. [51] in Figure 2.22 for arc welding, this changes in Cr/Ni=1.5.
Figure 2.20 Effect of the alloying elements on the solidification temperature range of carbon and low alloy steels. Modified from Principles and Technology of the fusion welding of metals [52].

Figure 2.21 Effect of impurity on grain boundary liquid of weld metal: (a) weld metal near pool; (b) no liquid on pure Fe; (c) some liquid with small amount of Si; (d) much more liquid with a small amount of sulfur [3].

Figure 2.22 Solidification crack susceptibility of austenitic stainless steels for arc welding [51].
The mechanism of solidification cracking in all systems is similar. This type of crack is formed during the terminal stages of solidification when liquid films are distributed along the solidification grain boundaries and, in some cases, interdendritic sites. At this stage, shrinkage strains across the partially solidified boundaries can become appreciable as a result of thermal contraction, solidification shrinkage. Thus, these strains in combination with external restraints (i.e. clamping or base metal constraint and thickness of base metal), induce tensile stresses across adjacent grains which can exceed the strength of the almost completely solidified weld metal [53-55]. If the distribution or wetting of the liquid film be high enough to form a continuous film along the boundaries, the strains cannot be accommodated and the boundaries separate to form a crack. It should be considered that the solidification cracks reside along solidification grain boundaries and interdendritic regions that are the last regions to solidify. Therefore, susceptibility to weld solidification cracking is a function of both metallurgical factors and local strain present at the end of the solidification. The metallurgical factors that affect the susceptibility to solidification cracking include (i) the solidification temperature range (STR), (ii) the amount and the distribution of the interfacial terminal liquid film, (iii) the primary solidification phase, (iv) the surface tension of the grain boundary liquid and (v) the grain structure. Solute redistribution (composition) plays an important role in solidification cracking as it affects the STR and amount of terminal liquid.

The distribution of liquid near the end of the mushy zone is controlled by the amount of terminal liquid and solid/liquid surface tension. When the amount of the terminal liquid is moderate, between approximately one-to-ten volume percent [56, 57] and/or the surface tension is low, the liquid tends to wet the boundary and forms a continuous film. This type of the morphology is most detrimental as it interferes with formation of solid/solid boundaries thus reducing the ability of the material to accommodate strain. In contrast, a small amount of terminal liquid, generally less than approximately one volume percent, that exhibits a high surface tension with solid will often exist as isolated globules and promote solid/solid bridging, thereby reducing cracking tendency. When the amount of terminal liquid is high (greater than approximately ten volume percent), it can often flow into the cracks and provide a “crack healing” effect.

For a given alloy system, the solidification temperature range and amount of the terminal liquid are primarily controlled by composition [56, 58]. Figure 2.23 schematically shows the effect of composition on crack susceptibility.

![Figure 2.23](image)

**Figure 2.23** Effect of composition on crack susceptibility: (a) weld; (b) crack susceptibility curve; (c) pure metal; (d) low solute; (e) more solute; (f) much more solute [3].
Effect of eutectic reaction during terminal solidification

Eutectic reactions can often occur during the terminal stage of solidification and extend the solidification temperature range in alloys for instance in the nickel superalloys. Dupont et al. [10] studied the effect of eutectic reactions on the solidification temperature range and solidification cracking. They investigated some superalloys and stainless steels containing niobium (Nb).

In their investigation of some superalloys, they assessed the two eutectic reactions $L \rightarrow \gamma + \text{NbC}$ and $L \rightarrow \gamma + \text{Laves}$.

They showed that Laves phase forms at lower temperature relative to NbC at interdendritic regions. Thus, Laves phase extends the solidification temperature range more. Table 2.4 shows the eutectic reactions for different superalloys with their associated temperature. Besides, M.J.CIESLAK [59, 60] conclude that iron and silicon are Laves phase promoters while the more carbon that present the tendency to form NbC is higher. Consequently, the NbC formation is preferred in order to reduce the solidification temperature range, thus, the susceptibility to solidification cracking decrease.

**Table 2.4** Summary of terminal solidification reactions and associated temperature for several commercial Nickel alloys [10].

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Terminal Solidification Reaction</th>
<th>Temperature, °C (°F)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B-2</td>
<td>$L \rightarrow L + \gamma + \text{M}_{\text{c}}\text{C}$</td>
<td>1277 (2330)</td>
</tr>
<tr>
<td>W</td>
<td>$L \rightarrow L + \gamma + \sigma$</td>
<td>1250 (2280)</td>
</tr>
<tr>
<td>C-22</td>
<td>$L \rightarrow L + \gamma + \text{P}$</td>
<td>1285 (2345)</td>
</tr>
<tr>
<td>C-276</td>
<td>$L \rightarrow L + \gamma + \text{P}$</td>
<td>1285 (2345)</td>
</tr>
<tr>
<td>242</td>
<td>$L \rightarrow L + \gamma + \text{M}_{\text{c}}\text{C}$</td>
<td>1260 (2300)</td>
</tr>
<tr>
<td>625 (0.03 Si, 0.009 C)</td>
<td>$L \rightarrow L + \gamma + \text{Laves}$</td>
<td>1150 (2100)</td>
</tr>
<tr>
<td>625 (0.03 Si, 0.038 C)</td>
<td>$L \rightarrow L + \gamma + \text{NbC}$</td>
<td>1246 (2275)</td>
</tr>
<tr>
<td>625 (0.38 Si, 0.008 C)</td>
<td>$L \rightarrow L + \gamma + \text{Laves}$</td>
<td>1148 (2100)</td>
</tr>
<tr>
<td>625 (0.46 Si, 0.035 C)</td>
<td>$L \rightarrow L + \gamma + \text{Laves}$</td>
<td>1158 (2115)</td>
</tr>
<tr>
<td>HR-160</td>
<td>$L \rightarrow L + \gamma + \text{(Ni,Co)}<em>{\text{a}}(\text{Ti,Cr})</em>{\text{b}}\text{Si}_{\text{c}}$</td>
<td>1162 (2125)</td>
</tr>
<tr>
<td>C-4 Gd</td>
<td>$L \rightarrow L + \gamma + \text{Ni}_{\text{a}}\text{Gd}$</td>
<td>1258 (2295)</td>
</tr>
</tbody>
</table>
2.8.2 Heat Affected Zone liquation crack

All engineering alloys melt and solidify over a range of temperatures. Generally, the higher alloying element content, the larger the melting/solidification temperature range. Thus, solid-solution strengthened Ni-base alloys or austenitic stainless-steels can particularly exhibit wide melting and solidification temperature range due to their high alloying content. During welding, the base metal adjacent to the fusion zone will experience a range of peak temperature that is between the liquidus and effective (non-equilibrium) solids temperature of the alloy. Consequently, at this region the microstructure of the base metal undergoes a partial and localizes melting. This region is described as the partially melted zone (PMZ) region of the heat affected zone (HAZ). Liquation cracking can occur in the PMZ in Ni-base alloys and aluminum alloys [61, 62] when the liquid within the locally melted region cannot sustain the applied strain and forms a crack usually along a grain boundary. The tendency for HAZ grain boundary liquation is increased with welding processes that are at high heat input, such as gas metal arc welding (GMAW) in the spray transfer mode [10].

Mainly, there are two basic mechanisms for liquation cracking: (i) a segregation mechanism and (ii) a penetration mechanism. Lippold et al. [63] proposed a segregation-induced liquation mechanism for austenitic and duplex stainless steels. In such a mechanism the alloy and/or impurity elements that depress the melting point segregate to GBs by a diffusion mechanism, lower the melting point, and cause the GB liquation. In other words, GB segregation takes place first and GB liquation next. They suggested that such GB segregation can be caused by (i) equilibrium diffusion of atoms of the elements to GBs, (ii) GB sweeping of such atoms into migrating GBs during grain growth, and (iii) “pipe line” diffusion of such atoms along GBs in the fusion zone that are continuous across the fusion boundary into the PMZ [63, 64].

- Penetration Mechanism

The penetration mechanism for HAZ liquation cracking involves the interaction of a migrating HAZ grain boundary with liquating matrix second phase particles such as carbides, sulphides, borides, Laves, etc. The metallurgical basis for this mechanism is a phenomenon known as constitutional liquation. Constitutional liquation is a non-equilibrium phenomenon associated with the rapid heating rates experienced in the HAZ adjacent to the fusion line. In this case, the rapid heating associated with the weld thermal cycle does not permit sufficient time for dissolution of the secondary phase within the matrix. Upon heating above the eutectic temperature, the secondary phase reacts with matrix to form an interfacial localize liquid film along the particle-matrix interface that is at the eutectic composition [65]. The amount of liquid that forms along the interface depends on the heating rate, the dissolution kinetics of the constituent particle, and the diffusivity of solute atoms in the matrix. For instance, when heating rates are extremely high, as in the HAZ of an electron beam or laser beam weld, particle dissolution is negligible and constitutional liquation would be minimized. Conversely, if heating rates in the HAZ are slow, as in electro slag weld, particle dissolution approaches equilibrium conditions. Solid-state particle dissolution thus precludes the onset of constitutional liquation. Generally, this mechanism is most commonly observed when susceptible materials like austenitic stainless steel are welded using the more conventional welding processes (SMAW, GTAW, and GMAW). This type of liquation has been observed at carbides and intermetallic phases in a number of Ni-base alloys, including the precipitation-strengthened alloys Udiment 700, Waspaloy, and Alloy 718 and the solid-solution strengthened alloys such as Hastelloy X and Alloy 625 [66-70]. This mechanism is more common in precipitation-strengthened alloys [10].

Another development of the penetration mechanism is the localized melting of the residual eutectic constituents. In this case, the rapid heating cycle does not permit sufficient time for dissolution of the eutectic constituent, and constituent simply melts when it exceeds the eutectic temperature. If the alloy is above its maximum solid solubility, the eutectic constituent cannot
dissolve regardless of the heating rate and localized melting will always occur. Figure 2.24 [71] shows the liquidation of a Ni-base alloy containing the NbC and Laves phase. The liquidation can be identified by partial dissolution near the edges of the secondary constituents.

For the penetration mechanism, localized melting is generally not sufficient in itself to promote cracking, as the locally melted regions are initially isolated. However, the interaction of the mobile grain boundary with liquid (due to grain growth) causes the liquid to penetrate and wet the boundaries, often leading to complete grain boundary coverage by the liquid film. The rate of the grain boundary migration increases as a function of proximity of the fusion line. In the region of the HAZ where constitutionally liquated particles are usually observed, grain boundaries are normally quite mobile and the likelihood of the interaction between a boundary and second phase particle is high.

As it was already mentioned above the two criteria for liquation cracking at HAZ by penetration mechanism is constitutional liquation (or eutectic melting) with simultaneous grain boundary migration, assuming that the liquating species are not already at the grain boundary sites. The penetration of the boundary by the liquid at the particle-matrix interface either pins the boundary or significantly slows its rate of migration and allows uniform wetting of the boundary by the liquid. These liquid films then serve to embrittle the HAZ grain boundaries in the presence of sufficient mechanically and/or thermally induced restraint imposed during the welding process.

![Figure 2.24 Example of HAZ liquation in Ni-base alloy containing NbC carbide and Laves phase [71].](image)

- **Segregation Mechanism**

Despite the utility of the grain boundary penetration mechanism, HAZ liquation cracking is often encountered in materials where constitutional liquation does not, or cannot, occur. (For example, in materials that does not contain intermetallic constituents). As a result, a liquation mechanism has evolved that is not dependent on the interaction of a mobile grain boundary with a liquated constituent particle. Many single-phase materials that are relatively free of intermetallic and constituent particles have exhibited susceptibility to HAZ liquation cracking. In general, the location of embrittled grain boundaries in the HAZ of these materials is nearly identical to that of materials which are embrittled by the penetration mechanism. As a result, another model that is known as a grain boundary segregation mechanism is often proposed to explain HAZ liquation cracking. The segregation model in its simplest form provides for solute/impurity element segregation to grain boundaries, thereby reducing the melting temperature of the boundary relative to the surrounding matrix. Above a critical temperature during the HAZ thermal cycle, preferential melting occurs along these boundaries effectively embrittling a narrow region of the HAZ adjacent to the weld fusion zone. The actual mechanism by which solute/impurity atoms segregate to the HAZ grain boundaries is not clear.

