Induction Bending of Internally Clad Steel Pipes: Failure Mechanisms & Processing
Parameter Optimization in Ni-base Alloy Weld Overlays

THESIS

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By

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Abstract

Cracking in corrosion resistant clad overlays on low alloy carbon steel pipes made with Alloy 825 has been experienced in the industry in an effort to reduce production costs by changing the cladding material from the more costly Alloy 625. A detailed metallurgical investigation was carried out to understand the root cause of the cracking phenomenon. Analysis consisting of optical and scanning electron microscopy along with hardness traverses and mapping revealed weld metal heat-affected zone liquation cracks in the second overlay after welding, as well as a region of high hardness in the planar growth region of the weld metal directly adjacent to the dissimilar metal weld interface. Serial sectioning shows that ductility-dip cracks form between the pre-existing weld metal liquation cracks and microcracks forming in the embrittled planar growth region, ultimately leading to through-thickness cracks of the overlay during induction bending. The strain-to-fracture test was modified to replicate the bending procedure, and an optimal parameter window consisting of bending temperature, total strain, and strain rate was identified based on test results.

ThermoCalc pseudo-binary phase diagrams were created using both the equilibrium and Scheil models. Neither diagram predicts the formation of any low melting eutectic constituents that could lead to liquation during welding. EDS results show spikes of titanium in the bulk weld metal, presumably due to the presence of
titanium carbide particles. A plot of solidus temperature versus weight percent titanium created in Thermocalc reveals severe melting point depression in the Alloy 825 matrix as the titanium content increases. It is hypothesized that the weld metal heat-affected zone liquidation cracking occurs via constitutional liquation of titanium carbide particles in close proximity to the fusion zone during welding of the second overlay.

The region of high hardness at the DMW interface was observed to correlate with microcracks forming in that region as initial straining began during induction bending. Gleeble testing showed that avoidance of microcrack formation by manipulation of bending parameters is not possible. A study on hardness at the interface during processes steps was performed, revealing that the hardness increase occurs during the normalizing post-weld heat treatment before bending. A DICTRA diffusion model was carried out to further understand the mechanism behind the increased hardness in the planar growth region. Results show a pile-up of carbon extending approximately 100-150 microns into the weld metal at the DMW interface. It is theorized that avoidance of microcracks at the DMW interface is best achieved by elimination of the PWHT.

Replication of the induction bending process in the Gleeble thermo-mechanical simulator was achieved by modification of the strain-to-fracture test. Results show that reducing the strain rate opens the safe bending parameter window in terms of temperature and total strain. A bending temperature of 975 ± 25 °C is suggested to successfully induction bend pipes without causing ductility-dip cracking.
To my wonderful wife, Delsi, and our future together.
Acknowledgments

I would like to thank my advisor, Professor Boian Alexandrov, for his wealth of knowledge and guidance throughout the duration of this project. Our research would not have been possible without support and materials provided ExxonMobil, particularly from Dr. Desmond Bourgeois, who met with us frequently to discuss our results and plans to move forward. I am also grateful for the patient assistance provided by Dr. Carolin Fink regarding Gleeble programming and SEM characterization, along with Dr. Tyler Borchers for his assistance with microhardness mapping. Finally, I would like to thank all of my professors and fellow students in the Welding Engineering program for being a constant sounding board and source of inspiration as we discussed our research and findings together.
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Chapter 1: Introduction

In an effort to reduce manufacturing costs of subsea pipelines in the Oil and Gas industry, low alloy carbon steel pipes are commonly clad with a corrosion resistant alloy (CRA) on the internal diameter. Historically, nickel-base Alloy 625 has been used as a cladding material. Work has been done recently to further decrease manufacturing costs by using Alloy 825 as an alternate cladding material. The appeal of Alloy 825 as a cladding material stems from its differences in chemical composition when compared to Alloy 625 (see Table 1). By using an alloy with a reduced amount of nickel and increased percentage of iron, a significant reduction in material cost can be achieved.
Table 1: Chemical composition ranges in weight percent of Alloys 625 and 825 (1) (2).

<table>
<thead>
<tr>
<th>Element</th>
<th>Alloy 625</th>
<th>Alloy 825</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>58.0 min.</td>
<td>38.0-46.0</td>
</tr>
<tr>
<td>Cr</td>
<td>20.0-23.0</td>
<td>19.5-23.5</td>
</tr>
<tr>
<td>Fe</td>
<td>5.0 max.</td>
<td>22.0 min.</td>
</tr>
<tr>
<td>Mo</td>
<td>8.0-10.0</td>
<td>2.5-3.5</td>
</tr>
<tr>
<td>Nb</td>
<td>3.15-4.15</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>0.10 max.</td>
<td>0.05 max.</td>
</tr>
<tr>
<td>Mn</td>
<td>0.50 max.</td>
<td>1.0 max.</td>
</tr>
<tr>
<td>Si</td>
<td>0.50 max.</td>
<td>0.5 max.</td>
</tr>
<tr>
<td>P</td>
<td>0.015 max.</td>
<td></td>
</tr>
<tr>
<td>S</td>
<td>0.015 max.</td>
<td>0.03 max.</td>
</tr>
<tr>
<td>Al</td>
<td>0.40 max.</td>
<td>0.2 max.</td>
</tr>
<tr>
<td>Ti</td>
<td>0.40 max.</td>
<td>0.6-1.2</td>
</tr>
<tr>
<td>Co</td>
<td>1.0 max.</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>1.5-3.0</td>
<td></td>
</tr>
</tbody>
</table>

CRA overlays are produced by using a tandem hot wire gas tungsten arc welding (GTAW) process. Two separate passes are performed in order to accomplish the desired dilution in the final overlay. Pipes are then penetrant tested and normalized at 1000 °C for 15 minutes prior to induction bending. After bending is complete, excess material is removed from the bends and the pipes are quenched and tempered to achieve desired properties in the base metal. An overview of the manufacturing process can be seen in Figure 1.
Severe cracking of corrosion-resistant nickel-base alloy 825 overlays has been observed after during the induction bending process. The bending temperature and initial metallography suggest ductility dip cracking (DDC), although the propagation of solidification cracks during bending may also present a potential cause. Further understanding of the cracking mechanism will allow industry to optimize the induction bending parameter window and produce crack-free CRA overlaid pipes for subsea service.
2.1 Solid Solution Strengthened Ni-base Alloys

2.1.1 Overview of Solid Solution Strengthened Ni-base Alloys

Development of commercial solid solution nickel based alloys first began in the early 1900s when Ambrose Monell first introduced nickel-copper alloy later known as MONEL® (3). Since then, a wide variety of nickel-base solid solution strengthened alloys have been produced, being well known for their strength and corrosion resistance at elevated temperatures. These nickel-base alloys are typically strengthened by the addition of copper, chromium, iron, tungsten, and molybdenum as alloying elements (4) (5). The addition of such elements in solid solution creates distortions within the nickel-rich fcc atomic lattice, thus inhibiting dislocation motion and providing a stiffening mechanism within the material. To date, a variety of alloy families exist among solid solution strengthened nickel-base alloys, including nickel-chromium, nickel-chromium-molybdenum, and nickel-chromium-iron.

The nickel-chromium-iron alloys are of most relevance to the research conducted in this investigation. This family of alloys was developed to bridge the performance and cost gaps between nickel-chrome alloys and austenitic stainless steels (6). In many cases,
nickel-iron-chromium alloys are compositionally very similar to high-nickel austenitic stainless steels, and although they can contain more iron than nickel in some cases, they are still commonly referred to as nickel-base alloys (6). The high iron content of such alloys allows for a drastic reduction in production expense, albeit at the cost of reducing solubility of molybdenum, subsequently reducing corrosion resistance. Despite a reduction in corrosion resistance, the low cost factor of this family of alloys has allowed it to find a variety of industrial applications.

2.1.2 Characteristics of Alloy 825

2.1.2.1 Applications and Alloying Elements

Incoloy® alloy 825 is a nickel-iron-chromium (Ni-Fe-Cr) alloy designed for superior corrosion resistance (1). Initially developed in the 1950’s (5), Alloy 825 is commonly used in applications involving chemical processing, pollution control, acid production, pickling operations, handling of radioactive waste, and oil and gas recovery, primarily in moderately sour oil and gas wells (1). While cold working can substantially strengthen the alloy (1), its predominant source of strength comes from solid solution strengthening by alloying elements such as copper, chromium, iron, and molybdenum (5). Corrosion resistance of Alloy 825 is achieved by adding a variety of alloying elements such as nickel, molybdenum, and chromium. These elements provide resistance to chloride-ion stress-corrosion cracking, reducing environments such as sulfuric and phosphoric acids, pitting and crevice corrosion, and oxidizing substances such as nitric
acid. Titanium is added to stabilize the material and prevent sensitization to intergranular corrosion (1).

2.1.2.1 Physical Properties and Hot Forming

In addition to excellent corrosion resistance, Alloy 825 has good mechanical properties in temperature ranging from -253 °C to approximately 500 °C (1). At room temperature, Alloy 825 has a yield strength of around 700 MPa with 40% elongation in the annealed state. Cold working of the alloy can increase yield strength up to 1000 MPa while dropping the elongation to 15% (1). The material exhibits no ductile-to-brittle transition temperature and has good impact strength from room temperature down to cryogenic temperatures (1). Extended time at temperatures over 540 °C can “result in microstructural changes that significantly lower ductility and impact strength” due to sigma phase embrittlement (1).

The stated hot-working temperature range for Alloy 825 is between 870 and 980 °C, as yield strength drops by a factor of three to five (see Figure 2) (1). Slow cooling of thick sections from temperatures in this range may result in sensitization to intergranular corrosion. An annealing heat treatment between 930 to 980 °C followed by fast cooling can be used to stabilize the material and restore resistance to intergranular corrosion.
In 2011, Man et al conducted a series of experiments to evaluate the hot plasticity of various corrosion-resistant alloys, including Alloy 825 (7). Hot compression and tension tests were performed between 1000 and 1300 °C at multiple strain rates. The resultant curves of maximum stress versus temperature and reduction in area versus area can be seen in Figure 3 and Figure 4, respectively. As temperature increased, ductility improved (Figure 3). No observations about the effects of strain rate on ductility were made. This research concludes that the ideal hot-working temperature range of Alloy 825 is between 1050 and 1240 °C, roughly 200 °C higher than the range previously stated by
Special Metals. Man et al. attribute the increase in the hot-working temperature range to a reduction in deformation resistance occurring upon complete dissolution of carbides, yielding a single austenitic phase. Additionally, the nil-ductility temperature of Alloy 825 was recorded as 1240 °C (see Figure 4).

Figure 3: Relationship between maximum stress and temperature for alloys 825, G3, and G3-Z (7).
2.1.2.2 Phases and Microstructure

On a microstructural level, Alloy 825 has an austenitic face-centered cubic matrix consisting predominantly of iron, nickel, and chromium (8). In the annealed condition, SEM examination has revealed three types of geometric precipitates evenly dispersed throughout the grains (9). These particles have been identified as $M_23C_6$ carbides with varying percentages of chromium and titanium. In an investigation by Shaikh et al (9), the Cr:Ti ratios were shown to be 1:1 in some particles, 10:1 in others, and 90-95% Ti with 2% Cr in the third type. Ageing did not have an effect on the shape, size, or composition of these precipitates.
Gradual precipitation of M\textsubscript{23}C\textsubscript{6} carbides occurs in Alloy 825 during extended periods of time at high temperatures. These precipitates tend to form at grain boundaries and are typically spherical in shape with an average diameter of 0.18 microns (8). Additional microanalysis studies have shown that two types of M\textsubscript{23}C\textsubscript{6} carbides form with the first consisting of 74% Cr and 10% Mo, and the second having an average composition of 45% Cr and 13% Mo (9). A time-temperature transformation diagram of M\textsubscript{23}C\textsubscript{6} carbides in Alloy 825 can be seen in Figure 5. The plot was produced by analyzing the content of carbon residue after varied aging times and temperatures, and relative amounts of M\textsubscript{23}C\textsubscript{6} carbides can be compared by the percentage of chromium shown on the plot. For reference, the carbon residue composition in the annealed condition is 3% Cr, 87% Ti (10).
Figure 5: Time-temperature transformation diagram for $M_{23}C_6$ carbides in Alloy 825 (10).

The maximum size of precipitates is reached after 100 hours of aging at 870 °C, although precipitate nucleation continues after well over 264 hours. Continuous films of grain boundaries carbides with an average width of 2 to 3 microns have been observed via transmission electron microscopy after an extended aging period (434 hours at 870 °C) (8). Various other authors have also reported similar observations of $M_{23}C_6$ carbides in Alloy 825 (11; 12). Extremely fine precipitates in the matrix have also been observed, appearing to be gamma prime (8), although absolute identification was not possible due to the minute nature of the particles.
2.1.2.3 Grain Boundary Sensitization

Local depletion of chromium in the austenitic matrix of Alloy 825 due to the formation of chromium carbides at grain boundaries when exposed to temperatures between 600 and 800 °C has been reported to reduce corrosion resistance in the material, commonly referred to as “grain boundary sensitization” (10; 12). Three primary mechanisms are used to combat sensitization in Alloy 825: 1) minimization of weight percent carbon to restrict carbide precipitation, 2) addition of titanium, which more readily form carbides than chromium, and 3) control of chromium carbide formation such that diffusion of chromium back in to the matrix is possible. All of the aforementioned mechanisms are employed to combat sensitization in Alloy 825, although the mode of precipitation has recently been shown to have the greatest effect of sensitization (10).

2.2 Dissimilar Metal Welds

2.1.1 Weld Metal Dilution

Weld metal dilution occurs during heterogeneous welding, and can be defined as “a change in composition of a filler metal due to its mixing with the base metal during the melting process” (3). Dilution is presented in terms of the amount of base metal in the fusion zone after welding. This can be calculated by using the formula below, where A
and C represent the area of base metal melted in to the fusion zone, and B represents the area of weld metal included in the fusion zone (see Figure 6).

![Figure 6: Schematic showing calculation of dilution in a fusion weld (3).](image)

\[
\text{Dilution (\%)} = \frac{A + C}{A + B + C} \times 100
\]

2.2.2 Fusion Boundary Transition Region

The fusion boundary transition region exists at the interface between the steel base metal and nickel-base weld metal in a dissimilar metal weld. This region represents the area where the composition of melted material changes rapidly from 100% unmixed base metal to the bulk composition of the fusion zone (3). When the base and filler metal compositions are drastically different (such as in a dissimilar metal weld, for example), the transition region can contain significantly different microstructure and properties when compared to the base and filler metals. In a study performed at the Ohio State University in 2012, the transition zone microstructure in a DMW performed between low alloy steel and nickel-base filler was noted to contain evidence of epitaxial nucleation and
planar growth at the fusion boundary, shifting to cellular dendritic growth as solidification continued in to the weld metal (13). This region has also been referred to as the “featureless zone” due to lack of clear microstructural features. Low angle microscopy was developed by Alexandrov et. al in order to more accurately observe and characterize this narrow region.

2.2.1 Carbon Diffusion during PWHT

When nickel-base cladding deposited on a steel substrate is subjected to a post-weld heat treatment to temper the steel’s coarse grain heat affected zone, the literature has shown that carbon migration can occur, depleting carbon from the steel (3). Carbon has been shown to migrate into the nickel-base filler metal and create build up locally in the planar growth region. A particular case of carbon diffusion during PWHT in a dissimilar metal weld between 8630 steel and Alloy 625 was observed by Alexandrov et al (13). Hardness mapping was conducted along the DMW interface, and regions of hardness in excess of 800 HV were recorded within the featureless zone after PWHT. Carbon migration was studied via ThermoCalc™ and Dictra™ computational software, with EPMA analysis for validation. Dictra™ simulations showed the formation of a carbon pile-up and carbide accumulation at the fusion boundary region, with a carbon depleted area in the CGHAZ. Good correlation was found with respect both to the peak concentration and width of carbon buildup when compared to EPMA results. The driving force behind carbon diffusion into the weld metal during PWHT was attributed to a
larger negative chemical potential and lower diffusivity in Alloy 625 than in 8630 steel at the PWHT temperature.

2.3 Basics of Weld Metallurgy

2.3.1 Regions of a Fusion Weld

Metallographic observation of a fusion weld reveals three general regions: the fusion zone, heat-affected zone (HAZ), and unaffected base metal (see Figure 7). The fusion zone is associated with melted and re-solidified material, while the HAZ is comprised of base metal which has undergone metallurgical changes due to the increase in temperature seen in the vicinity of the fusion zone (3).

