Friction Stir Welding and Microstructure Simulation
of
HSLA-65 and Austenitic Stainless Steels

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ABSTRACT

Friction stir welding has recently become an attractive process for the joining of steels. Interest in using this welding process to join steels has become popular due to advancements in friction stir welding tool development. Wear resistant - high temperature tools have been developed, which allow friction stir welding of high melting temperature materials. One such material the U.S. Navy is interested in joining with friction stir welding is a high-strength low-alloy steel (HSLA-65). The U.S. Navy plans to replace the current ship haul steel, DH-36, with HSLA-65, but conventional arc welding processes result with major distortion. A post-flame straitening process must be used to solve the distortion problem. Friction stir welding of HSLA-65 would result with less distortion, which would avoid subjecting the material to the flame straitening process.

The work presented here on friction stir welding of HSLA-65 is a continuation of previous investigation conducted by Norton and Sinfield (1; 2). From these previous two studies, it was suggested that austenitic stainless steel be friction stir welded to observe the high temperature behavior of the stir zone material. During this investigation Type 310 stainless steel was friction stir welded to observe the resulting microstructure.

A preheating method was tested during the friction stir welding of Type 310 stainless steel. Heat generation from frictional heating in austenitic stainless steel is difficult due to the low thermal conductivity. This is one of the reasons which contribute the difficulties of friction stir welding Type 310 stainless steel. The preheating method was used with successful results. A visually acceptable weld was produced with minimal
weld discontinuities and the discontinuities which were present originated from embedded thermocouples.

Friction stir welds were also conducted on HSLA-65 to determine the effects of various weld parameters on the resulting microstructure. A high and low tool rotational speed with other weld variables constant was tested. The resulting microstructures from these two welds were similar, which indicated that tool rotational speed is a robust weld parameter. A high and low travel speed with other weld variables constant was tested. The results indicated that travel speed affects the resulting microstructure much more than variations in tool rotational speed.

Hot torsion tests were conducted on Type 310 and 304L stainless steel using the Gleeble torsion unit. The purpose of the hot torsion tests were to simulate the microstructure which results from friction stir welding these materials and to collect torque data so estimated shear flow stress data could be calculated. The torsion tests successfully simulated the different regions of a Type 310 friction stir weld and simulated most regions of a Type 304L friction stir weld. Estimated shear flow stress values generated during the testing were calculated for both materials, with shear flow stress in Type 310 being greater than the shear flow stress in Type 304L.

The reported results from the friction stir welding of Type 310 and HSLA-65, along with the calculated shear flow stress value from the hot torsion testing are intended to aid in the development of the US Navy’s friction stir welding simulation model for HSLA-65.
DEDICATION

To my parents Dave and Vicki
for all their love, support, and encouragement
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During my graduate student career I have met numerous people who have helped me along the way that I would like to recognize here. I would first like to thank my advisor Dr. John Lippold for giving me the opportunity to further my knowledge in welding engineering and for getting me interested in welding metallurgy during my undergraduate studies.

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CHAPTER 1

INTRODUCTION

The solid state welding process known as friction stir welding (FSW) is relatively new when compared to fusion welding. Although these welding processes are implemented for similar applications, the major difference between the FSW process and fusion welding processes is that FSW is a solid state welding process. A solid state welding process is one where the material undergoing the fabrication does not reach its melting temperature. During the FSW process the material is heated to a temperature which allows plastic deformation to occur easily.

The ability to use friction stir welding for the joining of ferrous materials has only recently been brought to fruition because of the specialized tool material which first required development. Two types of FSW tools which provide necessary wear resistance and high temperature stability properties are tungsten based tools and a polycrystalline cubic boron nitride (PCBN) tool. Since the advent of these FSW tools, several types of ferrous alloys have successfully been friction stir welded.

The effects of a friction stir weld on the material properties are important to understand when determining the quality of weld produced. During a friction stir weld the material undergoes severe plastic deformation, which results in complex stresses and strains. These stresses and strains cannot be easily calculated due to the passing of the FSW tool through areas of interest and hindering current data acquisition techniques. The friction stir welding process affects the material not only thermally but also mechanically, which causes the creation of three microstructurally distinct regions of a friction stir weld: the stir zone (SZ), the transition region (TR), and the heat affected
zone (HAZ), each of these regions have unique microstructure and material properties. It has become desirable to understand the microstructure evolution in each of these regions so the resultant material properties of a FSW will be predictable.

A steel alloy which has undergone rapid development in determining its friction stir weldability is a high-strength low-alloy Grade 65 steel (HSLA-65). The shipbuilding industry is particular interested in this alloy because its high strength-to-weight ratio and reduced fabrication time provide a substantial cost savings compared to the currently used DH-36 steel. The Office of Naval Research (ONR) has been a driving force in the development of HSLA-65 and its fabrication.

To uncover the underlying principles in the microstructure evolution of an HSLA-65 FSW, an understanding of how the austenite phase is affected by the process must first be evaluated. The austenite phase has a face-centered-cubic (FCC) crystal structure and is the phase present in steels at elevated temperatures. The effects on austenite must be evaluated because this is the phase present in the material during the welding process, however, extensive conclusions on how the phase is affected by the process cannot be made due to the phase transformations the material undergoes during cooling. For this reason, austenitic stainless steels have been friction stir welded to determine the effects of the welding process on the austenite phase. The austenitic stainless steel friction stir welded microstructure has been examined and characterized by the utilization of optical microscopy, scanning electron microscopy, and orientation imaging microscopy.

To better understand microstructure evolution during a friction stir weld, physical simulations have been utilized. A physical simulation allows the material properties to be determined during processing through data acquisition and allows simulation variables to be independently varied. A physical simulation which has proven to simulate the microstructure which results from a ferrous alloy friction stir weld is the modified
Gleeble hot torsion test. During the hot torsion test the specimen is heated to a specific temperature, once the temperature is reached and held for a specified time, torsion is applied at a specific rotational speed and the specimen undergoes one revolution at this speed. The sample is then cooled using a helium quench to match the cooling rates observed during the FSW process.

During the hot torsion testing, data is gathered on the applied torque at a specific point in the revolution. This data allows the material’s shear flow stress and strain to be calculated. The shear stress generated in the material during friction stir welding is not readily available, therefore accurate process modeling cannot be achieved. This physical simulation allows the torque generated during the test to be acquired, which allows the shear stress to be calculated. In turn this shear stress can be correlated to the shear stresses which may be present during a friction stir weld.

The work described here is a continuation of an Office of Naval Research funded project at The Ohio State University which started in 2004. The original research on this project was conducted by Dr. Seth Norton, whose area of interest was HSLA-65 friction stir weldability and development of the hot torsion test (1). Matt Sinfield next became the principal investigator, whose area of interest was using the modified Gleeble hot torsion test for HSLA-65 (2). Next was the work presented here, in which the area of interest is using the hot torsion test for austenitic stainless steels. The next phase in the project is to develop material specific properties to facilitate process modeling. The aforementioned research was conducted under the advisory of Dr. John Lippold.
2.1 Ferrous Alloys

A ferrous alloy is one which contains Iron (Fe) as its main constituent. The ferrous alloys which will be discussed throughout this thesis include a high-strength low-alloy steel (HSLA-65) and two austenitic stainless steels, Type 310 and Type 304L. The U.S. shipbuilding industry is the main consumer of HSLA-65, where it will replace DH-36. The HSLA-65 steel provides a cost savings due to reduced fabrication costs and weight reduction cost since thinner gauge material may be used (3). This steel exhibits a minimum yield strength of 448 MPa (65 ksi) and a minimum tensile strength of 538 MPa (78 ksi).

Austenitic stainless steels are most often classified in the AISI 300 series. The alloy with the leanest composition in this series is Type 301 and use of this alloy is usually restricted to ambient temperature applications. The austenitic stainless steel which is most commonly used is Type 304, which exhibits high temperature and corrosion resistance properties. Stainless steel alloys with an ‘L’ designation indicate that the alloy contains low-carbon compared to its undesignated sister alloy. Alloys in this series whose primary application includes high temperatures are those with increased alloy content, such as Types 309, 310, and 314. Austenitic stainless steels are based on the Fe-Cr-Ni ternary system (4).

According to the Handbook of Stainless Steels, the leaner AISI Type 300-series stainless steels, such as Type 304L, would be predicted to be fully austenitic or within the
austenite-ferrite field. As well, these alloys usually contain high-temperature ferrite as a second constituent at very high temperatures. For this reason Type 304L would not contain a fully austenitic microstructure during the friction stir welding process. Another factor which contributes to the formation of ferrite in Type 304L is the reduction in carbon content also makes ferrite formation more likely. Highly alloyed austenitic stainless steels, such as Type 310, are shifted far enough to the right of the ferrite region on the Fe-Cr-Ni ternary phase diagram, that they have a fully austenitic structure (4).

2.2 Friction Stir Welding

Friction stir welding (FSW) is a solid state welding process used in the coalescences of similar and dissimilar materials. The tool used in the welding process consists of a larger cylindrical portion, which is termed the “shoulder,” and a smaller diameter pin portion which extends from the shoulder, which is termed the “probe” or “pin.” Common terminology used for the friction stir welding process is displayed below in Figure 2.1.

According to the original U.S. Patent filled by Wayne M. Thomas of The Welding Institute describes friction stir welding as (5):

A method of operating on a workpiece comprises offering a probe of material harder than the workpiece material to a continuous surface of the workpiece causing relative cyclic movement between the probe and the workpiece while urging the probe and workpiece together whereby frictional heat is generated as the probe enters the workpiece so as to create a plasticized region in the workpiece material around the probe, stopping the relative cyclic movement, and allowing the plasticized material to solidify around the probe.

The portion of this definition which should be noted is that there is not mention of melting metal. This indicates that this welding process is solid state in nature.
The weld depicted in Figure 2.1 is a butt-weld, which is a common joint configuration for friction stir welds. Other possible joint configurations include, but are not limited to T-joints, lap-joints, edge butt joint, and other custom joint configurations. Other key terminology which should be noted is the advancing side and retreating side of the weld, labels 7 and 9 in Figure 2.1. On the advancing side the tool rotation and travel directions are the same, and on the retreating side the tool rotation direction is opposite to the travel direction. These two terms are dependent upon tool rotation direction and travel direction and differ from each other in their resulting microstructure. The two
sides of the weld experience different heat inputs, therefore their material properties vary slightly.

The heat which is generated during the welding process is the result of the frictional contact between the shoulder and the material and the pin and the material. The frictional heat generated is produced by the rotation of the tool against the stationary work piece and the traversing of the tool along the joint. The welding process produces enough heat to cause the material to reach its plastic state, therefore the material is plastically deformed as the tool passes through the material. As the tool traverses through the material, the material is translated from one side of the tool to the other side. The friction stir welding process has previously been described as an extrusion process, based on evidence in which material flows around the tool.

Factors which influence heat generation described by Schneider, include weld parameters, thermal conductivities of the work piece, pin tool, and backing anvil, and the weld tool geometry (7). The weld parameters which largely affect heat generation during friction stir welding are travel speed and tool rotational speed. Higher tool rotational speeds or lower travel speed correlate to hotter welds, while lower tool rotational speeds or higher travel speeds correlate to colder welds (7). In a study by Chao et al, it was discovered that only about 5% of the heat generated by the FSW process flows to the tool and the rest flows to the workpiece (8). This heat efficiency is high when compared to conventional fusion welding.

There are several weld parameters which must be defined prior to welding. These parameters include the travel speed, tool rotational speed, axial downward force, and tool tilt angle. These parameters influence the heat generated during the FSW process. Other factors which influence the resulting weld include the pin features, the tool material, and diameter of the tool. These factors can control a large portion of the resulting weld, but
do not make up the entire sum of factors which influence the FSW. As with any welding process, there are many subtle nuances which contribute to a single weld.

The travel speed determines the rate at which the tool progresses along the joint, usually reported in either inches per minute or millimeters per second. The tool rotational speed controls the speed of the tool; speeds are on the order of 300-1000 RPM depending on the material. The axial downward force, also referred to as the z-axis load, controls the load of the tool against the work piece. The tool tilt angle refers to the degree the tool is tilted with respect to the workpiece. The material and characteristics of the tool are dependent upon the material; specific tool features for FSW of steel will be discussed later.

Several of these friction stir welding parameters along with other mechanisms generated during the welding process are responsible for the amount of torque generated. These variables, according to Nandan et al, include applied vertical pressure, tool design, the tilt angle, local shear stress at the tool material interface, the friction coefficient and the extent of slip between the tool and the material (9). The measured torque provides an idea about the average flow stress near the tool. The torque decreases with an increase in the heat generation rate, because it becomes easier for the material to flow at high temperatures and strain rates (9).

There are several advantages to the friction stir welding process and as with all welding processes there are disadvantages as well. Some advantages include low degree of distortion, a lack of fume generation, good mechanical properties, an absence of consumables, an ease of automation, and good weld surface aesthetics. Disadvantages include an exit hole, large axial downward force requiring large clamping force, and slower travel speeds than other automated welding processes.
2.2.1 Previous Studies on FSW of Steel

Friction stir welding of ferrous alloys has become a large area of interest for the department of defense, the aviation/aerospace industry, and the shipbuilding industry. These industries want to utilize the advantages of FSW on ferrous alloys because of its strengths. Several FSW studies have been performed on mild steel, high strength low alloy (HSLA) steels, and stainless steels.

One of the first records of FSW on ferrous alloys was a study performed with mild steel (10). This study was performed by Lienert et al, who investigated FSW on AISI 1018 steel, with a material thickness of 0.25 inches (6.35 mm). Two different tool materials were utilized during this work, a molybdenum based alloy and a tungsten based alloy. Travel speeds from 1 to 4 inches per minute and rotational speeds from 450 to 650 revolutions per minute were used during friction stir welding. Thermocouples along with an optical pyrometer to used to collect the thermal data. Thermocouples were placed in shallow grooves at several locations on the top and bottom surfaces to avoid to thermocouple shearing.

Peak temperatures on the order of 1000°C were recorded. Studies performed since this initial investigation have proven that FSW of steel produces temperatures well above 1000°C. Microhardness measurements were taken across a weld cross section. The hardest material was found to be located in the stir zone, with a hardness of 155-175 VHN. The as received base material had a hardness of 135 VHN. This investigation determined that mild steel could be successfully friction stir welded, and based on the work they proposed that FSW of HSLA steel and stainless steel could be feasible.

Several studies have been performed by Konkol pertaining to the feasibility of friction stir welding HSLA-65, particularly related to shipbuilding (11; 12). Friction stir welding on 0.25 inch HSLA-65 was performed and compared with the submerged arc
welding process. The friction stir welding process is desirable in ship production to combat distortion which results from current fabrication methods. Two different materials were studied by Konkol for possible friction stir welding tools. A tungsten-rhenium tool and one of polycrystalline cubic boron nitride (PCBN), each has their strengths and weaknesses for friction stir welding steel.

The tungsten-rhenium (W-Re) tool provides the thermal properties necessary for friction stir welding steel, but wore quickly during the friction stir welds preformed by Konkol. The PCBN tool provides the necessary wear resistance, but can undergo tool breakage easily and is more costly than the W-Re tool. An argon shielding gas was used during the friction stir welding; this shielding gas is not necessary but provides a more desirable surface finish. During the FSW of the HSLA-65 in Konkol’s study, a tool rotational speed of 400 revolutions per minute and a travel speed of 3 inches per minute (1.27 mm/s) were selected.

Konkol identified three microstructurally different regions of an HSLA-65 FSW, these are the stir zone (SZ), thermomechanically affected zone (TMAZ), and the heat affected zone (HAZ). It was observed that the microstructure of the SZ consisted of coarse ferrite grains, the TMAZ consisted of very fine ferrite grains, and the HAZ consisted of ferrite and small pockets of either carbides or martensite-austenite clusters which was observed in the base metal. A microhardness traverse was performed across the weld regions with a 100g load. The hardness was observed to be consistent across the FSW, with a Vickers hardness range between 190 and 230.

Additional feasibility studies on FSW HSLA-65 have been conducted by Posada et al (13) and Nelson et al (14). The investigation by Posada et al, compared HSLA-65 friction stir welds to DH-36 steel to determine if the HSLA-65 steel displayed the desired
properties to replace the latter in shipbuilding. It was shown that the mechanical properties of HSLA-65 were significantly higher than DH-36 steel.