A possible mechanism for grain boundary segregation invokes equilibrium diffusion, where the segregated region is localized within a few atom diameters on either side of the boundary. This type of grain boundary segregation is driven by the free energy difference between a solute or
impurity atom in a matrix site vs. a grain boundary site. Since the defect structure (in terms of atomic misfit and dislocations) in a grain boundary is greater than that in the adjacent matrix, a reduction in the local free energy can be achieved by diffusion of the atom to the boundary site. This segregation is further enhanced when the solute/impurity element is highly surface active and/or exhibits a low solubility in the base metal. In many of the high-performance structural steels and stainless steels, elements such as sulphur, phosphorus, carbon, antimony, tin and arsenic exhibit this behaviour. The behaviour of an individual element, however, is a strong function of the base metal composition, metallurgical condition (annealed, heat treated, cold worked, etc.) and thermal history.

An equilibrium grain boundary segregation model is most tractable when one assumes that atomic rearrangement occurs at a static boundary. In the HAZ of a typical fusion weld, grain growth begins above a threshold temperature during the on-heating portion of the thermal cycle and continues up to the local peak temperature. Under these conditions, grain boundary liquidation due to equilibrium segregation then occurs once boundary motion has essentially stopped during the on-cooling portion of the thermal cycle. In other words, grain boundary segregation takes place first and grain boundary liquidation next. The HAZ embrittlement window, via this mechanism, is thus quite narrow since the grain boundary velocity must go to zero at a temperature that is sufficient to promote melting along the boundary. From a strictly theoretical basis, grain boundary liquidation due to equilibrium segregation would be enhanced by moderate to slow HAZ heating and cooling rates. For the austenitic stainless steels, this is, in fact, what is normally observed; HAZ cracking is almost never observed in electron beam welds or low heat input arc welding processes.

Alternatively, it has been argued that equilibrium segregation to grain boundaries is not sufficient to support liquidation in the HAZ since the segregation field is extremely localized. As grain boundaries migrate upon heating above the threshold grain growth temperature, solute and/or impurity atoms are swept into the boundaries and are dragged along as grain growth proceeds. Again, elements that are surface active and/or exhibit a low solubility in the matrix would have the highest probability of being swept into and assimilated with the boundary. As the temperature in the HAZ increases above the local grain boundary melting temperature, liquidation occurs and the region of the HAZ within a critical embrittlement temperature range becomes susceptible to liquidation cracking.

Another mechanism for HAZ grain boundary segregation is described by the pipeline diffusion theory. Since weld solidification occurs via an epitaxial nucleation process, grain boundaries are continuous from the fusion zone across the fusion boundary into the HAZ. By the nature of the solidification process, partitioning of solute and impurity elements along the solidification grain boundary results in solute/impurity enrichment relative to the adjacent, epitaxial HAZ boundary. This boundary provides a diffusion pipeline for elemental segregation and subsequent enrichment of HAZ grain boundaries. Since the rate of diffusion along grain boundaries is greatly accelerated relative to bulk diffusion, this mechanism provides a probable explanation for grain boundary liquidation adjacent to the fusion boundary.

The mere occurrence of a liquidation reaction, either by constitutional or boundary segregation-induced liquidation is not sufficient to produce a crack susceptible microstructure. In order to embrittle the microstructure, it is essential that the liquid species penetrate and wet the boundary. If wetting is ineffective, liquid will exist in isolated pockets allowing substantial solid-solid contact along the grain boundaries. As wetting becomes more effective, boundaries will be uniformly coated with a thin liquid film. The wetting characteristics are a function of the composition of liquid and solid in contact, the crystal structure, or structures, of the solid substrate, the relative surface free energies and the temperature. It is frequently reported that Boundaries like (for example, austenite-austenite, ferrite-ferrite) wet much more effectively than two-phase boundaries. Figure 2.25 (a) and (b) schematically describe the liquidation cracking phenomena [65, 3, and 10].
In general, the two mechanisms of the penetration mechanism (constitutional liquation and eutectic melting) are more detrimental from a cracking prospective than the segregation mechanism. This is valid for two reasons. First, constitutional liquation and eutectic melting generally produce more residual liquid and second, the temperature at which these liquid films solidify can be substantially below the alloy solidus. Since the grain boundary liquid films can persist to temperatures well below the alloy solidus (depending on the eutectic temperature), the distance to which liquid films will be present along base metal grain boundaries can be significant and sufficient strain can promote cracking. This distance is also a function of the temperature gradient in the HAZ and can be influenced by welding process and process conditions, particularly heat input.

### 2.8.3 Ductility-Dip Cracking

Ductility dip cracking (DDC) is a solid-state phenomenon that has been observed in a number of structural materials, including austenitic stainless steels, copper alloys, titanium alloys and Ni-base alloys. In materials susceptible to DDC, a sudden drop in ductility occurs in the temperature range between the solidus ($T_s$) and approximately $0.5T_s$. Often this drop in ductility occurs over a very narrow temperature range, as shown in Figure 2.19. As the contraction stresses during welding can be large in this temperature range, this dip in ductility can result in local exhaustion of ductility and subsequent DDC. As it has already been mentioned in the section 2.8, Hemsworth et al. proposed three forms of DDC occurring in the weld metal, HAZ or reheated weld metal. This is consistent with observations of ductility-dip cracking in the weld structures, although in practice DDC is most often observed in the weld metal. Unfortunately the term ductility-dip cracking, as defined by Hemsworth et al. is not specific enough to define a
single cracking mechanism, since their broad definition would still include such forms of cracking as reheat cracking, stress-relief cracking and strain-age cracking. These latter cracking mechanisms all stress relaxation to promote cracking—normally in the base metal HAZ. Grain boundary impurity segregation also contributes to these forms of cracking [10]. DDC requires neither the formation of strengthening precipitates nor grain boundary impurity segregation. High purity, solid-solution strengthened alloys are often more susceptible to DDC than those that undergo precipitation reactions or have moderate impurity levels. Thus, the DDC that is described here represents a distinct form of HAZ and weld metal cracking resulting from grain boundary sliding, or separation, and elevated temperature ductility exhaustion. Theories that are describing the mechanism of DDC are summarized in the following section.

The presence of a ductility dip in austenitic alloys was reported as early as 1912 by Bengough [72]. Since that time, a number of theories have been proposed to explain the mechanism of DDC, as listed in Table … . In 1961, Rhines and Wray [73] showed that a ductility-dip occurs in copper, nickel alloys, austenitic stainless steels, titanium and aluminium. They believed that the ductility dip was caused by grain boundary shearing, similar to creep-rupture failures. At temperatures below the recrystallization temperature, the voids have time to join by grain boundary shearing and cause a fracture. Above the recrystallization temperature, the formation of new grain boundaries makes difficult the joining of the voids. This mechanism is in general agreement with that recently proposed by Ramirez and Lippold [74], and Noecker and DuPont [75, 76].

<table>
<thead>
<tr>
<th>Name</th>
<th>Description</th>
<th>Year</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rhines &amp; Wray</td>
<td>Grain boundary shearing up to recrystallization temperature.</td>
<td>1961</td>
</tr>
<tr>
<td>Yamaguchi et al.</td>
<td>Sulfur segregation and embrittlement.</td>
<td>1979</td>
</tr>
<tr>
<td>Zhang et al.</td>
<td>Combination of effects up to recrystallization temperature.</td>
<td>1985</td>
</tr>
<tr>
<td>Ramirez and Lippold</td>
<td>Grain boundary sliding and microvoid formation, effect of boundary tortuosity.</td>
<td>2005</td>
</tr>
<tr>
<td>Noecker and DuPont</td>
<td>Grain boundary sliding, carbide distribution and morphology.</td>
<td>2008</td>
</tr>
<tr>
<td>Young et al.</td>
<td>Precipitation-induced cracking</td>
<td>2008</td>
</tr>
</tbody>
</table>

Yamaguchi et al. [77] proposed that increases in sulfur content increased the tendency for a ductility dip between 950 and 1150°C, where sulfur segregated to and embrittled the grain boundaries which cracked under an applied stress. Similar sulfur segregation mechanisms have also been proposed by Matsuda [78] and more recently by Nishimoto et al. [79, 80]. Recent work by Collins et al. [81] with filler metal 82 also showed that sulfur additions increase susceptibility to DDC. However sulfur and other impurities may contribute to DDC, the work by Ramirez and Lippold concluded that differences in susceptibility could not be simply explained by impurity (S and P) content since many materials with very low impurity content were also susceptible [74]. Zhang et al. [82] reported that the combined effects of grain boundary precipitation, grain boundary sliding, grain boundary migration and grain boundary serration affect the DDC performance of the low expansion alloy INVAR (Fe-36 Ni). They also suggested that recrystallization and decreased flow stress were factors in the recovery of ductility at elevated temperatures [83]. In recent works by Ramirez and Lippold [74] have conducted a comprehensive study of DDC in Ni-base alloys and concluded that DDC is essentially an elevated temperature, grain boundary creep phenomenon. They found that impurity (P, S, O and H) segregation, grain boundary precipitation and boundary “tortuosity” affect DDC susceptibility and that controlling the nature of grain boundaries is the key to avoid DDC in Ni-base weld metals.

Finally, it would be mentioned that there is still not general agreement on the mechanism for DDC in Ni-base alloys (or other materials), and it is possible that DDC manifests itself differently in different materials. Remarkably, virtually all austenitic (fcc matrix) alloys exhibit a ductility dip. Even austenitic stainless steel weld metals that contain ferrite show a perceptible dip in ductility above 0.5T.
CHAPTER 3
3.0 EXPERIMENTAL PROCEDURES

In this chapter, the techniques which were used to assess the microstructure and solidification behaviour will be reviewed. An explanation of the various materials which were investigated, laboratory techniques, computational tools, and characterization methods will be described in detail.

3.1 Materials and deposition method

For the current study, the compatibility of dissimilar welds of carbon steel and Ni-base alloys is investigated. For this purpose three consumables are used which are pure nickel (UTP A 80 Ni), low carbon steel and Inconel 625. Tables 3.1, 2 and 3 show the chemical composition of these three consumables respectively. The Ni base consumables which are almost pure nickel and Alloy 625 are considered in order to evaluate the effect of alloying elements on the solidification behaviour and microstructure evolution following welding. In the case of crack formation in the microstructure, characterization is mainly focused to understand the possible mechanisms in formation of such cracks. In order to compare these two Ni base consumables, two samples are prepared where the nickel base metals are deposited on top of the two separate steel plates (21x6x2 cm) to provide layers of almost pure nickel and Alloy 625. On top of this Ni-based layer, a second layer of C-Mn steel is deposited. A schematic of the structured layers is shown in Figure 3.1. The depositions are performed by gas tungsten arc welding process (GTAW) and for feeding the consumables an automatic wire feeder machine is used. The more details of the process and deposition technique will be explained in the section 3.2.

As it was already mentioned in the section 1.0, in a preliminary study a sample cladded with Alloy 625 and C-Mn steel (see Figure 3.1) was investigated by optical microscopy and scanning electron microscope as a reference microstructure. For this sample the wire was deposited by gas metal arc welding process (GMAW) with cold metal transfer (CMT) mode. It was observed that specific elements such as Mo and Nb segregated to the interdendritic regions of the top C-Mn steel layer as a result of the dilution by the Alloy 625. These two elements form second phase constituents (i.e. carbides and Laves phases) at terminal stages of the solidification which increase the solidification temperature range and susceptibility to cracking. Figure 3.2 shows the microstructure and chemical distribution map of the steel top layer, diluted by the Alloy 625 substrate. Besides, almost pure nickel is also considered to assess the effect of alloying elements on formation of microstructure cracks.