Figure 7: Schematic showing basic regions of a fusion weld (14).
Both the fusion zone and HAZ can be further broken down into smaller sub-regions, as seen in Figure 8. The fusion zone is currently understood to contain three sub-regions: 1) the composite region, 2) the transition zone, and 3) the unmixed zone. The majority of the fusion zone is made up of the composite zone, where filler metal has become diluted and formed a “composite” composition due to mixing with the base metal. The transition zone is found in heterogeneous welds and is discussed in more detail in Section 2.2.2. It is defined as the thin region where fusion zone composition changes rapidly from that of the composite zone to that of the undiluted base metal (3). In some welds, an “unmixed” zone forms, where base metal melts and solidifies without mixing into the composite region. This can result in a drastic local change in properties.

The HAZ can be broken down into two distinct sub-regions: 1) the partially melted zone, and 2) the true heat-affected zone. The partially melted zone is a narrow region occurring in the HAZ closest to the fusion boundary, and can be characterized by localized melting along base metal grain boundaries. This region represents the portion of the HAZ that experiences a range of temperatures between the base metal’s liquidus and solidus during welding. The “true” HAZ is defined as the area surrounding the fusion zone where all metallurgical reactions occur in the solid state.
When multi-pass welds or weld overlays are performed, these distinct fusion weld regions overlap one another, making exact characterization of some regions complex. However, the general understanding of fusion weld regions provides sufficient background to analyze such welds, and is crucial to understanding the metallurgical reactions occurring throughout the weldment.

2.3.2 Microstructural Effects of Weld Solidification

2.3.2.1 Types of Grain Boundaries

Three types of grain boundaries can be observed in the fusion zone after weld solidification: 1) solidification subgrain boundaries, 2) solidification grain boundaries, and 3) migrated grain boundaries (see Figure 9) (3). Solidification subgrain boundaries are some of the smallest components of weld metal microstructure that can be observed via optical microscopy, and exist within individual grains in the fusion zone. These
subgrain boundaries are the result of microscopic solute redistribution during solidification, rendering local variances in composition between cells or dendrites. Solidification grain boundaries “[result] from the intersection of packets, or groups, of subgrains” and “are the direct result of competitive growth that occurs along the trailing edge of the weld pool” (3). Since each packet of solidification subgrain boundaries grows in a different direction, adjacent solidification grain boundaries typically show high-angle misorientation. Solidification grain boundaries also exhibit solute and impurity element redistribution, due to the macroscopic effects of weld solidification. Migrated grain boundaries occur when the crystallographic component of a solidification grain boundary shifts away from the compositional component in an attempt to lower grain boundary energy. This is most common in single-phase austenitic weld metals, and typically occurs during reheating of the weld metal. The formation of migrated grain boundaries can be inhibited by the formation of a second phase towards the end of solidification, resulting in pinning of the solidification grain boundary (3).
2.3.2.2 Solute Redistribution

During the rapid, non-equilibrium cooling rates experienced by the fusion zone during welding, local rejection of solute and impurity elements can occur during solidification both along solidification grain boundaries and inter-dendritically.
Redistribution of alloying elements along solidification grain boundaries can be attributed to the macroscopic effects of solidification, and inter-dendritic redistribution of alloying elements can be attributed to the microscopic effects of solidification.

As macroscopic solidification begins at the trailing edge of the weld pool, solute elements are initially rejected from the solid region and pushed in to the liquid ahead of the advancing solidification front. Steady-state solidification ensues, maintaining a consistent solute gradient in the liquid. Mixing of the solute in the liquid is not considered. When solidification grain boundaries meet, the solute-enriched liquid finally solidifies, causing the remaining solute to be “dumped” in to a narrow region at the grain boundary (3). On a microscopic level, complete mixing of the solute in the surrounding liquid can be considered, and solute redistribution along solidification subgrain boundaries can be estimated with the Scheil model. This can result in the formation of a low melting eutectic or simply depress the melting point of the grain or subgrain boundary, depending on the extent of segregation.
2.4 Liquation Cracking

2.4.1 Definition

Liquation cracking is a type of hot cracking characterized by local melting along grain boundaries in the HAZ or reheated weld metal during multi-pass welding (3). In both cases, liquation cracking results from a local reduction in melting temperature relative to the bulk material due to an increase in solute or impurity elements. The increase in solute and impurity elements can be attributed to two separate mechanisms, described in further detail in Section 2.4.3. When this reduction in melting temperature is combined with the resulting temperature gradient from the welding process and strain from thermal shrinkage on cooling, cracks form in the PMZ directly adjacent to the fusion boundary.

Weld metal liquation cracking is specific to multi-pass welds or weld overlays, and is most often observed in single-phase austenitic weld metal such as stainless steels or Ni-base alloys (3). While diffusion mechanisms are required to enrich solute and impurity concentrations in the base metal HAZ, the nature of the rapidly solidifying weld pool creates a microstructure that is inherently more susceptible to liquation cracking. Non-equilibrium solidification during welding results in the rejection of solute and impurity atoms into solidification grain boundaries, increasing solute and impurity element concentrations and locally reducing the melting temperature. When the next weld pass is applied, solidification grain boundaries are susceptible to liquation.
2.4.2 Key Variables Affecting Liquation Cracking

Liquation cracking is primarily influenced by material composition, grain size, and heat input (3). Materials containing niobium and titanium as carbide forming elements are susceptible to constitutional liquation of such particles. The presence of impurity elements such as phosphorus, sulfur, and boron increases the likelihood of cracking via the segregation mechanism. Large grains result in increased susceptibility, as less grain boundary area is available to accommodate strain and prevent wetting when compared to smaller grains. This is particularly detrimental in weld metal liquation cracking, as grains found in the fusion zone are typically larger than those in the base metal. Heat input is the most easily controlled factor. Lower heat inputs result in a steeper temperature gradient in the HAZ, reducing the size of the PMZ. High heat inputs result in a shallow temperature gradient extending further out in to the HAZ, increasing the size of the region susceptible to liquation cracking.

2.4.3 Liquation Mechanisms

Two distinct mechanisms can be used to identify the root cause of liquation cracking in susceptible alloys. Solid solution strengthened Ni-base alloys are typically susceptible to liquation cracking due to the segregation mechanism, as the high alloying content used to increase the materials strength results in a relatively wide solidification temperature range (5). On the other hand, liquation cracking in precipitation hardened Ni-
base alloys is commonly attributed to the penetration mechanism due to liquation of secondary constituents such as NbC and TiC (5).

The “segregation” mechanism occurs due to a build-up of solute or impurity elements in grain boundaries. Solid state grain growth occurs at temperatures above 50% of the melting temperature, resulting in movement of grain boundaries. As these boundaries move, smaller elements such as sulfur, phosphorus, oxygen, titanium, and silicon can be picked up and “swept” along. As growth continues, solute and impurity concentration increases, resulting in melting point depression (3). The level of heat input then determines the distance in to which local melting of the grain boundaries occurs.

Figure 10: Schematic representation of the liquation cracking via the segregation mechanism (3).

Diffusion of alloying elements can also occur along epitaxial grain boundaries in to the HAZ during weld metal solidification, commonly referred to as “pipeline diffusion”. When solute is rejected in to solidification grain boundaries, a diffusion
gradient exists between portions of the boundary in the fusion zone and HAZ. Due to increased diffusion rates along the grain boundary, alloying elements travel through the grain boundary into the HAZ, locally depressing the melting point and resulting in a crack that extends from the HAZ into the fusion zone (3).

The “penetration mechanism” occurs at high temperatures when a mobile grain boundary is intercepted by a liquated particle which then pins and wets or “penetrates” the grain boundary, thus inducing a liquation crack (Figure 11). This mechanism is known to exist in Ni-base alloys containing secondary constituents such as intermetallics, carbides, or residual eutectics (5). The presence and liquation of a secondary constituent is essential if liquation cracking is to occur via the penetration mechanism. Localized melting in the PMZ can also occur via incipient melting at grain boundaries or as the result of compositional banding due to thermomechanical processing, although neither of these is particularly common (3). In most cases, liquation cracking via the penetration mechanism is due to constitutional liquation of a secondary particle, as described below.

Figure 11: Schematic representation of liquation cracking via the penetration mechanism (3).

2.4.4 Constitutional Liquation
The theory of constitutional liquation was first suggested in the 1950s and then proven in the 1960s by Savage and Pepe (3). Constitutional liquation occurs due to a “reaction between a constituent particle and the surrounding matrix such that local melting occurs at the constituent/matrix interface” (3). Materials susceptible to constitutional liquation must exhibit a eutectic reaction between the elemental composition of the particle and the matrix phase. It should be noted that the particle itself does not liquate during the process, as particles involved in constitutional liquation typically have a melting point much higher than that of the surrounding matrix. During the rapid heating rates experienced in the welding process, partial dissolution of the constituent particle occurs. This causes the surrounding area at the interface to become enriched in solute. Once sufficient enrichment has occurred and the eutectic temperature has been reached, liquid forms between the particle and its surrounding matrix. Mobile grain boundaries can then become pinned by the liquated region, leading to wetting of the grain boundary resulting in liquation cracking via the penetration mechanism. A more complete explanation of constitutional liquation can be found in papers published by Savage and Pepe in the Welding Journal during the late 1960’s (16; 17).
2.4.5 Identification of Liquation Cracks

Liquation cracks are typically very small, making detection via non-destructive evaluation difficult. Metallographic sectioning can be used in part to identify liquation cracks by their presence in PMZ grain boundaries in close proximity to the fusion boundary. In some instances, liquation cracking can extend across the fusion boundary into the fusion zone. The formation of liquid films along grain boundaries can be evidenced by widening of grain boundaries during metallographic observation, as seen in Figure 12 (3). Complications can arise during characterization of liquation cracks via optical microscopy, since both weld metal liquation cracks and ductility-dip cracks are common in austenitic weld metal at high temperatures and both can occur along grain boundaries in similar regions of the HAZ. Thus, fractography via scanning electron microscopy is essential to properly distinguish between weld metal liquation cracking and DDC.
Analysis of liqation cracks via SEM can reveal a variety of fracture surfaces, depending on the amount of liquid present at failure (3). When liqation cracking occurs due to thin liquid films, clear intergranular features are observed on the fracture surface. Larger amounts of liquid on the crack surface can mask clean intergranular features (see Figure 13). Constitutional liqation can be evidenced by the presence of carbides on the fracture surface (see Figure 14).
Figure 13: SEM image of liquation cracking in Waspaloy showing masking of clear intergranular features due to liquid film along grain boundaries (18).

Figure 14: SEM image of liquation cracking in Waspaloy due to constitutional liquation of boron carbide (18).
2.4.6 HAZ Liqueation Cracks in Alloy 800

Liqueation cracking in Alloy 800 was observed by multiple researchers at Oak Ridge National Laboratory and The Ohio State University in the late 1970s and early 1980s (19) (20) (21). Alloy 800 is a Fe-Cr-Ni alloy with a composition very similar to that of Alloy 825 (see Table 2). Initial research conducted at Oak Ridge National Laboratory showed that heats of Alloy 800 where the ratio of Al+Ti to C+Si contents were greater than 2 had highest susceptibility to hot cracking in general (19). Additional work on a modified 800H alloy showed that a reduction in base metal grain size reduced the likelihood of HAZ liqueation cracking (21). It was theorized that dissolution of titanium carbides could result in dissolution of the carbides and redistribution of titanium along grain boundaries. Segregation of titanium to grain boundaries was observed, and it was postulated that a low melting titanium-enriched eutectic formed along grain boundaries, resulting in liquation on heating during welding. An investigation using EDS analysis of grain boundaries showed that segregation of sulphur and phosphorus to grain boundaries was a major factor in cracking (20).

Later characterization performed by Lippold showed intergranular cracks due to grain boundary liqueation in the coarse-grain heat affected zone with some cracks extending in to the fusion zone (21). Evidence of constitutional liqueation of titanium carbide particles was observed in a thin region of the PMZ directly adjacent to the fusion boundary, but was not found to be the sole mechanism behind crack formation. Electron probe microanalysis revealed titanium concentrations along CGHAZ grain boundaries.
that were 50 times higher than that of the bulk metal. The increase in titanium concentration was attributed to grain boundary sweeping and pipeline diffusion. Lippold theorized liquation could be due to the formation of a Ti-rich Laves phase along grain boundaries in the PMZ.

Table 2: Comparison of composition ranges between Inconel alloys 800 and 825 (22; 1).

<table>
<thead>
<tr>
<th>Element</th>
<th>Alloy 800</th>
<th>Alloy 825</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>30.0-35.0</td>
<td>38.0-46.0</td>
</tr>
<tr>
<td>Cr</td>
<td>19.0-23.0</td>
<td>19.5-23.5</td>
</tr>
<tr>
<td>Fe</td>
<td>39.5 min</td>
<td>22.0 min</td>
</tr>
<tr>
<td>Mo</td>
<td></td>
<td>2.5-3.5</td>
</tr>
<tr>
<td>C</td>
<td>0.10 max.</td>
<td>0.05 max.</td>
</tr>
<tr>
<td>Mn</td>
<td>1.50 max.</td>
<td>1.0 max.</td>
</tr>
<tr>
<td>Si</td>
<td>1.0 max.</td>
<td>0.5 max.</td>
</tr>
<tr>
<td>S</td>
<td>0.015 max.</td>
<td>0.03 max.</td>
</tr>
<tr>
<td>Al</td>
<td>0.15-0.60</td>
<td>0.2 max.</td>
</tr>
<tr>
<td>Ti</td>
<td>0.15-0.6</td>
<td>0.6-1.2</td>
</tr>
<tr>
<td>Cu</td>
<td>0.75 max.</td>
<td>1.5-3.0</td>
</tr>
</tbody>
</table>

2.5 Ductility Dip Cracking

2.5.1 Definition and History

Ductility-dip cracking is a solid-state phenomenon occurring intergranularly along solidification or migrated grain boundaries at high temperatures, typically in reheated single-phase austenitic weld metal (3). Although a drop in ductility at elevated
temperatures in some austenitic alloys was first noted in 1912, DDC was not recognized as an issue during welding until the 1970s. Early reports of DDC often mistook the cracking phenomena for a type of hot cracking, and referred to it as “microfissuring” (3). Extensive research on DDC with respect to welding was not carried out until the late 1990s when severe cases were observed in the nuclear power industry (3). Since then, the welding community’s understanding of the mechanism behind DDC has increased greatly, although it is still not fully understood.

A sharp drop in ductility of susceptible alloys typically occurs between one-half of the alloy solidus and the solidus temperature (see Figure 15). This temperature range typically corresponds to 800-1150 °C in stainless steels and nickel-base alloys (3). It should be noted that no liquation occurs during ductility-dip cracking, thus differentiating it from hot cracking (denoted by the brittle temperature range or “BTR” in Figure 15). In order for DDC to occur, a sufficient amount of restraint must exist in the weld metal, such that a “critical strain” is reached before cracking begins (\(E_{\text{min}}\) in Figure 15). Thus, if only low restraint exists in a given application of a susceptible alloy, DDC may not be observed. However, if strain is increased due to a change in geometry or welding conditions, DDC will occur. Accordingly, an understanding of both the susceptible temperature range and critical strain required to induce cracking is required to fully eliminate DDC in weldments.
2.5.2 Variables Affecting DDC

In 2002, Nissley and Lippold developed the Strain-to-Fracture test in order to more effectively understand the formation of ductility-dip cracks (23). Their research, along with that of many others, has shown that grain boundary character, material composition, temperature, and strain are some the most important variables affecting DDC (3). Weld metal with grain boundaries that are long and straight (typically the result of grain boundary migration during reheating of weld metal) are most susceptible, as DDC is currently understood as a grain-boundary sliding mechanism. If grain boundaries
can be pinned by the formation of a precipitate or second phase at the end of solidification, sliding can be prevented. In this case, grain boundary “tortuosity” (see Figure 16) provides a mechanical locking mechanism that prevents grain boundary sliding (3). However, not all precipitates are effective at reducing susceptibility to DDC, and some may even increase susceptibility. Ramirez and Lippold (24) reported that a “large number of small M_{23}C_{6} precipitates observed on… straight grain boundaries [were] more prone to void and microcrack formation than the tortuous grain boundaries with medium and small NbC-like precipitates” (24). They concluded that “the actual effect of intergranular particles on grain boundary sliding and void formation depends on the particle type, interfacial relationship with the matrix, particle size and distribution, and the temperature range of formation” (24). Thus, the composition of a given material and its ability to form precipitates or secondary phases plays a large role in both its resistance and susceptibility to DDC. At low temperatures, grain boundary sliding is not possible. DDC can be avoided at higher temperatures due to strain localization and recrystallization along grain boundaries, resulting in more grain boundary area available to accommodate strain (3).
2.5.3 Current Understanding of DDC Mechanism

While many mechanisms have been proposed (see Table 3), the most modern understanding of the mechanism behind DDC was provided by Ramirez and Lippold during the mid-2000s (24). Their work, along previous work carried out at The Ohio State University in the early 2000s (25) (26) (27), reviewed previous theories and revealed that while each theory explained a portion of the mechanism behind DDC, a complete and overarching explanation of the mechanism had not yet been developed. For example, both Yamaguchi and Nishimoto suggested that DDC was due to impurity segregation along grain boundaries leading to grain boundary embrittlement, but DDC was also reported to occur in materials with low levels of sulphur and phosphorus. In the same way, while Young et al. concluded that DDC could be induced by stress
accumulation at grain boundary precipitates in nickel-chromium alloys (28), it did not explain why DDC occurred in alloy systems where grain boundary precipitation did not occur.