<table>
<thead>
<tr>
<th>Material</th>
<th>YS MPa (ksi)</th>
<th>UTS MPa (ksi)</th>
<th>% RA</th>
<th>% EL</th>
<th>Charpy V-Notch Impact J (ft-lb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weld 652 HSLA-65</td>
<td>517 (75)</td>
<td>634 (92)</td>
<td>64</td>
<td>26</td>
<td>10.8, 28.4, 98.6, 105.3 (8, 21, 73, 78) @ -20°F</td>
</tr>
<tr>
<td>Weld 749-SH HSLA-65</td>
<td>537 (78)</td>
<td>654 (95)</td>
<td>61</td>
<td>24</td>
<td>143.1, 140.4, 79.7, 18.9, 50, 12.5 (106, 104, 59, 14, 37, 9) @ -20°F</td>
</tr>
<tr>
<td>Base Metal Material Code HIE</td>
<td>537 (78)</td>
<td>579 (84)</td>
<td>32</td>
<td>28</td>
<td>221.4, 159.3, 221.4 (164, 118, 164) @ -40°F</td>
</tr>
<tr>
<td>Base Metal ASTM A 945</td>
<td>448 (65) min</td>
<td>(537 – 689)</td>
<td>**</td>
<td>22</td>
<td>94.5 (70) @ -40°F</td>
</tr>
<tr>
<td></td>
<td></td>
<td>78 - 100</td>
<td></td>
<td></td>
<td>47.3 (35) @ -20°F**</td>
</tr>
<tr>
<td>Weld 648/649 DH-36</td>
<td>551 (80)</td>
<td>737 (107)</td>
<td>65</td>
<td>23</td>
<td>14.9, 18.9, 16.2, 13.5 (11, 14, 12, 10) @ -20°F</td>
</tr>
</tbody>
</table>

** HSLA-65 weld metal requirement

Table 2.1: Mechanical properties of FSW HSLA-65 compared to DH-36, by Posada et al (13).

A FSW process parameter window for HSLA-65 was constructed during the investigation by Nelson et al (14). The HSLA-65 plates used for friction stir welding were 0.25 in (6 mm) thick and the oxide was removed prior to welding. The material of the friction stir welding tool used was PCBN. The parameter window, Figure 2.2, illustrates FSW parameters in which fully consolidated welds may be produced (14).
Figure 2.2: FSW process parameter window for HSLA-65. Circles marked X indicate welds with small voids (14).

During this study, the range of thermal cycles which result from the different weld parameters included in the process parameter window were not collected. Microhardness traverses were conducted across the center of FSWs which used different weld parameters. It was found that at a constant rotational speed, the hardness across the weld nugget increases with travel speed. Also, peak hardness is observed on the advancing side of the weld and shifts toward the weld center with increasing weld travel and/or rpm (14).

An investigation of HSLA-65 single pass friction stir welds with a butt joint configuration was conducted by Pao et al (15). The weld parameters of a tool rotational speed of 600 rpm and travel speed of 5 mm/s (11.81 ipm) were utilized. Detailed optical microscopy was performed to determine the microstructural variations across the welds. It was found that the base metal consisted of small equiaxed ferrite grains with fine
particles of cementite dispersed throughout. Transmission electron microscopy (TEM) was also performed, which revealed dense dislocation population in the base metal.

In this study the HAZ was classified into the outer-HAZ (oHAZ) and inner-HAZ (iHAZ). The oHAZ was observed to be similar to the base metal, except contained fewer but coarser precipitates and also contained a lower dislocation density. The microstructure of the iHAZ consisted of finer equiaxed grains and some traces of retained austenite. This suggests the iHAZ region of the FSW reached temperatures above the $A_1$ temperature, where ferrite begins to transform to austenite.

The stir zone microstructure displayed Widmanstatten ferrite plates contained within large prior austenite grains. A TEM analysis confirmed the presence of martensite in the stir zone microstructure. A similar microstructure was observed in the TMAZ, except the prior austenite grains were much smaller.

Tensile samples cut from the transverse cross section were tested to determine the weakest part of the weld. All the tensile specimens were observed to fail in the region just outside the oHAZ on the advancing side. The observed tensile strength was 610 MPa and the yield strength was 523 MPa. It was also determined that the fatigue crack growth rate for the SZ and HAZ were significantly lower than the base metal due to the compressive stresses in these regions (15).

Bead-on-plate HSLA-65 friction stir welds were conducted by Sinfield with a W-25Re tool (2). The weld parameters used was a tool rotational speed of 850 rpm, travel speed of 6 ipm (2.54 mm/s), and a forging force of 3500 lbf (15.569 kN). These friction stir welding parameters were determined to be the nominal values by the Naval Surface Warfare Center, Carderock Division. During Sinfield’s investigation thermal-histories of the welds were collected, the weld microstructure was characterized, and SS-DTA was performed to determine phases present.
In Sinfield’s study the thermal-histories were collected by three different thermocouples to determine the best suited for friction stir welding. The thermocouples included Type-K wire and sheathed thermocouples and eroding thermocouples. It was found that they Type-K wire thermocouples collected better data than the sheathed. Also, by using eroding thermocouples in the center of the stir zone, an estimate on peak temperatures reached in this region could be collected. The thermal-history in Figure 2.3 was recorded from an eroding thermocouple. The microhardness map in Figure 2.4 shows the resulting hardness for the different weld regions.

![Figure 2.3: Thermal-histories collected by an eroding thermocouple on the advancing side of an HSLA-65 stir zone (2).](image-url)
2.2.2 Previous Studies on FSW of Type 304L Stainless Steel

Austenitic stainless steels are used in a variety of industries, especially where the materials need to exhibit good mechanical properties at high temperatures and maintain corrosion properties. One problem with fusion welding these austenitic stainless steels is their susceptibility to corrosion cracking and decay due to sensitization in the HAZ (16). Many of the studies which have been conducted on FSW of austenitic stainless steels tend to concentrate on the tool material, because by varying the tool material different material properties will result.
2.2.2.1 FSW of Type 304 with PCBN Tool

Friction stir welds on Type 304 stainless steel were conducted by Park et al, with a polycrystalline cubic boron nitride (PCBN) tool, a tool rotational speed of 550 rpm, and a travel speed of 1.3 mm/s (3.14 ipm) (17). The cross section of these welds displayed no defects. It was observed that the advancing side of the stir zone microstructure contained sigma phase. The rapid formation of sigma phase was due to the presence of delta-ferrite formation, high process temperatures, and large shear stress experienced in the stir zone.

Further investigations conducted by Park et al, determined that the ferrite which formed near a void defect in the stir zone consisted of grains on the order of 1 μm in size located along austenite grain boundaries. An example of this occurrence and other areas in the stir zone where ferrite was observed is shown in Figure 2.5 below (18). This was determined by the utilization of Electron Backscatter Diffraction (EBSD), which helped differentiate between the austenite and ferrite phases. In microstructural regions where sigma phase was observed during previous studies (17), there was no sigma phase present due to thinner material welded during this study. The lack of sigma formation is attributed to the faster cooling rate due to the difference in thickness. The welds without sigma phase formation were conducted on 2 mm thick material with a welding speed of 10.63 ipm (4.5 mm/s) and a rotation speed of 1300 rpm (18). Whereas the welds with sigma phase formation were conducted on 6 mm thick material with the welding parameters listed above (17).
Figure 2.5: EBSD maps of different stir zone regions (a) around void defect (b) near weld center and (c) on the advancing side of SZ. Grey and white colors indicate austenite matrix and ferrite which formed along boundaries, respectively (18).

2.2.2.2 FSW of Type 304 with W-Based Alloy Tool

An investigation on the influence of a W-based alloy tool to FSW Type 304 stainless steel was carried out by Sato et al (16). The focus of this study was to determine how the W-based FSW tool influenced the microstructure, mechanical properties, and corrosion properties of a Type 304 friction stir weld when compared to a Type 304 FSW completed with a PBCN tool. Bead-on-plate welds were conducted on 6 mm (0.24 in) thick Type 304 stainless steel with a travel speed of 1.33 mm/s (3.14 ipm) and a rotational speed of 550 rpm.

Examination of the weld cross section showed a tunnel defect at the bottom of the stir zone. The base material microstructure consisted of an annealed grain structure with a high density of annealing twins. The stir zone of was identified by two parts, the stir zone and the advancing side stir zone. The microstructure of the stir zone contained
slightly smaller grains than the base material and a lower density of twins. The microstructure of the advancing side stir zone consisted of two phases contained in small grains. The two phases were austenite and ferrite. The tungsten from the wear of the tool may have contributed to the formation of the ferrite since it contributes mildly to ferrite stabilization.

The research conducted by Sinfield also included 304L friction stir welds with a W-25Re tool. The friction stir weld parameters used were a tool rotational speed of 850 rpm, a travel speed of 2 ipm, and a forging load of 3500 lbf (2). The cross-section in Figure 2.6 is from a 304L friction stir weld. It was noticed that the resulting stir zone from a 304L FSW was circular in nature, whereas stir zones in other materials take the form of the tool geometry. Sinfield observed a difference in the retreating and advancing side stir zone microstructures. The advancing side of the stir zone contained more ferrite with finer austenite grains. It was believed the difference in the amount of ferrite present was due to the difference in temperatures between these two regions (2).

Figure 2.6: 304L friction stir weld cross-section from Sinfield (2).
2.2.3 FSW of Other Austenitic Stainless Steels

Several austenitic stainless steels other than Type 304 have been investigated to determine their friction stir weldability along with the resulting material properties. Friction stir welds on Type 316L stainless steel with a PCBN tool were conducted by Okamoto et al, to determine the stability of the austenite phase in the stir zone (19). Defect free bead-on-plate friction stir welds were completed on the Type 316L stainless steel. The stir zone consisted of a slightly refined equiaxed grain structure of austenite. A hardness traverse was conducted across the weld with 500 gram load, the resulting hardness profile was consistent due to homogeneous grain size (19).

Friction stir welds were performed on superaustenitic stainless steel AL-6XN with a tungsten alloy tool with a simple geometry by Reynolds et al (20). The composition of AL-6XN used during this study was 0.021%C, 0.36%Mn, 0.35%Si, 20.21%Cr, 22.63%Ni, 5.72%Mo, 0.003%S, 0.018%P, 0.28%Cu and 0.25%N. Several different weld parameters were tested and the acceptable welds were observed to be resultant of low tool rotational speed and high loading force. Grain refinement was observed in the stir zone of the FSW. Tensile testing was conducted on specimens taken perpendicular to the welding direction, and observations concluded that the tests failed in the base metal.

2.3 Physical Simulation

The physical simulation of friction stir welds is desirable to help determine the degree of stress and strain the material undergoes and thermal-history the material experiences. Hot compression and hot torsion tests utilizing the Gleeble have been investigated to determine their accuracy in simulating FSW resultant microstructure and providing the necessary mechanical data. A Gleeble based hot compression test on HSLA-65 was conducted by Forrest et al, which failed to simulate the microstructure resulting from friction stir welding (21). Studies have been performed by Norton (1) and
Sinfield, (2)(22) using a Gleeble based hot torsion test. This test has proven to accurately simulate the microstructure of a ferrous friction stir weld.

2.3.1 Gleeble Hot Torsion Tests

The Gleeble is a testing machine which allows the physical simulation of a material process to be conducted under controlled thermal and mechanical cycles. This machine is ideal for simulating various welding processes because of its capability to follow a thermal-history and perform high rate mechanical tests (23). The Gleeble hot torsion test utilizes the Gleeble 3800s’ mobile torsion unit.

The mobile conversion unit (MCU) provides a means to produce high strains while maintaining a temperature gradient across a torsion specimen gauge length (24). The torsion specimen is heated using direct current heating and the temperature is controlled by the use of Type K thermocouples. The specimen is heated to a peak temperature at a specific heating rate and held at this temperature for a user determined time, than torsion is applied at a specific rpm to one end of the specimen as the other end is held stationary.

A modified Gleeble hot torsion test was first conceptualized by Norton, which uses a slightly modified torsion sample and internal and external helium quenching system. The torsion sample was modified from the standard Gleeble torsion sample, which has a longer, solid gauge section. The modified samples contain a through-hole to allow internal quenching. The purpose of the internal and external quenching is to match the cooling rates observed in friction stir welding. This modified Gleeble simulation has successfully reproduced the microstructure of a ferrous friction stir welds during investigations by Norton and Sinfield.
2.3.2 Modified Gleeble Hot Torsion Test

The dimensions of the torsion specimen used in the modified Gleeble hot torsion test are displayed in Figure A.3 in appendix A. The hot torsion tests performed by Norton were conducted over a range of temperatures, number of revolutions, and speeds (rpm). The materials the torsion tests were conducted on were Armco iron and HSLA-65. To control the temperature of the specimens, an optical pyrometer was utilized. Norton found that certain microstructural regions of a friction stir weld could be simulated using the modified Gleeble hot torsion test. Shear strain for the torsion specimens was calculated by the following equation:

$$\gamma = \frac{r \cdot \theta}{l}$$

Where \( r \) is the outer radius of the gauge section, \( \theta \) is the rotation in radians of a specific point in the gauge section, and \( l \) is the length of the gauge section. The rotation in radians was measured by scribing a line on the gauge section before testing, then measure the amount of deflection in degrees after testing (1).

The modified Gleeble torsion tests which Sinfield conducted used a slightly modified technique. First, instead of using more than one rotation during the test, a single rotation was used. It was found that a single rotation was all that was needed to achieve the desired strain. A single rotation was also used because it was found that multiple rotations caused sample failures. This was due to the plastic instability of the torsion sample’s annular geometry at elevated temperatures (22). Second, instead of using a pyrometer to control the temperature, which was found to be imprecise, three Type K thermocouples were used to collect the thermal gradient across the gauge length. Each thermocouple was placed at a different location on the gauge length, one at the
center, one 3.175 mm (0.125 in) from the center, and one at the shoulder of the gauge. Based on the predetermined gradient, this allowed the temperature at the center of the gauge to be determined if the thermocouple placed there detached from the specimen (2).

With these refinements to the modified Gleeble torsion test, Sinfield was able to accurately simulate all microstructural distinct regions in a HSLA-65 friction stir weld. It was found though that the method Norton used for calculating shear strain was inaccurate because this method only accounted for the shear strain at the outer surface of the gauge. This is a problem because the area in which the simulated microstructure was occurring was not at the surface of the gauge. To combat this issue, Sinfield created a numerical model which simulated the hot torsion test and was able to calculate a more accurate shear strain and strain rate. The numerical model displayed a shear strain gradient through the thickness of the gauge (22).

The strain and temperature gradients existed along the gauge section due to the sample geometry and the heat flow from the center of the gauge to the sample grips. The model Sinfield used to analyze this gradient effect was created using DEFORM 3D (22). By modeling the hot torsion test, the effective strain and effective strain rate distribution could be visualized. The effective strain and strain rate gradients in Figure 2.7 show that these values are not uniform over the length or thickness of the gauge section.
Figure 2.7: Modified Gleeble hot torsion specimen which simulated FSW microstructure effective strain and effective strain rate from model (22)
2.4 Plasticity

During the modified Gleeble hot torsion test, the test specimen undergoes plastic deformation. The hot torsion test of a hollow specimen allows the analysis and prediction of metal flow (strain and strain rates), temperature and heat transfer, local variation in material strength or flow stress, and shear stresses to be investigated (25). The flow stress of a material during friction stir welding is desirable to know, because the flow stress is the required applied stress for metal to start flowing. Flow stress is the yield stress of a material and depends upon strain, strain rate, and temperature.

In hot torsion tests where the testing temperature is above the recrystallization temperature, the influence of strain on flow stress is insignificant and the influence of strain rate becomes increasingly important. The modified Gleeble hot torsion test allows stress to be obtained at higher strains, such as those observed in friction stir welding (25). The shear stress of a hot torsion test can be calculated as follows:

\[ \tau = \frac{T \cdot r}{J} \]

Where \( \tau \) is the calculated shear stress, \( T \) is the measure torque of the torsion test, \( r \) is the radius of the gauge, and \( J \) is the polar moment of inertia. For the modified Gleeble hot torsion test, the radius which was used for calculations was the center of the wall thickness. This radius was used since this is where the microstructure was simulated. For a hollow round shaft the polar moment of inertia is:

\[ J = \frac{\pi}{2} (r_o^4 - r_i^4) \]

Where \( r_o \) and \( r_i \) are the outer and inner radii of the specimen gauge (26).
2.5 Single Sensor Differential Thermal Analysis

During the friction stir welding of ferrous alloys it is desirable to observe the temperatures at which the solid-state phase transformations occur. A method used for in-situ determination of the transformation temperatures is Single Sensor Differential Thermal Analysis (SS-DTA). The FSW thermal history is first acquired by utilizing thermocouples. It is necessary to collect enough data during the welding cycle to generate an accurate cooling curve, to achieve this sampling rates of 500-1000 hertz are used (27; 28; 29).

The SS-DTA code creates a temperature profile of the welding cycle with respect to time. A reference curve is then generated and compared to the measured temperature, which allows local deviations between the curves to be observed. These local deviations can then be refined to determine the solid-state phase transformation temperatures (27). The SS-DTA method was utilized by Sinfield to measure the phase transformations in an HSLA-65 friction stir weld (2).