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<table>
<thead>
<tr>
<th>Table 3.1 Chemical composition of the almost pure nickel consumable (values are in weight percent).</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Element</strong></td>
</tr>
<tr>
<td>-----------------------------</td>
</tr>
<tr>
<td>UTP A 80Ni</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 3.2 Chemical composition of carbon steel consumable (values are in weight percent).</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Element</strong></td>
</tr>
<tr>
<td>-----------------------------</td>
</tr>
<tr>
<td>DIN 8559 SG 2</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 3.3 Chemical composition of Alloy 625 (values are in weight percent).</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Element</strong></td>
</tr>
<tr>
<td>-----------------------------</td>
</tr>
<tr>
<td>Alloy 625</td>
</tr>
<tr>
<td><strong>Nb (+Ta)</strong></td>
</tr>
</tbody>
</table>

1 If determined.
Figure 3.1 overall view of prepared samples by GTAW process: (a) schematic of layers deposited on steel plate; (b) top view of deposited layers.

Figure 3.2 SEM (EDS) images of top layer for Element distribution.
3.2 Welding process and sample deposition

Based on the information provided in chapter 2 section 2.2, in this study, for the deposition of the two layers the GTAW process is used instead of GMAW, because, the GTAW process has a lower heat input compared to the GMAW process. Eventually, the GTAW technique is more controllable process particularly in terms of feeding of filler wire and process parameters. The welding transformer is a TIG MIGATRONIC COMMANDER 400 AC/DC. For feeding the consumable wire to the weld pool an automatic wire feeder MIGATRONIC KT4 is used. The feeder head is connected at the front side of the welding torch. It should be mentioned in order to make a reasonable weld, the angle and position of consumables relative to tungsten electrode tip and base metal play a significant role during welding. Moreover, in order to have the same welding temperature for each pass, a FLUKE 2190A DIGITAL THERMOMETER is used. Eventually, the welding process is operated with the programmed DMC_21x2/21x3 software by Galil Motion Control, Inc. Figure 3.3 shows the welding torch and wire feeder position relative to torch and base metal.

![Welding torch and wire feeder head angle and position relative to TIG torch.](image)

**Figure 3.3** Welding torch and wire feeder head angle and position relative to TIG torch.

On plate one Inconel 625 is deposited and the second plate is deposited by almost pure nickel (UTP A 80 Ni). In both cases efforts have been made to minimize the dilution of the Ni layers by the substrate (steel plate) by reducing the heat input. However, there are some limitations for reducing the heat input. In order to deposit a layer of Ni layer there should be enough wetting between the molten droplets of the consumable and steel plate. Therefore, finding the optimum parameters to achieve both criteria, which are low dilution and appropriate wetting to obtain a suitable layer, are the most important factors during the welding. Several weld trials have been made in order to find the optimum parameters such as current (I), welding speed (ν), feeding rate (F.R), arc length (A.D), wire feeder head angle relative to substrate and gas flow rate. Table 3.4 and 3.5 shows the final deposition parameters that are used for both plates with different Ni consumables. In addition, for each of the plates two welding parameters are considered for two passes of whether almost pure nickel or Alloy 625 consumables as candidates for deposition, to evaluate the effect of welding parameters on dilution by the steel plate into nickel layer. In addition, in a same way, two weld passes of the steel consumables with different welding parameters of the nickel layer are deposited on Ni base layers on both plates in order to determine the dilution of the steel top layer by nickel layer. Table 3.6 and 3.7 shows the associating welding parameters for the four separate passes on each plate for the dilution measurement. However, in order to make a larger deposition, the candidates that gives the lower heat input is considered for deposition. The voltage and temperature profile measured by an oscilloscope YOKOGAWA DL716 (16CH DIGITAL SCOPE). Voltage measurement were started.
when the arc had ignited in order to avoid high frequency (HF) noise. The voltage values are depicted in Figure 3.4, 5, 6 and 7 for the first and second layer with the associated currents. It should be mentioned that also for the top layer, the main aim is to reduce the dilution by Ni layer. Thus, the lowest possible current is used to minimize the heat input for the C-Mn steel top layer. It was indeed found in the preliminary study that Alloy 625 dilutes the steel top layer, causing a particular microstructure evolution and cracking by the formation of low temperature eutectic phases at interdendritic regions. Consequently, in this experiment instead of the GMAW process, the GTAW process is examined and as an alternative solution almost pure nickel is investigated beside the Alloy 625.

### Table 3.4 Welding parameters for deposition of first and second layer on the plate one.

<table>
<thead>
<tr>
<th></th>
<th>Plate 1</th>
<th>First layer= Alloy 625</th>
<th>Second layer= steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Current I (A)</td>
<td>130</td>
<td>110</td>
<td></td>
</tr>
<tr>
<td>Average voltage (V)</td>
<td>12</td>
<td>11</td>
<td></td>
</tr>
<tr>
<td>Heat input (KJmm⁻¹)</td>
<td>0.312-0.499⁴</td>
<td>0.242-0.387</td>
<td></td>
</tr>
<tr>
<td>Welding speed (mms⁻¹)</td>
<td>2.5</td>
<td>2.5</td>
<td></td>
</tr>
<tr>
<td>Gas flow (L min⁻¹)</td>
<td>14</td>
<td>12</td>
<td></td>
</tr>
<tr>
<td>Feeding rate (mms⁻¹)</td>
<td>6.3</td>
<td>6.3</td>
<td></td>
</tr>
<tr>
<td>Arc distance (mm)</td>
<td>3</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>Interpass temperature (°C)</td>
<td>&lt; 40</td>
<td>&lt; 40</td>
<td></td>
</tr>
<tr>
<td>Shielding gas (%)</td>
<td>Argon 100%</td>
<td>Argon 100%</td>
<td></td>
</tr>
<tr>
<td>Pre-flow (s)</td>
<td>4</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td>Post-flow (s)</td>
<td>5</td>
<td>5</td>
<td></td>
</tr>
</tbody>
</table>

### Table 3.5 Welding parameters for deposition of first and second layer on the plate two

<table>
<thead>
<tr>
<th></th>
<th>Plate 2</th>
<th>First layer= UTP A80 Ni</th>
<th>Second layer= steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Current I (A)</td>
<td>130</td>
<td>110</td>
<td></td>
</tr>
<tr>
<td>Average voltage (V)</td>
<td>12</td>
<td>11</td>
<td></td>
</tr>
<tr>
<td>Heat input (KJmm⁻¹)</td>
<td>0.312-0.499</td>
<td>0.242-0.387</td>
<td></td>
</tr>
<tr>
<td>Welding speed (mms⁻¹)</td>
<td>2.5</td>
<td>2.5</td>
<td></td>
</tr>
<tr>
<td>Gas flow (L min⁻¹)</td>
<td>14</td>
<td>12</td>
<td></td>
</tr>
<tr>
<td>Feeding rate (mms⁻¹)</td>
<td>6.3</td>
<td>6.3</td>
<td></td>
</tr>
<tr>
<td>Arc distance (mm)</td>
<td>3</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>Interpass temperature (°C)</td>
<td>&lt; 40</td>
<td>&lt; 40</td>
<td></td>
</tr>
<tr>
<td>Shielding gas (%)</td>
<td>Argon 100%</td>
<td>Argon 100%</td>
<td></td>
</tr>
<tr>
<td>Pre-flow (s)</td>
<td>4</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td>Post-flow (s)</td>
<td>5</td>
<td>5</td>
<td></td>
</tr>
</tbody>
</table>

⁴ Based on the GTAW process efficiency (50-80%).
### Table 3.6 Welding parameters for lane depositions on the plate one for dilution measurement.

<table>
<thead>
<tr>
<th></th>
<th>Plate 1</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1\textsuperscript{st} pass= Alloy 625</td>
<td>2\textsuperscript{nd} pass= Alloy 625</td>
<td>1\textsuperscript{st} pass= steel</td>
<td>2\textsuperscript{nd} pass= steel</td>
</tr>
<tr>
<td>Current I (A)</td>
<td>130</td>
<td>145</td>
<td>100</td>
<td>110</td>
</tr>
<tr>
<td>Average voltage (V)</td>
<td>12</td>
<td>13</td>
<td>10.5</td>
<td>11</td>
</tr>
<tr>
<td>Heat input ((KJmm^{-1}))</td>
<td>0.312-0.499</td>
<td>0.377-0.603</td>
<td>0.21-0.336</td>
<td>0.242-0.387</td>
</tr>
<tr>
<td>Welding speed ((mms^{-1}))</td>
<td>2.5</td>
<td>2.5</td>
<td>2.5</td>
<td>2.5</td>
</tr>
<tr>
<td>Gas flow ((L min^{-1}))</td>
<td>14</td>
<td>14</td>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>Feeding rate ((mms^{-1}))</td>
<td>6.3</td>
<td>8.8</td>
<td>6.3</td>
<td>6.3</td>
</tr>
<tr>
<td>Arc distance ((mm))</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

### Table 3.7 Welding parameters for lane deposition on the plate two for dilution measurement.

<table>
<thead>
<tr>
<th></th>
<th>Plate 2</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1\textsuperscript{st} pass= UTP A80 Ni</td>
<td>2\textsuperscript{nd} pass= UTP A80 Ni</td>
<td>1\textsuperscript{st} pass= steel</td>
<td>2\textsuperscript{nd} pass= steel</td>
</tr>
<tr>
<td>Current I (A)</td>
<td>130</td>
<td>145</td>
<td>100</td>
<td>110</td>
</tr>
<tr>
<td>Average voltage (V)</td>
<td>12</td>
<td>13</td>
<td>10.5</td>
<td>11</td>
</tr>
<tr>
<td>Heat input ((KJmm^{-1}))</td>
<td>0.312-0.499</td>
<td>0.377-0.603</td>
<td>0.21-0.336</td>
<td>0.242-0.387</td>
</tr>
<tr>
<td>Welding speed ((mms^{-1}))</td>
<td>2.5</td>
<td>2.5</td>
<td>2.5</td>
<td>2.5</td>
</tr>
<tr>
<td>Gas flow ((L min^{-1}))</td>
<td>14</td>
<td>14</td>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>Feeding rate ((mms^{-1}))</td>
<td>6.3</td>
<td>8.8</td>
<td>6.3</td>
<td>6.3</td>
</tr>
<tr>
<td>Arc distance ((mm))</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

**Figure 3.4** Voltage measurement for I= 145 (A).
**Figure 3.5** Voltage measurement for $I = 130$ (A).

**Figure 3.6** Voltage measurement for $I = 110$ (A).

**Figure 3.7** Voltage measurement for $I = 100$ (A).
3.3 Thermal analysis

The temperature of the first layer (Ni-layer) and second layer (steel top layer) are recorded. First, the temperature profile of the Ni-base weld on the steel plate at the edge of the plate is measured by thermocouples attached at specific positions. Besides, a type K (chromel- alumel) thermocouple is manually plunged into the solidifying weld pool immediately behind the welding torch while it is moving. Temperature data is then recorded during the molten stage and during solidification. The same procedure is performed for the second layer (C-Mn steel-layer). Table 3.8 states the welding parameters for thermal analysis measurement.

Table 3.8 Welding parameters for thermal analysis.

<table>
<thead>
<tr>
<th>Welding parameters</th>
<th>Current (A)</th>
<th>Weld time (s)</th>
<th>Welding speed (mms⁻¹)</th>
<th>Arc length (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni weld (Pure and Alloy 625)</td>
<td>130</td>
<td>21.6</td>
<td>2.5</td>
<td>3</td>
</tr>
<tr>
<td>Steel on Ni weld</td>
<td>110</td>
<td>21.6</td>
<td>2.5</td>
<td>3</td>
</tr>
</tbody>
</table>

Thermocouples are attached at three different positions. One of the thermocouples is plunged into the solidifying weld pool the other three are attached to the steel substrate edge at the middle way of the welding direction from 2.5, 4 and 7 mm from the surface toward the depth of the plate. In order to protect them from the arc they are covered by the ceramic cup. Following the attachment of thermocouples the welding is started for the first lane (Alloy 625 wire). After cooling of the first lane to room temperature, the second lane (steel wire) is deposited on top of the Alloy 625 lane. Figure 3.8 roughly gives an indication of the location of the thermocouples.