Table 3: Summary of ductility-dip cracking theories (3).

<table>
<thead>
<tr>
<th>Author(s)</th>
<th>Theory</th>
<th>Year</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rhines and 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, microvoid formation, boundary tortuosity</td>
<td>2004</td>
</tr>
<tr>
<td>Nishimoto et al.</td>
<td>Impurity segregation</td>
<td>2006</td>
</tr>
<tr>
<td>Noecker II and DuPont</td>
<td>Grain boundary sliding, carbide distribution and morphology</td>
<td>2007</td>
</tr>
<tr>
<td>Young et al.</td>
<td>Precipitation-induced cracking</td>
<td>2008</td>
</tr>
</tbody>
</table>

Ramirez and Lippold surmised that the overarching mechanism behind DDC was comprised of a combination of previous findings. Their work has shown that DDC is a creep-like grain boundary sliding phenomenon occurring along migrated grain boundaries in Ni-base alloys, similar to the mechanism initially proposed by Rhines and Wray in 1961 (24). At the low end of the ductility-dip trough (Figure 15), grain boundary sliding cannot occur. At the high end of the ductility-dip trough, recrystallization begins
to occur, thus eliminating accumulation of deformation along grain boundaries. At intermediate temperatures where the material is most susceptible, grain boundary sliding causes strain concentration at grain boundary triple points and other types of grain boundary irregularities such as intergranular precipitates or grain boundary steps (24). Such strain concentrations along the grain boundaries can lead to intergranular cavity formation around grain boundary irregularities. Under adequate temperature and strain conditions, the intergranular cavities will grow and link up, resulting in microcrack formation and DDC. Figure 17 provides a graphical explanation of how various grain boundary irregularities can affect strain concentrations. Straight grain boundaries result in severe grain boundary sliding and strain concentration at the triple points. The presence of intergranular precipitates locks the grain boundaries, reducing grain boundary sliding and causing strain concentration around the precipitates. Tortuous grain boundaries and intergranular precipitates efficiently lock the grain boundaries, reducing GB sliding, causing further strain distribution, and reducing the likeliness of void formation (24).
2.5.4 Identification of DDC

Although ductility-dip cracks can form in both the HAZ and weld metal, they can most often be characterized by their presence along migrated grain boundaries in reheated weld metal (3). As a result of DDC occurring along migrated grain boundaries, the resulting cracks are typically very straight (see Figure 18) (3). When the MGB has not seen a significant shift in crystallographic location and is still in close proximity to the solidification grain boundary, it can be difficult to distinguish between DDC and
solidification or liquation cracking when only optical microscopy is used. In some cases, careful distinction can be made between the three types of cracking by the following features: 1) cracks resulting from DDC are typically long and straight, 2) solidification cracks are frequently wavy and show signs of liquid films, and 3) weld metal liquation cracks are usually very short and form only in close proximity to the fusion boundary. When precise characterization of crack type cannot be achieved optically, it is necessary to evaluate the fracture surface of the crack using scanning electron microscopy.

Figure 18: DDC along straight, migrated grain boundaries in weld metal (25).

The fracture surface of ductility-dip cracks can vary as a function of material type and temperature, although intergranular features can always be seen on the microscopic scale (3). Work completed by Collins, Ramirez, and Lippold has shown that distinct
fracture features can be seen at low (625-800 °C), medium (850-1000 °C), and high (1050-1200 °C) temperature ranges (26). At both low and high temperatures, intergranular features with a significant amount of sharp ductile dimples were observed. Fractures occurring within the intermediate temperature range revealed “ductile intergranular morphology with a flat appearance macroscopically” (see Figure 19) (26). When magnification was increased, a “wavy” pattern was observed microscopically on the fracture surface (see Figure 20). Such features were characterized as rounded ductile dimples, suggesting that microductility was limited in the temperature region. The size of these wavy features increases as a function of temperature, and it is believed that they are associated with the formation of voids along grain boundaries, which ultimately link together to form ductility-dip cracks.
Figure 19: Macroscopic DDC fracture surface showing flat appearance in 310 stainless steel at 1100 °C (18).

Figure 20: Microscopic DDC fracture surface showing "wavy" features in 310 stainless steel at 1100 °C (18).
2.6 The Strain-to-Fracture Test

The Strain-to-Fracture test was developed to “in order to better quantify susceptibility to DDC” at The Ohio State University by Nathan Nissley and Dr. John Lippold in 2002 (3). The Strain-to-Fracture test “employs a ‘dogbone’ tensile sample with a GTA spot weld applied in the center of the gauge section” (see Figure 21) (3). Solidification conditions of the spot weld are controlled, resulting in a radial array of grain boundaries extending from the center. The Gleeble® thermo-mechanical simulator is used to test each dog bone under varying temperatures and strains. Standard temperatures range from 650 to 1200 °C, with strain ranging from 0-20%. Average strain is determined by using a Rockwell C indenter to produce gauge marks, which are measured before and after testing. The number of cracks induced in the spot weld is then counted under a binocular microscope at 30x magnification. The resulting data can be used to produce plots of temperature versus strain with the number of cracks indicated for each data point, thus revealing the DDC susceptibility envelope (23).
The strain-to-fracture test was initially developed to overcome some of the difficulties present in other DDC testing methods used at the time. The goal while developing the Strain-to-Fracture test at The Ohio State University in the early 2000s was to “develop a test technique that is robust and reproducible, that [also] quantifies DDC susceptibility” (23). The geometry of the strain-to-fracture sample was based on that of the double-spot varestraint test, which previously yielded acceptable results during DDC. It was decided to use the Gleeble thermo-mechanical simulator to heat and strain the sample in order to reduce the number of experimental variables. The effects of the
remaining experimental variables were then evaluated to ensure a robust testing technique.

Experimental variables evaluated during development of the strain-to-fracture test included peak temperature, heating rate, hold time, the effect of stroke rate, the effect of multiple heating cycles, and the relationship between stroke and strain. Neither peak temperature, heating rate, hold time, nor the number of heating cycles were found to have an effect on crack susceptibility. Higher stroke rates were found to increase the amount of strain required to initiate DDC. Ultimately, heating rate, hold time, and the number of heating cycles were chosen as constants during strain-to-fracture testing. Final experimental variables were narrowed down to test temperature, stroke length, and stroke rate. During a comparison of three separate materials, stroke rate was fixed at 0.06 cm/s while test temperature and stroke length were varied.

2.6.2 Strain-to-Fracture Test Results

Once the strain-to-fracture tests are completed and the number of cracks measured, strain-to-fracture plots can be produced to show the DDC susceptibility envelope (see Figure 22). A standard strain-to-fracture plot shows the test temperature on the x-axis, with total strain on the y-axis. A data point is plotted for each strain-to-fracture sample, and the total number of cracks is shown by the type of marker used for each point. A curve can then be drawn between the points at each temperature where the number of cracks changes from zero to one, thus representing the DDC susceptibility
range. The ductility-dip temperature range can be determined by reading the points on the curve that intersect with a horizontal line placed at 15% strain. A “threshold strain” can also be determined from the strain-to-fracture plot by simply observing the lowest amount of strain required to induce cracking at any temperature.

Figure 22: Typical strain-to-fracture plot showing susceptibility curve (25).

2.7 Computational Modelling

2.7.1 Abaqus FEA Modelling of DMW Interface on Heating

Due to differences in the coefficient of thermal expansion between different materials in a dissimilar metal weld, a thermally induced strain field can exist at the
DMW interface upon heating. Multiple researchers have shown that finite element analysis can be used to efficiently evaluate the level of strain induced by the difference in thermal expansion (29) (30) (31) (32) (33). The literature shows that the most relevant material properties to effectively model strain on heating are the Young’s modulus, Poisson’s ratio, thermal conductivity, and coefficient of thermal expansion (29) (30) (31) (32). In 2013, both Zhang and Miller (29), as well as Wang et. al (32) showed that the mismatch in coefficient of thermal expansion can dominate the contributions to total strain when the temperature change is large. Stress is typically largest at the DMW interface, requiring the finest mesh in the region (31) (29). For simplicity, uniform heating is assumed and residual stresses from welding are be ignored so that the weldment is stress-free at 0 °C (31) (29).

2.7.2 ThermoCalc Pseudo-Binary Dual-Alloy Phase Diagrams

ThermoCalc™ thermodynamic simulation software is a powerful resource commonly used to predict phase equilibria in alloy systems. Calculations can be performed under both equilibrium and non-equilibrium (Scheil) solidification conditions. However, the software is currently unable to produce a phase diagram based on dilution between two separate alloys. In 2012, Alexandrov et. al utilized Thermo-Calc™ to analyze a nickel-iron dissimilar metal weld (13). Simulations were conducted with dilution steps of 1% between 100 and 95% dilution, 5% between 95 and 60% dilution, and 10% between 60 and 0% dilution. When the dilution composition contained over
50% iron, the TCS Steels/Alloys Database v5 was used. The TT Ni-Alloys Database was used for all other dilution ranges. It should be noted that neither database produces accurate predictions when the content of the main element (Fe or Ni) nears 50%.

Individual simulations were run at each dilution composition and the results recorded. The Scheil-Gulliver module was used to calculate the solidification temperature range and predict the solidification mode. Values were obtained when the system was 99% solid. Since then, further work has shown that extracting data at 98% solid gives more accurate results (34).

2.7.3 Dictra Modelling of DMW PWHT

Dictra™ computational kinetics simulation software was used by Alexandrov et al (13) at the Ohio State University in 2012 to simulate the effect of diffusion on the distribution of carbon across the fusion boundary in a dissimilar metal weld during PWHT. The Solid Solutions Database v.4 was used to determine phase equilibrium along with the Mobility Database v.2 to determine diffusion coefficients of matrix phases. The homogenization model within Dictra™ was then used to solve the diffusion problem at the given heat treatment time and temperature. EPMA was used to validate simulation results, which were found to accurately model carbon diffusion and build-up in a dissimilar metal weld between AISI 8630 steel and Alloy 625, both in terms of peak concentration and the width of build-up.
Chapter 3: Objectives

Failure of Alloy 825 overlaid pipes due to overlay cracking has been observed after induction bending. The initial goals of this project were to identify the metallurgical phenomena of the cracking mechanism and determine an optimal bending parameter window to successfully manufacture induction bent pipes. It was also found desirable to evaluate the effects of the welding process and post weld heat treatment (PWHT) on crack susceptibility. As such, the objectives of this study are as follows:

1. Identify the nature of cracking in Alloy 825 overlays of induction bent carbon steel pipes.
2. Optimize the bending parameter window (temperature, total strain, and strain rate) in order to avoid cracking of the weld overlay.
3. Understand the effects that the welding process and PWHT have on cracking susceptibility.
4.1 Materials

Three sections of pipe were received from industry for metallurgical characterization of the cracking mechanism. Each section of pipe represented a different stage in the manufacturing process, as outlined in Chapter 1 (see Figure 1). All pipes were made of X65 steel with an outer diameter of 16 inches and wall thickness of 16.66 mm, internally clad with Alloy 825. The exact compositions of the base and filler metals provided in the material certifications for the pipes received from industry can be seen in Table 4. Two layers of cladding were applied by using the GTAW tandem hot wire process in a helical fashion around the internal diameter. The second layer of cladding was not applied until after the first layer was complete. Measurement of the maximum interpass temperature (215 °C) was taken in to account after the first layer was made, and before welding of the second layer had begun.
Table 4: Alloy compositions (wt%) provided with pipes received from industry.

<table>
<thead>
<tr>
<th>Element</th>
<th>X65</th>
<th>Alloy 825</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>97.7672</td>
<td>26.4</td>
</tr>
<tr>
<td>Ni</td>
<td>0.36</td>
<td>43.5</td>
</tr>
<tr>
<td>Cr</td>
<td>0.12</td>
<td>22.5</td>
</tr>
<tr>
<td>Mo</td>
<td>0.12</td>
<td>3.1</td>
</tr>
<tr>
<td>C</td>
<td>0.09</td>
<td>0.01</td>
</tr>
<tr>
<td>Si</td>
<td>0.25</td>
<td>0.3</td>
</tr>
<tr>
<td>Mn</td>
<td>1.08</td>
<td>0.7</td>
</tr>
<tr>
<td>P</td>
<td>0.012</td>
<td>0.014</td>
</tr>
<tr>
<td>S</td>
<td>0.002</td>
<td>0.002</td>
</tr>
<tr>
<td>V</td>
<td>0.04</td>
<td></td>
</tr>
<tr>
<td>Nb</td>
<td>0.0024</td>
<td></td>
</tr>
<tr>
<td>Ti</td>
<td>0.003</td>
<td>0.8</td>
</tr>
<tr>
<td>Al</td>
<td>0.0034</td>
<td>0.2</td>
</tr>
<tr>
<td>Cu</td>
<td>0.15</td>
<td>2.2</td>
</tr>
</tbody>
</table>

After cladding, all pipes were normalized at 1000 °C for 15 minutes, using a ramp-up rate of 150 °C per hour followed by air cooling to room temperature. A 5” section was received in the normalized condition. A small portion of this normalized section was used for metallurgical characterization while the majority of the material was machined into samples for Gleeble testing. The remaining two sections of pipe were taken from pipes subjected to induction bending at temperatures between 1000 and 1050 °C after normalization. One section was supplied from a 48” radius bend, and the second section was taken from an 80” bend. Each section supplied was 3 inches wide and contained cracks through the overlay thickness, with dye penetrant indications marking crack locations. Bent samples were used solely for metallurgical characterization of the cracking mechanism.
4.2 Metallurgical Characterization

4.2.1 Sample Preparation

Cross sections transverse to the welding direction were removed from internally clad pipes received from industry. Serial sections of cracked pipes were obtained by taking six transverse sections spaced 0.125 inches apart. A Leco PR-36 mounting press was used to encapsulate each sample in a 1.25 inch Bakelite mold. Each sample was polished using 240, 620, 400, 600, 800, and 1200 grit sandpaper. Final polishing was accomplished by using 3µm and 1 µm diamond paste. For low angle section microscopy, 1” thick transverse sections were removed and placed longitudinally on the mounting plate (see Figure 23). A piece of 0.045” filler wire was used to create the desired angle. Samples were then ground with 180 grit sandpaper until the desired interface was revealed. Polishing then continued per the steps outlined above.
Etching of the dissimilar metal welds was achieved via a two-step process involving immersion in 5% Nital for 45 to 60 seconds to reveal the steel microstructure, followed by the use of a 10% chromic acid electrolytic etch at 6 volts and 1 amp for 3 to 10 seconds. Preferential corrosion of the second layer of cladding prevented the first layer of cladding from etching in the same manner. When characterization of the first overlay was desired, the two-step process detailed above was first performed, after which the etched regions of the second layer of overlay were passivated by the application of clear nail polish. The 10% chromic acid electrolytic etch was then performed a second time for 10 to 15 seconds. After etching was completed, samples were placed in a heated ultrasonic bath of ethanol to remove nail polish while avoiding pitting.
4.2.2 Optical Microscopy

Light optical microscopy was conducted on an Olympus GX51 microscope with magnification ranging from 12.5x to 1000x. Polarized light and a differential interference contrast (DIC) lens were used at times to increase contrast between dendrites and grain boundaries in the first layer of cladding and to observe carbides.

4.2.3 Scanning Electron Microscopy

Scanning electron microscopy, including energy dispersive spectroscopy (EDS) was performed using a Quanta 200 scanning electron microscope. Since no overlay cracks were open to the surface, cracks were opened for analysis by notching a transverse section of pipe adjacent to a crack and cooling it in liquid nitrogen before inducing strain sufficient to fully fracture the sample. SEM characterization was done with a spot size of 4, 20 kV accelerating voltage, and 10 mm standoff distance. Dwell time during EDS scans varied according to scan length.

4.2.4 Hardness Measurements

Hardness traverses were initially conducted across the dissimilar metal weld interface, spanning 1 mm in to the base metal and through both weld metal overlays (4 mm). Hardness mapping was also performed on selected samples. In both cases, a LECO
LM100AT microhardness tester was used with a load of 100 grams and indent spacing of 100 micrometers. All hardness measurements were recorded on the Vickers scale (HVN).