There were two solid-state phase transformations observed in an HSLA-65 FSW. The transformations were concluded to correlate with Widmanstatten ferrite and bainite (2). The start and stop phase transformation temperatures for bainite were observed to be 646°C and 459°C respectively. The austenite to ferrite transformation was also observed in the friction stir welds, with corresponding start and stop temperatures to be 906°C and 804°C respectively. The $A_{c3}$ and $A_{c1}$ temperatures along with the bainite transformation temperatures are listed in Table 2.2.
<table>
<thead>
<tr>
<th></th>
<th>HSLA-65 SS-DTA</th>
<th>Temperature °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_{c3}$</td>
<td></td>
<td>906</td>
</tr>
<tr>
<td>$A_{c1}$</td>
<td></td>
<td>804</td>
</tr>
<tr>
<td>Bainite Start</td>
<td></td>
<td>646</td>
</tr>
<tr>
<td>Bainite Stop</td>
<td></td>
<td>459</td>
</tr>
</tbody>
</table>

Table 2.2: Ferrite and bainite start-stop temperatures of HSLA-65 FSW(2)
CHAPTER 3

OBJECTIVES

Project objectives

1. Evaluate the impact of friction stir welding on the microstructure of AISI Type 310 austenitic stainless steel.
   a. Determine the microstructurally distinct regions resulting in the Type 310 friction stir weld
   b. Characterize the microstructure of the Type 310 friction stir weld using common metallographic techniques.

2. Determine how the material properties of HSLA-65 are affected by varying friction stir welding parameters.
   a. Characterize the microstructure resulting in the microstructurally distinct regions of the different HSLA-65 friction stir welds.

3. Estimate the shear flow stress in ferrous alloys by utilizing the modified Gleeble hot torsion test.
   a. Develop method of utilizing generated torque data from the Gleeble hot torsion test to determine shear stress
   b. Compare the resulting torsion specimen microstructure with the microstructure found in ferrous friction stir welds to determine a rough estimate of shear stress generated during the welding process.
CHAPTER 4

EXPERIMENTAL PROCEDURES

4.1 Materials

The material which was the main focus of this study was a high-strength low-alloy steel classified in the United States as, AISI HSLA-65. Two other steels were investigated to determine how an HSLA-65 friction stir weld microstructure evolves. These two steels were austenitic stainless steels classified in the United States as, AISI Type 304L and AISI Type 310. The modified Gleeble hot torsion tests were conducted on Type 304L and Type 310 stainless steel. Bead-on-plate friction stir welds were performed on HSLA-65 and Type 310. Bead-on-plate friction stir welds on Type 304L and HSLA-65 modified Gleeble hot torsion tests were conducted in a previous study performed by Sinfield (2).

4.1.1 HSLA-65 Material Properties

AISI HSLA-65 was the main focus of the study because this is the material the US Navy would like to integrate into their fleet. The Navy Surface Warfare Center, Carderock Division (NSWCCD), supplied AISI HSLA-65 0.5 in (12.7 mm) rolled plate. The microstructure of the plate is displayed in Figure 4.1. The microstructure of the plate consisted of equiaxed ferrite grains, fine bands of pearlite were observed along the rolling direction. The chemical composition of the HSLA-65 plate is listed in Table 4.1. Friction stir welds were performed on 6x12x0.5 in (147x304.8x12.7 mm) HSLA-65 plates. All six sides of the plates were machined to remove the mill scale which was present.
Table 4.1: Material compositions for HSLA-65, Type 310 stainless steel, and Type 304L stainless steel

<table>
<thead>
<tr>
<th></th>
<th>HSLA-65</th>
<th></th>
<th>304L</th>
<th></th>
<th>310</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Range</td>
<td>Actual</td>
<td>Range</td>
<td>Actual</td>
<td>Range</td>
</tr>
<tr>
<td>Fe</td>
<td>96.7</td>
<td>97.396</td>
<td>65.0-71.0</td>
<td>70.139</td>
<td>48.0-53.0</td>
</tr>
<tr>
<td>Cr</td>
<td>0.25</td>
<td>0.144</td>
<td>18.0-20.0</td>
<td>18.676</td>
<td>24.0-26.0</td>
</tr>
<tr>
<td>Ni</td>
<td>0.25</td>
<td>0.34</td>
<td>8.0-12.0</td>
<td>8.262</td>
<td>19.0-22.0</td>
</tr>
<tr>
<td>C</td>
<td>0.2</td>
<td>0.074</td>
<td>0.03</td>
<td>0.024</td>
<td>0.25</td>
</tr>
<tr>
<td>Cu</td>
<td>0.35</td>
<td>0.252</td>
<td>-</td>
<td>0.391</td>
<td>-</td>
</tr>
<tr>
<td>Mn</td>
<td>0.7-1.6</td>
<td>1.35</td>
<td>2</td>
<td>1.848</td>
<td>2</td>
</tr>
<tr>
<td>Mo</td>
<td>0.08</td>
<td>0.064</td>
<td>-</td>
<td>0.247</td>
<td>-</td>
</tr>
<tr>
<td>Co</td>
<td>-</td>
<td>0.007</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Si</td>
<td>0.15-0.5</td>
<td>0.24</td>
<td>1</td>
<td>0.314</td>
<td>1.5</td>
</tr>
<tr>
<td>P</td>
<td>0.04</td>
<td>0.011</td>
<td>0.045</td>
<td>0.028</td>
<td>0.045</td>
</tr>
<tr>
<td>S</td>
<td>0.05</td>
<td>0.006</td>
<td>0.03</td>
<td>0.002</td>
<td>0.03</td>
</tr>
<tr>
<td>V</td>
<td>-</td>
<td>0.058</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Nb</td>
<td>-</td>
<td>0.018</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Al</td>
<td>-</td>
<td>0.017</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Ti</td>
<td>-</td>
<td>0.012</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Ca</td>
<td>-</td>
<td>0.001</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Sn</td>
<td>-</td>
<td>0.01</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>N</td>
<td>-</td>
<td>0.069</td>
<td>-</td>
<td>0.064</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 4.1: Material compositions for HSLA-65, Type 310 stainless steel, and Type 304L stainless steel
4.1.2 Type 310 Stainless Steel Material Properties

Austenitic stainless steel alloys with increased alloy content, such as Type 310, are primarily used in elevated temperature applications. This microstructure was the reason for testing Type 310 stainless steel. It was suggested by Sinfield that Type 310 could be used to examine the effect of thermomechanical processing on austenite grain size evolution, which was obscured by ferrite formation in Type 304L. AISI Type 310 stainless steel plate was supplied in the form of 0.5 in (12.7 mm) rolled plate, the microstructure of this plate is shown in Figure 4.2. The microstructure of the supplied plate consisted of a mixture of medium to large twinned austenite grains. The chemical
composition of the Type 310 stainless steel plate is listed in Table 4.1. Friction stir welds were performed on 6x12x0.5 in (147x304.8x12.7 mm) plates, in which all six sides of the plates were machined to remove any oxides and scale which were present.

Figure 4.2: AISI Type 310 stainless steel base metal microstructure, 50 ml HNO\textsubscript{3} + 50 ml H\textsubscript{2}O electrolytic etch, org. mag. 200x

4.1.3 Type 304L Stainless Steel Material Properties

Norton was the first to propose the idea of using Type 304L to examine the austenite field and how it was affected during friction stir welding (1). Sinfield then conducted friction stir welds on Type 304L and after examination realized that ferrite
formed during the process \( (2) \). As a result of the ferrite formation in the microstructure, the grain size evolution of the austenite grains was hindered.

Type 304L was examined in this study for microstructure replication using the modified Gleeble hot torsion test. The resulting microstructures were compared to those observed by Sinfield’s Type 304L friction stir welds. Supplied AISI Type 304L stainless steel was in the form of 0.5 in \((12.7 \text{ mm})\) rolled plate and consisted of austenite grains dispersed with twinning and ferrite stringers from the rolling process in the microstructure, which is presented in Figure 4.3. The chemical composition of the Type 304L stainless steel is listed in Table 4.1.

![Figure 4.3: AISI Type 304L stainless steel base metal microstructure. Aligned phase along rolling direction is ferrite. Oxalic electrolytic etch, org. mag. 200x](image-url)
4.2 Thermocouples

Before performing the bead-on-plate friction stir welds, thermocouples were welded to plates in various locations. Two different plate designs were used for the placement of the thermocouples. The difference in the plate designs was the type of thermocouples used and the placement of the thermocouples. The thermocouple locations in the plates were based on Sinfield’s results. The thermocouples were placed in locations were temperatures from the bottom of the stir zone, advancing and retreating sides of the stir zone, and near the shoulder of the tool would be collected. The eroding thermocouples were placed in the center of the predicted stir zone.

One of the plate designs utilized eight Omega, Type K, 0.01” diameter, fine gage thermocouple wire with special limits of error. Special limits of error refer to the accuracy of the calibration of the thermocouple, which provide the thermocouple couples with less deviation in temperature than the standard calibration. These thermocouple wires were welded into predetermined hole placements using an Omega TL-Weld thermocouple and fine wire welder. Three wires were welded into each hole, a positive, negative, and grounding wire, which provided accurate data collection. The thermocouple wires were then cemented into place with Omega high temperature cement.

The second plate design utilized four welded thermocouples, the same type mentioned above, and two Nanmac, Type-K, 0.125” diameter, 304L E-12 series pencil probe eroding thermocouples. Shrink fitting with liquid nitrogen of the eroding thermocouples was tested, but the circumference of the thermocouple did not reduce enough to fit the pre-drilled hole. Therefore, the holes had to be bored to accept the eroding thermocouples. The two different plate designs are displayed in the Figure 4.4. For exact locations of the machined holes, refer to Figure A.1 and Figure A.2 provided in appendix A.
Figure 4.4: Backside of plates, (a) Thermocouple variation along path of predicted FSW; (b) Thermocouple placement with eroding thermocouples
4.3 **Friction Stir Welding**

All friction stir welding was performed at the Naval Surface Warfare Center Carderock Division (NSWCCD) on an MTS ISTIR PDS Intelligent Stir Welding machine (see Figure 4.5). Displayed in Figure 4.6 is the W-25Re featureless pin tool which was used to perform the welding. The pin of the tool was not redressed between welds, but the shoulder of the tool was filed down if metal from the previous weld had become attached to the tool. Prior to the weld initiation, the surface of the plates were wiped down with acetone to ensure there was no residual oil present from the machining process.

![MTS ISTIR PDS Intelligent Stir Welding machine at NSWCCD](image)

*Figure 4.5: MTS ISTIR PDS Intelligent Stir Welding machine at NSWCCD*
4.3.1 FSW HSLA-65

A total of four HSLA-65 friction stir bead-on-plate welds were completed, each with a weld length of 10.5 in (265 mm). The tool tilt angle was -3.5°, the forge load (z-load) was 3500 lbf (15.569 kN), and the tool rotation was clockwise for each weld. Before the bead-on-plate weld was performed, a pilot hole 0.200 in (5.08 mm) in diameter and 0.1875 in (4.7625 mm) in depth was drilled in the location of the start of the weld. This pilot hole reduced the wear on the tool, because the hole provided a location for the displaced material to flow during the plunge. The pin of the tool was not plunged into the pilot hole, but directly in front of the hole.

Each of the four HSLA-65 friction stir welds was conducted using a different set of parameters. These parameters were selected from a HSLA-65 friction stir welding operating window complied at NSWCCD. Two of the welds were completed with varying tool rotational speed and a constant travel speed, and the other two welds were completed with varying travel speed and a constant tool rotational speed. The varying
rotational speeds and travel speeds were selected from the high and low ends of the operating window. The weld parameters for each weld are listed in Table 4.2.

<table>
<thead>
<tr>
<th>Rotational Speed</th>
<th>Weld #426</th>
<th>Weld #427</th>
<th>Weld #430</th>
<th>Weld #431</th>
</tr>
</thead>
<tbody>
<tr>
<td>Travel Speed</td>
<td>1100 rpm</td>
<td>700 rpm</td>
<td>850 rpm</td>
<td>850 rpm</td>
</tr>
<tr>
<td></td>
<td>6 ipm (2.54 mm/s)</td>
<td>6 ipm (2.54 mm/s)</td>
<td>4 ipm (1.69 mm/s)</td>
<td>8 ipm (3.39 mm/s)</td>
</tr>
<tr>
<td>Forging Load</td>
<td>3500 lbf (15.569 kN)</td>
<td>3500 lbf (15.569 kN)</td>
<td>3500 lbf (15.569 kN)</td>
<td>3500 lbf (15.569 kN)</td>
</tr>
</tbody>
</table>

Table 4.2: HSLA-65 friction stir welding parameters

Before each weld was performed, a practice weld was completed on an HSLA-65 test plate to confirm that a visually acceptable weld would be constructed using the specific set of weld parameters.

4.3.2 FSW Type 310 Stainless Steel

No readily available weld parameters for friction stir welding Type 310 stainless steel were known, therefore several practice welds on Type 310 test plates were performed before a plate with thermocouples embedded was welded. All the Type 310 friction stir welds were performed using -3.5° tool tilt angle and a clockwise tool rotation. Pilot holes with the same dimensions as the HSLA-65 pilot holes were used in the location of the tool plunge. Three practice welds were completed each with differing weld parameters and each of these welds were visually unacceptable. The welds were determined unacceptable because of a visual worm-hole defect present on the surface of the weld.
A second Type 310 test plate was then preheated to 400°F (204°C) and then a friction stir weld was performed on the material. The resulting weld was visually acceptable, no surface defect was observed. Preheat was applied by wrapping the plates in a ceramic blanket, the preheat temperature was determined by using a portable thermocouple thermometer. A second test weld was performed using a preheat of 500°F (260°C), the result was the same as the first preheated test weld. Two 6 in (152.4 mm) bead-on-plate friction stir welds were then conducted on Type 310 plates which had embedded thermocouples. The weld parameters for each of these bead-on-plate welds are listed in Table 4.3

<table>
<thead>
<tr>
<th></th>
<th>Weld #437</th>
<th>Weld #438</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rotational Speed</td>
<td>500 rpm</td>
<td>500 rpm</td>
</tr>
<tr>
<td>Travel Speed</td>
<td>1 ipm (0.423 mm/s)</td>
<td>1 ipm (0.423 mm/s)</td>
</tr>
<tr>
<td>Forging Load</td>
<td>3500 lbf (15,569 kN)</td>
<td>3700 lbf (16,458 kN)</td>
</tr>
<tr>
<td>Preheat</td>
<td>500°F (260°C)</td>
<td>500°F (260°C)</td>
</tr>
<tr>
<td>Weld Length</td>
<td>7 inches (177.8 mm)</td>
<td>6.5 inches (165.1 mm)</td>
</tr>
</tbody>
</table>

Table 4.3: Type 310 stainless steel friction stir weld parameters

The welds constructed with the weld parameters displayed in the table above were visually acceptable, meaning there were no visual worm holes.

4.4 Modified Gleeble Torsion Tests

A Gleeble 3800 mobile torsion unit was used to perform the hot torsion tests on Type 310 and 304L stainless steel. The dimensions of the modified torsion sample can be found in Figure A.3 in the appendix A. The modified Gleeble hot torsion test setup is displayed in Figure 4.7.
Three Type-K thermocouples were welded onto the test specimen to monitor and collect the thermal data. The thermocouples were placed 0 inches (TC3), 0.375 in (9.525 mm) (TC2), and 0.5 in (12.7 mm) (TC1) from the left shoulder of the gauge section. The thermocouple placed 0.375 in (9.525 mm) from the shoulder was used to control the heating and cooling rates of the torsion test. This thermocouple was chosen because the low probability it would detach during testing. The thermocouple placed at the shoulder
was used to provide a safe guard for the possibility that the thermocouple placed 0.5 in (12.7 mm) from the shoulder would fall off, the temperature at the center of the gauge section could still be determined by interpolating the thermal data gathered from the remaining two thermocouples based on the knowledge of the thermal gradient along the specimen axis.

The hot torsion testing was performed in a chamber under a $4.2 \times 10^{-1}$ torr (56 Pa) vacuum, this constitutes a rough vacuum. Before the rough vacuum was pulled, the chamber was first double back purged with argon to try and displace any air in the chamber.

The modified torsion test samples were heated at a rate of $40^\circ$C/s. The Type 310 stainless steel samples were first heated to a temperature of $260^\circ$C and held at this temperature for 30 seconds, this was to simulate the preheat which was used during the FSW. After the 30 second hold, the sample was heated to the predetermined peak testing temperature and held at this temperature for 5 seconds before the torsion was applied. The Type 304L stainless steel samples were heated to the predetermined peak testing temperature and held at this temperature for 15 seconds before the torsional loading was applied. This allowed the temperature of the entire gauge section to become uniformly distributed.

Once the modified torsion test samples were heated to the peak temperature, the samples underwent one revolution of torsion at a specified rpm. The specimens were then cooled at a specified rate which matched the cooling rates obtained from thermal data collected during friction stir welding. To match the cooling rates produced by friction stir welding, a helium quench was utilized. The modified torsion samples were cooled both internally and externally.
A design of experiments (DOE) was constructed for the modified torsion tests, since testing several different peak temperatures and rpms was desirable. When a specified temperature and specified rpm are paired together during the test, this constitutes a single run in the DOE. During this experiment, each run was repeated twice, creating a total of 32 runs. The peak temperatures and rpms used in the Type 310 stainless steel modified Gleeble hot torsion tests are listed in Table 4.4. The full DOE for the torsion testing of Type 310 stainless steel is listed in Table B.1 in the appendix B.