Apart from the temperature measurements, a FEM model was built to obtain a global overview of temperature profile as a function of time during the deposition of both layers. This modeling was carried out by ABAQUS software version 6.12. Furthermore, reasonable predictions of heat dissipation and solidification direction were seen. Generally, the input data for the thermal analysis by ABAQUS software are the position of thermocouples, arc diameter, heat input, process efficiency (0.7) and the amount of heat that is observed by the consumable. The more details of the software code and parameters are mentioned in the appendix A.
3.4 Phase diagram and Solidification Temperature Range (STR)

Various phase diagrams are constructed and Solidification Temperature Range (STR) are calculated by using Thermo-Calc Classic version R (TCCR), a commercially available software package from Thermo-Calc software AB of Sweden. TCCR is a powerful software platform that can be used for many thermodynamic calculations [84]. Thermodynamic calculations are performed using thermodynamic information taken from databases that have been developed by Thermo-Calc software. The various data bases are suitable for a wide range of materials systems and simulations.

In this investigation, TCCR is used primarily to perform solidification simulations with the SCHEIL module and TCS Steels/Fe –Alloys databases, version 6 and 7 (TCFE6 and 7). Two simulations are performed for plate one and two to obtain the equilibrium ternary phase diagram using the (POLY) module and TCFE6 and 7 databases. The TCFE6 (7) data base is used since the most dominant element in the top layer (steel layer) is iron, which is more than 50 percent. However, this top layer is strongly diluted by nickel from lower layer. Based on the average chemical composition at the diluted steel layer, simulations are performed to computationally identify the compositional influence of various alloying additions on solidification temperature range (STR) during solidification of the steel (DIN 8559 SG2) top layer.
3.5 Characterization

3.5.1 Light Optical Microscopy

Samples which are characterized via light optical microscopy (LOM) are prepared using standard metallographic supplies and procedures. Sectioning of samples is performed by using a Struers Discotom-6 sectioning machine. The machine is equipped with abrasive wheel and high speed cutting liquid. From each plate, four transverse and one longitudinal sectioning relative to weld direction are considered for crack detection in the microstructure. In addition, in order to measure the dilution two transverse sections are prepared for Ni layer dilution and steel dilution by the nickel layer. Following sectioning, samples are directly grinded without mounting since the size of samples is large enough for ease of grinding. Samples are sequentially ground using 80, 180, 320, 800, 1200 and 2000 grit SiC abrasive paper disks on a rotating plate with continuous water cooling. Following grinding, ultrasonic cleaning is done in ethanol liquid for 5 minutes to prevent scratches from remained particles between grinding and polishing. Polishing is then sequentially performed using 3 µm and 1 µm diamond particles in an oil-based suspension. Once removed from polishing samples are washed by water and ethanol respectively and dried using cool air.

Following polishing, samples of both plates were first analyzed without etching under the optical microscope Olympus BX60M equipped with an OLYMPUS U-TV1X color view soft imaging Digital camera in order to detect the possible microstructural cracks and their position relative to the weld bead. After detection of cracks in all the samples, etching is performed for microstructural characterization. Etching is done by Kalling which the composition is provided in table 3.9. Ethanol is used to rinse samples once etching finished. Table 3.9 contains a summary of the chemical etchant used. The etching time is approximately 10 seconds.

<table>
<thead>
<tr>
<th>Etch</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kalling’s</td>
<td>CuCl₂ (5 grams)+ Hydrochloric acid (100 ml)+ Ethanol (100 ml)</td>
</tr>
<tr>
<td>No.2</td>
<td></td>
</tr>
</tbody>
</table>

3.5.2 Scanning Electron Microscopy

Sample preparation for scanning electron microscopy (SEM) was very similar to that previously described for LOM. However, for orientation contrast microscopy (Electron Backscatter Diffraction-EBSD) an extra preparation step is suggested after polishing with 1 µm suspension. Therefore, the abrasive oxide polishing suspension, *i.e.* colloidal silica (OP-S) with a particle size of 0.25 µm is operated by DiaPro Mol cloth for 10 minutes approximately. Besides, for EBSD operation a dimension restriction is present for the thickness of the sample. The maximum thickness should be 10 mm. SEM samples were often analyzed in both the etched and non-etched conditions.

The SEM FEI quanta 450® with Field Emission Gun (FEG) filament is used for various measurements. Images for fracture surface analysis were acquired using a secondary electron detector. The backscattered electron detector of type EDAX-TSL® is used for quantitative analysis of crystallographic orientations of aggregates. In addition, the FEI quanta 450 also equipped with an energy dispersive spectrometer (EDS) for qualitative and semi-quantitative chemical analysis.
**EBSD measurement**

In this investigation, for the EBSD measurements the sample is mounted with a tilt angle of 70° (i.e. the angle between the sample surface normal and the incident beam) and a working distance of 18 mm and acceleration voltage of 20 kV is employed. In EBSD operating condition, a medium to high acceleration voltage of 20 to 30 kV is applied with an incident beam current from 1 to 15 nA. Consequently, the resolution quality from the backscattered electrons is in the order of 20-30 nm, while secondary electrons give a better resolution in the order of approximately 1 nm. The scanning step size also plays a significant role during scanning. This parameter determines the distance between two points in the scan. A proper step size leads to an appropriate balance between scanning time and the image resolution. In this current study a general step size of 1 µm is chosen.

A series of data points is obtained by the scanning beam over the sample. Each of these data points reveals specific information [85]:

- X, Y coordinates of the data point;
- The three Euler angles that characterize the crystallographic orientation of the data point;
- A specific number that refers to the crystal phase of the data point;
- The image quality of the data point;
- The confidence index of the data point;

There are two parameters that normally should be considered during EBSD measurement in order to achieve consistent results. The first parameter is called *image quality* (IQ) which quantifies the sharpness of the EBSD pattern. This parameter is dependent on different factors such as; sample preparation, chemical composition, crystallographic phase, crystal orientation and most important the dislocation density. The second parameter is the *confidence index* (CI) that is a measure of the reliability of the orientation determination (i.e. calculation of Euler angles on an indexed pattern representing a solution of the pattern). As the EBSD pattern contains excessive redundant information for orientation determination, it implies that the Euler angles associated with Kikuchi pattern can be calculated many times over. The CI is defined in the following equation 3.1:

\[
CI = \frac{V_1 - V_2}{V_{tot}}, \tag{3.1}
\]

with \(V_{tot}\) the total number of solutions, \(V_1\) and \(V_2\) are the largest and the second largest number of identical solutions, respectively [85]. CI usually ranges from 0 to 1. As the CI becomes more close to 1, the pattern reliability is high. In practice, it was shown that solutions have more than 95% reliability of the CI value exceed 0.1.

The EBSD software controls the data acquisition, solves the diffraction patterns and stores the data. However, further software is required to analyze and display the data. For instance, the Orientation Imaging Microscopy (OIM®) is a fully automated data acquisition program that collects and processes EBSD patterns. The principle of OIM program is schematically shown in Figure 3.9.
Figure 3.9 Principle of the OIM measurement [86].

As the electron beam scans the microstructure (Figure 3.9a), the EBSD patterns (Figure 3.9b) are collected, stored and analyzed. The expected phases (*i.e.* FCC and/or BCC, *etc.*) are initially defined by the user. Following, the diffraction patterns appear, the crystal structure is identified and then the patterns are indexed (*CI*).

Moreover, the crystallographic planes and orientations are calculated. During post processing these data are converted into an image in which each crystallographic system is represented by a particular color code (Figure 3.13c). One of the most used presentations of processed data is called Inverse Pole Figure (IPF). This figure shows the distribution of sample direction with respect to crystal reference system. In order to distinguish crystallographic orientations (grains), each grain has given a specific color with respect to sample reference system. This is used to clarify if the cracks are intergranular or transgranular. Other colored maps are also possible, like *e.g.* phase map in which the fractions and positions of considered phases are depicted.
3.5.3 Chemical analysis by EDS

For qualitative and semi quantitative chemical analysis, the Energy Dispersive Spectrometer (EDS) is preferred relative to Wavelength Dispersive Spectrometer (WDS). WDS is generally the technique for *quantitative* analysis, especially minor and trace element quantitation. However, the EDS technique is more *qualitative* and determines the approximate weight fraction. EDS can either qualitatively determine the elements that should be measured by displaying them, or it can qualitatively identify the phase (mineral) thereby leading the analyst toward which elements (major, minor and trace) should be measured. Meanwhile, in the WDS technique it is necessary to know *a priori* which elements to measure. While a high resolution WDS wavelength scan take up to 30 minutes to complete, the EDS spectrum is acquired in a few seconds. Since the solid-state EDS detector was first introduced in 1968 it has been improved considerably. Resolution has been improved from 500 eV to 150 $^{5}$ eV to less than 115 eV today, thus it can be used as sensitive and quantitative as WDS. In this study the EDS technique is used to analyze the general bulk chemical composition of the samples since the whole thickness of depositions are almost equal to 2 mm. In addition, for local and chemical analysis and segregation behavior of the alloying elements the EDS technique is the best option. By EDS it is possible to make line scans in order to understand the segregation behavior at dendritic and interdendritic regions, while with area map scans, the phases and accumulation of specific elements is detectable.

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$^{5}$ EDS resolution has been standardized for the FWHM of Mn Kα or an Fe$^{55}$ source (5.9 KeV).
4.0 RESULTS

4.1 Dilution measurement

As it was mentioned in the section 2.5.2, the dilution of the steel layer by the nickel underneath layer either Alloy 625 or pure Ni on the plate one and two respectively, plays an important role in the microstructure evolution of fusion zone. Dilution of the weld metal microstructure can act as one of the mechanisms for hot cracking phenomena particularly by segregation of the low melting point alloying elements. Therefore, in this investigation the effort has been made to minimize the dilution by lowering the heat input, while it should be noted that on the other hand lowering the heat input can lead to formation of other weld defects or particularly lack of sufficient fusion and wetting for good deposition.

For dilution measurement, the samples are sectioned perpendicular to the welding direction and equation 2.6 is used (section 2.5.2). In addition, for dilution measurement on both plates, as it was mentioned in the section 3.2, four welding parameters are considered that could balance the two most important criteria; first good deposition and second low dilution. Figure 4.1 represents schematically the part of both plates used for dilution measurements indicated by the red brackets. The welding parameters for dilution measurements are mentioned in the section 3.2, tables 3.6 and 7 for plate one and two, respectively.

![Figure 4.1 Schematic of four weld passes for dilution measurement.](image)

For the ease and accuracy of the dilution measurement the nickel base layer particularly at the location of the two passes of C-Mn steel, was milled in order to have a flat surface. The steel substrates (plate 1 &2) initially were also milled in order to have really flat surface in order to keep the arc length constant during each weld pass. The measurements of molten base metal area and filler metal were carried out by Photoshop software by measuring the number of pixels in each area with the help of histogram to increase the accuracy. Besides, to minimize the deviation of the result each measurement was repeated 3 times. Table 4.1 and 2 demonstrate the dilution numbers for the first and second plate with Alloy 625 and pure Ni respectively.

<table>
<thead>
<tr>
<th>Material</th>
<th>Dilution (%)</th>
<th>Current (A)</th>
<th>Feeding rate (mms⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy 625 on the steel substrate</td>
<td>52.5</td>
<td>130</td>
<td>6.3</td>
</tr>
<tr>
<td>Alloy 625 on the steel substrate</td>
<td>50</td>
<td>145</td>
<td>8.8</td>
</tr>
<tr>
<td>Steel on the Alloy 625</td>
<td>46</td>
<td>100</td>
<td>6.3</td>
</tr>
<tr>
<td>Steel on the Alloy 625</td>
<td>60</td>
<td>110</td>
<td>6.3</td>
</tr>
</tbody>
</table>

The deviations in the values are maximum 1%.

Other welding parameters are mentioned in the table 3.6 (particularly the key parameter is feeding rate).
Table 4.2 Dilution results for plate two (Pure Ni).