4.3 Gleeble Testing

4.3.1 Procedure Development

The Gleeble test developed for this project was based on the strain-to-fracture test designed at OSU, as outlined in Chapter 2. The strain-to-fracture test was modified to economically and effectively simulate the induction bending process. Changes to the strain-to-fracture test include sample geometry, the method of quantifying cracking response, along with different heating, cooling, and stroke rates.

Sections of internally clad steel pipe received from the industry were removed from the internal diameter and machined into dog bones (see Figure 24). The gauged section of the sample was cut transverse to the welding direction, as the pipe was clad in a helical fashion. This allowed for application of a tensile load during testing in the same direction that the weld overlay experiences during induction bending. The final geometry resulted in a dog bone that was 60 percent weld metal and 40 percent base metal through the thickness of the sample (see Figure 25). Temperature measurements at varying locations were taken on both sides of the dissimilar metal combination during resistance heating of the sample in the Gleeble to ensure that the two metals heated equally.
Figure 24: Schematic of strain-to-fracture sample removal from received pipes.

Figure 25: Dimensions of modified strain-to-fracture sample.
A heating rate of 7.9 °C/s and free air cooling were chosen based on values known in industry for induction bending of similarly sized pipes. The primary strain rate was determined based on the actual bending speed (Equation 1). It should be noted that the standard heated area of a pipe with a wall thickness less than 1” is 1.5” wide, according to industry sources. Two additional stroke rates were chosen to evaluate the effects of strain rate on crack susceptibility by both doubling and halving the primary stroke rate. Total strain targets were established by calculating the maximum bending strain experienced between two different radii bends (Equation 2). The target strain calculated for a 48” radius bend was 15.3% at the DMW interface, and 15.0% at the pipe ID. Similarly, the target strain for an 80” radius bend was 9.2% at the DMW interface and 9.0% at the pipe ID. Target strains of 15% and 9% for the 48” and 80” radius bends were chosen, respectively. The time at temperature varied depending on the total strain and strain rate chosen for each sample. A final range of parameters can be seen in Table 5.

Equation 1: Strain rate as a function of known induction bending parameters.

\[
\text{Strain Rate} = \frac{\text{Bending Strain}}{\text{Heated Area} \times \frac{1}{\text{Travel Speed}}}
\]
Equation 2: Bending strain as function of bend radius and distance from neutral axis.

\[ Bending \, Strain = \frac{-1 \times (Distance \, from \, Neutral \, Axis)}{Bend \, Radius} \]

Table 5: Range of key strain-to-fracture parameters.

<table>
<thead>
<tr>
<th>Strain Rates (mm/s)</th>
<th>0.0125</th>
<th>0.025</th>
<th>0.05</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Strain (%)</td>
<td>3-23</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bending Temperatures (°C)</td>
<td>900</td>
<td>950</td>
<td>1000</td>
</tr>
<tr>
<td></td>
<td>1050</td>
<td>1000</td>
<td>1050</td>
</tr>
<tr>
<td>Heating Rate (°C/s)</td>
<td>7.9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cooling Rate</td>
<td>air cool</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

4.3.2 Sample Preparation

Prior to testing, samples were cleaned with acetone to remove any films resulting from machining. Five indents spaced 0.100 inches apart were made in the weld metal on each side of the gauged section using a Rockwell C indenter with 150 kg load (Figure 26). The exact distance between each indentation was then measured using an Olympus GX51.
optical microscope at 12.5x magnification. For all samples, a type K thermocouple was placed at the center of the gauged section on the weld metal side. This thermocouple was used as the control temperature in all tests. When additional temperature distribution data was desired, four type K thermocouples were placed on the weld metal spaced 0.125 inches apart as shown in Figure 27.

Figure 26: Schematic of gauge mark locations on modified strain-to-fracture sample.
4.3.3 Testing Procedure

The Gleeble thermo-mechanical simulator with high force jaws was used to perform each individual strain-to-fracture test. A tension preload of 100 kilograms was applied using the air ram to ensure the sample was seated in the jaws. After tightening the sample, the preload was removed. The chamber was then evacuated with a roughing pump to $4 \times 10^{-1}$ Torr and backfilled with argon to 10 in Hg. The evacuation and backfilling process was repeated twice to minimize oxidation of the sample.

A heating rate of 7.9 °C/s was used to bring samples up to the desired bending temperature, during which time the jaws were allowed to move freely to accommodate for thermal expansion. Upon reaching the desired temperature, the stroke was zeroed
before a constant stroke rate was applied to reach the intended total strain. A plot showing temperature and stroke recorded during the modified strain-to-fracture test can be seen in Figure 28. Once the total strain was achieved, the sample was allowed to freely cool until the control thermocouple reached 150 °C. The chamber was then vented and the sample removed.

Figure 28: Plot of temperature and stroke during modified strain-to-fracture test at 950 °C.
4.3.4 Quantification of Cracking Response

In order to quantify the cracking response in each sample, three cross sections from the gauged section were taken transverse to the welding direction. Sectioning was achieved by removing the center 20 mm of the gauged section and making 3 cuts spaced at 0.125”, transverse to the weld bead (see Figure 29). The three faces revealed by the transverse sections were mounted in Bakelite, then polished and etched as discussed previously. The central portion of each cross section was observed via light optical microscopy at 50 to 100x magnification. The total crack length within a 0.100 inch region to either side of the center line in each cross section was measured and counted. The number of interfacial micro-cracks in the observed region was also recorded.

Figure 29: Sectioning of modified strain-to-fracture samples for metallographic observation. Sections mounted for crack counting are shown as “A”, “B”, and “C”. Central 0.200” portion was monitored for cracks.
4.3.5 Strain-to-Fracture Plots

Two separate strain-to-fracture plots were created for each strain rate: one showing the total crack length, the other showing the number of interfacial micro-cracks. Each plot was constructed using temperature on the x-axis and total strain on the y-axis. For plots showing the total crack length, the total length was enumerated next to each data point. On plots showing the number of interfacial micro-cracks, the total number of cracks was denoted next to each data point.

4.4 Replication of GTAW Process and PWHT

4.4.1 GTAW Overlays

Small scale samples of the weld metal overlays were created at The Ohio State University using a Miller Dynasty 280 power supply hooked up to a Jetline linear welding system. Welding parameters were determined based on those used in industry. It should be noted that while a tandem hot wire process was used in production, a single cold wire was used at OSU. Communication with industry suggests that the single cold wire process would not change the heat input drastically, and that the only major change between processes is the deposition rate. The interpass temperature was followed only between the first and second layers of the CRA alloy, as the helical application of the
overlay allowed for approximately two minutes between passes based on travel speed and
the internal diameter.

4.4.2 Post Weld Heat Treatment

The post weld heat treatment performed in industry was recreated by using a
Lindberg horizontal furnace. The chamber was filled with a continuous flow of argon
during the heat treatment. Samples were heated to 1000 °C at a rate of 2 °C per minute
and then held at 1000 °C for 15 minutes. Upon completion of the PWHT, samples were
removed from the furnace and allowed to air cool.

4.4.3 Metallographic Preparation

Samples were sectioned, mounted, and polished as described in Section 4.2.1.
After the hardness measurements were taken, samples were etched using the two-step
Nital and 10% chromic electrolytic process. Metallurgical characterization was done
using the Olympus GX51 as explained in Section 4.2.2.

4.4.4 Hardness Measurements

Hardness traverses were performed across the dissimilar metal weld interface,
spanning 3 mm in to the base metal and through both weld metal overlays (3 mm). Four
traverses were conducted on each sample. Two traverses passed through center of the weld beads, while the two other traverses passed through the region of bead overlap (see Figure 30). A LECO LM100AT microhardness tester was used with a load of 100 grams and indent spacing of 100 μm. Microhardness maps were performed using the same parameters. All hardness measurements were recorded on the Vickers scale (HVN).

Figure 30: Hardness traverse locations; A & C represent the region of bead overlap, B & D represent the bead centers.
4.5 Numerical Modeling/Simulation

4.5.1 Abaqus FEA

A 2-D axisymmetric model of the pipe cross section was developed. Model portioning and meshing were created based on a micrograph taken at 12.5x magnification (see Figure 31). Separate regions were created for the base metal, first weld metal overlay, and second weld metal overlay. Material properties including coefficient of thermal expansion, modulus of elasticity, Poisson’s ratio, and yield strength as a function of temperature were gathered according to the literature (2) (1) (35). Where experimentally determined properties were not available, JMatPro software was used to predict material properties based on composition. Uniform heating was applied from 20 °C to 1000 °C and strain accumulation in the weld overlays at the DMW interface was observed. The simulation was first run using Alloy 825 weld metal, and then repeated using Alloy 625. A comparison of maximum strain at the dissimilar metal weld interface was made between overlays of each alloy.
4.5.2 ThermoCalc Simulations

Alloy compositions were obtained from material certifications provided for the pipes received from industry. Dilution compositions were calculated in five percent increments from zero to one hundred, where one hundred percent dilution was considered pure base metal. Impurity elements were not included in any of the calculations.
4.5.2.1 X65/825 Equilibrium Pseudo-Binary Phase Diagram

Using the equilibrium module in ThermoCalc, a step diagram of the molar fraction of each phase was produced for each dilution increment. Temperature was plotted on the x-axis, ranging from 700-1600 °C. Each set of phases and their respective temperature ranges were recorded in a separate table. Data from the table was used to plot phases as a function of temperature (on the y-axis) and dilution (on the x-axis), resulting in a pseudo-binary phase diagram between the two materials. Regions of the phase diagram corresponding to the planar growth region and each overlay dilution range were marked, according to measurements from EDS traverses.

4.5.2.2 X65/825 Non-Equilibrium Pseudo-Binary Phase Diagram

The ThermoCalc Scheil module was used to create a phase diagram based on the non-equilibrium cooling conditions seen during welding. The TCFE8 database was used when the dilution was comprised of primarily iron and the TCNI8 was used when the dilution contained more nickel than iron. No phases were eliminated from the calculation, and carbon was included as a fast-diffusing element. The resulting plot of phases as a function of temperature and mole fraction solid was used to capture phase data for each dilution increment, up to 99% solid. The data recorded from each dilution increment was combined to create a pseudo-binary phase diagram between the two materials, with temperature on the y-axis and dilution on the x-axis. Regions of the phase diagram
corresponding to the planar growth region and each overlay dilution range were marked, according to measurements from EDS traverses.

4.5.2.3 Calculation of X65/825 Partitioning Coefficients Based on Dilution

Partitioning coefficients of each alloying element included in the Scheil module calculations were determined for each dilution increment. Two additional plots were created for each dilution: one with weight percent of each alloying element in the matrix phase on the y-axis and the mole fraction solid on the x-axis, and the other with weight percent of each alloying element in the liquid phase plotted against the mole fraction solid. The weight percent of each element at 99% was recorded, and the partitioning coefficient was calculated as the ratio of weight percent in solid to weight percent in liquid. A final plot was constructed showing the partitioning coefficient of each element as a function of dilution.

4.5.2.4 Pseudo-Binary 825 Phase Diagram with Varying Titanium Concentration

Pseudo-binary phase diagrams were generated for Alloy 825 as a function of weight percent titanium using the ThermoCalc equilibrium module. These phase diagrams were created for three separate compositions: pure Alloy 825, 21% dilution (representing the first cladding layer), and 7% dilution (representing the second cladding layer). The map function of ThermoCalc was utilized, generating data for a 2-D plot with
temperatures ranging from 227 to 1727 °C on the y-axis, and weight percent titanium ranging from 0 to 30% on the x-axis. Data from the pseudo-binary phase diagrams was used to create a plot showing the respective solidus temperature for each composition.

4.5.3 Dictra Simulations

Dictra was used to simulate the normalizing PWHT performed on the cladding prior to induction bending. Thermodynamic data was gathered for a system consisting of chromium, iron, nickel, and carbon. Only the FCC_A1 and BCC_A2 phases were considered, along with M\textsubscript{23}C\textsubscript{6} carbides. The ramp-up rate and hold time chosen for the simulation were identical to those seen in production. Two regions were defined in the model: a ferrite matrix 1x10\textsuperscript{-3} meters wide on the left hand side, and an austenite matrix 250x10\textsuperscript{-6} meters wide on the right side. The composition of the X65 base metal was entered into the ferrite matrix, and a 21% dilution of Alloy 825 representing the first overlay selected for the austenite matrix. Once the simulation was complete, a plot was constructed of weight percent carbon in 5000 second increments as a function of distance.
5.1 Metallurgical Analysis of Cracking Mechanisms

5.1.1 Characterization of Normalized Pipes

Optical Microscopy

Macroscopic observation of X65 steel pipes internally clad with Alloy 825 in the normalized condition shows three distinct regions of material: 1) the steel base metal and heat affected zone (HAZ), 2) the first layer of Alloy 825 cladding, and 3) the second layer of Alloy 825 cladding (Figure 32). The exact location of fusion boundaries from the GTAW overlay process can be seen in red. Note that the bead location is staggered by approximately 50% between overlays, and that the welding process results in a macroscopically wavy interface between the first overlay and base metal. Some difficulty was encountered in obtaining uniform etching throughout the second overlay due to the change in composition between the two layers, thus a two-part etching process was used to fully etch both layers of the corrosion-resistant weld metal overlay (Section 4.2.1). It may be noted that the second overlay etches in a darker fashion than the first overlay, and that some narrow bands of over-etched and un-etched material exist between the two
overlays. These bands are due to the exact placement of the inert nail polish applied to the second overlay, which neutralized the upper layer and allowed for complete etching of the first overlay.

Figure 32: Macrograph of Alloy 825 cladding on X65 steel in the normalized condition.

Microscopic observation of the dissimilar metal weld interface between the X65 steel and first overlay of Alloy 825 shows four distinct regions: 1) the partially mixed zone, 2) the planar growth region, 3) the cellular growth region, and 4) the dendritic growth region (Figure 33). It is believed that the partially mixed zone is comprised
primarily of martensite, as typically seen in the literature regarding dissimilar metal welds between carbon steels and nickel-base materials (5). It is also possible that a thin layer of M$_{23}$C$_6$ carbides is present, starting at the partially mixed zone and moving in to the planar growth region, according to kinetics and thermodynamics simulations conducted as part of this study (Figure 43 and Figure 61). Additional characterization is required to fully understand the nature of the partially mixed zone in this dissimilar metal weld interface.

Figure 33: Micrograph of DMW interface between Alloy 825 cladding and X65 steel in the normalized condition.
Weld Overlay Composition and Compositional Gradients

An EDS traverse performed across the DMW interface reveals the width of the transition zone and average dilution in the first overlay. The transition zone is denoted by a sharp decrease in the amount of iron content present in the material, along with a sharp increase in nickel and chromium content. The width of the transition zone is approximately 30 micrometers, and corresponds to the planar and cellular growth regions at the dissimilar metal weld interface. The weld metal composition was found to be relatively consistent throughout the bulk of the first overlay once dendritic growth began (Figure 34). Occasional spikes in titanium of 8 to 10 weight percent were found throughout the overlay. This represents a nearly ten-fold increase in titanium in these areas, as the bulk weld metal in the first overlay nominally contains 0.7 weight percent titanium. The literature has shown that titanium carbide particles are known to exist within the grains Alloy 825 as a result of casting (9). Thus, such spikes in titanium concentration are attributed to the presence of titanium carbides within the first overlay. Base metal dilution in the first overlay was calculated to be 21% on average.
Figure 34: Plot of EDS traverse results across the DMW interface.

Figure 35: SEM micrograph showing the location of EDS traverse across the DMW interface.
EDS results from a scan across the weld metal interface between the two weld metal layers show that the average dilution of base metal changes from 21% in the first layer to approximately 7% dilution in the second layer (Figure 36). A narrow transition region 15 microns wide can be seen between the two layers, denoted by a decrease in iron and increase in nickel and chromium content. A large spike in titanium can be seen about 30 microns from the second overlay fusion boundary in resulting HAZ of the first layer, likely due to intersection with a titanium carbide particle. The nominal content of titanium does not appear to change between the layers. However, it should be noted that EDS is not particularly sensitive to elements with low atomic numbers such as titanium. As such, EPMA is recommended to properly characterize the levels of titanium in the material.
Figure 36: Plot of EDS traverse results across the weld metal interface.

Figure 37: SEM micrograph showing location of EDS traverse across weld metal interface.
An EDS traverse was also conducted through the bulk weld metal of the second layer, across solidification grain boundaries (Figure 38 and Figure 39). Note that no major change in composition occurs at the solidification grain boundaries. Small spikes of 5 to 9 weight percent titanium were also observed in the second overlay, similar to those seen in the bulk metal of the first overlay. These points have been attributed to the intersection of the EDS traverse with titanium carbide particles, in the same fashion as those observed previously (Figure 34 and Figure 36).
Figure 38: Plot of EDS traverse results across solidification grain boundaries in the second overlay.