<table>
<thead>
<tr>
<th>Temperature °C</th>
<th>RPM</th>
</tr>
</thead>
<tbody>
<tr>
<td>900</td>
<td>250</td>
</tr>
<tr>
<td>1000</td>
<td>550</td>
</tr>
<tr>
<td>1100</td>
<td>850</td>
</tr>
<tr>
<td>1200</td>
<td>1150</td>
</tr>
</tbody>
</table>

Table 4.4: Peak temperatures and rpm's values for the Type 310 torsion DOE

The peak temperatures and rpm’s used in the Type 304L stainless steel modified Gleeble hot torsion tests are listed in Table 4.5. The DOE constructed for the Type 304L torsion tests consisted of 24 runs. The full DOE for the modified torsion testing of Type 304L stainless steel is listed in Table B.2 in the appendix B.

<table>
<thead>
<tr>
<th>Temperature °C</th>
<th>RPM</th>
</tr>
</thead>
<tbody>
<tr>
<td>975</td>
<td>250</td>
</tr>
<tr>
<td>1075</td>
<td>550</td>
</tr>
<tr>
<td>1250</td>
<td>850</td>
</tr>
</tbody>
</table>

Table 4.5: Peak temperatures and rpm's values for the Type 304L torsion DOE
During the hot Gleeble hot torsion testing, torque data was acquired. The torsion of the sample was also acquired during testing. This allowed a specific point during the one revolution the sample underwent to be matched with the corresponding torque generated. These two values allow hot torsion shear stress-strain curves to be generated. The acquired torque data allowed the shear stress to be determined with the following equation.

\[ \tau = \frac{T \cdot r}{J} \]

The shear stress, \( \tau \), is dependent upon torque \( T \), radius of the gauge length \( r \), and the polar moment of inertia \( J \). The radius which was during the shear stress calculations was the radius to the mid-point of the wall thickness. This radius was use since this is the area in which the simulated microstructure was observed. The polar moment of inertia used was for a hollow round shaft. The polar moment of inertia equation is shown below, where \( r_o \) and \( r_i \) are the outer and inner radii of the specimen gauge.

\[ J = \frac{\pi}{2} (r_o^4 - r_i^4) \]

The shear strain was calculated for a specific point during the samples one revolution. Shear strain for the torsion specimens was calculated by the following equation:

\[ \gamma = \frac{r \cdot \theta}{l} \]
Where \( r \) is the radius of the gauge section, \( \theta \) is the rotation in radians of a specific point in the gauge section, and \( l \) is the length of the gauge section. Similar to the shear stress calculation, the radius which was used for the shear strain calculations was the radius to the mid-point of the wall thickness. The gauge rotation, \( \theta \), was determined from the acquired rotation values from the torsion test. The rotation data had to first be converted to units of radians by multiplying the acquired data by \( 2\pi \).

4.5 Material Characterization

Several different analytical methods were utilized to characterize the microstructure of the ferrous friction stir welds and the hot torsion tests. The techniques used to examine the microstructure include optical microscopy (OM), scanning electron microscopy (SEM), and electron backscatter diffraction (EBSD). To confirm the observations, Vickers hardness maps were created of the friction stir welds and phase transformations were analyzed using SS-DTA.

4.5.1 Metallographic Techniques

The friction stir welds were cross sectioned, using a vertical band saw, in specified areas where the welding process reached steady state. For the hot torsion specimens, the gauge section was first isolated, then cross sectioned, using a precision diamond saw, parallel to the longitudinal axis. The samples were mounted and prepared using a semi-automatic grinder/polisher. The following grinding and polishing steps were used: 120 grit SiC grinder paper, CAMEO Platinum #1 disk, CAMEO Silver disk with 6 micron diamond in suspension, ultra-silk cloth with 3 micron diamond paste and microid extender, and imperial cloth with colloidal silica.

After polishing, if the samples were to be used for SEM or EBSD analysis, they were placed on a vibratory polisher with colloidal silica for 24 hours. The samples
prepared for OM and SEM were etched using the following solutions: HSLA-65 was etched with 2% nital for 20 seconds, Type 310 was electrolytically etched with 50ml Nitric acid and 50 milliliters distilled water for 30 seconds with a current greater than 0.5 amps, the voltage was greater than 10 volts to act as a constant voltage power source. The Type 304L was electrolytically etched with 10g oxalic acid and 90ml distilled water for 30 seconds with a voltage of 3.5V. Each of the aforementioned etchants revealed the general microstructure of the material.

4.5.2 Instrumentation and Software

To conduct the optical microscopy, a Nikon Epiphot-TME inverted microscope with PAX-it! version 6 software was utilized. The images for SEM and EBSD were collected with two different microscopes, a Quanta 200 SEM and a XL30F ESEM. The SEM parameters use for the ferrous alloys were 20 kV and a spot size of either 4 or 5. The samples prepared for SEM analysis were mounted in Konductomet, had standard metallographic procedures, and etched with the solutions mentioned previously. The etching times were extended to provide a deep etch to increase image quality.

The EBSD data was collected and analyzed with TSL OIM Data Collection version 5.2 and OIM Analysis version 5.2 respectively. The samples prepared for EBSD analysis were the same as those for SEM analysis, except the samples were not etched. Different step sizes, from 10 to 0.3 microns, were experimented with to examine the resulting quality. It was found that a step size of less than 1 micron produced the best images. The microhardness maps were performed and constructed on a LECO LM 100AT microhardness tester and Amh43 version 1.56 software. The microhardness tests were conducted with a 300g load, 13 second dwell time, and a 250 micron indent spacing.
5.1 HSLA-65 Friction Stir Weld Regions

The friction stir weld region nomenclature which will be used throughout the results and discussion includes the stir zone (SZ), transition region (TR), and heat affected zone (HAZ). These three weld regions are depicted in Figure 5.1. The stir zone includes the region of material in which the probe of the tool passed through and the surrounding material which underwent recrystallization. The material which was not disturbed by the probe of the tool is included in the stir zone region because this material (1) reached temperatures which resulted in a phase transformation upon cooling and (2) underwent recrystallization due to the working of the material.

The transition region is the material between the stir zone and the heat affected zone. This region includes the friction stir weld region commonly referred to as the thermomechanically affected zone (TMAZ). The term transition region has been used by others investigating steel friction stir welds. It was observed by Konkol, et al that no thermomechanically affected zone was found in HSLA-65 friction stir welds because the material in this region was heated above the ferrite-austenite transition temperature (12). This region was classified as the transition region in HSLA-65 friction stir welds conducted by Sinfield, who after using a prior austenite etch found shear bands characteristic of the TMAZ. The region which contained the shear bands was narrow and only found on the advancing side (22).
The microstructure of the transition region is different from the microstructure of the HAZ; this is the division between these two regions. The microstructure in the TR underwent recrystallization because the ferrite grains are refined compared to the base metal. The TR also contains a larger distribution of Fe₃C located between the grains. The heat affected zone is the region in which the material only experienced a thermal cycle.

![Steel friction stir weld region nomenclature](image)

Figure 5.1: Steel friction stir weld region nomenclature
5.2 HSLA-65 FSW Acquired Thermal-Histories

The thermal-histories were acquired from various locations in the HSLA-65 friction stir welds. These thermal-histories recorded from different locations allowed the generation temperature distribution through the weld to be examined. This also provided a basis for a correlation between peak temperature and welding parameters. Each of the HSLA-65 was conducted with a different set of welding parameters; only one parameter was varied for each weld. Two different types of thermocouples were utilized during the HSLA-65 friction stir welding, (1) eroding thermocouples, and (2) Type-K wire thermocouples.

The eroding thermocouples allow the temperature to be acquired after the thermocouple has been sheared. For this reason, the eroding thermocouples were placed in the center of the stir zone. The eroding thermocouples were only embedded in the weld plates for weld 427 (700 rpm, 6 ipm, 3500 lbf) and weld 430 (850 rpm, 4 ipm, 3500 lbf). The acquired thermal-histories for this two HSLA-65 friction stir welds are shown in Figure 5.2. An image of the exact locations of the eroding thermocouples is also shown in Figure 5.2. The eroding thermocouple in weld 427 was placed 3 mm below the weld surface, while the eroding thermocouple placed in weld 430 was 4.5 mm below the weld surface.

Both of these thermocouples recorded peak temperatures above 1100°C. HSLA-65 friction stir weld 427 recorded a peak temperature of 1116°C and weld 430 recorded a peak temperature of 1180°C, both these value along with the respective cooling rates (30.9°C/s; 21.2°C/s) are listed in Table 5.1. From previous friction stir welding studies it is known that higher tool rotation speeds and slower welding speeds produce higher welding temperatures (7). The friction stir weld with a slower welding speed produced a higher peak temperature, 64°C higher, as predicted, although the difference in welding
speeds was only 2 ipm. The difference in welding speed was not the only parameter which affected the peak temperature, the difference in rotation rate contributed to this difference as well. Although, it will later be discussed that varying the tool rotation speed does not influence the peak temperature as much as varying the welding speed. From these peak temperatures it can be estimated that the peak temperature generated in the higher travel speed FSW, 8 ipm, would be lower than the slow travel speed of 4 ipm. This difference in temperatures would also contribute to a difference in resulting microstructures between the friction stir welds.

<table>
<thead>
<tr>
<th>Weld</th>
<th>Rotation Rate</th>
<th>Welding Speed</th>
<th>Thermocouple Location</th>
<th>Peak Temperature</th>
<th>Cooling Rate (800-500°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>427</td>
<td>700 rpm</td>
<td>6 ipm</td>
<td>eroding thermocouple; 3 mm below weld surface in center of probe path</td>
<td>1116°C</td>
<td>30.9°C/s</td>
</tr>
<tr>
<td>430</td>
<td>850 rpm</td>
<td>4 ipm</td>
<td>eroding thermocouple; 4.5 mm below weld surface in center of probe path</td>
<td>1180°C</td>
<td>21.2°C/s</td>
</tr>
</tbody>
</table>

Table 5.1: Peak temperature and cooling rates acquired from the eroding thermocouples placed in the HSLA-65 friction stir welds
Figure 5.2: HSLA-65 FSW thermal-histories acquired from eroding thermocouples
Each of the four HSLA-65 friction stir welds had a Type-K wire thermocouple located near the bottom of the stir zone. These wire thermocouples were 0.65 mm below the bottom of the tool. The temperature profiles in Figure 5.3 are the thermal-histories acquired from these thermocouples. Also shown in Figure 5.3 is an image of the exact locations of these thermocouples with respect to the friction stir welding tool and stir zone. The peak temperatures acquired from each of these thermocouples along with their measured cooling rates are listed in Table 5.2. The highest peak temperatures recorded in this weld region were from friction stir welds conducted with either a high tool rotation rate (weld 426) or a high welding speed (weld 430).

<table>
<thead>
<tr>
<th>Weld</th>
<th>Rotation Rate</th>
<th>Welding Speed</th>
<th>Thermocouple Location</th>
<th>Peak Temperature</th>
<th>Cooling Rate (700-300°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>426</td>
<td>1100 rpm</td>
<td>6 ipm</td>
<td>0.65 mm below probe</td>
<td>905°C</td>
<td>12.4°C/s</td>
</tr>
<tr>
<td>427</td>
<td>700 rpm</td>
<td>6 ipm</td>
<td>0.65 mm below probe</td>
<td>779°C</td>
<td>14.5°C/s</td>
</tr>
<tr>
<td>430</td>
<td>850 rpm</td>
<td>4 ipm</td>
<td>0.65 mm below probe</td>
<td>910°C</td>
<td>8.7°C/s</td>
</tr>
<tr>
<td>431</td>
<td>850 rpm</td>
<td>8 ipm</td>
<td>0.65 mm below probe</td>
<td>744°C</td>
<td>14.8°C/s</td>
</tr>
</tbody>
</table>

Table 5.2: Peak temperatures and cooling rates acquired from wire thermocouples located near the bottom of the welding tool in HSLA-65 friction stir welds
The peak temperature recorded from weld 426 was 905°C and the peak temperature of weld 430 was 910°C. The peak temperatures recorded from welds 426 and 431 were 779°C and 744°C, respectively. The peak temperature acquired from the thermocouple near the bottom of the stir zone in weld 430 was 270°C lower than the temperature recorded in the center of the stir zone. The difference of 337°C was recorded from thermocouples in similar locations in weld 426. Again, this difference can be attributed to the different welding parameters.

The two HSLA-65 welds conducted with a constant welding speed but different tool rotational speeds had a difference of 126°C in temperature. The cooling rates of these two welds were close. Weld 426 had a measured cooling rate of 12.4°C/s, while weld 427 had a measured cooling rate of 14.5°C/s. The difference in temperature between the welds conducted with a constant tool rotational speed but varying welding speed, welds 430 and 431, was 166°C. The difference between their cooling rates was larger than the difference between the cooling rates of weld 426 and 427. Weld 430 had a cooling rate of 8.7°C/s and weld 431 had a cooling rate of 14.8°C/s. These differences in temperature contribute to the theory that varying the tool rotational speed has less influence on peak temperature than varying the welding speed.
Figure 5.3: HSLA-65 FSW thermal-histories acquired from wire thermocouples near the bottom of the stir zone
Type-K wire thermocouples were placed on the advancing and retreating sides of welds 426 and 431. This allowed the peak temperatures generated on the different sides of the weld to be compared. The thermal-histories in Figure 5.4 were acquired from these thermocouples in welds 426 and 431. The imaging showing the exact location of the thermocouples is also shown in Figure 5.4. The thermocouples were located in the stir zone boundary and were either 4 mm or 5.5 mm from the tool center line. The thermocouples 5.5 mm from the tool center line were only located on the advancing side and were 2 mm from the weld surface. The thermocouples 4 mm from the tool center line were located on both the advancing and retreating sides, and were 3 mm from the weld surface.

From the plot of the thermal-histories, it was noticed that the peak temperatures on both sides of the weld were close for a particular weld. The peak temperatures for each of these welds are listed in Table 5.3. The thermocouple located closer to the surface on the advancing side of weld 426 recorded a peak temperature of 1095°C and the thermocouple located 3 mm from the weld surface on the advancing side recorded a peak temperature of 904°C. The peak temperature recorded on the retreating side of weld 426 was 988°C. The difference in peak temperatures measured from the thermocouples on the advancing side of weld 426 may be due to the higher temperature thermocouple being located near the weld surface. The majority of the heat generated during friction stir welding is generated at the tool shoulder, base metal interface (7). The difference in temperature between the advancing and retreating sides of weld 426 was 84°C. This difference is similar to advancing and retreating side differences in literature, in which a difference of 100°C has been observed (7).

The peak temperature acquired on the advancing side of weld 431, from the thermocouple 2 mm from the weld surface and 5.5 mm from the center of the probe was
867°C. The other advancing side thermocouple in weld 431 recorded a peak temperature of 988°C. The latter thermocouple was 3 mm from the weld surface and 3.5 mm from

<table>
<thead>
<tr>
<th>Weld</th>
<th>Rotation Rate</th>
<th>Welding Speed</th>
<th>Thermocouple Location</th>
<th>Peak Temperature</th>
<th>Cooling Rate (800-500°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>426 (1)</td>
<td>1100 rpm</td>
<td>6 ipm</td>
<td>Advancing side, 5.5 mm from center of probe path, 2 mm for weld surface</td>
<td>1095°C</td>
<td>29.4°C/s</td>
</tr>
<tr>
<td>426 (3)</td>
<td>1100 rpm</td>
<td>6 ipm</td>
<td>Advancing side, 3.5 mm from center of probe path, 3 mm from weld surface</td>
<td>904°C</td>
<td>28.5°C/s</td>
</tr>
<tr>
<td>426 (4)</td>
<td>1100 rpm</td>
<td>6 ipm</td>
<td>Retreating side, 3.5 mm from center of probe path, 3 mm from weld surface</td>
<td>988°C</td>
<td>27.7°C/s</td>
</tr>
<tr>
<td>431 (2)</td>
<td>850 rpm</td>
<td>8 ipm</td>
<td>Advancing side, 5.5 mm from center of probe path, 2 mm for weld surface</td>
<td>867°C</td>
<td>39.3°C/s</td>
</tr>
<tr>
<td>431 (5)</td>
<td>850 rpm</td>
<td>8 ipm</td>
<td>Advancing side, 3.5 mm from center of probe path, 3 mm from weld surface</td>
<td>988°C</td>
<td>41.8°C/s</td>
</tr>
<tr>
<td>431 (6)</td>
<td>850 rpm</td>
<td>8 ipm</td>
<td>Retreating side, 3.5 mm from center of probe path, 3 mm from weld surface</td>
<td>895°C</td>
<td>30.3°C/s</td>
</tr>
</tbody>
</table>

Table 5.3: Peak temperatures and cooling rates acquired from thermocouples located on both the advancing and retreating sides of weld 426 and 431
the center of the probe. The difference in peak temperatures between these two thermocouples on the advancing side of weld 431 was also observed in their respective cooling rates. The thermocouple closer to the weld surface had a cooling rate of 39.3°C/s and the thermocouple further below the weld surface had a cooling rate of 40.8°C. This difference in temperature may be due to the thermocouple which recorded the higher temperature being closer to the stir zone. Since weld 431 was conducted with a high travel speed, more friction heat may have been generated in this region due to the material undergoing severe plastic deformation.