<table>
<thead>
<tr>
<th>Material</th>
<th>Dilution (%)</th>
<th>Current (A)</th>
<th>Feeding rate (mms⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure (A 80 Ni) on the steel substrate</td>
<td>48</td>
<td>130</td>
<td>6.3</td>
</tr>
<tr>
<td>Pure (A 80 Ni) on the steel substrate</td>
<td>40.5</td>
<td>145</td>
<td>8.8</td>
</tr>
<tr>
<td>Steel on the pure Ni</td>
<td>39</td>
<td>100</td>
<td>6.3</td>
</tr>
<tr>
<td>Steel on the pure Ni</td>
<td>52.5</td>
<td>110</td>
<td>6.3</td>
</tr>
</tbody>
</table>

As it can be seen, the dilution of Alloy 625 and pure nickel welds by the steel substrate show a contradictory behavior relative to the applied currents. Normally, by increasing the current the dilution increases since more heat input is released by the arc, thus, the more base material is melted. However, in this case by increasing the current the dilution reduced. The reason for this phenomenon is related to the amount of feeding rate of the consumables that is injected to the weld pool which is higher for the current of 145(A).

Consequently, the heating power of the arc is considerably observed by the consumable which causes a lower amount of heat consumed by the base material. Therefore, the dilutions are lower for an applied current of 145 (A) relative to 130 (A) with lower feeding rate. Figure 2.7 in the section 2.5.2 shows this effect clearly for stainless steel.

4.2 Thermal analysis

The details of the experimental procedure of the thermal analysis were mentioned in the section 3.3. However, it should be mentioned that the experiment was carried out only for one plate where a single pass of Alloy 625 deposited after which a second pass of C-Mn steel is placed on top of the Alloy 625. As the plate size and all welding parameters are the same for both plates except the chemical composition of the first layer, the temperature profile and distribution for the other situation is considered not to change considerably. The welding parameters for these two pass depositions are mentioned in the section 3.3, Table 3.8.

Figure 4.2 and 3 demonstrate the temperature profile of the first layer and second layer, respectively, for the four thermocouples at various positions. The “top” position stands for the thermocouple that was plunged into the solidifying weld pool.

![Figure 4.2](image)

Figure 4.2 Temperature profile of the first lane deposition (Alloy 625).

---

8 Other welding parameters are mentioned in the table 3.7 (particularly the key parameter is feeding rate).
Figure 4.3 Temperature profile of the second lane deposition (steel).

Obviously, the “top” thermocouple shows the highest peak temperature in the both layers. Peak temperatures reduce with increasing distance. The all peak temperatures of the Figure 4.2 are higher than the peak temperatures of the Figure 4.3 as a result of higher heat input of the first layer and the larger distance of the second layer to the thermocouples positions. In order to have an approximate calculation for the cooling rate of the weld pool the curve of the “top” position is considered for both layers. It can be calculated from Figure 4.2 and 4.3 that the cooling rate for the first pass (Alloy 625) and the second pass (steel) are 291.6°C/s and 255°C/s respectively. For the cooling rate calculation, data are taken from the third data point from the peak temperature and the temperature after one second from the previous temperature (third data point).
4.2.1 ABAQUS simulation

In order to understand how the arc heat is dissipated in the plate by different mechanisms like convection, conduction and radiation, simulation provide a proper understanding. The simulation and experimental results are in the good agreement (see Figure 4.4). It should be mentioned that the K-type thermocouple can reliably measure up to maximum temperature of 1350°C, while in the simulation the top position peak temperature exceeds this limit. Therefore, the “top” position did not considered for the simulation.

![Figure 4.4 Temperature profile of the simulation and experiment for both lane one and two](image)

Figure 4.4 Temperature profile of the simulation and experiment for both lane one and two [Solid lines show the simulation data and points show the experimental values].

It was assumed during the simulation the second layer, deposited after 604 seconds (≈ 10 minutes) at a temperature of almost 37°C. Besides, the model predicts heat flow direction reasonable, from which microstructural evolutions including grain growth can be derived. Figure 4.5 and 4.6 depict the heat flow and temperature distribution in the sample initially with first layer and after the deposition of second layer (lane).

![Figure 4.5 Heat flow and temperature distribution in the plate with first layer](image)
Figure 4.6 Heat flow and temperature distribution after deposition of second layer on the first layer.

Figure 4.5 and 4.6 represent the direction of the heat flow which is in the direction of maximum temperature gradient. The legend shows the temperatures in degree Celsius associated to specific colours. Areas exceeding 1455°C (melting point of pure nickel⁹), indicating that the temperature is higher than the liquidus temperature. This temperature (1455°C) is only considered as a standard value to show how the temperature proportionally varies at different positions by the heat flow mechanisms. The input values for the modelling were already mentioned in the section 3.3 and the codes in the appendix A.

⁹ Close to the melting point of the steel DIN 8559.
4.3 Phase diagrams simulation

In this section, the equilibrium binary and ternary phase diagrams that obtained by Thermo-Calc software for both plate two and one are presented respectively. The binary phase diagram is considered for plate two, as the two main elements are iron and nickel. The carbon and nickel are both austenite stabilizing elements and the steel layer is diluted by pure nickel layer. In addition, the carbon contents in the steel layer DIN 8559 is relatively low (see Table 3.2). Therefore, considering the carbon content as an independent element axis in the phase diagram would not release informative results. However, the carbon content and the possible carbide phases were considered during the coding and calculation of equilibrium phases (i.e. Carbide) of the binary phase diagram. Figure 4.7 (a) and (b) is the binary phase diagram of Fe-Ni with 0.08 wt% carbon.

(a) Binary phase diagram of Fe-Ni.

(b) Magnified red rectangular part of Figure 4.7 (a).

Figure 4.7 Binary phase diagram of Fe-Ni with 0.08% carbon content.
The binary phase diagram of iron-nickel is limited to a nickel content of 40 wt%. This amount of nickel was chosen being the maximum limit of TCFE6 database, which is the iron and/or steel data base. Consequently, the iron must be higher than 50 wt% of the total nominal composition. In addition, the chemical analysis of C-Mn steel layer showed that the nickel content is in the range of 25 to 35 wt%. Therefore, the binary phase diagram can show the equilibrium phases in the range of the nominal composition of the diluted steel layer by pure nickel layer.

Moreover, for plate one with first layer of Alloy 625, the ternary phase diagram is considered. The three main elements in the ternary phase diagram are iron (Fe), nickel (Ni) and chromium (Cr) in decreasing order of alloying content, based on the chemical analysis by the EDS. Generally, the chromium is a BCC stabilizer (α-phase) while nickel is an austenite stabilizer. Therefore, considering Cr and Ni as independent elements beside the iron would be informative to predict which phases are formed during solidification and following that to the room temperature. Besides, the niobium (Nb), molybdenum (Mo) and carbon (C) are also considered as the main alloying elements with constant amount (based on the chemical analysis results from the EDS) for plotting the ternary phase diagram in order to see if Laves and carbide phases could form. Table 4.3 demonstrates the primary conditions for plotting the ternary phase diagram of Fe-Ni-Cr. Figure 4.8 depicts the ternary phase diagram of Fe-Ni-Cr at different temperatures.

<table>
<thead>
<tr>
<th>Conditions</th>
<th>C (wt %)</th>
<th>Nb (wt%)</th>
<th>Mo (wt %)</th>
<th>Pressure (Pa)</th>
<th>N (mole)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Values</td>
<td>0.08</td>
<td>1.05</td>
<td>2.4</td>
<td>1.E5</td>
<td>1</td>
</tr>
</tbody>
</table>

![Diagram](image)

(a) At T=1500 °C

(b) At= 1300 °C
As is shown in Figure 4.8, the ternary phase diagram of Fe-Ni-Cr reveals different phases at various temperatures. These equilibrium phases based on the thermodynamic and kinetic reasons form and transform to other phases at the specific temperatures. By comparing the Figure 4.8 (b) and (d), it is clear that the Laves phase forms at lower temperature relative to the carbide. These two phases can extend the solidification temperature range, particularly Laves phase which forms at a lower temperature. In addition, apparently in Figure 4.8 (e) a new BCC phase has formed, as indicated by $\alpha'$. This phase ($\alpha'$) is a Fe rich phase in opposition of the $\alpha$ (BCC) phase rich in Cr. See appendix B for the fractions of the phases and the chemical compositions.
4.4 Solidification Temperature Range (STR)

In order to calculate the solidification temperature range (STR= $T_L - T_S$) of the plate one and two as a function specific element that form eutectic phases, the Thermo-Calc software has been used. These elements normally segregate to grain and sub-grain boundaries during the solidification and hence form liquid films at these regions. Therefore, they can extend the solidification temperature range. For the STR measurement specific elements have been chosen based on the chemical analysis that has been done for each of the two plates. For the plate one with three main elements of Fe, Cr and Ni, two alloying elements which are niobium (Nb) and molybdenum (Mo) are chosen to calculate their concentration effect on the STR. Besides, for the plate two that has two main elements, iron and nickel, three alloying elements are selected to assess their concentration effect on the STR.

• **STR of plate one**

Figure 4.9 (a) and (b) demonstrate the concentration effect of the Nb and Mo alloying elements on the solidification temperature range (STR). The amount of chromium and nickel assumed to be constant and considered as 6 and 15 (wt%) respectively.

![STR as a function of Nb (wt. %)](image)

(a) STR as a function of Nb (wt. %).

![STR as a function of Mo (wt. %)](image)

(b) STR as a function of Mo (wt. %)

**Figure 4.9** STR as a function of two alloying elements; (a) Nb, (b) Mo for the ternary Fe-Ni-Cr alloy.

It is clearly seen that the higher concentrations of the both alloying elements increases the STR, thus, the crack susceptibility. In addition, the effect of Nb in increasing the STR is remarkably higher than Mo.
Figure 4.10 (a) and (b) demonstrate the concentration effect of the C, Si and Ti alloying elements on the solidification temperature range (STR). The amount of nickel assumed to be constant and considered as 35 (wt%).

Figure 4.10 STR as a function of two alloying elements; (a) C, (b) Si and (c) Ti for the binary Fe-Ni alloy.

From Figure 4.10, it is concluded that all of these three alloying elements increased the STR. However, in terms of the severity or effectiveness in the increasing the STR, carbon has the highest influence and subsequently Ti and Si.
4.5 Characterization

The first step for characterizing the cracks is their detection. Therefore, as it was mentioned in the section 3.5.1 from each plate four transverse sectioned samples were analyzed under the optical microscope without etching for two purposes. First, detecting the cracks and counting them. The second reason is finding the relative position of the cracks to the weld bead. Two longitudinal cuts from the both plates could not reveal appropriate indication of cracks and their relative position.

The four transvers sectioned samples of plate one (Alloy 625\textsuperscript{10}) showed several cracks. Each samples of plate one had 15 welding passes next to each other. The four samples of the plate two (Pure Ni\textsuperscript{11}) had 22 passes. Table 4.3 and 4 represent statistically the number of cracks in each samples for plate one and two respectively. The crack probability is defined in order to compare the crack susceptibility of the two plates. This parameter is calculated by the following equation:

\[ Crack\ susceptibility = \frac{\text{Total number of cracks (all pieces)}}{\text{number of pieces} \times \text{number of passes}} \]  

\[ (4.1) \]

<table>
<thead>
<tr>
<th>Material: plate one (Alloy 625)</th>
<th>Number of passes</th>
<th>Number of cracks</th>
<th>Crack probability</th>
</tr>
</thead>
<tbody>
<tr>
<td>Piece 1</td>
<td>15</td>
<td>4</td>
<td>42%</td>
</tr>
<tr>
<td>Piece 2</td>
<td>15</td>
<td>7</td>
<td></td>
</tr>
<tr>
<td>Piece 3</td>
<td>15</td>
<td>7</td>
<td></td>
</tr>
<tr>
<td>Piece 4</td>
<td>15</td>
<td>8</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Material: plate two (Pure Ni)</th>
<th>Number of passes</th>
<th>Number of cracks</th>
<th>Crack probability</th>
</tr>
</thead>
<tbody>
<tr>
<td>Piece 1</td>
<td>22</td>
<td>10</td>
<td>26%</td>
</tr>
<tr>
<td>Piece 2</td>
<td>22</td>
<td>7</td>
<td></td>
</tr>
<tr>
<td>Piece 3</td>
<td>22</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>Piece 4</td>
<td>22</td>
<td>1</td>
<td></td>
</tr>
</tbody>
</table>

4.5.1 Optical microscopy (OM)

The optical microscopy showed the position of the weld cracks clearly. It appears that no liquation cracks were formed in the HAZ for both plates. This might be as a result of the low heat input that was used during the depositions. Thus, this can minimize the susceptible region (partially melted zone) for the liquation cracking. Particularly, for plate one with Alloy 625 deposition, even in the presence of niobium (Nb) and molybdenum (Mo) as eutectic phase formers (Laves and carbides), HAZ liquation cracks could not be found. All cracks found were located at centre of the weld bead or at the bulk of the fusion zone. Figures 4.11 and 4.12 show the cross sections of piece 2 from the middle part of plate one and two, respectively. The position of the cracks relative to the weld bead in the both figures reveals that they are solidification cracks. Figure 4.11 depicts almost two passes of the weld in plate one, while

\textsuperscript{10} Alloy 625 deposited as a middle layer under the steel layer.