Figure 39: SEM micrograph showing EDS traverse location across solidification grain boundaries in the second overlay.
Liquation Cracking

Work performed at Oak Ridge National Laboratory has shown that alloys of a similar composition to Alloy 825 are susceptible to hot cracking, and that the ratio of Al + Ti to C + Si contents played a role in determining susceptibility to HAZ liquation cracking (19). The Al + Ti to C + Si ratio for the material in the received pipes was calculated to be 3.23, suggesting that Alloy 825 would have a high potential for hot cracking. Weld metal HAZ liquation cracks were found primarily in second layer of the weld overlay in the normalized condition (see Figure 40). These cracks were typically very short and narrow, making them difficult to find when performing microscopy on weld cross sections. Note that the crack forms perpendicularly to the fusion boundary, extending slightly into the fusion zone.
Figure 40: Weld metal HAZ liquation crack in second overlay; normalized condition.

Figure 41 delineates the fusion boundary between two weld beads in the second overlay. The lower portion was deposited first, followed by the upper portion. This crack can be characterized as a weld metal HAZ liquation due to: 1) its short length, 2) its location in the heat-affected zone in proximity to the fusion boundary, 3) the fact that it extends in to the second pass, and 4) the rough nature of the crack edges, indicating that the crack occurred while liquid was still present. The depth to which the crack appears to penetrate in to the second pass results from pulsing of the arc during the welding process. Evidence of the pulsed arc can be observed in the form of a fusion boundary concentric to the one highlighted in the macrograph at the tip of the liquation crack (Figure 40).
In order to verify that the cracks observed via optical microscopy were truly liquation cracks, additional characterization was performed using a scanning electron microscope. Due to the small nature of the cracks, they could not be opened for direct observation of the fracture surface. Figure 42 shows a portion of the fracture surface visible after sectioning and polishing. Note the inter-dendritic morphology of the fracture surface, suggesting that liquid was present when the crack formed. This evidence of liquid formation, along with the short length and location in the HAZ close to the fusion boundary, suggest that such cracks are the result of weld metal liquation cracking during the initial GTAW cladding process.
Figure 42: SEM image of weld metal HAZ liquation crack (3000x).
The equilibrium pseudo-binary phase diagram produced between X65 steel and Alloy 825 shows no low melting eutectic constituents that could result in solidification or liquation cracking during the welding process (Figure 43). Ferritic solidification occurs only at the highest dilution levels, while the majority of the cladding solidifies in the fully austenitic mode between 95 to 0% dilution. It should be noted that the dilution range between 25 to 45% contains compositions where the iron and nickel contents near 50 weight percent, rendering the results seen in this region less accurate than the rest of the diagram. The transition region at the DMW interface is expected to form a layer of $\text{M}_{23}\text{C}_6$ carbides at a dilution range between 70 to 85%. The formation of such carbides was observed via optical microscopy. The presence of these carbides observed in the normalized metallographic sections is believed to be enhanced due to carbon diffusion during the normalizing post-weld heat treatment, as shown in Figure 61.

ThermoCalc predictions suggest that sigma phase may form in both the first and second overlays, but etching with NaOH did not reveal the presence of any remaining sigma phase particles after normalization or bending. The maximum temperature range of sigma phase formation in both overlays is 846 °C, well under the minimum temperature experienced in the induction bending temperature range during manufacturing (1000 to 1050 °C). Thus, sigma phase embrittlement is not expected to play a factor in the cracking mechanism.
Figure 43: Equilibrium Pseudo-Binary Phase Diagram between X65 steel and Alloy 825.
The Scheil module pseudo-binary phase diagram between X65 steel and Alloy 825 predicts similar solidification modes to those seen in the equilibrium pseudo-binary phase diagram (Figure 44). This lower temperature range of this particular diagram was produced using 99% solid as the ending point, instead of the 98% previously suggested by the literature (34). A phase diagram was also produced using 98% solid as the ending point for calculations, but lowering the amount of solid present at the end of the calculation eliminated the presence of any phases other than austenite at all points during solidification. Scheil model results do not show the formation of any low melting eutectic constituents, similar to equilibrium predictions. No direct comparison of the solidification temperature range between the two overlays can be made, as the thermodynamic database used for calculations changed between 10 and 15% dilution.

No carbides are expected to form in the transition region during welding according this model. The formation of carbides observed in the normalized cross sections can be attributed in part to their equilibrium thermodynamic stability. This model suggests that $M_{23}C_6$ carbides would form in the first overlay during initial solidification, and that no such carbides would form in the second overlay. Metallographic analysis of cross sections removed from pipes received from the manufacturer present a stark contrast to these predictions (see Figure 48 and Figure 49). More carbides were found in the second layer than the first layer, although the prediction of carbides in the first layer was correct.
Figure 4. Schel Module Pseudo-Binary Phase Diagram between X65 and Alloy 825.
Partitioning Coefficients

Partitioning coefficients for each alloying element in Alloy 825 as a function of dilution can be found in Figure 45. It can be clearly seen that titanium and carbon segregate most heavily throughout the entire dilution range. A large drop in the partitioning coefficient of carbon occurs at 10% dilution, close to the 7% dilution observed in the second overlay. This drop results in heavier segregation of carbon to solidification grain boundaries as the weld metal of the second overlay solidifies, forming more titanium carbides in the grain boundary. Carbon and titanium are also expected to segregate in the first overlay at approximately 21% dilution, although not as strongly as in the second overlay. The increased presence of titanium in the grain boundaries of the second overlay could lead to grain boundary liquation due to segregation during solidification, or cause constitutional liquation of titanium carbides, ultimately leading to the weld metal HAZ liquation cracking observed in the normalized condition. Based on the partitioning coefficients shown here, it is hypothesized that the carbides predicted by ThermoCalc and observed via optical microscopy at the DMW interface could be titanium carbides as well.
In order to verify partitioning of titanium to grain boundaries during solidification of the weld pool in the second overlay, EDS traverses were conducted across solidification grain boundaries in the bulk weld metal of the second overlay (Figure 38) and in the weld metal directly adjacent to fusion boundaries in the second overlay where liquation cracks were found to form (Figure 46). The titanium concentration was nominal through the traverses, and no increase in weight percent titanium was found at the grain boundaries, despite the prediction of heavy partitioning by ThermoCalc. It should be noted that EDS is not highly sensitive to lightweight elements such as titanium. In work conducted by Lippold on liquation cracking in Alloy 800, Electron Probe Micro Analysis was found to be effective in identifying titanium that had segregated to grain boundaries.
Thus, EPMA of solidification grain boundaries is recommended to further validate the model.

Figure 46: EDS traverse across solidification grain boundaries outside of a fusion boundary in the second overlay.

Solidus Temperature Depression due to Titanium Enrichment

In order to more effectively understand the effects that an increased content of titanium along solidification grain boundaries has on the solidus temperature, a series of
pseudo-binary phase diagrams were developed for different dilution levels seen in Alloy 825 weld overlays. The plot depicted in Figure 47 was produced from the solidus data generated via ThermoCalc in the pseudo-binary phase diagrams. The nominal solidus temperature for each level of dilution is approximately 1320 °C, based on a nominal level of 0.7 weight percent titanium present in the bulk weld metal. As the level of titanium increases up to 5 weight percent, the first and second overlays undergo a drop in solidus temperature down to roughly 1135 and 1115 °C, respectively. At 10 weight percent titanium, the solidus temperatures for each overlay drops to 1110 and 1095 °C, respectively. The solidus temperature in both overlays drops most substantially at a titanium level of 20% in the second overlay. Although EDS results showed spikes in titanium of up to 10 weight percent, it is possible that the matrix surrounding the titanium carbide particles could see a substantial local increase in titanium concentration from diffusion of the particle during the rapid heating experienced during welding, leading up to constitutional liquation. The lower level of dilution in the second overlay results in an additional 50 °C decrease in solidus temperature when compared to the first overlay.
Figure 47: Plot of solidus temperature in varying dilution compositions of Alloy 825 with respect to titanium content.

Carbide Distribution in Weld Metal

Micrographs taken with polarized light and a DIC filter emphasize the presence of carbides in the weld metal at the DMW interface (Figure 48). Carbides were found to form in a thin, continuous layer ranging from approximately 50 to 100 microns in the weld metal of the first overlay directly adjacent to the fusion boundary. These carbides are believed to be M$_{23}$C$_6$, chromium- or titanium-rich particles based on a survey of the literature (9; 10) and the ThermoCalc results shown previously (Figure 43, Figure 44, and Figure 45). Additional carbides can be seen extending along grain boundaries in to the...
weld metal. Note that the presence of carbides in the bulk metal of the first overlay is sporadic.

Figure 48: Micrograph of DMW interface with carbides emphasized via polarized light and DIC filter.

A clear distinction can be made between magnitude and organization of the carbide distribution in the first and second overlays (see Figure 49). As previously noted, the bulk metal of the first overlay contains evenly distributed carbides throughout. The presence of carbides along solidification grain boundaries in the second overlay can be clearly noted in the upper half of Figure 49, in distinct contrast to the evenly distributed
carbides in the first overlay. Concentration of carbide formation along solidification grain boundaries in the second layer can be attributed to strong partitioning of carbon at the dilution range encountered in this region compared to that of the first overlay (see Figure 45).

Figure 49: Micrograph of weld metal interface with carbides emphasized via polarized light and DIC filter.
Hardness measurements recorded across the DMW interface in a low angle metallographic section reveal the significant increase in hardness occurring in the planar growth zone directly adjacent to the fusion boundary after normalization (Figure 50). Pattern 1 was taken in the center of a weld bead, and Pattern 3 was taken in the region of overlap. Pattern 2 shows an intermediate point between the two other traverses. Each traverse was performed with the first twelve indents in the base metal. The peak hardness shown was 367 Vickers. The enlarged high hardness region in Pattern 3 can be attributed to a local variation in the width of the planar region induced during the welding process. The region of increased hardness was found to correlate with the thin layer of carbides and planar/cellular growth regions observed at the DMW interface (Figure 51).
Figure 50: Plot of low angle section hardness traverse across DMW interface in the normalized condition. Pattern 1: weld bead center, Pattern 3: weld bead overlap, Pattern 2: intermediate point between 1 and 3.

Figure 51: Micrograph of hardness indents on low angle section of normalized pipe. Hardness shown in Vickers.
In order to increase resolution while performing hardness mapping, low angle sections of the DMW interface were used. A region of high hardness (approximately 350 Vickers) can be seen in the planar growth region of the weld metal (see Figure 52). The maximum hardness recorded at the interface was 406 Vickers. The average weld and base metal hardness in the normalized condition is approximately 200 Vickers. Increased hardness near the regions of weld bead overlap is due to the angle at which the sample was sectioned. Idents performed in this region penetrated into the high hardness layer at the interface directly below the weld metal in the area.

Observation of a micrograph taken at the dissimilar metal weld interface where hardness mapping was performed shows that peak hardness measurements in the planar/cellular growth regions is highly dependent on the exact location at which the indents were performed (Figure 53). Hardness appears to reach its maximum values in the indentations made closest to the dissimilar metal weld interface. Hardness remains elevated throughout the planar and cellular growth regions, and then returns to around 200 Vickers in the bulk weld metal. It should be noted that as a result, peak hardness measured from simple hardness traverses conducted across the interface may not be fully representative of the peak hardness existing in the weldment. The true peak hardness at the dissimilar metal weld interface is best represented by the maximums found via performing hardness mapping on low-angle sections.
Figure 52: Low angle section hardness map of DMW interface in the normalized condition.

Figure 53: Micrograph of low angle section hardness map.
Effects of the Normalizing Post-Weld Heat Treatment

Hardness traverses along with accompanying micrographs taken in varying stages of the manufacturing process prior to induction bending can be seen in Figure 54 Figure 59. Measurements taken after the first clad layer is applied show the highest hardness occurring in the base metal, just outside of the dissimilar metal weld interface (Figure 54). Note that the traverses labelled “Overlap” 1 and 2 show some effects of bead tempering in the base metal closest to the fusion boundary due to additional heat input from the next weld pass. At this point in processing, the average base metal hardness is approximately 220 Vickers, and the average weld metal hardness is around 190 Vickers. The micrograph shown in Figure 55 clearly shows that the region of highest hardness at this step of processing is located in the base metal. While there is no clear evidence of a coarse-grained heat affected zone in the base metal, it is probable that base metal in this region was heated in to the austenitic field and rapidly cooled during welding, resulting the formation of some martensite in the microstructure.
Figure 54: Plot of hardness traverses across DMW interface after one layer of 825 cladding was applied.

Figure 55: Micrograph of hardness indents after one layer of GTAW cladding.
Once the second clad layer is applied, the full effects of bead tempering can be seen in the X65 steel base metal (Figure 56). The hardness in the majority of the CGHAZ is lowered to approximately 220 Vickers, close to the initial hardness measured when the first overlay was applied. It should be noted, however, that the additional heat input from welding of the second overlay is not sufficient to fully temper the base metal and create a fully uniform hardness near the dissimilar metal weld interface. The micrograph in Figure 57 shows that the peak hardness at this stage of manufacturing is still in the base metal, as evidenced by the size of the hardness indents.
Figure 56: Plot of hardness traverses across DMW interface after two layers of 825 cladding were applied.

Figure 57: Micrograph of hardness indents after two layers of GTAW cladding.
The most important change in hardness occurs after the welded pipe undergoes a normalizing PWHT at 1000 °C for 15 minutes. The PWHT was originally selected by the manufacturer to equalize the hardness measurements across the DMW interface in preparation for bending. This is a common manufacturing process when preparing pipes clad with Alloy 625 in order to reduce cracking during induction bending. However, our study has shown a distinct spike in hardness in the zone at the DMW interface after normalization. This drastic increase in hardness was seen to occur only after the normalizing PWHT was applied at OSU (Figure 58). It should also be noted that a drop in hardness occurred in the base metal after normalization, which is not seen in the pipes received from the manufacturer. The micrograph of the dissimilar metal weld interface shown in Figure 59 clearly shows the region of highest hardness to be present near the planar and cellular growth regions of the weld metal, in parallel with observations made about hardness in normalized pipes received from the industry. Note that at this stage of processing, a thin layer of carbides can be seen in the weld metal directly adjacent to the steel base metal, which was previously non-existent. It is hypothesized these carbides form due to carbon diffusion from the base metal into the weld metal at the interface during the normalizing post-weld heat treatment.
Figure 58: Plot of hardness traverses across DMW interface after normalizing PWHT.

Figure 59: Micrograph of hardness indents after normalizing PWHT.
A comparison of hardness traverses taken from both industry samples and the overlays performed at OSU across the dissimilar metal weld interface in the normalized condition can be seen in Figure 60. Both traverses appear to show similar widths of the high-hardness region in the weld metal. Although the traverse performed across the normalized overlay made at OSU shows a peak hardness nearly 60 points lower than the corresponding peak hardness in the pipe received from industry, previous work has shown that the exact value of peak hardness is highly dependent on the precise location at which the indent was made in the planar growth region. It is likely that both overlays have similar peak hardness values, but verification of this statement can only be achieved by performing a hardness map on a low-angle microsection of the overlay made at OSU.