The thermocouple located on the retreating side of weld 431 recorded a peak temperature of 895°C and had a cooling rate of 30.3°C/s. The peak temperature difference between thermocouples located in similar locations on the advancing and retreating sides of weld 431 was only 98°C. This difference is comparable to the difference observed in weld 426, except in weld 431 the advancing side experienced a higher peak temperature.
Figure 5.4: HSLA-65 FSW thermal-histories acquired from the advancing and retreating sides of the weld
All four HSLA-65 friction stir welds contained wire thermocouples located on the retreating side of weld. These thermocouples were either close to the shoulder of the tool or located in the transition region. The exact locations of the thermocouples, along with the acquired thermal-histories are shown in Figure 5.5. The thermocouples which were located close to the shoulder recorded a higher peak temperature than the thermocouples located in the transition region of the weld. This may be due to the higher heat generation which occurs between the tool shoulder and base metal interface. Cooling rates were not measured for these thermocouples if temperatures above 700°C were not recorded, because the material would not have undergone a phase transformation.

The peak temperatures acquired from each of these thermocouples are listed in Table 5.4. The thermocouple located on the retreating side of weld 426 was located in the transition region, which was 1 mm from the stir zone boundary. The peak temperature recorded from this thermocouple was 480°C. HSLA-65 weld 431 had a thermocouple in a similar location on the retreating side of the weld. A peak temperature of 416°C was recorded from this thermocouple. The thermocouples located on the retreating sides of welds 427 and 430 were positioned close to the shoulder of the tool. The peak temperature acquired the thermocouple in weld 427 was 608°C and the peak temperature in this location of the retreating side of weld 430 was 780°C.
<table>
<thead>
<tr>
<th>Weld</th>
<th>Rotation Rate</th>
<th>Welding Speed</th>
<th>Thermocouple Location</th>
<th>Peak Temperature</th>
<th>Cooling Rate (700-300°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>426</td>
<td>1100 rpm</td>
<td>6 ipm</td>
<td>1 mm from stir zone boundary, 2 mm from weld surface</td>
<td>480°C</td>
<td>N/A</td>
</tr>
<tr>
<td>427</td>
<td>700 rpm</td>
<td>6 ipm</td>
<td>1.5 mm from stir zone boundary, 1 mm from weld surface</td>
<td>608°C</td>
<td>N/A</td>
</tr>
<tr>
<td>430</td>
<td>850 rpm</td>
<td>4 ipm</td>
<td>1 mm from stir zone boundary, 2 mm from weld surface</td>
<td>780°C</td>
<td>9.1°C/s</td>
</tr>
<tr>
<td>431</td>
<td>850 rpm</td>
<td>8 ipm</td>
<td>1.5 mm from stir zone boundary, 1 mm from weld surface</td>
<td>416°C</td>
<td>N/A</td>
</tr>
</tbody>
</table>

Table 5.4: Peak temperatures and cooling rates acquired from thermocouples located on the retreating side of HSLA-65 friction stir welds
Figure 5.5: HSLA-65 FSW thermal-histories acquired from the retreating side of the welds
5.3 Effects of Varying Tool Rotational Speed

One of the weld parameters which affects the amount of heat generated during a friction stir weld is tool rotational speed. To determine how HSLA-65 is affected by the differences in heat generation due to tool rotational speed, high and low rotational speeds were used during friction stir welding. The friction stir weld numbered 426 was conducted with the following parameters: tool rotational speed of 1100 rpm, travel speed of 6 ipm (2.54 mm/s), and forging load of 3500 lbf. (15.569 kN). The surface finish of the weld is displayed in Figure 5.6. The surface weld width is uniform over the length of the weld. There are burrs present along the length of the weld on both the advancing and retreating sides of the weld; although, they are more consistent along the advancing side.

![Figure 5.6: Surface finish of HSLA-65 FSW #426](image)

The HSLA-65 friction stir weld which was conducted with a low rotational speed was numbered 427 and conducted with the following parameters: tool rotational speed of 700 rpm, travel speed of 6 ipm (2.54 mm/s), and forging load of 3500 lbf. (15.569 kN).
The surface in Figure 5.7 is of this FSW, which had uniform weld width and contained burrs along the length of the weld. The burrs resulting from the slower tool rotational speed are smaller and more uniform than the burrs resulting from the higher speed. The difference in observed burr size could be due to the shoulder losing contact while welding with the high tool rotational speed.

Figure 5.7: Surface finish of HSLA-65 FSW #427

The weld conducted with a tool rotational speed of 1100 rpm had a plate design with all Type-K wire thermocouples. The weld with lower tool rotational speed, 700 rpm, had a combined plate design with eroding and Type-K thermocouples. To ensure the location of thermocouples, radiography was performed on the friction stir welds (see Figure 5.8). These images were also used to determine if any weld defects were present. After examining the radiography images, it was determined that no weld defects were present, and the locations of the thermocouples in the all-welded Type-K thermocouple design were in the weld path. In the radiography image, it was noticed that the eroding thermocouple on the advancing side of the weld may not have extended into the center of
the stir zone. Also, the location of the thermocouple placed on the shoulder of the advancing side may not have been in the correct location.

Figure 5.8: Radiography images of the HSLA-65 FSW with varying tool rotational speed

5.3.1 Microstructure Characterization

The weld microstructure of welds 426 and 427 did not vary significantly in areas where the material is only affected by heat generation, such as the heat affected zone. This may be due to both welds experiencing similar thermal-histories. The measured cooling rates from thermocouples located below the stir zone of both welds (Table 5.2) are comparable. The cooling rate of the weld conducted with a tool rotational speed of 1100 rpm was 12.4°C/s. The cooling rate of the weld conducted with a tool rotational
speed of 700 rpm was 14.5°C/s. In microstructural regions where the material not only experienced a thermal cycle but also a mechanical cycle, a difference in microstructure was observed between the two tool rotational speeds. This difference in microstructure can be attributed to the amount of stress and strain generated in the material during the friction stir welding process. When austenite grains experience severe deformation upon cooling, the bainite start temperature is suppressed while the formation of ferrite is promoted due to an increase in nucleation sites (30). This phenomenon will be talked about in more detail later in the chapter.

The heat affected zones generated during the HSLA-65 friction stir welding with varying tool rotational speeds are approximately the same size (see Figure 5.9 and Figure 5.10). The friction stir welding tool profile is overlaid on the weld cross-sections in Figure 5.9 and Figure 5.10. After examining the weld cross-sections, the only observable difference is in the weld shape. The FSW conducted with a tool rotational speed of 1100 rpm is slightly broader at the base of the stir zone. The cross-section in Figure 5.9 also shows the location of the Type-K thermocouple at the bottom of the stir zone.
Figure 5.9: HSLA-65 FSW with a tool rotational speed of 1100 rpm cross section, 2% nital etch

Figure 5.10: HSLA-65 FSW with a tool rotational speed of 700 rpm cross section, 2% nital etch
The stir zone microstructures have a slight difference due to the different tool rotational speeds used during the welding process. The stir zone microstructure resulting from the weld conducted with a tool rotational speed of 1100 rpm contained large prior austenite grains that have transformed to a ferrite and bainite/Widmanstatten ferrite microstructure. The stir zone microstructure of this weld is displayed in Figure 5.11. The stir zone microstructure resulting from the weld conducted with a tool rotational speed of 700 rpm also contained large prior austenite grains that have transformed into a ferrite and bainite/Widmanstatten ferrite microstructure (see Figure 5.12).

Figure 5.11: HSLA-65 FSW stir zone microstructure resultant a tool rotational speed of 1100 rpm, 2% nital, org. mag. 400x
The amount of ferrite contained in the stir zone of the 700 rpm weld is greater than the amount in 1100 rpm stir zone. This may be due to the higher temperatures generated in the weld with a tool rotational speed of 1100 rpm, because the material becomes softer, reducing the amount of stress and strain. The bainite/Widmanstatten ferrite lath spacing is greater in the stir zone microstructure of the weld with a lower tool rotational speed. Again, this may be attributed to the higher amount of stress and strain experienced in the stir zone of this weld. These results observed in the micrographs were confirmed by SEM analysis. The microstructures of the stir zones with a prior austenite grain outlined for the high and low tool rotational speed welds are shown in Figure 5.13 and Figure 5.14, respectively. Both stir zone microstructures contained prior austenite grains on the order of 50 µm in size.
Figure 5.13: HSLS-65 FSW SZ microstructure resultant of a tool rotational speed of 1100 rpm, 2% nital etch

Figure 5.14: HSLA-65 FSW SZ microstructure resultant of a tool rotational speed of 700 rpm, 2% nital etch

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The microstructure of the transition region which resulted in each of these welds was similar. The TR of the HSLA-65 welds lacked shear bands, which are the main characteristic in the TMAZ of other material friction stir welds (7). The microstructure of the TR contained refined ferrite grains with dispersed colonies of Fe₃C. The ferrite grain size in the TR was greatly reduced when compared to the ferrite grain size in the base metal. This is caused by the mechanical stresses and strains experienced in this region during friction stir welding. The transition region microstructures for the welds conducted with tool rotational speeds of 1100 rpm and 700 rpm are displayed in Figure 5.15, and Figure 5.16, respectively.

Figure 5.15: HSLA-65 FSW TR microstructure resultant of a tool rotational speed of 1100 rpm, 2% nital etch, org. mag. 400x
The grain size in the TR of the weld with a tool rotational speed of 1100 rpm is slightly larger than the grain size in the TR of the weld with a tool rotational speed of 700 rpm. This difference is the result of the thermal cycle experienced in this region of each weld and the generated stress and strain of the welding process. Since the weld conducted with a higher tool rotational speed generated slightly more heat, the required flow stress of the material should be slightly lower than the weld which was executed with the lower tool rotational speed. Therefore, less dynamic recrystallization occurred during the welding process.

Since the material experienced a similar thermal cycle during the friction stir welding with varying tool rotational speeds, there was not a difference in the microstructure of the heat affected zone. The HAZ microstructure consisted of larger
ferrite grains due to ferrite grain growth during the thermal cycle. In Figure 5.17, which is a micrograph of the resulting HAZ, the upper left corner is the end of the TR which transitions into the HAZ.

![Figure 5.17: HSLA-65 FSW HAZ microstructure which was characteristic of both high and low tool rotational speeds, 2% nital etch, org. mag. 400x](image)

To aid in the investigation of how friction stir welding parameters affect HSLA-65 material properties, microhardness measurements were taken. From the measurements, microhardness maps were created. From these maps, it is easy to distinguish the different microstructural regions of the friction stir welds and their corresponding hardness; with higher hardness corresponding to a bainite/Widmanstatten ferrite microstructure. Figure 5.18 is the hardness map for the HSLA-65 FSW conducted with a tool rotational speed of 1100 rpm with the tool profile overlaid. By examining the
hardness map, it can be concluded that the hardest material is located in the stir zone, followed by the transition region, and then the heat affected zone.

![Microhardness map of HSLA-65 FSW with a tool rotational speed of 1100 rpm](image)

Figure 5.18: Microhardness map of HSLA-65 FSW with a tool rotational speed of 1100 rpm

The hardness of the stir zone, about 280-300 VHN, is evenly distributed across the weld region. Although there is a slightly harder area on the right side of the stir zone, which corresponds to the advancing side of the weld, the uniform distribution of hardness correlates to a uniform heat gradient generated in the stir zone. This also means that there is a smaller difference in material properties between the advancing side and retreating side of the weld. This is not true for the HSLA-65 FSW with a tool rotational speed of 700 rpm whose hardness map is located in Figure 5.19.
The hardness of the stir zone of the HSLA-65 FSW with a tool rotational speed of 700 rpm was between 275-300 VHN, which is slightly lower than the FSW with a tool rotational speed of 1100 rpm. The hardness of the lower tool rotational speed weld is not evenly distributed, with a higher hardness occurring on the advancing side of the weld. The weld shape is wider for this weld than the weld shape of the higher tool rotational speed weld. The transition region and heat affected zone dimensions are about the same for both welds.

The uniform distribution of hardness in the higher tool rotational speed weld compared to the lower tool rotational speed weld can be attributed to the higher stir zone peak temperature. Since a higher peak temperature was achieved during welding along with low stresses, more material reached a uniform temperature in the stir zone before undergoing a phase transformation. In the lower tool rotational speed weld, the highest
temperature was on the advancing side of the weld. Unlike the weld conducted with 1100 rpm, the weld conducted with 700 rpm generated higher stresses on the advancing side of the weld resulting in an asymmetric heat generation across the stir zone.

5.4 Effects of Varying Welding Speed

When the welding speed was decreased from 8 ipm (3.39 mm/s) to 4 ipm (1.69 mm/s) during friction stir welding, an increase in heat generation occurred. This was observed from the measure thermal-histories of each weld, and is consistent with previous studies (7). One reason for the increase in heat generation during the slower travel speeds is due to the increase in time for conduction to occur between the FSW tool and the work piece. Two different travel speeds with constant tool rotational speed were used during the HSLA-65 friction stir welding. The low travel speed was 4 ipm (1.69 mm/s), the high travel speed was 8 ipm (3.39 mm/s), a constant tool rotational speed of 850 rpm, and forging load of 3500 lbf. (15.569 kN) was used during the friction stir welding of weld 430 and 431, respectively.

The lower travel speed of 4 ipm (1.69 mm/s) generated more heat during friction stir welding, which was apparent from examining the heat affected zone from the top surface of the weld (see Figure 5.20). From this image, large burrs present on the advancing side of the weld were noticed. This may be due to the higher heat input raising the material’s temperature to a degree in which the softened material became highly plasticized, and more material is displaced to the shoulder of the weld. The top surface of friction stir weld 430 is wider than friction stir weld 431 which again is due to the higher heat generation.

The higher travel speed of 8 ipm (3.39 mm/s) generated a narrow heat affected zone which is observed from the top surface of the weld (see Figure 5.21). The lower heat generation during this friction stir weld caused almost no burrs to form on the
surface of the weld. Again, this may be due to the material in this friction stir weld becoming highly plasticized compared to the FSW with a lower travel speed.

Figure 5.20: Surface finish of HSLA-65 weld #430

Figure 5.21: Surface finish of HSLA-65 weld #431
To ensure the thermocouples were in the correct location and to identify any weld defects that may have been present, radiography was performed on the welded plates (see Figure 5.22). The radiographic image of weld 430 shows all the thermocouples to be in the correct location except the thermocouple which was supposed to be placed at the advancing side shoulder. After examination of this image, it was determined that there were not any visible weld defects present. All the Type-K thermocouples in weld 431 were determined to be placed in the correct location, and no visible weld defects were detected after examining the radiographic image.

Figure 5.22: Radiographic images of HSLA-65 FSW conducted with varying tool travel speed
5.4.1 Microstructure Characterization

The difference in generated heat and resulting cooling rates during the HSLA-65 friction stir welds was noticeable from the examination of their resulting microstructures. The higher heat generation in the FSW with a low travel speed produced a larger transition region and heat affected zone than the FSW with a high travel speed. Figure 5.23 and Figure 5.24 are the macro views of the respective weld cross section.

![Image of weld cross section](image)

**Figure 5.23**: HSLA-65 FSW with a travel speed of 4 ipm (1.69 mm/s) cross-section, 2% nital etch

From studying Figure 5.24, it was noticed that a possible void defect may have formed during the FSW with a high travel speed. This void is located in the lower corner of the advancing side stir zone, and was not observed in the radiography image of this weld. This void may be the result of the FSW tool contacting an eroding thermocouple.
causing a disruption in material flow, or this defect could be the result of the increased travel speed. Too fast of a travel speed is known to result in void formations, because the material cools before this region becomes filled with stirred material.

Figure 5.24: HSLA-65 FSW with a travel speed of 8 ipm (3.39 mm/s) cross-section, 2% nital etch

The stir zones which resulted from the friction stir welds performed with low and high travel speeds exhibit slightly different microstructures. The stir zone microstructure from the FSW with a travel speed of 4 ipm (1.69 mm/s), Figure 5.25, contains mostly a bainite/Widmanstatten ferrite with dispersed acicular ferrite microstructure. Whereas, the FSW with a travel speed of 8 ipm (3.39 mm/s) resulted in a stir zone microstructure made up of ferrite and bainite/Widmanstatten ferrite grains (see Figure 5.26). The difference in microstructure was due to the different thermal-cycle and mechanical cycle the material experienced in this weld region. The higher heat generation along with a lower stresses
generated in the low travel speed FSW allowed a more equiaxed structure to form. The high travel speed FSW experienced a greater mechanical cycle which resulted in much more deformed grains containing a fine bainite/Widmanstatten ferrite structure.