\textsuperscript{11} Pure Ni deposited as a middle layer under the steel layer.
Figure 4.12 shows 3 passes. The more detail description and characterization of the microstructure and its crack will be mentioned in the following sections and will be discussed in the chapter 5.

The initial investigation of the microstructure by LOM on the etched samples showed a dendritic sub-grain microstructure for both plates particularly in the second layer. This dendritic microstructure in the steel top layer is a result of the presence of alloying elements in the steel wire and/or by the dilution with Ni layer. Consequently, as it was already mentioned in the section 2.6.2, the constitutional super cooling phenomenon occurs. Depending on the position in the weld pool, the constitutional supercooling may vary which leads to the formation of columnar and equiaxed substructures. Each groups or packets of dendrites with the same growth direction can be seen in Figure 4.13 and 4.14. These groups or packets form one solidification grain as it is indicated roughly by red lines in Figure 4.13 and 4.14 (b).

![Figure 4.11](image1.png)

**Figure 4.11** Two passes of the steel weld of plate one (etched with Kallings).

![Figure 4.12](image2.png)

**Figure 4.12** Three passes of the steel weld of plate two (etched with Kallings).
Moreover, the microstructure assessment by LOM in both plates within the steel layer could distinguish the grains clearly while their growth directions were toward the weld pool centre line. Figure 4.15 shows the grains with their subgrain microstructure and associating growth direction toward weld centre line. It is wise to note that grains close to the weld bulk show more equiaxed dendrite sub-grain microstructure compared to the grains at the fusion line which have a columnar dendrite substructure as a result of variation in the constitutional supercooling. However, it should be noted that this kind of changes in the sub-structure might be also as a result of the particular cross section that has been made (perpendicular to the welding direction).
Figure 4.15 Grain growth toward weld center line from plate two with pure nickel (Arrows show the fusion line or the interface of two weld pass).
4.5.2 Scanning electron microscopy (SEM)

In this section, the fractography of the cracks is described. In first instance, the crack surface is studied in order to analyze if the cracks have a smooth or brittle surface. Generally, by fractography or assessing the morphology of the crack surface, it is possible to confirm whether hot cracking has occurred during the solidification temperature range. For instance, the secondary electron images of the plate one deposited with alloy 625 are shown in Figure 4.16. All cracks are depicted at two different magnifications. The fractography clearly reveals fine protrusions which correspond to primary and secondary cellular-dendritic arms associated with a solidification fracture surface of a dendritic microstructure. The relatively smooth, dendritic and wavy appearance of the crack surface shows the typical morphology of the solidification cracks.

(a) Crack one (Mag= 300X)  
(b) crack one (Mag= 2000X)  
(c) crack two (Mag= 80X)  
(d) crack two (Mag= 600X)
The SEM images of plate two deposited with pure nickel almost show the same behavior. However, since the amount of alloying elements is lower than the alloying elements in the plate one, the fractographs of the cracks surfaces in the plate two demonstrate a more flat crack surface with less protrusions as a result of less segregation at grain the boundaries. Figure 4.17 depicts two cracks from plate two for two different magnifications.

**Figure 4.16** SEM images of three cracks from piece 2 of the plate one (Deposited with alloy 625).

**Figure 4.17** SEM images of two cracks from piece 3 of the plate one (Deposited with pure Ni).
4.5.3 Electron Backscatter Diffraction

The EBSD technique is able to reveal the microstructure aggregates with their associating crystallographic orientation in a quantitative manner. The Invers Pole Figure map can distinguish each grain with specific color codes via the crystallographic orientation changes (see section 3.5.2). Therefore, this map (IPF map) reveals that cracking occurs at interdendrite regions within one grain (transgranular) or at solidification grain boundaries (intergranular). In addition, the phase fractions and positions of these phases are determined within the microstructure by two different colors (blue and red) in the phase map (see Figure 4.18 (c)). However, the phase fractions cannot represent the global microstructure phase fractions and distributions since this analysis is operated locally. Besides, it was also possible to determine the easy growth direction of dendrites. For the present BCC and FCC phases it has been measured that the dendrite easy growth directions were always <100> that oriented closest toward the maximum heat flow direction. Table 4.6 shows these easy growth directions. Moreover, the most interesting outcome of the EBSD analysis achieved when the orientation relation between austenite (FCC) and ferrite (BCC) determined. By considering the Kurdumov-Sachs orientation relation which is \( \{110\}_{BCC} // \{111\}_{FCC} \), \( <111>_{BCC} // <110>_{FCC} \), it is possible to understand that the ferrite phase has been transformed from parent austenite phase. This relation exist by rotating the FCC crystal structure around \( <112> \) axis by 90°. This analysis has been done by OIM software that was already mentioned in the section 3.5.2.

In this investigation totally six EBSD measurements have been done in which three of these measurements are corresponding to plate one deposited with alloy 625 and the other three are conducted on plate two deposited with pure nickel. It should be mentioned for both plates only one of the results is presented here for description purposes. The other analysis are included in the appendix C. Figures 4.18 and 4.20 show the EBSD results for plate one and two, respectively.

- **Plate one (With Alloy 625):**

![Plate one (With Alloy 625) - Image quality (IQ) map.](image1)

![Plate one (With Alloy 625) - IPF map.](image2)
Figure 4.18 EBSD quantitative analysis; (a) IQ map, (b) IPF map, (c) Phase map with K-S relation boundaries, (d) magnification of image (c) and (e) Phase fractions and K-S boundary fraction.

on first sight, the IPF map of Figure 4.18 (b), it is clearly shown that the crack initiated and propagated along the solidification austenite grain boundary. It should be noted that the dominant phase at this position of the weld bead is austenitic with a FCC crystal structure. In addition, Figure 4.18 (c) and (d) demonstrate how the red austenite (FCC) phase transforms to a blue ferritic phase (BCC). It can be seen that all the bcc regions are surrounded by yellow boundaries which describe the K-S orientation relation between the parent austenite phase that transformed into the ferrite phase. Quantitative analysis of the EBSD can precisely give statistical data regarding to the fraction of the phases present and the crystallographic orientation of each aggregate in Figure 4.18 (e) and (b) respectively. Figure 4.19 shows the position of the crack in figure 4.18 that has been analyzed by EBSD.
Figure 4.19 OM image of the crack position relative to weld bead for the EBSD analysis in figure 4.16 [etched with Kallings].

- **Plate two (With pure Ni):**

  ![Image](image_url)

  (a) Image quality (IQ) map.

  (b) IPF map.

  (C) Phase map with K-S orientation.

  (d) Magnified of the image (c) considering K-S orientation shown with yellow boundaries.
Figure 4.20 EBSD quantitative analysis; (a) IQ map, (b) IPF map, (c) Phase map with K-S relation boundaries, (d) magnified of the image (c) and (e) Phase fractions and K-S boundary fraction.

The same measurement has been done for a crack in the plate 2 (deposited with pure nickel). Figure 4.20 indicates that the crack is along the parent austenite grain, however, the ferritic subgrain structure (BCC) can be seen, colored in blue, within the prior austenite grain. The solidification subgrain boundaries are dominantly austenitic with a FCC structure colored with red. Figure 4.20 (d) again proves the K-S orientation relationship between austenite and ferrite phases. In addition, the most dominant phase at the location of the analysis is iron-alpha (ferrite). Figure 4.21 illustrates the position of the crack where the EBSD analysis was conducted.

![Image of crack position](image)

**Figure 4.21** OM image of the crack position relative to weld bead for the EBSD analysis in figure 4.18 [etched with Kallings].

In order to statistically describe the EBSD analysis data for the whole measurements, Table 4.6 is drawn. There are only two crack regions that their easy growth directions are tabled below. The reasons to have only two data for easy growth family directions were related to the local microstructure features around the cracks such as equiaxed dendrites, fully austenitic solidification grains with no subgrain microstructure. Besides, it is seen on the Table 4.6 that the total phase fractions is not precisely hundred percent (100%) which is as a result of data points with low confidence index (CI). These data points were not analyzed by the software because of...
low reliability. Therefore, it should be expected that the total phase fraction be a little bit lower than one hundred percent.

The whole EBSD measurements with their associating maps are mentioned in the Appendix C.

<table>
<thead>
<tr>
<th>Samples from 2nd piece</th>
<th>Local phase fractions (ς and α)</th>
<th>Crack position</th>
<th>&lt; easy growth &gt;</th>
</tr>
</thead>
</table>
| 625 crack 1            | ς (FCC)= 14%  
                         α (BCC)= 80%  | Intergranular (SGB)  
                         | -              |
| 625 crack 2            | ς (FCC)= 89%  
                         α (BCC)= 3%  | Intergranular (SGB)  
                         | -              |
| 625 crack 3            | ς (FCC)= 51.2%  
                         α (BCC)= 40.9%  | Intergranular (SGB)  
                         | [-1,-27,3],[0,-29,3],[1,-27,3],… |
| Pure crack 1           | ς (FCC)= 88.4%  
                         α (BCC)= 7.5%  | Intergranular (SGB)  
                         | -              |
| Pure crack 2           | ς (FCC)= 45.7%  
                         α (BCC)= 51.5%  | Intergranular (SGB)  
                         | [0.7, -23],[26.2,-3],[-1,-3,16], … |
| Pure crack 3           | ς (FCC)= 31.6%  
                         α (BCC)= 65.7%  | Intergranular (SGB)  
                         | -              |

It should be mentioned that the phase fractions are accurately measured by excluding the cracks from the local analysis in order to skip the error by considering the blue or red points within the cracks.

12 SGB = Solidification Grain Boundary.
4.5.4 Chemical analysis (EDS)

The chemical analysis by energy dispersive X-ray spectrometry (EDS) could specify the alloying element distribution and their segregation behavior. Several line and area map scans were conducted on both plate one and two at various regions. This analysis could also provide a phase map based on the chemical compounds and their associating elements fractions. In order to classify the analysis of the plate one and two, from each plate one are map scan is considered in this section in order to understand the distribution and segregation of elements at the steel weld bead (second layer).

- **Plate one (with Alloy 625)**

Figure 4.22 illustrates the fraction of the red and yellow regions around the crack (see figure 4.22 (a)). These regions are classified based on the elements concentration and distribution and distinguished by different colors. This classification with itself cannot be considered as the phase map. However, it can clearly state the distribution of elements and the tendency for the formation of the specific phase at the local region with the same color. By the help of the EBSD and EDS analysis, it would be possible to understand the microstructure evolution and stable phases at the room temperature. Figure 4.22 (b) represents the two dominant regions around the crack. Both yellow and red regions have similar main chemicals but with different fractions. Table 4.7 (a) and (b) show the concentration of all elements within these two red and yellow regions respectively.

![Regional map by EDS and associating fractions.](image)

(a) Regional map.  
(b) Fractions of two main regions.