Figure 60: Plot comparing hardness traverses on normalized overlays received from industry and performed at OSU.
Carbon Diffusion during Normalization

Carbon diffusion during PWHT was suspected to be the mechanism behind weld metal embrittlement in the transition zone at the DMW interface, based on previous results reported in similar dissimilar metal welds from the literature (13). Results from a DICTRA simulation of the normalizing PWHT process performed by the manufacturer can be seen in Figure 61. A distinct spike in carbon concentration initially develops at the DMW interface due to a chemical potential gradient present between the X65 steel and Alloy 825. As the PWHT continues, carbon slowly begins to diffuse into the FCC matrix of the weld metal. The DICTRA model suggests that the width of the carbon-rich region in the transition zone is between 100-150 microns, showing good correlation with the width of the high hardness region previously observed during metallographic characterization. The final peak carbon concentration calculated by DICTRA is roughly 10-15 times higher than the nominal carbon composition of Alloy 825. Verification of carbon composition via EPMA was planned, but was not possible due to time and budget constraints on the project. The literature shows that similar simulations have produced highly accurate results regarding carbon diffusion, both in terms of width and peak concentration (13). It can thus be concluded that the increased hardness in the transition zone after PWHT is due to carbon enrichment of the austenitic matrix and/or the formation of M₇C₃ carbides at the interface, as the literature has shown formation of M₇C₃ carbides in the transition zone of 8630 steel to Alloy 625 filler metal where local hardness increased up to above 860 Vickers (13).
Figure 61: Plot of carbon concentration at the DMW interface as a function of time during the normalizing PWHT.
5.1.2 Characterization of Induction Bent Pipes

Optical Microscopy

A macrograph of a failed induction bent weld overlay is shown in Figure 62. It can be noted that the second overlay etches in a darker fashion than the first overlay, and that some narrow bands of over-etched and un-etched material exist between the two overlays. These bands are due to the exact placement of the inert nail polish applied to the second overlay, which neutralized the upper layer and allowed for complete etching of the first overlay. Fusion boundaries have been highlighted in red for clarification. All cracks form in the same region, spanning perpendicularly from the fusion boundaries of the uppermost weld beads down towards the geometric stress concentrators at the dissimilar metal weld interface. It is observed that cracks form consistently at a roughly 60° angle from the plane of the DMW interface. Note that cracks do not penetrate into the base metal, but form only through the weld metal overlay.
At higher magnification, it can be seen that cracks form intergranularly along long, straight grain boundaries in the bulk weld metal (Figure 63). The intergranular ductility-dip cracks can be seen to link up with a pre-existing weld metal heat-affected zone liquation crack that occurred during the cladding process. Although it is difficult to optically distinguish ductility-dip cracks from liquation cracks, the jagged edges of the first 120 microns of the crack extending outwards from the fusion boundary suggest that this portion of the propagated crack is indeed the result of liquation during welding. Figure 64 shows ductility-dip cracks occurring along long, straight grain boundaries in the bulk weld metal of the second overlay.
Figure 63: Micrograph of ductility-dip crack passing through a fusion boundary in the second overlay.

Figure 64: Micrograph of ductility-dip cracks in the bulk weld metal of the second overlay.
A series of intergranular cracks can be found in the planar growth zone in proximity to the geometric stress concentrator formed between weld beads (Figure 65). These intergranular micro-cracks are typically very short, ranging from 20 to 40 microns in length. Higher magnification of an intergranular micro-crack reveals that the cracks form directly in the planar growth region at the DMW interface and propagate along the grain boundaries in the cellular/dendritic region of the overlay. (Figure 66). Low-angle sectioning of the dissimilar metal weld interface shows the presence of multiple microcracks forming along grain boundaries in the planar growth region and propagating along the cellular growth region and up in to the dendritic growth region of the bulk weld metal in the presence of a geometric stress concentrator formed by overlapping weld beads at the fusion boundary (Figure 67).
Figure 65: Micrograph of interfacial microcracks at DMW interface after induction bending.

Figure 66: Micrograph of interfacial microcrack propagating along grain boundaries through the planar and cellular growth region of the weld metal at the DMW.
Serial Sections

Serial sectioning was used to understand the sequence of crack formation and propagation during induction bending. The serial sections presented here were obtained creating six transverse cuts through overlaid pipes, each spaced 0.100” apart. Separate cracks were identified in each cross section, and images of the individual cracks at each cross section were compiled into composite micrographs showing how each crack propagated through the weld overlays.

Figure 68 shows a series of small, un-propagated overlay cracks. Note that all cracks are located in the HAZ of subsequent passes in the second weld overlay. These
cracks can be categorized as weld metal HAZ liquation cracks, given the small size and proximity to the fusion boundary. It should be noted that these liquation cracks are similar to those seen in the normalized condition, despite the fact that these images were taken from a bent pipe. Cracks in sections B and D have begun to grow along grain boundaries in both directions. The pre-existing weld metal HAZ liquation cracks seen in sections A, C, and F act as stress concentrators. Once strain is applied during the induction bending process, the liquation cracks can begin propagating via DDC along susceptible grain boundaries in close proximity.
Figure 68: Serially sectioned macrographs of un-propagated cracks in weld metal.
Sections A, C, and D of Figure 69 clearly show the initial stages of crack growth and propagation. Crack growth occurs from left to right (section A to section F). Note that crack growth occurs in a relatively vertical (but slightly diagonal) fashion. The thin cracks in section A extend outward from fusion boundaries in the second overlay. These cracks propagate outwards from HAZ liquation cracks down towards two locations: 1) neighboring weld metal HAZ liquation cracks in the next fusion boundary (typically above the existing crack), and 2) a stress concentrator formed at the DMW interface due to weld bead overlap as the first overlay is deposited. As shown previously, such a stress concentrator creates local interfacial micro-cracks, further increasing the ease of crack propagation.

Cracks can be seen to increase in width and length in sections D through F. By this stage, the cracks tend to be long and straight, running along epitaxially nucleated grain boundaries in the second overlay. In the first overlay, the cracks run along similarly shaped grain boundaries, from pre-existing weld metal liquation cracks toward the pre-existing micro-cracks at the DMW interface. Crack growth is complete once it has propagated through the full overlay thickness.
Figure 69: Serially sectioned macrographs showing initial stages of crack growth and propagation. Red box denotes region shown microscopically in Figure 70.
Figure 70 shows the multiple types of cracks occurring in an induction bent pipe. Two separate types of cracking can be seen in two distinct locations: 1) weld metal liquation cracking in the HAZ of the second layer fusion boundaries, and 2) ductility dip cracking in the fusion zone of the bulk weld metal. The liquation cracks are typically short (approximately 100 microns) and can be characterized by their occurrence just outside of the fusion boundary of subsequent passes in the second overlay. Note that most of these cracks have extended down along long, straight grain boundaries in the bulk weld metal in this sample via DDC. The ductility-dip cracks in Figure 70 can be characterized by their presence along long, straight grain boundaries in the bulk portion of the fusion zone. Examples of each type of cracking at higher magnification can be observed in Figure 71 and Figure 72.
Figure 70: Micrograph of Section A from Figure 69. Boxes 1 and 2 are shown at higher magnification in Figure 71 and Figure 72, respectively.

Figure 71: Micrograph of weld metal HAZ liquation crack from Figure 69.
Figure 72: Micrograph of ductility-dip from Figure 69.

An additional example of crack propagation can be seen in Figure 73. This serial section clearly shows how weld metal HAZ liquation cracks connect through the bulk weld metal via DDC. In section A, a liquation crack between the two weld overlays has been widened by the strain imposed during bending. Section B shows an additional liquation crack in the heat affected zone of the weld bead in the uppermost section of the sample. A long, straight ductility dip crack can be seen extending between the two weld metal HAZ liquation cracks in the second overlay. At this point, the crack also begins to extend down towards the geometric stress concentrator at the DMW interface. Again, this cracking follows long, straight grain boundaries, indicative of DDC. Section C shows the crack widening in both layers of the overlay. An interfacial micro-crack has also grown.
significantly as it begins to connect with the ductility dip crack in the adjacent weld metal. In section D, multiple ductility dip cracks have formed in the second overlay, and a small series of weld metal HAZ cracks can be seen in the first overlay just to the left of the largest ductility dip crack. Note that the fully propagated crack in section F extends from the HAZ of the uppermost weld bead down through to the DMW interface, connecting all three types of cracks.
Figure 73: Serially sectioned macrographs showing all stages of crack growth and propagation. Red box denotes region shown microscopically in Figure 74.
Figure 74 shows the liquation cracks and ductility dip cracks from Section B of Figure 73 at higher magnification. Two HAZ liquation cracks can be seen in the second overlay: one close to the interface with the first layer, and the second towards the top of the overlay. Note that both cracks have the same orientation as a result epitaxial nucleation from each subsequent weld pass. Due to an increase in stress concentration between the two liquation cracks, a ductility dip crack has formed in the bulk weld metal.

Figure 74: Micrograph of weld metal HAZ liquation cracks connected by DDC from Section B of Figure 73.
Identification of the exact metallurgical nature of the overlay cracks with certainty required SEM fractography of the fracture surfaces. SEM fractography was carried out on cracks found in both the first and second overlays of a bent pipe received from the industry. Since cracking was found to occur inside the weld overlays and no fracture surfaces were readily available for characterization, fracture surfaces were obtained by breaking open cracks in each layer, as explained in Section 4.2.3.

Analysis of the fracture surface from a crack in the first overlay reveals two distinct features: 1) microvoid coalescence due to ductile rupture and overload during opening of the cracks, and 2) macroscopically flat surfaces indicative of ductility-dip cracking (Figure 75). Observation of the fracture surface obtained from the second overlay shows similar macroscopically flat features throughout the entire region (Figure 76). At higher magnification, the fracture surfaces in both layers show microscopically wavy features (Figure 77 and Figure 78). These wavy features form as strain is accumulated around grain boundary carbides, opening up voids which ultimately link together to form ductility-dip cracks (3; 26). No evidence of liquid films was found at any point during analysis of the fracture surfaces, suggesting that the weld metal is susceptible enough to ductility-dip cracking that DDC may initiate without the added stress concentration of a pre-existing liquation crack.
Figure 75: SEM macro image of opened ductility-dip crack in the first overlay. Red box denotes region shown at higher magnification in Figure 77.

Figure 76: SEM macro image of opened ductility-dip crack in the second overlay. Red box denotes region shown at higher magnification in Figure 78.
Figure 77: SEM micro image of opened ductility-dip crack in first overlay.

Figure 78: SEM micro image of opened ductility-dip crack in the second overlay.
Expected Phases at Bending Temperatures

In order to understand the phases that could be present during induction bending, the induction bending temperature range of 1000 to 1050 °C was overlaid on the equilibrium and Scheil pseudo-binary phase diagrams generated by ThermoCalc (Figure 79 and Figure 80). The equilibrium phase diagram predicts that the weld metal will be fully austenitic during induction bending, with no additional secondary phases present. It should be noted that the bending temperature could come very close to the solidus temperature predicted in the Scheil diagram at 15% dilution in the transition region between the weld beads. At this point, the solidus is predicted to be 1060 °C, only 10 degrees above the maximum bending temperature. It is possible that some liquation could occur during bending, since the exact temperature during bending could vary depending on the exact location of the pipe within the induction coil. However, no concrete evidence that liquation occurred during bending has been found.

The equilibrium phase diagram produced in ThermoCalc suggests that no carbides are present at the bending temperature (Figure 79). The Schiel pseudo-binary phase diagram in Figure 80 predicts the formation of M$_{23}$C$_6$ carbides in the first overlay between 1120 and 1060 °C on cooling. However, the microscopic “wavy” features present along the fracture surfaces of both the first and second overlay suggest that some sort of fine carbides must exist along the grain boundaries of both overlays at elevated temperatures (26). Thus, it is hypothesized that the presence of fine M$_{23}$C$_6$ carbides could
cause strain concentrations along grain boundaries, resulting in an increase in susceptibility of the material to ductility-dip cracking.

Figure 79: Equilibrium phase diagram showing expected phases at bending temperatures.
Figure 80: Scheil module phase diagram with expected phases at bending temperatures.

The induction bending temperature range was also overlaid on the plot of solidus temperature versus weight percent titanium for completeness in understanding if liquid could be present at the bending temperature (Figure 81). The plot shows that local instances of liquation could potentially occur in the second overlay, if the titanium concentration were 17 to 26 weight percent. EDS traverse results showed that the typical titanium concentrations found at titanium carbides were only 5 to 10 weight percent, which would not cause liquation during bending. However, if the weld metal heat-affected zone cracks found in the normalized condition were caused by constitutional liquation, it is hypothesized that the local titanium concentration in the matrix surrounding the titanium carbide particles could be sufficiently enriched enough to cause
additional liqation during bending. It should be noted that concrete evidence of this has not yet been found.

Figure 81: Plot of solidus temperatures versus weight percent titanium showing bending temperature range.
Finite element analysis of the strain induced by heating of the dissimilar metal weld joint was carried out on overlays comprised of both Alloy 825 and Alloy 625 in order to understand if the cracking mechanism observed in induction bent Alloy 825 pipes was caused by the larger coefficient of thermal expansion found in Alloy 825 versus Alloy 625. It was found that the maximum strain induced on heating in the Alloy 825 overlay was 2.56% (Figure 82), while the maximum strain in the Alloy 625 overlay was 2.05% (Figure 83). As the total bending strain experienced by the pipe during manufacturing ranges between 9 to 15%, it was concluded that the 0.5% additional thermal strain seen in pipes clad with Alloy 825 was negligible and potentially did not contribute to the overall cracking mechanism.

During the analysis, it was found that stress concentrators exist at the dissimilar metal weld interface due to weld beads in the first clad layer overlapping each other. These stress concentrators correlate with the regions where interfacial microcracks have been observed to form. Optical microscopy has also shown that ductility-dip cracks propagate towards this region. It is believed that the increase in stress at regions of weld bead overlap contributes to the formation of microcracks during initial bending, forming sharper stress concentrators which promote the formation of ductility-dip cracks along nearby grain boundaries.
Figure 82: FEA results showing strain induced in an Alloy 825 overlay on heating.

Figure 83: FEA results showing strain induced in an Alloy 625 overlay on heating.
Figure 84 shows relatively similar hardness values at each location across a low-angle section of the dissimilar metal weld interface in the bent condition. The dip in hardness seen in Pattern 1 can be attributed to the presence of a crack extending towards the hardness traverse. Peak hardness recorded in the bent condition was 320 Vickers, located in the planar/cellular growth region of the weld metal. The micrograph shown in Figure 85 clearly shows the correlation between high hardness in the weld metal at the dissimilar metal weld interface and the formation of intergranular microcracks in the region. Note that the traverse and cracks represented in the micrograph also occur in the presence of a stress concentrator, promoting microcrack formation in the embrittled region.
Figure 84: Plot of low angle section hardness traverse across DMW interface in the bent condition. Pattern 1: weld bead center, Pattern 3: weld bead overlap, Pattern 2: intermediate point between 1 and 3.

Figure 85: Micrograph of low angle section hardness traverse in the bent condition. Hardness values shown in Vickers.
Hardness mapping was also performed on the same low angle section in order to more accurately measure the peak hardness (Figure 86). After bending, the hardness in the planar growth region increases from approximately 350 Vickers in the normalized condition to 400-450 Vickers in the bent condition. The maximum hardness recorded in the planar growth region was 481 Vickers. Observation of the indent size in Figure 87 shows that the peak hardness measurements were obtained from the weld metal indentations placed in closest proximity to the dissimilar metal weld interface, particularly in the planar growth region. It can be clearly seen that increased hardness occurs only through the planar and cellular growth regions, and that the weld metal resumes its average value upon entering the dendritic growth region. The average hardness of the base metal and weld metal were found to be around 200 Vickers.
Figure 86: Hardness map of DMW interface in the bent condition.

Figure 87: Micrograph of low angle section hardness map in the bent condition.
5.2 Discussion of Metallurgical Cracking Mechanisms

5.2.1 Weld Metal HAZ Liquation Cracking

Optical microscopy has shown the infrequent presence of short cracks (typically under 100 microns in length) forming outside of the fusion boundary between subsequent passes in the second layer of Alloy 825 cladding (Figure 41). SEM fractography revealed that liquid films were present along the faces of these cracks, suggesting that the cracks observed in the normalized overlays are weld metal heat-affected zone liquation cracks (Figure 42). Although these cracks were observed in the normalized condition, the initial formation of these cracks occurred during welding. Two mechanisms may be proposed to explain the formation of these weld metal liquation cracks: 1) segregation of solute atoms to the grain boundaries resulting in melting point depression, or 2) constitutional liquation of titanium carbide particles resulting in grain boundary wetting.

Non-equilibrium cooling rates experienced during welding result in partitioning of solute elements to inter-dendritic regions and solidification grain boundaries. Calculation of partitioning coefficients in ThermoCalc has shown that titanium and carbon partition most heavily in Alloy 825 throughout both layers of cladding (Figure 45). The literature has attributed liquation cracking in a similar Ni-Fe-Cr alloy (Alloy 800) to local reduction in the melting temperature of the matrix caused by an increase in titanium concentration during solidification (19) (20) (21). However, EDS traverses performed across solidification grain boundaries in the second overlay did not show increased levels of titanium as predicted by ThermoCalc (Figure 46). Despite the fact that
EDS is not highly sensitive to light elements such as titanium, it is theorized that the formation of weld metal liquation cracks is not due to segregation of titanium in the solidification grain boundaries.

The literature has also surmised that constitutional liquation of titanium carbide particles could play a role in liquation cracking of Alloy 800, which has a very similar composition to that of Alloy 825 (21). Characterization of Alloy 825 presented in the literature has shown that titanium carbides are present throughout the austenitic matrix (10) (11) (12). Calculation of partitioning coefficients reveals that titanium and carbon segregate most during solidification, and that carbon segregates even more heavily in the second overlay, where liquation cracks have been observed. Optical microscopy via polarized light with a DIC filter has shown continuous layers of carbides throughout the grain boundaries (Figure 49). It is hypothesized that the rapid heating experienced during welding causes partial dissolution of grain boundary titanium carbides in close proximity to the fusion boundary, resulting in weld metal liquation cracking.