Figure 5.25: HSLA-65 FSW stir zone microstructure from weld performed with a travel speed of 4 ipm (1.69 mm/s), 2% nital etch, org. mag. 400x
Figure 5.26: HSLA-65 FSW stir zone microstructure from weld performed with a travel speed of 8 ipm (3.39 mm/s), 2% nital etch, org. mag. 400x

The microstructures observed in the stir zones of the friction stir welds conducted with varying travel speeds was confirmed by SEM and analysis (see Figure 5.27 and Figure 5.28). Prior austenite grains are outlined in both stir zone microstructures in the SEM images. The weld with a welding speed of 4 ipm had a prior austenite grain size of 50 µm and the stir from the weld with a welding speed of 8 ipm had a prior austenite grain sized of 25 µm. Again, the big difference between the stir zone microstructure of these two welds is the amount of ferrite which formed. The weld performed with a high travel speed contained much more ferrite in the stir zone, which is primarily due to the mechanical cycle this material experienced. The higher strains produced in this material during friction stir welding caused the bainite start temperature to be suppressed and the formation of ferrite to be promoted during the cooling cycle.
Figure 5.27: SEM image of stir zone microstructure from HSLA-65 FSW with low travel speed, 2% nital etch

Figure 5.28: SEM image of stir zone microstructure from HSLA-65 FSW with high travel speed, 2% nital etch
The stir zone of these two welds was not the only weld region which exhibited a difference in structure. The transition regions of each weld resulted in differing microstructures, which was determined after the examination of Figure 5.29 and Figure 5.30. The TR resulting from the FSW performed with a low travel speed contained refined ferrite grains with dispersed Fe₃C throughout the microstructure. The microstructure found in this same region of the weld with a high travel speed contained larger ferrite grains and less Fe₃C. Again, these observed differences in microstructures were the direct result of the thermal cycle and mechanical cycle the material experienced. The ferrite grain refinement which occurred in this weld region indicates the material experienced recrystallization. When material experiences strains the resulting microstructure has usually recrystallized.

Figure 5.29: HSLA-65 FSW TR from weld performed with a travel speed of 4 ipm (1.69 mm/s), 2% nital etch, org. mag. 400x
Figure 5.30: HSLA-65 FSW TR from weld performed with travel speed of 8 ipm (3.39 mm/s)

Unlike the HSLA-65 friction stir welds conducted with varying tool rotational speeds, the heat affected zone microstructures which resulted from the FSW with varying tool travel speeds were dissimilar (see Figure 5.31 and Figure 5.32). The HAZ was only dependent upon the heating cycle experienced in this region. The FSW conducted at a low travel speed resulted in a HAZ containing a mixture of large and small ferrite grains. The HAZ which resulted from the FSW with a higher travel speed, contained ferrite grains along with a small population of Fe₃C clusters. The HAZ from the low travel speed weld resembles the base metal microstructure much more than the HAZ resulting from the high travel speed weld. This is due to the HAZ material in the weld conducted with a 8 ipm welding speed experiencing a lower peak temperature.
Figure 5.31: HSLA-65 FSW HAZ from weld performed with a travel speed of 4 ipm (1.69 mm/s), 2% nital etch, org. mag. 400x

Figure 5.32: HSLA-65 FSW HAZ from weld performed with travel speed of 8 ipm (3.39 mm/s), 2% nital etch, org. mag. 400x
To further quantify the difference between microstructures generated during the HSLA-65 friction stir welding with varying tool travel speeds, microhardness maps were constructed. A peak of 300 VHN was observed in the stir zone of the FSW conducted with a low travel speed. The hardness map is shown in Figure 5.33. The peak hardness is uniformly distributed through the stir zone. A of 240 VHN resulted in the TR of this weld which makes up a large portion of this weld. The HAZ of this weld had a Vickers hardness of 220 and was noticeably smaller than the transition region.

![Microhardness map of HSLA-65 FSW with a tool travel speed of 4 ipm (1.69 mm/s)](image)

Figure 5.33: Microhardness map of HSLA-65 FSW with a tool travel speed of 4 ipm (1.69 mm/s)

A much higher hardness was observed in the stir zone of the HSLA-65 friction stir weld conducted with a high travel speed; this weld’s microhardness map is presented in Figure 5.34. This weld’s stir zone had a hardness of 320 VHN. The majority of this hardened material was on the advancing side of the weld. The Vickers hardness values of the TR and HAZ of this weld are 260 and 240, respectively; these regions were much
thinner than those observed in the low travel speed weld. The high travel speed weld resulted in a much narrower weld shape which is noticeable in the microhardness map. This difference in weld shape is contributed to the heat generated during the welding process and the shear forces present at the tool/workpiece interface. The possible shear mechanisms are either sticking or sliding, and from the resulting weld shapes, it seems a sticking mechanism was present during the low travel speed weld and a sliding mechanism present during the high travel speed weld (7).

Figure 5.34: Microhardness map of HSLA-65 FSW with a tool travel speed of 8 ipm (3.39 mm/s)

5.5 HSLA-65 FSW EBSD

The microstructurally different regions of an HSLA-65 friction stir weld were analyzed by EBSD to determine if a preferred orientation existed in any of the regions. In previous studies, it has been found that the grains located in the transition region of a
friction stir weld tend to align in a preferred orientation. This is due to the grains in this region all undergoing the same amount of strain and adiabatic shear bands forming. The EBSD scan in Figure 5.35 contains the different microstructural regions of an HSLA-65 friction stir weld. There was not a noticeable difference in grain size between the base metal, TR, and the stir zone. This was due to the large step size which was used over a large sample area. In the TR of this HSLA-65 weld, there seems to be a slightly noticeable preferred grain orientation.

The grains in the transition region were confirmed to have a preferred orientation by examining the pole figures associated with the EBSD images. The pole figures in Figure 5.36 correspond to the different regions in the prior EBSD image. The [001] direction pole figure for the heat affected zone shows the typical pattern associated with rolled plate. The intensity of the preferred grain orientation is less than normal preferred grain orientation observed in rolled plate. This may be due the thermal cycle the HAZ experienced. The [101] direction pole figure for the TR shows less random grain orientation than the stir zone. Even though shear bands are not present in this weld region, this confirms the grains in this region were aligning in a similar orientation. Both pole figures for the stir zone show that a random grain orientation exits in this region, which was expected.
Figure 5.35: EBSD scan of an HSLA-65 FSW
Figure 5.36: Pole figures for the different regions of an HSLA-65 FSW
The EBSD scan in Figure 5.37 was performed on the microstructure of an HSLA-65 FSW stir zone. This scan had the common grain orientations highlighted to aid in the observation of the prior austenite grains. The prior austenite grains contained laths of bainite/Widmanstatten ferrite. The prior austenite grains are quite large compared to the grains in the base metal. The grain boundaries are somewhat distorted, a characteristic which has been observed in the Type 310 FSW stir zone. The smaller grains located at the lath grain boundaries are cementite (Fe₃C) which have formed. Also, the laths in this area of the stir zone do not seem to have a preferred grain orientation.

Figure 5.37: EBSD scan of an HSLA-65 FSW stir zone with grain orientation highlighted
The EBSD scan in Figure 5.38 was conducted over an area in the transition region from an HSLA-65 friction stir weld. The grains in this area of the TR have a random orientation. The larger grains in this image are ferrite grains and dispersed between these grains are iron carbides.

Figure 5.38: EBSD scan of an HSLA-65 FSW TR/HAZ
5.6 Phase Transformations

To confirm the phases present in the microstructure of the HSLA-65 friction stir welds, SS-DTA was performed on the collected thermal-histories to determine the temperatures at which the transformations were occurring. The transformations start and finish temperatures for each weld, along with the thermocouple locations in the weld and their respective $T_{8.5}$ cooling rates, are listed in Table 5.5. In general, the bainite start temperature was between 500°C and 600°C as shown in Figure 5.39. It was noticed that the bainite start temperature was influenced more by the peak temperature than by the cooling rate. Welds with a higher peak temperature should have higher start and finish temperatures. From observing the transformation start and finish temperatures, it was noticed that this trend to not occur. This may be due the plastic deformation which occurs in friction stir welds.

<table>
<thead>
<tr>
<th>Weld</th>
<th>Tool Rotation, rpm</th>
<th>Travel Speed, ipm</th>
<th>Location and Peak Temperature</th>
<th>Start</th>
<th>Finish</th>
<th>$T_{8.5}$, °C/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>426</td>
<td>1100</td>
<td>6</td>
<td>AS, 904°C</td>
<td>560</td>
<td>407</td>
<td>10.5</td>
</tr>
<tr>
<td>431</td>
<td>850</td>
<td>8</td>
<td>AS, 988°C</td>
<td>585</td>
<td>383</td>
<td>7.2</td>
</tr>
<tr>
<td>426</td>
<td>1100</td>
<td>6</td>
<td>AS Shoulder, 1095°C</td>
<td>590</td>
<td>466</td>
<td>10.2</td>
</tr>
<tr>
<td>431</td>
<td>850</td>
<td>8</td>
<td>AS Shoulder, 867°C</td>
<td>560</td>
<td>398</td>
<td>7.6</td>
</tr>
<tr>
<td>426</td>
<td>1100</td>
<td>6</td>
<td>Bottom SZ, 905°C</td>
<td>549</td>
<td>389</td>
<td>12</td>
</tr>
<tr>
<td>426</td>
<td>1100</td>
<td>6</td>
<td>RS, 988°C</td>
<td>552</td>
<td>402</td>
<td>10.8</td>
</tr>
<tr>
<td>427</td>
<td>700</td>
<td>6</td>
<td>SZ, 1116°C</td>
<td>602</td>
<td>394</td>
<td>9.7</td>
</tr>
<tr>
<td>430</td>
<td>850</td>
<td>4</td>
<td>SZ, 1180°C</td>
<td>590</td>
<td>449</td>
<td>14.1</td>
</tr>
</tbody>
</table>

Table 5.5: Phase transformation temperatures for the HSLA-65 friction stir welds
Figure 5.39: Bainite transformation temperatures for the various HSLA-65 friction stir welds cooling rates

The phases which form in ferritic steels are dependent on the cooling rate; therefore, continuous-cooling-transformation diagrams are referenced to determine the phases which are favored during cooling. JMatPro v.4.1 software was used to construct a rough estimate of the CCT diagrams for HSLA-65 microstructure with different austenite grain sizes. A rough estimate of how the ferrite and bainite start temperatures shift in a
CCT diagram is shown in Figure 5.40. The CCT diagram shows that when austenite grain size is reduced, the formation of ferrite is promoted. This was observed in the HSLA-65 friction stir welds which experienced a lower peak temperature in the stir zone. The stir zone microstructure of these welds contained more ferrite. Along with a decrease in austenite grain size, when the austenite grains experience plastic deformation the formation of ferrite is promoted. This explains why the stir zone microstructure of HSLA-65 weld 431 contains more ferrite grains than the other welds.

Figure 5.40: Shifts in ferrite and bainite start temperatures in a CCT diagram for HSLA-65 when the austenite grain size is decreased from 50 µm to 25 µm
5.7 Microstructure Evolution

The microstructure observed in the various HSLA-65 friction stir welds SZ consisted of large prior austenite grains. The prior austenite grains were approximately 50 μm in size. The only HSLA-65 FSW which did not possess prior austenite grains this large was the weld conducted with a welding speed of 8 ipm. The prior austenite grains in this weld were approximately 25 μm in size. These prior austenite grains evolved from the high peak temperatures experienced in this region. The peak temperatures observed in the stir zone were above 1100°C, which allowed the SZ microstructure to become austenitized. The prior austenite grains observed in the stir underwent grain growth which is known to occur when recrystallization is complete. The new structure is metastable, which allowed these grains to grow to reduce the grain boundary energy per unit volume (31).

The stir zone microstructure of the HSLA-65 FSW with a travel of 8 ipm (3.39mm/s) contained smaller prior austenite grains than the other HSLA-65 friction stir welds. This may be the result of higher strains experienced in this region due to the high travel speed and lower peak temperature. The higher strains and lower peak temperature in the high travel speed HSLA-65 FSW may retard extensive dynamic recrystallization to occur. This phenomenon where higher strain rates can block the start of dynamic recrystallization has been documented in other research (31).

The stir zone microstructures which experienced higher strains contained a higher ferrite content. This ferrite is the result of a phenomenon known as mechanical stabilization and the lower peak temperatures experienced by the stir zone material. Mechanical stabilization occurs when the material experiences severe plastic deformation and the phase transformation becomes hindered (30). It has been noted by Bhadeshia that displacive transformations occur by the advance of glissile interfaces which can be
rendered sessile when they encounter dislocation debris. Therefore, the serve plastic deformation aids in the decomposition of austenite becoming prolonged.

Mechanical stabilization not only retards the displacive transformation, but promotes the ferrite transformation. This is due to an increased in dislocation density resulting from the working of the material (32). These grains observed in the stir zone of the highly strained HSLA-65 were confirmed to be ferrite by microhardness measurements. The higher cooling rates caused the stir zone material to experience faster cooling rates and smaller prior austenite grain formation, which causes the ferrite to form first in the CCT diagram. The microhardness measurements for the ferrite and bainite/Widmanstatten grains found in the SZ are shown in Figure 5.41.

The transition region of the HSLA-65 friction stir welds experienced a portion of the strain generated in the stir zone. This strain caused the microstructure in the transition region to undergo dynamic recrystallization. Grain growth did not occur, which may be due to lower amount of time this region sustained elevated temperatures. The lack of adiabatic shear bands forming may be due to the phase transformations which take place in HSLA-65 during cooling (12).
Figure 5.41: Microhardness measurements for (a) ferrite and (b) bainite/Widmanstatten ferrite observed in the stir zone
CHAPTER 6

FRICION STIR WELDING TYPE 310 STAINLESS STEEL

6.1 Stainless Steel FSW with Preheat

At first two bead-on-plate friction stir welds without preheat were conducted to determine if acceptable welds could be produced on Type 310 by adjusting the welding parameters. After examining the surface of these two trial welds, it was determined that void defects were present on the advancing side of the welds (see Figure 6.1). It was then decided to use preheat by wrapping the plates to be welded in a ceramic blanket. The void defects which were generated during the Type 310 friction stir welds may be due to the low thermal conductivity of stainless steel. This is the reason for using preheat during the welding process.

Two more trial welds were conducted using the preheat method. During the first trial weld, a preheat of 150°C was used, and for the second trial weld, a preheat of 260°C was used. The resulting welds we deemed acceptable from visual inspection (see Figure 6.2). It was later determined, by radiographic inspection, that these welds contained subsurface void defects on the advancing side. After conducting the trial welds with preheat and obtaining visually acceptable welds, the plates with embedded thermocouples were welded to obtain the thermal-histories for Type 310 stainless steel friction stir welds. The weld parameters along with the amount of preheat used for each of these welds and the trial welds are list in Table 6.1. Each of these welds was visually acceptable as shown in Figure 6.3.
Figure 6.1: Type 310 trial friction stir welds without preheat

Figure 6.2: Type 310 trial friction stir welds with preheat
Figure 6.3: Type 310 friction stir welds with preheat and embedded thermocouples

<table>
<thead>
<tr>
<th>Weld Number</th>
<th>Tool Rotational Speed</th>
<th>Tool Travel Speed</th>
<th>Forging Force</th>
<th>Pre-Heat</th>
</tr>
</thead>
<tbody>
<tr>
<td>432</td>
<td>500 rpm</td>
<td>0.423 mm/s (1 ipm)</td>
<td>15.569 kN (3500 lbf)</td>
<td>N/A</td>
</tr>
<tr>
<td>433</td>
<td>800 rpm</td>
<td>0.423 mm/s (1 ipm)</td>
<td>15.569 kN (3500 lbf)</td>
<td>N/A</td>
</tr>
<tr>
<td>435</td>
<td>500 rpm</td>
<td>0.423 mm/s (1 ipm)</td>
<td>15.569 kN (3500 lbf)</td>
<td>149°C (300°F)</td>
</tr>
<tr>
<td>436</td>
<td>500 rpm</td>
<td>0.423 mm/s (1 ipm)</td>
<td>15.569 kN (3500 lbf)</td>
<td>260°C (500°F)</td>
</tr>
<tr>
<td>437</td>
<td>500 rpm</td>
<td>0.423 mm/s (1 ipm)</td>
<td>15.569 kN (3500 lbf)</td>
<td>260°C (500°F)</td>
</tr>
<tr>
<td>438</td>
<td>500 rpm</td>
<td>0.423 mm/s (1 ipm)</td>
<td>16.458 kN (3700 lbf)</td>
<td>260°C (500°F)</td>
</tr>
</tbody>
</table>

Table 6.1: Type 310 FSW parameters
The image of the surface of the stainless steel FSW numbered 437 shows a surface discontinuity towards the beginning of the weld. It is not believed that this surface discontinuity was a result of the welding process, since it corresponds with the location of an eroding thermocouple. The eroding thermocouple material which was in contact with the FSW tool produced inclusions and voids in the weld. In the weld numbered 438, it is apparent from the exit hole of the weld that a void defect was present on the advancing side of the weld. This defect was determined to be a void because of the channel which extends from the exit hole back into the advancing side of the weld.