**Figure 4.22** Regional map by EDS and associating fractions.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Weight %</th>
<th>Fe</th>
<th>Ni</th>
<th>Cr</th>
<th>Co</th>
<th>Nb</th>
<th>Mo</th>
<th>S</th>
<th>Mn</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>71.28</td>
<td>13.68</td>
<td>12.83</td>
<td>0.1</td>
<td>0.14</td>
<td>0.94</td>
<td>0.29</td>
<td>0.13</td>
<td>0.01</td>
</tr>
<tr>
<td>(a) Red region.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Elements</th>
<th>Weight %</th>
<th>Fe</th>
<th>Ni</th>
<th>Cr</th>
<th>Co</th>
<th>Nb</th>
<th>Mo</th>
<th>S</th>
<th>Mn</th>
<th>Ti/Al</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td>63.27</td>
<td>16.14</td>
<td>15.37</td>
<td>0.11</td>
<td>1.3</td>
<td>1.52</td>
<td>0.6</td>
<td>0.24</td>
<td>0.1</td>
</tr>
<tr>
<td>(b) Yellow region.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Table 4.7** Chemical composition of the two regions: (a) Red region, (b) Yellow region.

It can be predicted that at low temperatures red regions in the Figure 4.22 (a) forms the ferrite phase in which it has higher iron content. This prediction has been proved by the EBSD analysis.
that those red regions (dendrite cores) mostly form the ferrite phase with BCC structure and the yellow regions (interdendritic regions) form the austenite phase (FCC). Moreover, the area map scan around the crack could reveal the segregation behavior of alloying elements, particularly the low melting point eutectic phase formers. Figure 4.23 depicts the segregation behavior of the alloying elements at the dendrite and interdendritic regions specifically for those eutectic phase formers at the steel layer (second layer).

Figure 4.23 Chemical maps of alloying elements in the plate one at steel layer (deposited with Alloy 625).
By comparing the Figure 4.23 with 4.18 (b), it can be seen that the S, Nb and Mo segregate at interdendritic and grain boundaries regions while Fe, Ni and Cr did not segregate at interdendritic regions. However, it is clear that Cr and Ni behave in a same manner that at regions where iron accumulated, chromium and nickel depleted.

- **Plate two (with pure Ni)**

The same sort of chemical analysis results is provided in this section in Figures and Tables below. By assessing the chemical analysis of plate one and two, it is possible to compare the severity of the elements segregation behavior. These segregations would lead to formation of microstructural features such as second phase constituents and defects particularly crack.

![Phase map](image)

(a) Phase map.

![Fractions of four main regions](image)

(b) Fractions of four main regions.

**Figure 4.24** Regional map by EDS and associating fractions.

**Table 4.8** Chemical composition of the four regions; (a) Red region, (b) Green region, (c) Brown region, (d) Blue region.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Fe</th>
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<th>Mn</th>
<th>Ti</th>
<th>S</th>
<th>P</th>
<th>Al</th>
<th>Si</th>
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</thead>
<tbody>
<tr>
<td>Weight %</td>
<td>77.3</td>
<td>20.23</td>
<td>1.19</td>
<td>0.59</td>
<td>0.02</td>
<td>0.03</td>
<td>0.08</td>
<td>0.56</td>
</tr>
<tr>
<td>(a) Red region.</td>
<td></td>
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<td></td>
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</tr>
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<table>
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<th>Mn</th>
<th>Ti</th>
<th>S</th>
<th>P</th>
<th>Al</th>
<th>Si</th>
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</thead>
<tbody>
<tr>
<td>Weight %</td>
<td>70.95</td>
<td>25.94</td>
<td>1.37</td>
<td>0.96</td>
<td>0.04</td>
<td>0.04</td>
<td>0.01</td>
<td>0.69</td>
</tr>
<tr>
<td>(b) Green region.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
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<table>
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<tr>
<th>Elements</th>
<th>Fe</th>
<th>Ni</th>
<th>Mn</th>
<th>Ti</th>
<th>S</th>
<th>P</th>
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<td>(c) Brown region.</td>
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<td>(d) Blue region.</td>
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By considering the binary phase diagram of Fe-Ni (see Figure 4.7) and the chemical composition of different regions in Figure 4.24 (a), it would be expected that the primary solidification mode is indeed austenitic. However, following cooling to the room temperature the austenite transforms to ferrite at localize regions that the nickel concentration is low relative to the other regions. This can be seen in the red regions of Figures 4.24 (a) and 4.20 (c) in which the dendrite cores are ferritic (BCC) while the interdendritic regions (green regions) are austenitic (FCC) with higher concentration of nickel. In addition, the blue and brown regions have relatively higher nickel concentration have fully austenitic microstructure consisting of prior austenite grains with no sub-grain microstructure (see Figure 4.20 (b)).
Figure 4.25 clearly illustrates the distribution and segregation behavior of the chemical within the steel layer diluted with pure nickel. By comparing each chemical map of the elements in Figure 4.25 (b, c, and d) with (a), it can be seen that iron (Fe) concentrates at the dendrite cores while nickel (Ni) concentrates more at dendrite boundaries. Therefore, it was analyzed by the EBSD that dendrite cores transformed to ferrite while dendrite boundaries with higher accumulation of nickel remained austenitic. Besides, it can be mentioned that titanium (Ti) segregates at interdendrite regions and particularly at prior austenite boundary in which the crack initiated and propagated. In order to prove this statement Figure 4.20 (b) can describe that the crack is within the prior austenite grain boundary. In addition, this has been also described by chemical composition of the red and green regions in Figure 4.24 (a) that have been tabulated in Table 4.8 (a) and (b). Therefore, the highest amount of segregations always occurs at solidification grain boundaries (SGB) compare to solidification sub-grain boundaries (SSGB). Consequently, the crack susceptibility is higher at the SGBs relative to interdendrite regions (SSGBs).

Although, P could hardly show its segregation behavior clearly, sulfur slightly segregated at the sub-grain boundaries. These two elements (S and P) have unclear chemical maps (see Figure 4.25 (e) and (f)) since they have relatively low concentration in the microstructure.
CHAPTER 5
5.0 DISCUSSION

In this chapter, based on the theoretical background and the result that have been mentioned in the chapter 2 and 4 respectively, the microstructure evolution and particularly the crack formation mechanisms are discussed. First the microstructure of the both plates is characterized in the following section in order to have better understanding about the microstructural features that can lead to the crack formation. Subsequently, the solidification crack formation mechanisms based on the existing results from the chapter 4 and corresponding theories are described.

5.1 Microstructure characterization

As it was already discussed in the section 4.1, the effort was to minimize the dilution and mixing of the first and the second layer by optimizing the welding parameters. However, the dilution measurements showed relatively high amount of the dilution in the second layer by the Ni-base layer. The solidification mode of the diluted C-Mn steel weld pool was assessed by the EBSD and EDS analysis (see section 4.5.3 and 4.5.4). Both plates showed austenitic solidification. In addition, the ternary and binary phase diagrams in the section 4.3 for the plate one and two also represented the same solidification mode and the phase transformation sequences according to the nominal chemical composition measured by the EDS. The solidification starts with the heterogeneous nucleation of the austenite grains at the fusion line. Subsequently, since both the diluted second layer (C-Mn steel) and the first layer (Ni-base) have the FCC crystal structure, epitaxial growth of the nuclei occurs from the Ni-base layer toward the center of the weld bead (parallel to maximum temperature gradient). The large EBSD scan of both layers in the appendix C also illustrates this fact. The FEM model gave general information regarding to the heat flow direction (opposite to the maximum temperature gradient) and the temperature distribution. This model validated by the experimental temperature profile measurements that was mentioned in the section 4.2. Eventually, the thermal analysis by the experiment and the simulation helped to understand the microstructure evolution and particularly the grain growth directions.

Following the solidification, prior austenite grains based on their position relative to the weld bead and the local constitutional super cooling showed either sub-grain or sub-grain free microstructure. After cooling to the room temperature, the EBSD results (see section 4.5.3) showed those prior austenite grains that had sub-grain microstructure, consist of ferritic dendrite cores and the austenitic interdendrites. The EBSD also confirmed that the ferrite dendrites have been transformed from the parent austenite by the K-S orientation relation between FCC and BCC crystal structure. The EDS analysis in the plate one unraveled that the ferritic dendrite cores have higher iron content relative to the austenitic interdendrites which have lower iron content. Besides, the Cr and Ni segregate more at the interdendrite regions with austenitic microstructure (FCC crystal structure). The similar behavior was seen by the EDS in the plate two, in which the iron content of the dendrite cores was high while the nickel concentration (austenite stabilizer) was low. Besides, by the segregation of nickel into the interdendritic region austenitic microstructure was seen. Therefore, from the EDS analysis on the both plates, it is concluded that iron and nickel showed contrary segregation behavior. Furthermore, it should be considered that in the plate one as of the higher amount of alloying elements, more segregation behavior was seen particularly Nb, Mo and S. These elements form low eutectic point constituents at the sub-grain boundaries that increase the solidification temperature range. However, in the plate two the amount of alloying elements are remarkably lower than the plate one, but segregation was occurred particularly for Ti. It has been reported in the literatures that titanium also forms eutectic phases at the interdendritic regions that can extend the solidification temperature range.
5.2 Solidification crack characterization

As it was indicated in the section 4.5.1, all the cracks were observed by the LOM. All those cracks in the both plates were located at the bulk of the fusion zone, thus, they are considered as the solidification cracks. The formation mechanism of the solidification cracks was already mentioned in the section 2.8.1.

In this section, the all solidification cracking theories that have been proposed over the years are discussed briefly. Subsequently, based on the observed microstructural features in the current study, the most probable mechanisms that may lead to the solidification cracking are discussed. There is a common factor between all of the solidification cracking theories in which the low temperature liquid films exist at the end of the solidification and may be pulled apart due to the stresses experienced during cooling. These liquid films that exist at the terminal stage of the solidification are a direct result of solute segregation originating from non-equilibrium cooling conditions.

The commonly recognized theories for discussing solidification cracking behavior include (in chronological order): Shrinkage – Brittleness Theory [87, 88], Strain Theory [89], Generalized Theory [90], Technological Strength Theory [91], and Modified Generalized Theory [92].

The Shrinkage-Brittleness Theory proposed in the late 1940’s by Bochvar for the cracks in aluminum casting above the solidus temperature [87]. Following the Bochvar’s work with aluminum casting, Pumphrey completed the theory to describe the cracking behavior of aluminum alloy welds during solidification [88]. The basis of this theory is that a solidifying alloy exposes a temperature in which dendrite interlocking occurs during cooling. This temperature is called coherence temperature. The proportion of the liquid to solid above the coherence temperature is large enough that cracking will not occur. However, below the coherence temperature, solid-solid interaction begins to take place between adjacent dendrites and the remaining liquid films may rupture due to the strain accommodation. If sufficient amount of liquid exist (such as in alloys with the large amount of eutectic formation), crack healing may occur where liquid flow into crack or freshly formed void.

In the early 1950’s Pellini, developed the Strain Theory to describe the observation of hot tears in castings and then for the occurrence of solidification cracking in weld metals. He explained solidification process undergoes into two stages; first a “mushy condition” and then followed by a “film stage” where solidification cracking may occur. The mushy condition is considered by simultaneous presence of solid and liquid at the solidifying weld pool, but the strain at this stage is uniformly distributed and prevents cracking. During the film stage, the continuous liquid film exists along interdendritic region while strain accumulation at interdendritic regions occurs due to the temperature gradient in the solid. Consequently, the liquid film can be separated at interdendritic regions.