All weld metal heat-affected zone liquation cracks that were found via optical microscopy were observed in the second layer of Alloy 825 cladding. No liquation cracking was observed in the first overlay. The presence of liquation cracks in only the second overlay can be explained by two theories: 1) heavier partitioning of carbon in the second overlay leads to formation of a larger amount of titanium carbides, increasing susceptibility to constitutional liquation, and 2) an increased heat input in the second overlay due to residual heat from the first overlay expands the temperature gradient in to the weld metal heat affected zone, increasing susceptibility to liquation cracking in
general. First, it has been observed that carbon segregates more heavily in the second overlay than in the first layer, and that continuous grain boundary carbides are present in the second overlay. Assuming that these particles are chromium- and titanium-rich $\text{M}_{23}\text{C}_6$ carbides as the literature suggests are present in the material, the susceptibility to constitutional liquation of such titanium carbides would be much higher in the second overlay than in the first, due to the higher concentration of carbides in the grain boundaries. It has also been shown that the level of dilution found in the second overlay shows a higher response to melting point depression with respect to the level of titanium present in the surrounding matrix, further increasing the susceptibility to liquation cracking the second overlay (Figure 47).

Second, the residual heat from the application of the first overlay combined with short cooling times between individual beads in the second overlay increases the temperature gradient in to the weld metal heat affected zone, detrimentally affecting the susceptibility to liquation cracking. Lower temperatures between individual beads in the first layer would be experienced, as there is no preheat applied, and the initial temperature of the pipe is that of the ambient environment, or room temperature. The interpass temperature recorded in the WPS supplied by industry partners was 215 °C, substantially higher than room temperature. This interpass temperature was observed between cladding layers, but not between the applications of individual beads in each layer. Given the helical nature and rapid travel speed of the GTA weld head around the internal diameter of the pipe, approximately two minutes of time passed before the arc returned to its initial position within the pipe. Combined with preheating of the pipe from
the application of the first cladding layer, the temperature gradient outside of the fusion zone would be longer and shallower in the second overlay than in the first overlay. Literature has shown that a long, shallow temperature gradient increases susceptibility to liquation cracking by heating a larger area outside of the fusion zone and increasing the temperature through susceptible grain boundaries (Figure 10). It is also probable that a higher level of restraint exists within the pipe at the point in time where the second overlay is applied, due to residual stress from the first overlay, thus increasing susceptibility to liquation cracking even further.

These results provide considerable evidence that the cracks observed in the normalized overlays are likely due to constitutional liquation of titanium carbide particles, leading to a drastic local reduction in the solidus temperature of the material. Combined with a higher level of restraint resulting from residual stress in the pipe after application of the first overlay and a shallow temperature gradient resulting from the higher heat input of the second overlay, this reduction in solidus temperature and increase in restraint ultimately lead to the formation of weld metal heat-affected zone liquation cracks in the second layer of weld overlays.

5.2.2 DMW Interface Embrittlement

Initial characterization of the dissimilar metal weld interface in both the normalized and bent conditions showed a narrow region of high hardness in the planar and cellular growth regions of the weld metal (Figure 51). Small interfacial microcracks
were found to form intergranularly in the planar growth region of the weld metal, ending at the dendritic growth region on one end and at the base metal on the other end of the embrittled region of the bent pipes (Figure 85). Reproduction of the cladding process performed at OSU allowed for characterization of the dissimilar metal weld interface after each stage of manufacturing: application of the first clad layer, application of the second clad layer, and the normalizing post-weld heat treatment performed on the completed overlay. Hardness traverses and accompanying micrographs have shown that embrittlement of the planar and cellular growth regions of the weld metal does not occur until after post-weld heat treatment (Figure 54 through Figure 59).

DICTRA kinetics modeling of the normalizing heat treatment shows that the slow ramp-up rate experienced by the pipes before reaching the normalizing temperature of 1000 °C results in an extended period of time at high temperature, allowing for carbon diffusion from the steel in to the weld metal (Figure 61). The literature has shown that a chemical potential gradient exists in dissimilar metal welds between 8630 steel and Alloy 625, and that carbon is drawn towards the chromium-rich nickel material, resulting in embrittlement due to the formation of carbon-enriched austenite or $M_7C_3$ carbides (13). Micrographs of Alloy 825 overlays produced at The Ohio State University show that a thin layer of carbides forms at the dissimilar metal weld interface only after normalizing post-weld heat treatment has taken place (Figure 55, Figure 59). Hardness maps of overlays received from the industry show that hardness reaches peak values closest to the layer of carbides, suggesting that they do play a role in embrittlement of the region (Figure 52, Figure 86).
Ultimately, it is believed that the normalizing post-weld heat treatment performed by the manufacturer is detrimental to crack susceptibility. Removing the post-weld heat treatment would eliminate carbon diffusion and embrittlement of the weld metal at the dissimilar metal weld interface, thus preventing the formation of microcracks. Since ductility-dip cracks have been observed to link up with microcracks at the DMW interface, elimination of microcrack formation could help reduce DDC susceptibility and severity.

5.2.3 Ductility-Dip Cracking

Optical microscopy of cracked induction bent Alloy 825 overlays has shown large cracks extending through the full thickness of the weld overlays, terminating at the dissimilar metal weld interface (Figure 62). These cracks tend to form at an approximately 60 degree angle with respect to the dissimilar metal weld interface and the axis through which bending strain is applied. Microscopically, the cracks run along long, straight grain boundaries in the bulk weld metal (Figure 64). Analysis of fracture surfaces in the first and second overlays obtained by breaking open cracks found in a bent pipe reveals macroscopically flat fracture surfaces with microscopically wavy features, indicative of ductility-dip cracking (Figure 75 through Figure 78).

FCC nickel-base alloys are generally known to be most susceptible to ductility-dip cracking at temperatures 800 to 1150 °C (3). The induction bending temperature range of 1000 to 1050 °C, falls well within this range of susceptible temperatures. DDC
also requires that a certain level of threshold strain be present before strain concentrations build up and initiate sliding along susceptible grain boundaries (3) (26). No work has been recorded in the literature regarding the threshold strain of Alloy 825, but it is believed that the bending strains of 9 to 15% induced during the induction bending process are more than sufficient to overcome the threshold strain required to initiate ductility-dip cracking within the overlays.

The presence of carbides along grain boundaries has shown to affect ductility-dip cracking susceptibility in both advantageous and deleterious manners (24) (26). The presence of large, blocky carbides can pin grain boundaries and DDC. On the other hand, small, evenly distributed carbides have been recorded to have a detrimental effect on DDC susceptibility by acting as strain concentrators and initiating voids that link up to form ductility-dip cracks. No evidence of tortuous grain boundaries formed from the presence of blocky carbides that would prevent ductility-dip cracking was found during metallographic characterization of the Alloy 825 weld overlays. The carbides observed along solidification grain boundaries in the Alloy 825 clad layers were found to be continuous chains of fine carbides, suggesting that their presence may adversely affect the ductility-dip cracking susceptibility of the material (Figure 49).

Based on crack morphology, location, the amount of strain induced by bending, and the elevated bending temperature at which cracks occur, it is evident that through-thickness cracking leading to ultimate failure of the weld overlay occurs via ductility-dip cracking.
5.2.4 Sequence of Metallurgical Cracking Mechanisms Leading to Final Failure

A variety of metallurgical cracking mechanisms observed in Alloy 825 overlays on X65 steel pipes has been presented in this study, but no explanation has yet been given to link them together and explain the complete sequence of the overall failure mechanism and how the individual cracking phenomena work together to form the final cracks observed in the induction bent pipes received from the industry. It is the intention of this section to explain how each metallurgical cracking mechanism assists in the final failure of the Alloy 825 weld overlay during each step of the manufacturing process.

The first of the cracks to appear in the overlay are the weld metal heat-affected zone liquation cracks during application of the second GTAW layer of cladding. The presence of these cracks along in the un-bent pipe would be no cause for alarm with regards to mechanical properties or corrosion resistance of the overlaid pipe, as they are typically very small, infrequent, and self-contained within the weld overlay. However, these small cracks act as stress concentrators once induction bending begins, causing local increases in strain along the grain boundaries in which they are present. This in turn facilitates the formation of ductility-dip cracks as the induction bending process continues.

Once welding is complete, the Alloy 825 clad pipes undergo a normalizing post-weld heat treatment, which is a standard industry practice used on pipes clad with Alloy 625 in order to obtain consistent hardness measurements across the dissimilar metal weld interface. However, carbon migration from the steel in to the planar and cellular growth
regions of the weld metal causes embrittlement of the region, leading to poor mechanical properties at the dissimilar metal weld interface. During initial application of strain during induction bending, microcracks begin to form along grain boundaries in the planar growth region through the cellular growth region, around the geometric stress concentrators at the DMW interface caused by the overlap of weld beads in the first clad layer. These microcracks further concentrate the strain along solidification grain boundaries in the cellular and dendritic regions, facilitating the formation of ductility-dip cracks in the bulk weld metal.

Thus far, it can be seen that both the pre-existing weld metal heat-affected zone liquation cracks and the interfacial microcracks act as stress concentrators as the initial strain during induction bending begins. As the bending process continues, additional strain is accumulated along the long, straight, epitaxial solidification grain boundaries running through both layers of the weld metal cladding. Ductility-dip cracks begin to extend outward from heat-affected zone liquation cracks in the middle of the overlay towards liquation cracks in the upper portion of the second overlay and towards the geometric stress concentrators and microcracks at the DMW interface. Once the cracks have extended through the full thickness of the overlay, the cracks begin to widen and pull apart as the final amount of strain is applied during bending.
5.3 Strain-to-Fracture Testing

5.3.1 Procedure Development

5.3.1.1 Resistance Heating of a Dissimilar Metal Joint

This was the first time that a dissimilar metal weld overlay had been resistively heated in the Gleeble™ at The Ohio State University. Type K thermocouples were placed on both sides of an initial modified strain-to-fracture sample in order to understand the effects that a through-thickness change in thermal conductivity would have on temperature distribution. Two thermocouples were placed at the center of the sample, and two more thermocouples were offset by 0.125” from the center. Tests showed that the steel side of the sample was consistently 7-11 °C cooler than the nickel overlay over a through-thickness distance of 0.25 inches (see Figure 88). The full temperature profile throughout the test can be seen in Figure 89. Such a small change in temperature through the thickness of the sample was believed to have an insignificant effect on the cracking mechanism. Since the temperature of the weld overlay was to be correlated with the cracking mechanism, thermocouples were placed solely on the weld metal for the remainder of all tests.
Figure 88: Schematic of modified strain-to-fracture sample showing average temperature distribution at 950 °C.

Figure 89: Plot of though-thickness temperature profile of modified strain-to-fracture sample held at 950 °C.
5.3.1.2 Temperature Distribution

Knowledge of the temperature distribution within the resistively heated sample was required to ensure that metallographic observation of cracks was carried out within an evenly heated region. Temperature distribution was recorded as explained in Section 4.3.2 (see Table 6). An example of the exact temperature profile can be seen in Figure 91. It was found that the average holding temperature at the center of the sample varied by no more than 9.0 ± 7.7 °C within 0.125 inches in both the x and y directions (Table 7). Average variance at 0.25 inches in the x direction from the sample center was found to be 23.8 ± 4 °C. The temperature gradients in both the x and y directions at all holding temperatures were relatively consistent. A holding temperature of 1050 °C produced the most even temperature distribution within the sample. Temperature varied most widely when the target temperature was 950 °C. It was decided that the maximum distance from the sample center observed during metallographic crack counting would be 0.125 inches, resulting in a total observed length of 0.25 inches.
Figure 90: Thermocouple locations during temperature distribution tests. Spacing between thermocouples is 0.125”.

Figure 91: Temperature profile plot in modified strain-to-fracture sample held at 900 °C.
Table 6: Recorded temperature distributions from tests run between 900-1100 °C.

<table>
<thead>
<tr>
<th>Target Temperature (°C)</th>
<th>T1</th>
<th>T2</th>
<th>T3</th>
<th>T4</th>
</tr>
</thead>
<tbody>
<tr>
<td>900</td>
<td>900</td>
<td>896</td>
<td>891</td>
<td>876</td>
</tr>
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<td>950</td>
<td>939</td>
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<td>1050</td>
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<td>1049</td>
<td>1033</td>
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<tr>
<td>1100</td>
<td>1100</td>
<td>1093</td>
<td>1091</td>
<td>1077</td>
</tr>
</tbody>
</table>

Table 7: Variance from target temperature as a function of distance from center.

<table>
<thead>
<tr>
<th>Target Temperature (°C)</th>
<th>T2</th>
<th>T3</th>
<th>T4</th>
</tr>
</thead>
<tbody>
<tr>
<td>900</td>
<td>4</td>
<td>9</td>
<td>24</td>
</tr>
<tr>
<td>950</td>
<td>11</td>
<td>23</td>
<td>26</td>
</tr>
<tr>
<td>1000</td>
<td>6</td>
<td>3</td>
<td>29</td>
</tr>
<tr>
<td>1050</td>
<td>-5</td>
<td>1</td>
<td>17</td>
</tr>
<tr>
<td>1100</td>
<td>7</td>
<td>9</td>
<td>23</td>
</tr>
</tbody>
</table>

Average

<table>
<thead>
<tr>
<th>Standard Deviation</th>
<th>4.6</th>
<th>9</th>
<th>23.8</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.3</td>
<td>7.7</td>
<td>4.0</td>
<td></td>
</tr>
</tbody>
</table>
An understanding of the relationship between stroke applied by the Gleeble and the average strain induced in the modified strain-to-fracture samples was desired in order to accurately produce the desired data points for the strain-to-fracture plots developed. Development of this relationship began with an extension rate of 0.025 mm/s and temperature of 950 °C, where both low and high stroke values were chosen to initially explore the relationship. After testing was complete, average strain in the central region of each sample was measured, and a linear interpolation was made between the two points. An additional data point at 950 °C between the two initial points show that the assumption of a linear relationship between stroke and strain was correct (Figure 92). Low and high stroke lengths were then chosen for each testing temperature, and linear interpolations were made for each temperature in the same manner previously described. A general trend of lowered stroke at higher temperatures to achieve the desired amount of strain can be observed in the plot.

The relationship between stroke and strain at varied stroke rates was also evaluated at 950 °C, and can be observed in Figure 93. It may be noted that as strain rate increases, the total stroke required to achieve a desired amount of total strain increases.
Figure 92: Plot of total strain versus stroke at 0.025 mm/s extension rate.

Figure 93: Plot showing effect of strain rate on stroke versus total strain at 900 °C.
5.2.2 Strain-to-Fracture Plots

Strain-to-fracture plots were produced based on data gathered from each induction bend simulation performed in the Gleeble (see Section 4.3 for details). The total crack length in mm found in the central region of each sample is noted next to each data point. Target strains of 9 and 15 percent are denoted on each strain-to-fracture plot based on expected manufacturing strain to two separate radii bends of 48” and 80”. Tests performed at low temperatures and strains were most resistant to cracking at each strain rate. A small amount of variability in crack length was observed at lower temperatures. In most cases, crack length increases with bending temperature and total strain. However, short cracks were observed in a sample pulled to 12% strain at 950 °C with a 0.05 mm/s extension rate, but no cracks were found when a sample was strained to nearly 20% elongation at the same temperature and strain rate (Figure 94). This can be attributed to the irregular presence of weld metal liquation cracks acting as stress concentrators in the weld metal. When weld metal liquation cracks existed in the central region of the strain-to-fracture sample, such cracks were found to open and enlarge during initial straining, ultimately leading to the formation of DDC along nearby grain boundaries in the bulk weld metal. Samples that exhibited no cracks in the weld metal during the induction bending simulation did not have any small pre-existing cracks which could open or initiate additional fracture paths.

The typical length of a heat affected zone weld metal liquation crack was found to be approximately 50 to 100 micrometers (Figure 95). In more rare and extreme cases,
some larger liquation cracks were measured up to 290 microns (Figure 96). Thus, it can be assumed that any total crack length under approximately 300 microns can be attributed to the presence of 2-3 small weld metal liquation cracks. This can be verified by optical characterization of transverse sections taken from the Gleeble samples (see Figure 97 and Figure 98). Some samples were found to deviate from the general trend of total crack length increasing at a constant temperature and increased total strain (Figure 96, 950 °C). This can also be attributed to a variation in the number of pre-existing weld metal HAZ liquation cracks. As such, it was decided that a total crack length of less than one millimeter would be used to distinguish the threshold strain between opening of liquation cracks and the onset of ductility-dip cracking.