The heat affected zones observed from the surface of the welds are much smaller than those observed in the HSLA-65 friction stir welds. In both of these welds, the flash generated during the welding process was confined to the retreating side, and the weld (438) with the larger forging force generated a larger amount of flash. The Type 310 FSW (438) with the larger downward force also generated a wider weld. To determine if any internal weld defects were present, radiography was performed on the welded plates (see Figure 6.4).
As observed from the radiographic images, both of the Type 310 friction stir welds contain void defects. In the image of weld 437, it was noticed that the start of the defect is inline with the first eroding thermocouple and the end of the defect is well before the end of the weld. This defect was observed to be located in the same area as the surface discontinuity, which supports the reasoning that this defect was the result of the eroding thermocouple. The image of weld 438 shows a void defect starting at the point of a wire thermocouple, but this defect extends to the end of the weld. Therefore, it was not believed that the defect was the result of the thermocouple but could be the result of the hole machined for the thermocouple.
As with the HSLA-65 friction stir welds, during Type 310 friction stir welding thermocouples became detached, were sheared, or were not in the correct position. The thermal data which was successfully collected during the friction stir welding is shown in Figure 6.5 and Figure 6.6. The difference in generated heat in the different regions of the friction stir welds can be attributed to the low thermal conductivity of stainless steel. Since the heat generated during the welding process was not easily conducted into the material surrounding the stir zone, lack of bonding (kissing bond) discontinuities could result. These discontinuities would result due to the material in these weld regions not sustaining temperatures in the plasticity regime.

The thermal-histories shown in Figure 6.5, along with an image of their exact location, were acquired from thermocouples located near the bottom of the stir zone boundary. The peak temperature for each thermocouple is listed in Table 6.2. The highest peak temperature recorded was 958°C from a thermocouple located 0.5 mm below the stir zone boundary. The other two thermocouples were located

<table>
<thead>
<tr>
<th>Weld</th>
<th>Pre-Heat</th>
<th>Rotation Rate</th>
<th>Welding Speed</th>
<th>Forging Load</th>
<th>Thermocouple Location</th>
<th>Peak Temperature</th>
<th>Cooling Rate (700-300°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>437 (1)</td>
<td>260°C</td>
<td>500 rpm</td>
<td>1 ipm</td>
<td>3500 lbf</td>
<td>2 mm below probe, 3 mm towards RS from stir zone boundary</td>
<td>795°C</td>
<td>3.6°C/s</td>
</tr>
<tr>
<td>437 (2)</td>
<td>260°C</td>
<td>500 rpm</td>
<td>1 ipm</td>
<td>3500 lbf</td>
<td>2 mm below probe, 2.5 mm towards AS from stir zone boundary</td>
<td>726°C</td>
<td>3.2°C/s</td>
</tr>
<tr>
<td>438 (3)</td>
<td>260°C</td>
<td>500 rpm</td>
<td>1 ipm</td>
<td>3700 lbf</td>
<td>0.5 mm below stir zone boundary</td>
<td>958°C</td>
<td>3.5°C/s</td>
</tr>
</tbody>
</table>

Table 6.2: Peak temperatures and cooling rates acquired from thermocouples located near the bottom of the stir zone boundary in Type 310 friction stir welds
Figure 6.5: Type 310 FSW thermal-histories and thermocouple locations for thermocouples located near the bottom of the stir zone
3 mm from the stir zone boundary on the retreating side of the weld, and 2.5 mm from the stir zone boundary on the advancing side of the weld. The peak temperature recorded from the thermocouple placed on the retreating side of the weld was 795°C and the peak temperature on the advancing side was 726°C. This difference in peak temperatures on the advancing and retreating sides of the weld is consistent with temperature differences listed in literature. It has been noted that the retreating side of an aluminum friction stir weld experiences a slightly higher temperature than the advancing side (7).

The thermal-histories and respective thermocouple locations in Figure 6.6 were acquired from thermocouples located on the advancing and retreating sides of Type 310 friction stir welds. The recorded peak temperatures along with the respective cooling rates for each thermocouple are listed in Table 6.3. On the retreating side of the weld a peak temperature of 1074°C was recorded from a thermocouple 1 mm from the stir zone boundary and 1 mm from the weld surface. The other retreating side thermocouple was located in the stir zone, 3 mm from the probe center line. This thermocouple recorded a peak temperature of 958°C. The thermocouple closer to the shoulder of the tool may have measured a higher peak temperature due to the higher heat produced at the tool shoulder and base metal interface.

The thermocouples located on the advancing side of the weld were 1 mm and 3 mm from the stir zone boundary, their respective peak temperatures were 1027°C and 1021°C. Even though these thermocouples were 2 mm apart, their peak temperatures were similar. This contributes to the idea that a step temperature gradient does not exist from the stir zone extending out into the heat affected zone.
<table>
<thead>
<tr>
<th>Weld</th>
<th>Rotation Rate</th>
<th>Welding Speed</th>
<th>Forging Load</th>
<th>Thermocouple Location</th>
<th>Peak Temperature</th>
<th>Cooling Rate (700-300°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>437 (1)</td>
<td>260°C</td>
<td>500 rpm</td>
<td>1 ipm</td>
<td>Retreating side, 1 mm below weld surface, 1 mm from stir zone boundary</td>
<td>1074°C</td>
<td>3.5°C/s</td>
</tr>
<tr>
<td>438 (2)</td>
<td>260°C</td>
<td>500 rpm</td>
<td>1 ipm</td>
<td>Retreating side, in stir zone, 3 mm from probe center line</td>
<td>958°C</td>
<td>3.8°C/s</td>
</tr>
<tr>
<td>438 (3)</td>
<td>260°C</td>
<td>500 rpm</td>
<td>1 ipm</td>
<td>Advancing side, 2 mm below weld surface, 1 mm from stir zone boundary</td>
<td>1027°C</td>
<td>3.8°C/s</td>
</tr>
<tr>
<td>438 (4)</td>
<td>260°C</td>
<td>500 rpm</td>
<td>1 ipm</td>
<td>Advancing side, 4 mm below weld surface, 3 mm from stir zone boundary</td>
<td>1021°C</td>
<td>3.6°C/s</td>
</tr>
</tbody>
</table>

Table 6.3: Peak temperatures and cooling rates acquired from thermocouples located on the advancing and retreating sides of Type 310 friction stir welds
Figure 6.6: Type 310 thermal-histories and locations of thermocouples positioned on the advancing and retreating sides of the weld
6.2 Stainless Steel FSW Metallurgy

The Type 310 stainless steel base metal was analyzed because stringers were present. The SEM image in Figure 6.7 is the Type 310 base metal. The stringers, which were observed, are the white boundaries passing through multiple austenite grains. It was desired to know the composition of these particles and how they may influence a Type 310 FSW. The particles in Figure 6.8 were analyzed by energy-dispersive X-ray spectroscopy (EDS). The resulting composition of the particle and the surrounding matrix is shown in Figure 6.9; in which it was determined that the particle was a Chromium-rich oxide. The Fe-Cr-Ni-rich matrix surrounding the Cr-rich oxide stringers are the main components of Type 310 stainless steel.

Figure 6.7: Base metal of Type 310 stainless steel with Cr-rich oxide stringers. Etchant: 50 ml HNO₃ + 50 ml H₂O
Figure 6.8: Cr-rich oxide particles found in the Type 310 stainless steel. Etchant: 50 ml HNO$_3$ + 50 ml H$_2$O
From the analysis of the weld surfaces, radiography images, and thermal-histories, it was expected that the resulting microstructure of the Type 310 friction stir welds would be similar to those observed in previous stainless steel friction stir welds. After examining the Type 310 FSW cross-section in Figure 6.10, which also contains an overlay of the welding tool profile, it was determined that a void discontinuity was
present on the advancing side of the weld. The weld shape is visibly different from those observed in the HSLA-65 friction stir welds.

On the advancing side of the weld, there is a distinct boundary between the stir zone and the transition region. There were particles found on this boundary which can be observed in Figure 6.10. They are contained in the white boundary near the top of the weld where the material has been affected by the shoulder of the tool. It was found by EDS analysis that these particles were Si-rich oxides, which resulted from the cement used to secure the welded thermocouples in place. A higher magnification image of this boundary where the particles were observed is shown in Figure 6.11. This contributes to the idea that the void discontinuities found in this weld may have evolved from contact with a pre-machined thermocouple hole. On the retreating side of the weld, this boundary is more diffuse and similar to the macrostructure found in carbon steel friction stir welds.

The weld shape is also distinctly different from the weld shape produced in a Type 304L FSW. The stir zone weld shape of a Type 304L FSW was circular in nature. Whereas, the Type 310 FSW has stir zone weld shape more similar to the shape of the welding tool. The differences can be contributed to the amount of heat generated and the flow of the material which occurs during austenitic stainless steel friction stir welding.
Figure 6.10: Cross-section of a Type 310 friction stir weld. Etchant: 50 ml HNO₃ + 50 ml H₂O

Figure 6.11: Distinct boundary on the advancing side of a Type 310 FSW where Si-rich particles were observed
The advancing side of the weld in Figure 6.12 shows the formation of shear bands near the void observed on the advancing side of the friction stir weld. These shear bands are a characteristic of the TMAZ in friction stir welds, and form in regions of high strains and areas where voids have formed. This image also shows the amount of grain refinement which occurred between the base metal and the stir zone. From the image of the retreating side of the weld in Figure 6.13, it is evident that this side of the weld experienced less deformation based on the orientation of the shear bands. The degree of shear band formation can be attributed to the different amount of shear stress and strain generated on the respective sides of a friction stir weld.

Figure 6.12: Advancing side of a Type 310 FSW. Etchant: 50 ml HNO₃ + 50 ml H₂O
It was desired to determine the amount of tool wear which occurred during the friction stir welding of Type 310 stainless steel. The microstructure near the surface of the weld was analyzed and is shown in Figure 6.14. Particles were found near the surface of the weld, also shown in Figure 6.14, and EDS was performed to determine their composition. The EDS scan in Figure 6.15 shows that these particles were rich in Tungsten. From this, it was concluded that some tool wear occurs during the stainless steel welding, but not enough wear to be significant.
Figure 6.14: (a) Surface of a Type 310 FSW were tungsten inclusions from tool wear was found (b) white particles are tungsten, Etchant: 50 ml HNO₃ + 50 ml H₂O

Figure 6.15: EDS scan of a tungsten particle near the surface of Type 310 FSW
The stir zone microstructure consists of refined austenite grains with deformed grain boundaries (see Figure 6.16). The stir zone austenite grains are about 10-20 microns; whereas, the base metal grains were around 50-70 microns. The distorted grain boundaries can be attributed to material being displaced by the friction stir welding tool. Almost no twin boundaries were observed in the stir zone microstructure, this can be attributed to the thermal and mechanical cycle this material experienced, which caused dynamic recrystallization to occur. Dynamic recrystallization is known to occur in stainless steel friction stir welds, and is evident because the twin boundaries which were present in the base metal are not present in the resulting weld microstructure.

The SEM images in Figure 6.17 and Figure 6.18 illustrate the degree of grain deformation which occurred in the stir zone during the friction stir welding process. The SEM image in Figure 6.19 is the microstructure of Type 310 base metal. The base metal microstructure provides a comparison for the degree of grain boundary distortion which occurred in the stir zone. The grain boundaries of the austenite in the base metal are ordered, whereas the austenite grains in the stir zone have jagged grain boundaries. From these images, the amount of grain refinement which has occurred was noticed. The pits, which were observed in the SEM image of the stir zone, may be the result of carbide formation. The microstructure which formed in the transition region is similar to that found in the stir zone.
Figure 6.16: Stir zone microstructure of a Type 310 FSW. Etchant: 50 ml HNO$_3$ + 50 ml H$_2$O, org. mag. 200x

Figure 6.17: SEM image of the stir zone microstructure from a Type 310 FSW. Etchant: 50 ml HNO$_3$ + 50 ml H$_2$O
Figure 6.18: Austenite grains in the stir zone of a Type 310 FSW. Etchant: 50 ml HNO₃ + 50 ml H₂O

Figure 6.19: SEM image of Type 310 stainless steel base metal. Etchant: 50 ml HNO₃ + 50 ml H₂O
The microstructure of the transition region consists of refined austenite grains, as shown in Figure 6.20. These austenite grains are more refined compared to those observed in the stir zone microstructure. Unlike the stir zone, the material in TR experienced less time at elevated temperatures, therefore hindering substantial grain growth. The refined grains were the result of the mechanical cycle causing recrystallization to occur during the welding process. The grain boundaries in this region of the weld are not as deformed as those observed in the stir zone. The images in Figure 6.21, Figure 6.22, and Figure 6.23 display the amount of grain refinement which occurred in the transition region of the Type 310 FSW. Again, it was noticed that these grains experienced less deformation than those in the stir zone. The EBSD scan in Figure 6.24 shows that the grains in the TR of a Type 310 FSW have a random orientation.

Figure 6.20: Transition region of a Type 310 FSW. Etchant: 50 ml HNO₃ + 50 ml H₂O, org. mag. 200x
Figure 6.21: SEM image of TR microstructure from a Type 310 FSW. Etchant: 50 ml HNO₃ + 50 ml H₂O

Figure 6.22: Austenite grains in the TR of a Type 310 FSW. Etchant: 50 ml HNO₃ + 50 ml H₂O
Figure 6.23: Refined austenite grains found in the TR of a Type 310 FSW. Etchant: 50 ml HNO₃ + 50 ml H₂O

Figure 6.24: EBSD scan of the transition region for a Type 310 FSW
The heat affected zone microstructure was similar to the microstructure of the base metal. The microstructure of the HAZ consisted of large austenite grains with twinning, as shown in Figure 6.25. As expected, grain growth occurred in the HAZ which resulted with grains as large as 100 microns and larger. One of these large austenite grains is shown in Figure 6.26 which is from the HAZ of the Type 310 FSW. The twin boundaries are still present because dynamic recrystallization did not occur since the material in this weld region only experienced a thermal-cycle during the welding process. The EBSD scan in Figure 6.27 was conducted over the HAZ of a Type 310 friction stir weld. There Cr-rich oxide stringers are present in this area of the HAZ and are distinguishable by the black lines which traverse the large austenite grains.

![HAZ microstructure](image)

Figure 6.25: HAZ microstructure of a Type 310 FSW. Etchant: 50 ml HNO₃ + 50 ml H₂O, org. mag. 200x
Figure 6.26: Large austenite grains in the HAZ of a Type 310 FSW. Etchant: 50 ml HNO$_3$ + 50 ml H$_2$O

Figure 6.27: EBSD scan of the HAZ from a Type 310 FSW
A hardness map was constructed of Type 310 friction stir weld to determine if a difference was present between the stir zone and the base metal. By examining Figure 6.28, it was concluded that only a small difference in hardness existed between these two regions. The Type 310 FSW microhardness map in Figure 6.28 contains an overlay of the welding tool profile. The stir zone had a hardness of 300 VHN while the base metal had a hardness of 225 VHN. The difference in hardness is due to the difference in grain size and the material in this region experiencing warm working. The Type 310 base metal had an austenite grain size of 100 µm and the stir zone had an austenite grain size of 25 µm. The smaller grains located in the stir zone and the transition region contributes to a higher hardness. The area in the stir zone where the hardness drops to around 100 VHN is the void defect which formed during welding.

![Hardness map of a Type 310 FSW](image)

Figure 6.28: Hardness map of a Type 310 FSW
6.3 Microstructure Evolution

The large austenite grains observed in the Type 310 base metal contributed to a large strain being produced prior to material flow, because it is known that the larger the grains the more difficult is becomes to generate material flow (33). This large strain aids in the difficulty of friction welding Type 310 stainless steel. The microstructure in the stir zone of the Type 310 friction stir welds did not experience grain growth. The high flow stress generated in the Type 310 friction stir welds causes a large dislocation density to form in the strained material. Along with dislocation generation, cross-slip occurs, which both contribute to work-hardening. The higher strains experienced during the work hardening causes the grains to be deformed or distorted (33).

During the friction stir welding process, a critical strain is achieved, which causes plastic strain energy saturation and recovery and recrystallization. The recovery and recrystallization allows for a large shear to occur through the grains or extensive grain refinement which facilitates large shear strains by grain sliding (33). The large flow stress achieved in the Type 310 friction stir welds were observed during the modified Gleeble torsion tests.

The heat affected zone of the Type 310 friction stir welds underwent grain growth, which was determined by examining the large austenite grains. The HAZ contains austenite grains approximately 125-150 µm in size, while the Type 310 base metal contains austenite grains approximately 100 µm in size. The larger austenite grains in the HAZ have grain boundaries which are convex in shape; while the smaller grains have concave grain boundaries. These grain boundary shapes are characteristics of grain growth.
CHAPTER 7

MODIFIED GLEEBLE HOT TORSION TESTS

7.1 Type 310 FSW Microstructure Simulation

The various microstructural regions of a Type 310 friction stir weld were simulated by the modified Gleeble hot torsion test. The microstructure which resulted from the hot torsion tests matched the microstructure from the friction stir welds in that the grain size which was produced and grain boundary distortion were similar. The torsion test which produced a similar microstructure to that found in the stir zone of a Type 310 FSW was done so with a programmed temperature of 1000°C and a rotational speed of 1150 rpm. This torsion specimen microstructure is compared with the microstructure of the stir zone in Figure 7.1.