In 1960 Borland introduced the Generalized Theory to explain the solidification cracking phenomenon and overcome the apparent shortcomings of both the Shrinkage – Brittleness and Strain Theory. Borland classified the solidification process into four stages based on the quantity and distribution of liquid. During the first stage dendrites are widely dispersed by large amount of liquid (stage 1). Following the first stage, at the second stage Borland defined the coherent temperature that upon crossing this temperature dendrite interlocking occurs. If below coherent temperature the amount of liquid was not enough, cracking might occur just below the coherent temperature (stage 2). Upon further cooling, a critical temperature is reached in which dendrite formation has progressed to the point that interdendritic liquid networks becomes isolated and cracking healing is not possible (stage 3). Borland referred to this region as the critical solidification range (CSR). It is in this region, below the critical temperature and above the solidus temperature (complete solidification), that solidification cracking occurs. Following the third stage, at the final stage of the solidification, no liquid present any more and cracking
cannot occur (stage 4). It should be noted that under non-equilibrium condition that constitutional super cooling occurs, the solidus temperature is depressed due to the limited diffusion. Therefore, the critical solidification range (CSR) is extended and crack susceptibility is increased. In addition, Borland proposed new concepts regarding to solidification cracking that can stimulate cracking. The new concept was interface energies either the grain boundary or solid-liquid interface energy that plays a role in solidification cracking behavior by controlling wetting characteristics.

In 1962 Prokhorov proposed a solidification cranking theory based on the mechanical prospective. This theory is known as the Technological Strength Theory. He explained the solidification cracking as a result of decrease in the ductility through a temperature range, known as the brittle temperature range (BTR). During the solidification of the weld pool, the strain accumulation as a result of thermal contraction and external applied load occurs. If the strain reaches a critical value within the solidifying weld pool and within the BTR, solidification cracking will happen.

The most recent Theory was introduced by Matsuda in the early 1980’s. This theory was modification of Generalized Theory by Borland. Matsuda and his associates assessed the solidification cracking behavior by in-situ microscopic observations.

Matsuda modified the two concepts of Borland based on his observations. First, Matsuda declared that the coherent temperature was higher than originally proposed by Borland, which was indicated by extensive solid network formation early in the solidification process. Secondly, the CSR can be divided into two regions in itself; region one which is at higher temperature in the CSR (stage 3h) that solidification cracking initiation occurs due to the presence of the liquid film along the grain boundary. Upon further cooling, the region two of the CSR (stage 3l) is reached which is not susceptible to crack initiation, but to the propagation due to the presence of the liquid droplets along the grain boundary.

In the current study, the microstructure was investigated carefully by various techniques. The results were show and discussed in the chapter 4 and also in the chapter 5 (see section 5.1). As it was mentioned in the section 5.1, the microstructure showed dendritic structure with remarkable segregation of the alloying elements. In the plate one, by segregation of Nb, Mo and S at interdendritic regions the eutectic phases formed such Laves, carbide and FeS (see section 4.3). These eutectic phases increase the critical solidification range (CSR) and hence increase the crack susceptibility. Besides, during cooling the thermal contraction (shrinkage) occurs with the external clamping load which introduce the tensile stresses (see Figure 5.1). The response to this microscopic stresses would be localize strain on the liquid film along the prior austenite grain boundaries which lead to separation of the liquid film and initiation of the crack. Similar mechanisms occurs in the plate two, but the segregation of the detrimental elements (i.e. Nb, Mo) as in the plate one were not occurred. Instead the Ti and S segregated to the sub-grain boundaries. In addition, it was shown in the section 4.4 that Ti increase the critical solidification range (CSR) which is within the STR region. Hence, titanium can increase the solidification crack susceptibility. Eventually, the susceptibility calculation (see section 4.5) of the both plates revealed that the plate one has 1.6 times higher crack susceptibility compares to the plate two.
Figure 5.1 Schematic representation of the solidification crack with corresponding strain direction.
5.3 Recommendation

In this study the pure nickel wire was used in order to minimize the chemicals within the weld pool. However, the pure nickel consumable had 3 (wt. %) titanium that can extend the solidification temperature range and increase crack susceptibility. Besides, the steel wire had its own alloying elements and stray elements such as S, P and Si that increase crack susceptibility. Consequently, in terms of chemicals modification, studying the more pure nickel layer with pure iron wire would be still important to assess the effect of alloying elements in introducing the solidification crack in the microstructure. In order to study particularly the effect of the chemicals in formation of the microstructure cracks, it is recommended to use the same welding technique as it was used for this study which was GTAW technique. Based on the achieved results from the purification of the chemicals, changing the welding technique in order to minimize the heat input to avoid cracks is also recommended. In this study, although the lowest heat input was used, the dilution numbers were relatively high. Therefore, changing the deposition technique would be helpful to minimize the dilution and to change the weld pool shape. Moreover, it is widely accepted that the stresses always present during the terminal stages of solidification, thus, deposition techniques can play significant role in inducing the stresses in the microstructure and subsequently the crack formation.
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### Appendix A

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<tr>
<td>2.1e+11, 1470.</td>
<td>1509.</td>
</tr>
</tbody>
</table>

**Specific Heat**

<table>
<thead>
<tr>
<th>Specific Heat</th>
<th>T</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.5e+08, 25.</td>
<td></td>
</tr>
<tr>
<td>5.5e+08, 600.</td>
<td></td>
</tr>
<tr>
<td>6.6e+08, 1450.</td>
<td></td>
</tr>
</tbody>
</table>

*Physical Constants, absolute zero=-273.**

** PREDEFINED FIELDS**

** Name: Predefined Field-1  Type: Temperature
** Initial Conditions, type=TEMPERATURE Part-1-1.base, 25.

** Name: Predefined Field-2  Type: Temperature
** Initial Conditions, type=TEMPERATURE Part-1-1.layer1, 25.

** Name: Predefined Field-3  Type: Temperature
** Initial Conditions, type=TEMPERATURE Part-1-1.layer2, 25.

** STEP: Step-1

**

*Step, name=Step-1, inc=1001
*Heat Transfer, end=PERIOD, deltmx=100.
0.01, 4., 4e-05, 0.1,

**

*model change, remove Part-1-1.layer2

** LOADS**

**

Name: Load-1  Type: Body heat flux
*oflux, amplitude=Amp-1
Part-1-1.layer1, BF, 18000.

** Name: Load-3  Type: Surface heat flux
*dstflxSurf-3, SNU, 0.**

** INTERACTIONS**

** Interaction: Int-1
*sflmlayer1, FNU, 15., 0.

** Interaction: Int-2
*sflmlayer11, F, 15., 0.05

**

** OUTPUT REQUESTS

**

*restart, write, frequency=0

**

** FIELD OUTPUT: F-output-1

**

*output, field, variable=PRESELECT
*output, history, frequency=0

.*
**STEP: Step-2
**
*Step, name=Step-2, inc=1001
*Heat Transfer, end=PERIOD, deltmx=100. 0.01, 600., 0.0006, 30.,
**
** LOADS
**
** Name: Load-1    Type: Body heat flux
*Dflux, op=NEW
** Name: Load-3    Type: Surface heat flux
*Dflux, op=NEW
**
** OUTPUT REQUESTS
**
*Restart, write, frequency=0
**
** FIELD OUTPUT: F-Output-1
**
*Output, field, variable=PRESELECT
*Output, history, frequency=0
*End Step
**

** STEP: Step-3
**
*Step, name=Step-3, inc=1001
*Heat Transfer, end=PERIOD, deltmx=100. 0.01, 4., 4e-05, 0.1,
**
*model change,add
Part-1-1.layer2
** LOADS
**
** Name: Load-2    Type: Body heat flux
*Dflux, amplitude=Amp-1
Part-1-1.layer2, BF, 23000.
** Name: Load-4    Type: Surface heat flux
*DfluxSurf-6, SNU, 0.
**
** INTERACTIONS
**
** Interaction: Int-1
*Sfilm, op=NEW
** Interaction: Int-2
*Sfilm, op=NEW
layer11, F, 15., 0.05
** Interaction: Int-3
*Sfilm, op=NEW
layer2, FNU, 15., 0.
**
** OUTPUT REQUESTS
**
*Restart, write, frequency=0
**
** FIELD OUTPUT: F-Output-1
**
*Output, field, variable=PRESELECT
*Output, history, frequency=0
*End Step
** STEP: Step-4
**
*Step, name=Step-4
*Heat Transfer, end=PERIOD, deltmx=100.
 0.01, 600., 0.0006, 30.,
**
** LOADS
**
** Name: Load-2 Type: Body heat flux
*pflux, op=NEW
** Name: Load-4 Type: Surface heat flux
*dsflux, op=NEW
**
** OUTPUT REQUESTS**
*Restart, write, frequency=0
**
** FIELD OUTPUT: F-OUTPUT-1
**
*Output, field, variable=PREAD
*Output, history, frequency=0
*End Step

sub
  SUBROUTINE DFLUX(FLUX,SOL,KSTEP,KINC,TIME,NOEL,NPT,COORDS,JLTYP,
  1      TEMP,PRESS,SNAME)
C
C      INCLUDE 'ABA_PARAM.INC'
C
parameter (ZERO=0.D0,ONE=1.D0,TWO=2.D0,THREE=3.D0)
DIMENSION COORDS(3),FLUX(2),TIME(2)
 CHARACTER*80 SNAME
C
  v=2.5
  d=v*TIME(1)
C
  x=COORDS(1)
y=COORDS(2)
z=COORDS(3)
  if(KSTEP.EQ.1) THEN
    q=0.7*130*12*1000*0.4
    x0=-4
    y0=1.005365
    z0=5
  END IF
  if(KSTEP.EQ.3) THEN
    q=0.7*110*12*1000*0.4
    x0=-4
    y0=2.003347
    z0=5
  END IF

  a=5
  PI=3.1415
  heat=3.0*q/(2*a*PI)
  shape=exp(-3.0*(x-x0)**2/a**2+3.0*(z-z0+d)**2/a**2)

  FLUX(1)=heat*shape

RETURN
END
### Appendix B

**Figure 6.1** FCC and carbide phase in the ternary Fe-Cr-Ni phase diagram at 1300°C.

**Figure 6.2** FCC and carbide phase in the ternary Fe-Cr-Ni phase diagram at 800°C.
Conditions: 
\[ T = 773.15 \, \text{K}, \, \omega(\text{Cr}) = 0.65, \, \omega(\text{Ni}) = 0.75 \]

DEGREES OF FREEDOM 0

Temperature 773.15 K < 500.00 K, Pressure 1.000000E+05

Number of moles of components 1.000000E+00, Mass in grams 5.604000E+01

Total Gibbs energy -3.146222E+04, Entropy 1.79114E+04, Volume 7.23934E-06

<table>
<thead>
<tr>
<th>Component</th>
<th>Molar</th>
<th>W-Fraction</th>
<th>Activity</th>
<th>Potential</th>
<th>Ref.</th>
<th>Stat</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>5.6590E-01</td>
<td>3.1340E-01</td>
<td>5.6915E-01</td>
<td>Mass Fractions:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cr</td>
<td>1.0869E+01</td>
<td>1.0925E-00</td>
<td>2.9581E-00</td>
<td>FE 7.4552E-01</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ni</td>
<td>1.1718E-01</td>
<td>1.2359E-01</td>
<td>1.2938E-01</td>
<td>5.3281E-04</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>1.5018E-02</td>
<td>1.5018E-02</td>
<td>1.5018E-02</td>
<td>1.5018E-02</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Figure 6.3** BCC (α'), BCC (α), FCC, Laves and carbide phase in the ternary Fe-Cr-Ni phase diagram at 500°C.
• Appendix C

• Plate one (with Alloy 625)

(a) Image quality (IQ) map.  
(b) IPF map.  
(c) Phase map at right side of the crack with K-S orientation relationship.

Figure 6.4 EBSD quantitative analysis; (a) IQ map, (b) IPF map, (c) Phase map with K-S relation boundaries.
Figure 6.5 EBSD quantitative analysis; (a) IQ map, (b) IPF map, (c) Phase map with K-S relation boundaries.
Plate two (with pure Ni)

Figure 6.6 EBSD quantitative analysis; (a) IQ map, (b) IPF map, (c) Phase map with K-S relation boundaries.
Figure 6.7 EBSD quantitative analysis; (a) IQ map, (b) IPF map, (c) Phase map with K-S relation boundaries.
REFERENCES


Lippold, J. C., in Technology and Advancements and New Industrial Applications in Welding, Proceedings of the Taiwan International Welding Conference '98, Eds. C. Tsai and H. Tsai, Tjing Ling Industrial Research Institute, National University, Taipei, Taiwan, 1998.