![Total Crack Length](image)

Figure 94: Strain-to-fracture plot of simulated induction bend at 0.0125 mm/s.
Figure 95: Strain-to-fracture plot of simulated induction bend at 0.025 mm/s.

Figure 96: Strain-to-fracture plot of simulated induction bend at 0.05 mm/s.
Figure 97: Opened weld metal HAZ liquation crack in Gleeble sample pulled to 15.7% strain at 1000 °C, 0.0125 mm/s extension rate. Red line denotes fusion boundary.

Figure 98: Opened weld metal HAZ liquation crack in Gleeble sample pulled to 11.2% strain at 1000 °C, 0.025 mm/s extension rate. Red lines denote fusion boundaries.
Total crack length was found to increase exponentially after a threshold of approximately 1 mm total aggregate length was reached. In all cases, this exponential growth is correlated to the initiation of ductility-dip cracks in the weld metal (Figure 99 and Figure 100). The threshold strain required for ductility-dip crack initiation is marked on each strain-to-fracture plot with a gray line. This threshold strain level is only an approximate number, as the number of samples available for testing was limited. An extension rate of 0.025 mm/s was found to have the lowest threshold strain at all temperatures up to 1050 °C. Doubling the extension rate to 0.05 mm/s increased the threshold strain by 5% at 950 °C and 2% at 1050 °C. Halving the extension rate to 0.0125 mm/s increased the threshold strain substantially at temperatures near 950 °C, but the threshold strain at 1050 °C remained relatively constant.
Figure 99: Ductility-dip cracks in Gleeble sample pulled to 22.5% strain at 950 °C, 0.025 mm/s extension rate. Red lines denote fusion boundaries.

Figure 100: Ductility-dip cracks in Gleeble sample pulled to 16.0% strain at 1050 °C, 0.0125 mm/s extension rate.
The fracture surface of cracks induced during Gleeble simulation of the induction bending process closely mirror the features seen on the pipes received from the manufacturer (Figure 101). Note that the fracture surface is relatively flat and shows wavy features indicative of DDC. Based on these observations and the similarities found via optical microscopy between Gleeble samples and pipes received from industry, it is surmised that the induction bending simulation performed in the Gleeble accurately reproduces the bending and fracture conditions seen during manufacturing (compare to Figure 77 and Figure 78).

Figure 101: SEM image of DDC fracture surface in a Gleeble sample strained at 1100 °C.
5.2.3 Interfacial Micro-crack Plots

Strain-to-fracture plots depicting the number of interfacial micro-cracks that formed during Gleeble simulation were also developed (Figure 102, Figure 103, and Figure 104). The total number of micro cracks counted in each sample is listed next to each data point. Total strain was found to have a greater effect on micro crack formation than the bending temperature at each strain rate. A small number of micro cracks were found to form even at low temperatures and strain rates, suggesting that complete elimination of micro crack formation would not be possible by simply modifying the induction bending parameters. These results prompted an investigation of the effects caused by PWHT in an effort to minimize the formation of interfacial microcracks.
Figure 102: Strain-to-fracture plot of simulated induction bend showing the number of interfacial microcracks forming at a strain rate of 0.0125 mm/s.

Figure 103: Strain-to-fracture plot of simulated induction bend showing the number of interfacial microcracks forming at a strain rate of 0.025 mm/s.
Figure 104: Strain-to-fracture plot of simulated induction bend showing the number of interfacial micro cracks forming at strain rate of 0.05 mm/s.
The micrograph below shows the location of microcracks that formed during the modified strain-to-fracture test (Figure 105). It can be seen that the cracks form through the planar and cellular growth regions, in the same location as those observed in cracked overlays received from the industry.

Figure 105: Micrograph of interfacial microcracks induced during Gleeble testing.
5.4 Discussion of Strain-to-Fracture Plot Results

5.4.1 Effects of Bending Parameters on Ductility-Dip Crack Susceptibility

5.4.1.1 Total Strain

Since ductility-dip cracking is largely affected by the amount of applied strain, the value of total strain induced during the modified strain-to-fracture test is a key factor in determining the bending parameter window. As total strain increases at all strain rates and temperatures, the total crack length increases proportionately (Figure 94 through Figure 96). Eliminating ductility-dip cracking through reducing the total strain by changing the bend radius of pipes is possible, but not ideal. It is desirable to maintain the current bend radii used in the industry, thus an understanding of the effects of bending temperature and strain rate are crucial to successfully bending pipes overlaid with Alloy 825.

5.4.1.2 Bending Temperature

Bending temperature also plays a large role in determining the ideal bending parameter window for pipes clad with Alloy 825. A consistent pattern can be seen at all strain rates, in that the threshold strain required for onset of ductility-dip cracking is lowered as the bending temperature increases (Figure 94 through Figure 96). It is interesting to note that the threshold strain at all temperatures appears to be linear, and that no trough exists such as those seen in standard strain-to-fracture plots. It can thus be
seen that a reduction in bending temperature is ideal in order to avoid the formation of ductility-dip cracks.

5.4.1.3 Strain Rate

Modification of the strain rate during induction bending can have a great effect on the cracking response of Alloy 825 cladding. It was found that at a stroke rate of 0.025 mm/s, the bending parameter window in terms of temperature and total strain was smallest (Figure 106). Doubling the stroke rate increased the level of threshold strain to induce ductility-dip cracks, resulting in an increase of the maximum bending temperature at a 15% target strain from 1000 to 1025 °C. Limiting the bending temperature to 1000 °C shows that doubling the stroke rate to 0.05 mm/s also allows for a total strain of approximately 18%, up 4% from the threshold strain at 0.025 mm/s. Slowing the stroke rate to 0.0125 mm/s shows an increase in the threshold strain similar to that seen at a high stroke rate, but with a steeper slope. This is advantageous, as it opens the induction bending parameter window slightly more at temperatures below 1025 °C. It should also be noted that bending the pipes at a slower stroke rate also results in fewer opened weld metal heat-affected zone liquation cracks. Thus, it can be seen that the slowest strain rate which could be accommodated by the manufacturer would be ideal for induction bending pipes clad with Alloy 825.
The literature has shown that strain rate plays a role in the susceptibility of a material to ductility-dip cracking (23). Observation of the total crack length in the ductility-dip cracking regime of the strain-to-fracture plots shows that increasing the strain rate results in lowering the total crack length, while lowering the strain rate results in a large increase in the total crack length. These observations directly correlate with the generally accepted understanding that increasing strain rate reduces ductility-dip crack susceptibility. Despite the fact that ductility-dip cracks will grow more at slower strain rates, it is still recommended that the strain rate be reduced in order to successfully
manufacture pipes clad with Alloy 825, as the ductility-dip cracking regime can be avoided by simply modifying bending parameters such as temperature and total strain.

5.4.2 Optimization of the Induction Bending Parameter Window

The industry currently uses a bending temperature between 1000 and 1050 °C to obtain total strains of 9 and 15% at a strain rate equivalent to 0.025 mm/s in the Gleeble samples. The work completed in this project shows that these parameters push the cladding over the threshold strain, making them susceptible to ductility-dip cracking. This was verified by metallurgical characterization of bent pipes received from the industry in Section 5.1.2. As such, it is recommended that changes are made to the parameters currently used, in order to prevent failure of pipes overlaid with Alloy 825.

Given the fact that reducing the stroke rate results in the largest possible parameter window, it is recommended that the manufacturer form the pipes at as low a strain rate as possible. However, a balance must be kept between reducing the strain rate to eliminate DDC and keeping up with the demands on the rate of pipe production. At a stroke rate of 0.0125 mm/s, the maximum temperature used to achieve a total strain of 15% is shown to be 1025 °C. However, this puts the pipe precisely on the line of threshold strain. Results have shown that lowering the bending temperature substantially increases the threshold strain, and greatly opens the induction bending parameter window. A drastic reduction in bending temperature is not recommended, as a balance must be maintained between elimination of DDC and the yield strength of the X65 base metal as a function of temperature. In order to minimize yield strength of the base metal
and include a margin of safety while completely avoid ductility-dip cracking during induction bending, it is recommended that the bending temperature be limited to a maximum of 1000 °C. Maintaining the weld overlays below 1000 °C also completely eliminates the possibility of any further liquation, if there are any regions in which the local concentration of titanium in the matrix exceeds 17-18% in the second overlay. In order to retain the same manufacturing tolerances currently used with regards to temperature, a bending temperature of 975 ± 25 °C is suggested (see Figure 107). At this temperature and strain rate, it is expected that bends of both 48” and 80” radii can be readily performed, as the threshold strain for DDC at this temperature and strain rate is above 20%. This leaves a sizeable margin of safety with regards to the stress state in the pipes versus the Gleeble sample, assuming that in a worst case scenario the modified strain-to-fracture samples undergo only uniaxial stress, while the bent pipes experience tri-axial stresses due to constraints resulting from a larger geometry and thicker cross section.
5.4.2 Further Understanding of Cracking Mechanism

Optical microscopy and characterization of bent pipes received from industry has shown that the final cracks observed after bending are the result of a combination of pre-existing weld metal heat-affected zone liquation cracks, embrittlement leading to microcracking at the interface, and ultimately the formation of ductility-dip cracks. Based on comparison of the threshold strains required to induce microcracks and ductility-dip cracks, further observations can be made regarding the sequence in which each type of crack forms.
At the strain rate and bending temperature currently used in the industry, the threshold strain defined for microcrack formation will be surpassed at approximately 12% total strain. As the amount of total strain increases during bending, ductility-dip cracks will begin to form at 13% total strain. Under industry bending parameters, microcracks will form immediately before ductility-dip cracking takes over. Once the threshold strain for each type of crack is surpassed, the number and size of cracks begins to grow rapidly. Given the close correlation between the threshold strains for each type of crack, and the fact that microcracks immediately precede the onset of ductility-dip cracking, it is surmised that microcrack formation causes additional stress concentrators at the dissimilar metal weld interface, facilitating DDC propagation.

A similar correlation between the threshold strain required for microcrack formation and that required for onset of ductility-dip cracking can be observed at the recommended bending parameters of 975 °C at a 0.0125 mm/s extension rate. Microcrack formation is expected at 13% total strain, while the onset of DDC will not occur until approximately 21% total strain. It can thus be seen that while modification of the bending parameters can reduce susceptibility to ductility-dip cracking, a different approach must be taken to eliminate microcracking unless it is decided to make all bends with an 80” radius, lowering the total strain to 9% and avoiding the threshold strain for microcracks.
Chapter 6: Conclusions

6.1 Metallurgical Cracking Mechanism

Initial studies conducted in the industry suggested that overlay failure in Alloy 825 pipes was due to ductility-dip cracking. The metallurgical characterization of pipes received from industry, along with Gleeble simulation results from the modified strain-to-fracture test performed at OSU, show that multiple cracking mechanisms contribute to final failure of the overlay during induction bending. Weld metal heat-affected zone liquation cracks caused by the welding process act as stress concentrators at grain boundaries in the weld metal during induction bending. The normalizing post-weld heat treatment allows for carbon diffusion from the X65 steel in to the Alloy 825 weld metal at the dissimilar metal weld interface. This increases hardness of the weld metal at the interface, consequently causing embrittlement of the material. As strain is induced during induction bending, microcracks form along grain boundaries in the planar and cellular growth regions of the overlay, contributing additional stress concentrations along grain boundaries. Ductility dip cracks then propagate between liquation cracks and microcracks in the weld overlays as the level of strain increases during induction bending, ultimately resulting in final failure of the part.
6.2 Suggested Bending Parameters

Results from the modified strain-to-fracture test replicating the induction bending process show that changing process parameters such as bending temperature and strain rate can potentially eliminate ductility-dip cracking in Alloy 825 overlays. It is recommended that the strain rate be reduced by a factor of two in order to open the safe parameter window as much as possible in terms of bending temperature and total strain. At this strain rate, a bending temperature of 975 ± 25 °C is suggested in order to avoid the onset of DDC while still maintaining a reduction in yield strength of the X65 steel pipe. This will allow for a margin of 25 °C before the threshold strain for onset of ductility-dip cracking is reached at a maximum target strain of 15% for a 48” bend radius. At 975 °C, 6% more strain can be accommodated on top of the target strain of 15% before DDC begins. The additional strain available at this temperature is critical, as the Gleeble samples undergo a uniaxial strain, while the actual pipe experiences a more severe tri-axial stress state during bending. It is believed that bends of both 48” and 80” radii can be achieved with these bending parameters.

6.3 Microstructural Effects of the Normalizing PWHT

A region of high hardness in the planar and cellular growth regions in the weld metal at the dissimilar metal weld interface was found. Replication of the cladding and normalizing procedure at OSU has shown that the formation of this region of high
hardness is related to the normalizing post-weld heat treatment. DICTRA kinetics simulations show that the mechanism behind this embrittlement is carbon diffusion from the base metal into the weld metal during the heat treatment. Metallurgical characterization of bent pipes has shown that microcracks form along grain boundaries in this region, acting as stress concentrators to promote the onset of ductility-dip cracking leading to final failure of the weld overlay during induction bending. Changing bending parameters was not found to be an effective method of preventing microcrack formation in the dissimilar transition zone. Elimination of the normalizing post-weld heat treatment is recommended for pipes clad with Alloy 825 in order to perform bending successfully.

6.4 Final Recommendations

Overlay failure is not due simply to ductility-dip cracking during induction bending. While reducing the strain rate and bending temperature can reduce DDC susceptibility, modification of the bending parameters will not eliminate weld metal heat-affected zone liquation cracks or DMW interfacial microcracks, which act as stress concentrators and facilitate the onset of ductility-dip cracking. Liquation cracking can potentially be avoided by changing the welding process to reduce heat input and partitioning of alloying elements. Microcracking at the DMW interface can be avoided by elimination of the normalizing post-weld heat treatment. Thus, further work is required to define a new welding procedure and to test induction bending of weld overlays in the as-welded condition. For the time being, use of the current GTA welding process and
normalizing heat treatment may be continued, and DDC could be potentially avoided by halving the strain rate and lowering the bending temperature by 50 °C to 975 °C.
Chapter 7: Future Work

While the quality and quality of work performed in this study was sufficient to obtain the results outlined in the project’s scope of work, a number of additional tasks could be performed to further characterize the weldability issues, refine the suggested bending parameter window, and test the feasibility of recommended changes to the manufacturing process. Characterization of the weld overlays and liquation cracks could be furthered by EPMA analysis of grain boundaries in the second overlay in order to verify titanium segregation, which EDS traverses have been unable to detect. Low angle microscopy performed through the cracking plane in the weld metal may be a more effective method of opening fracture surfaces for observation, and could potentially reveal regions along the fracture surface where the cracking mechanism transitions from liquation cracking to DDC. Low angle microscopy could also be performed longitudinally at the DMW interface, allowing for characterization of the fracture surface of interfacial microcracks.

SSDTA analysis could be performed on samples removed from the first and second overlays in order to determine the temperatures at which phase changes occur in the material. It would be particularly advantageous to know the temperatures at which carbides begin to dissolve and/or precipitate in order to verify ThermoCalc results, and to understand exactly how carbides might play a role with regards to DDC in Alloy 825.
overlays. Producing equilibrium Alloy 825 phase diagrams in ThermoCalc with varying weight percent carbon and titanium (the alloying elements most prone to segregation) could be used to show formation of carbides in second layer grain boundaries under the non-equilibrium cooling conditions experienced during welding. True strain-to-fracture testing of undiluted Alloy 825 weld metal is also recommended, in order to fully understand the DDC susceptibility of Alloy 825 compared to other alloys, as no such information is currently available in the literature.

Additional testing using the modified strain-to-fracture test developed in this study is also suggested. Due to the limited amount of material available in this study, the full range of temperature and total strain could not be completely explored at high and low strain rates. If more material were available for testing, further exploration of these windows is suggested. An Abaqus FEA model showing the difference in stress triaxiality between the modified strain-to-fracture samples and the actual pipe during bending would also be beneficial in order to more effectively relate test results to the actual manufacturing process. The modified strain-to-fracture test should also be carried out on Alloy 825 overlays in the as-welded condition, in order to test the hypothesis that the normalizing PWHT is detrimental to crack susceptibility. Additional tests could also be carried out on samples with overlays performed using the GMAW CMT process, which could reduce crack susceptibility by refining the grain structure. For comparison purposes, it would also be useful to repeat the modified strain-to-fracture test on overlays made with Alloy 625, which is most commonly used in the industry at present.


27. An Investigation of Ductility-Dip Cracking in Nickel-Based Weld Metals, Part III.  


29. Theoretical Assessment of Dissimilar Metal Joint of Titanium to Stainless Steel.  


34. Daniels, Thomas W. Applicability of Cold Metal Transfer for Repair of Dissimilar Metal Welds in Stainless Steel Piping in Nuclear Power Plants. The Ohio State University. 2015.