Both the FSW and torsion specimen microstructures contain refined austenite grains with distorted grain boundaries. The torsion specimen microstructure is not an exact replica of the FSW stir zone microstructure because the torsion specimens contain twin boundaries. The twin boundaries may have remained in the microstructure of the torsion specimen because the hot torsion test does not displace the material unlike the friction stir welding process. The material in the stir zone of a friction stir weld is displaced by the passing of the tool, which may have caused the twin boundaries to annihilate.
The microstructure of a Type 310 friction stir weld was simulated in a modified Gleeble hot torsion test which was conducted with a programmed temperature of 1100°C and a torsion rate of 550 rpm. The microstructure of the transition region from the FSW is compared with the microstructure produced during the torsion test in Figure 7.2. Both microstructures contain refined austenite grains with a small dispersion of larger grains. The structure which resembles shear bands observed in the micrograph of the torsion specimen microstructure are most likely remnants of the chromium-oxide segregation bands observed in the base metal. These bands become more prevalent if the sample is over-etched, which may have occurred to this torsion specimen.

The hot torsion test which simulated the microstructure of the transition region was conducted at a slightly higher temperature, 1100°C, than the torsion specimen that simulated the microstructure of the stir zone, 1000°C. This is related to the low torsion rate which was used during the torsion test that simulated the transition region. The higher torsional speed used during the torsion test which simulated the stir zone caused

Figure 7.1: (a) Microstructure produced in the stir zone of a Type 310 FSW (b) Microstructure resulting from a Type 310 hot torsion test at 1000°C and 1150 rpm (a, b etchant: 50 ml HNO₃ + 50 ml H₂O, org. mag. 200x)
more adiabatic heating to occur during the test, and in turn causing the temperature of the specimen to reach a temperature higher than the programmed temperature. The lower torsion rate allowed the test specimen to remain closer to the programmed temperature, and the higher testing temperature provided the material to become more plasticized.

The heat affected zone of a Type 310 friction stir weld was simulated in all of the modified Gleeble hot torsion tests. This is due to the HAZ only depending upon the peak temperature reached during the test and the cooling rate from this temperature. Since all the torsion tests were conducted with the same cooling rate and with temperatures equal to or greater than those observed in an actual friction stir weld, all tests resulted with an area similar to the HAZ. The microstructure of the HAZ from a FSW was compared with the simulated microstructure from a torsion test in Figure 7.3. This microstructure resulted from a torsion test which was conducted with a programmed temperature of 1100°C and a torsion rate of 850 rpm.

Figure 7.2: (a) Microstructure produced in the TR of a Type 310 FSW (b) Microstructure resulting from a Type 310 hot torsion test at 1100°C and 550 rpm (a,b etchant: 50 ml HNO₃ + 50 ml H₂O, org. mag. 200x)
The heat affected zone microstructure of the FSW and the simulated microstructure from the hot torsion test contain large austenite grains with twin boundaries. This microstructure is similar to that observed in the base metal, but some of the grains have grown larger due to the thermal cycle. The HAZ and simulated microstructure contain smaller grains located between these large grains.

Figure 7.3: (a) Microstructure produced in the HAZ of a Type 310 FSW (b) Microstructure resulting from a Type 310 hot torsion test at 1100°C and 850 rpm (a,b etchant: 50 ml HNO$_3$ + 50 ml H$_2$O, org. mag. 200x)

7.2 Type 304L FSW Microstructure Simulation

The different metallographic regions of a Type 304L friction stir weld were not simulated as accurately with the modified Gleeble hot torsion test as were to the Type 310 and HSLA-65 torsion tests. This was primarily due to ferrite stringers which were present in the Type 304L base metal, but did not display the same behavior in the torsion tests as in the friction stir welds. The hot torsion test did not work the material in the same manner as a friction stir welding tool passing through the material. The
microstructures are similar in that the austenite grains behaved similarly from both the friction stir welding process and the hot torsion testing.

The microstructure comparisons between the Type 304L friction stir welds and the torsion tests are slightly contrasting because of the etching time used for each specimen. The Type 304L stir zone microstructure in Figure 7.4 was compared with the microstructure from a torsion test which was conducted at a temperature of 1250°C and a torsion rate of 550 rpm. Both microstructures exhibit austenite grains that are around 15-20 microns in size and surrounded by a network of ferrite stringers. The order and size of the ferrite was different between the FSW microstructure and the torsion specimen microstructure.

Figure 7.4: (a) Microstructure produced in the SZ of a Type 304L FSW (b) Microstructure resulting from a Type 304L hot torsion test at 1250°C and 550 rpm (a,b oxalic etch, org. mag. 400x)
The microstructure which was produced in the transition region of the Type 304L friction stir weld was different on the retreating side and the advancing side of the weld. The Type 304L FSW retreating side transition region in Figure 7.5 is compared with the microstructure from a Type 304L torsion test conducted at a temperature of 1250°C and a rotational speed of 850 rpm. Both microstructures contain refined austenite grains with ferrite at the grain boundaries. The ferrite stringers have a different appearance which may be the result of etching parameters.

The microstructure in Figure 7.6 was produced on the advancing side transition region of a Type 304L friction stir weld. This microstructure is noticeably different from the transition region produced on the retreating side of the weld. This may be due to higher shear stresses generated on the advancing side of the weld which could promote the formation of ferrite. There was not a match in microstructure between the advancing side transition region and a torsion specimen. This may be attributed to the different stress mechanisms generated during friction stir welding which cannot be simulated by the hot torsion test.
Figure 7.5: (a) Microstructure produced in the TR on the retreating side of a Type 304L FSW (b) Microstructure resulting from a Type 304L hot torsion test at 1250°C and 850 rpm (a,b oxalic etch, org. mag. 400x)

Figure 7.6: Microstructure produced in the TR on the advancing side of a Type 304L FSW (400x; oxalic etch)
The microstructure which resulted in the HAZ of a Type 304L FSW was similar to that found in the base metal, except the size of the austenite grains were slightly larger. The Type 304L friction stir weld HAZ in Figure 7.7 is compared with the microstructure generated during a Type 304L torsion test at a temperature of 1250°C and a torsion rate of 550 rpm. Both microstructures contain larger austenite grains with ferrite stringers. The sizes of the ferrite stringers were larger in the friction stir weld microstructure than in the torsion test microstructure.

Figure 7.7: (a) Microstructure produced in the HAZ of a Type 304L FSW (b) microstructure resulting from a Type 304L torsion test at 1250°C and 550 rpm (a,b oxalic etch, org. mag. 400x)
7.3 Shear Flow Stress Generated During Hot Torsion Testing

From the torque data collected during the modified Gleeble hot torsion testing, shear flow stress was calculated. The shear flow stress was calculated for the Type 310 and Type 304L torsion test conducted in this study, and for the HSLA-65 torsion test conducted by Sinfield (2). Shear stress-strain curves were created for the hot torsion test specimens, which simulated the stir zone of the friction stir weld. By analyzing these shear stress-strain curves, the shear stress which was generated during the hot torsion tests which simulated the microstructure of the different materials stir zones could be used to get an idea of the amount of shear stress in a friction stir weld stir zone.

The equation to calculate the shear stress was:

\[ \tau = \frac{T \cdot r}{J} \]

The shear stress, \( \tau \), is dependent upon torque (\( T \)), radius of the gauge length (\( r \)), and the polar moment of inertia (\( J \)). The radius which was during the shear stress calculations was the radius to the mid-point of the wall thickness. This radius was use since this is the area in which the simulated microstructure was observed. The polar moment of inertia used was for a hollow round shaft. The polar moment of inertia equation is shown below, where \( r_o \) and \( r_i \) are the outer and inner radii of the specimen gauge.

\[ J = \frac{\pi}{2} (r_o^4 - r_i^4) \]
The strain calculated for the shear stress-strain curves does not correlate to the amount of strain experienced by the resulting microstructure. This strain is the angular displacement of the torsion specimen for a particular point during the single revolution. Since the strain is dependent upon the amount of angular displacement at a particular point during the revolution, all samples have the same calculated shear strain since each specimen only underwent one revolution. If the amount of strain experienced by the resulting microstructure is desirable, then a computational model or compression tests should be conducted. The peaks and valleys which are visible in the shear stress-strain curves were caused by the torque generated by the Gleeble.

The shear stress-strain curves in Figure 7.8 all generated around 120 MPa during the hot torsion tests. These curves were generated from Type 310 hot torsion tests conducted at 1000°C and various strain rates (rotational speeds). The torsion specimen which simulated the stir zone microstructure was conducted with a torsion rate of 1150 rpm. From the shear stress-strain curve, a flow stress of about 120 MPa was generated during this sample’s torsion test. Two of the other torsion tests conducted at this temperature generated the same amount of stress. The hot torsion test with a lower torsion rate was only slightly lower than the higher torsion rates.
Figure 7.8: Shear stress-strain curve for Type 310 hot torsion test which simulated the SZ microstructure

The shear stress-strain curves in Figure 7.9 were calculated from torque data gathered from Type 304L torsion tests conducted at a temperature of 1250°C and various torsion rates. The Type 304L torsion specimen which closely simulated the stir zone microstructure was conducted with a torsion rate of 550 rpm. From the shear stress-strain curves, a shear stress around 35 MPa was generated. The Type 304L torsion specimen which closely simulated the retreating side TR microstructure was conducted with a torsion rate of 850 rpm. A shear stress of 45 MPa was generated during this Type 304L torsion test. The calculated shear stress values for the Type 304L torsion tests are
significantly lower than those for the Type 310 torsion tests, but on the same order of magnitude as the HSLA-65 torsion tests.

The Type 304L and HSLA-65 shear stress values which simulated the stir zone are lower than the amount of shear stress in the Type 310 torsion specimen because the latter was conducted at a lower temperature. The shear stress generated in a Type 310 specimen at a temperature comparable to the Type 304L and HSLA-65 torsion specimen produced a similar amount of shear stress. The shear stress generated for each of the materials hot torsion specimens are listed in Table 7.1.

Figure 7.9: Shear stress-strain curve for a Type 304L hot torsion test which simulated the SZ microstructure
The shear stress-strain curves in Figure 7.10 were calculated from torque data collected during HSLA-65 hot torsion tests conducted at 1275°C and various strain rates. The HSLA-65 torsion specimen which simulated the stir zone was conducted with a torsion rate of 1150 rpm. From the shear stress-strain curve, an average shear flow stress of 35 MPa was generated during the test. All other hot torsion tests conducted at this temperature have about the same shear flow stress. The initial large peak is related to the force which was first generated to allow the material to begin flowing. The force was dependent upon the elevated temperature yield stress of the material. The shear flow stress for HSLA-65 was much lower than those generated in Type 310 stainless steel. Additional shear stress-strain curves for these three materials are provided in appendix C.

Figure 7.10: Shear stress-strain curved from HSLA-65 hot torsion test which simulated the SZ microstructure
<table>
<thead>
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<th>Torsion Speed</th>
<th>Steady State Shear Stress</th>
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<td>40 MPa</td>
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Table 7.1: Shear stress values for each of the materials hot torsion specimens
CHAPTER 8

CONCLUSIONS

Friction Stir Welding of HSLA-65 with Various Welding Parameters

1. HSLA-65 friction stir welds performed with welding parameters which result in a peak temperature around 1100°C or less produces a stir zone microstructure containing a significant portion of ferrite.

2. It was determined that ferrous friction stir welds consist of three distinct microstructural regions: (1) stir zone, (2) transition region, (3) heat affected zone.

3. HSLA-65 friction stir welds conducted with a welding speed of 4 ipm produced a peak temperature of 1180°C in the stir zone.

4. Friction stir welding HSLA-65 with a tool rotational speed greater than 850 rpm or a welding speed less than 6 ipm, produces a weld which experiences a high peak temperature.

5. In general, the stir zone microstructure of an HSLA-65 friction stir weld contains ferrite grains and grains containing a bainite/Widmanstatten ferrite structure.

6. The average hardness resulting in the stir zone of an HSLA-65 friction stir weld is 300 VHN.

7. Friction stir welding HSLA-65 with a welding speed of 8 ipm produced a stir zone microstructure containing 25 μm prior austenite grains. Whereas, the other tested welding conditions produced a stir zone microstructure containing 50 μm prior austenite grains.

Friction Stir Welding Type 310 Stainless Steel

8. A peak temperature of 1074°C was measured from a thermocouple 1 mm from the stir zone boundary on the retreating side of a Type 310 friction stir weld.
9. The Type 310 stainless steel base metal contained austenite grains 100 μm in size, while the stir zone of a Type 310 friction stir weld contained austenite grains 25 μm. The transition region of a Type 310 friction stir weld contained austenite grains 10 μm in size. The heat affected zone of a Type 310 friction stir weld contained austenite grains 150 μm in size.

10. A distinct boundary, possibly a “kissing bond,” was produced on the advancing side of the Type 310 friction stir welds.

11. A peak hardness of 300 VHN was measured in the stir zone of a Type 310 friction stir weld. The Type 310 stainless steel had a hardness of 200 VHN.

Gleeble Hot Torsion Testing

12. The Type 310 hot torsion specimen conducted at 1000°C and 1150 rpm produced a microstructure with similar characteristics as a Type 310 friction stir weld stir zone microstructure.

13. The Type 304L hot torsion specimen conducted at 1250°C and 550 rpm produced a microstructure with a similar austenite grain size as a Type 304L friction stir weld microstructure.

14. The shear stress equation:

\[ \tau = \frac{T \cdot r}{J} \]

Can be used in conjunction with the generated torque data from the Gleeble hot torsion test to provide a rough estimate of the shear stress experienced by the hot torsion test specimen.

15. The Type 310 hot torsion specimen which produced a microstructure similar to the Type 310 friction stir weld stir zone microstructure experienced a shear stress of 120 MPa.
16. The Type 304L hot torsion specimen which produced a microstructure similar to the Type 304L friction stir weld stir zone microstructure experienced a shear stress of 35 MPa.

17. The HSLA-65 hot torsion specimen which produced a microstructure similar to the HSLA-65 friction stir weld stir zone microstructure experienced a shear stress of 40 MPa.
CHAPTER 9

SUGGESTIONS FOR FUTURE WORK

Based on the success of using preheat before friction stir welding Type 310 stainless steel, this preheating method should be tested with other difficult FSW materials. This preheating method needs to be refined to determine the minimum amount of heating required before welding takes place. If the Type 310 friction stir welds in this study would have been preheated with a higher temperature, acceptable welds may have been produced. Also, the method of preheating should be modified to better accommodate the friction stir welding process.

During this study, the Type 310 plates were preheated using a ceramic blanket, which required transportation of the plates from the area of heating to the FSW machine. Then the plate had to be clamped down before the welding could proceed. This caused much of input heat to be lost. A preheating method where the plate is already under the clamping force should be developed. This would decrease the amount of preheating time and the temperature used during preheating.

The modified Gleeble torsion test specimen should be refined to produce more accurate torque data. The more accurate torque data will provide a better estimation of shear flow stress occurring in a friction stir weld. The current torsion specimen design has too long of a gauge length. The long gauge length causes buckling to occur during some of the torsion testing; therefore, producing unreliable torque data. A shorter gauge length could solve this problem.
The long gauge length also contributes to the temperature and strain gradient produced in the specimen. If the gauge length is shortened, this would allow more accurate temperature and strain data to be collected during the torsion test. Currently, to calculate accurate strain and strain rate values produced during the torsion testing, a computer simulation model of the test has to be created. A simulation model of the torsion testing conducted during this study should be created to provide accurate strain data.
References


Appendix A

Engineering Drawings of Parts
Figure A.1: Engineering drawing of thermocouple plate design utilizing all Type-K wire thermocouples.

- All Dimensions are inches
- Not to scale
- All views are referenced to the top view (i.e. left side is left side of top view)
- All grooves same width and depth
- All holes same diameter

Drawing Name: HSLA-65 Plate with all welded thermocouples
Figure A.2: Engineering drawing of thermocouple plate design utilizing Type-K wire and eroding thermocouples.
Figure A.3: Modified Gleeble hot torsion test specimen
Appendix B

Design of experiments run order for modified Gleeble hot torsion tests
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Table B.1: Type 310 stainless steel DOE for torsion
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Table B.2: Type 304L stainless steel DOE for torsion
Appendix C

Shear Stress-Strain Curves Generated From Modified Gleeble Hot Torsion Tests
Figure C.1: Shear stress-strain curve for Type 310 at various strain rates and 1100°C

Figure C.2: Shear stress-strain curve for Type 310 at various strain rates and 1200°C
Figure C.3: Shear stress-strain curve for Type 304L at various strain rates and 975°C

Figure C.4: Shear stress-strain curve for Type 304L at various strain rates and 1075°C
Figure C.5: Shear stress-strain curve for HSLA-65 at various strain rates and 975°C

Figure C.6: Shear stress-strain curve for HSLA-65 at various strain rates and 1125°C