Advanced Characterization of Solid-State Dissimilar Material Joints

THESIS

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Abstract

Dissimilar materials are commonly used in various industries to create efficient joints that can maintain functionality and cut cost or weight. The energy industry commonly combines cost-effective steel with corrosion resistant alloys in particularly demanding service environments. The transportation industry has increasingly pushed for light-weighting vehicles by means of introducing aluminum-alloys into the steel-dominated industry using dissimilar joints. However, these material combinations come with many weldability issues, including embrittlement, solidification cracking, or intermetallic compounds formation. Solid-state welds have been developed to overcome such issues, but some challenges have persisted. This study examines friction stir welds (FSW) between Ni-base alloy to carbon steel and aluminum alloy to carbon steel as well as an impact vaporized foil actuator weld (VFAW) between aluminum alloy and a dual phase carbon steel. Electron microscope techniques such as transmission Kikuchi diffraction (TKD), a microscopy method akin to electron backscatter diffraction (EBSD), and energy dispersive X-ray spectroscopy (XEDS) are used to characterize weld interfaces. Regions within micrometers of the weld interface are characterized; Al-steel welds are examined for the presence of intermetallic compounds. The matrix and precipitates are identified in the Ni-steel FSW; intermetallic compounds are
confirmed in the VFAW weld but not in the Al-steel FSW. Texture analysis is also conducted, and the data correspond with reports of FSW and simple shear textures of similar alloys. Additionally, nanoindentation of join interfaces also corroborates current studies of strengthening mechanisms of the solid-state weld methods.
To my mom, for her never-ending support and advice.
To my dad, for inspiring me to pursue engineering.
I would like to express my sincere gratitude to my advisor Antonio J. Ramirez for his guidance on my work and for pushing me into countless opportunities to showcase my work and abilities. His support has been a driving factor in understanding the passion and talent I have for teaching and presenting my work.

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<th>Full Form</th>
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<tbody>
<tr>
<td>BCC</td>
<td>body-centered cubic</td>
</tr>
<tr>
<td>BCT</td>
<td>body-centered tetragonal</td>
</tr>
<tr>
<td>BSE</td>
<td>backscatter electron</td>
</tr>
<tr>
<td>CI</td>
<td>confidence interval</td>
</tr>
<tr>
<td>EBSD</td>
<td>electron backscatter diffraction</td>
</tr>
<tr>
<td>EMRF</td>
<td>electron microscope reference frame</td>
</tr>
<tr>
<td>FCC</td>
<td>face-centered cubic</td>
</tr>
<tr>
<td>FIB</td>
<td>focused ion beam</td>
</tr>
<tr>
<td>FSP</td>
<td>friction stir processing</td>
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<tr>
<td>FSW</td>
<td>friction stir welding</td>
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<tr>
<td>FZ</td>
<td>fusion zone</td>
</tr>
<tr>
<td>HAZ</td>
<td>heat-affected zone</td>
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<tr>
<td>IMC</td>
<td>intermetallic compound</td>
</tr>
<tr>
<td>IPF</td>
<td>inverse pole figure</td>
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<tr>
<td>IQ</td>
<td>image quality</td>
</tr>
<tr>
<td>LSDF</td>
<td>local shear deformation frame</td>
</tr>
<tr>
<td>PCBN</td>
<td>polycrystalline cubic boron nitride</td>
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<tr>
<td>PF</td>
<td>pole figure</td>
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<tr>
<td>SAD</td>
<td>selected area diffraction</td>
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<tr>
<td>SD</td>
<td>shear direction</td>
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<tr>
<td>SE</td>
<td>secondary electron</td>
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<td>SEM</td>
<td>scanning electron microscopy</td>
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<tr>
<td>Abbreviation</td>
<td>Full Form</td>
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<tr>
<td>SFE</td>
<td>stacking fault energy</td>
</tr>
<tr>
<td>SPN</td>
<td>shear plane normal</td>
</tr>
<tr>
<td>SRF</td>
<td>specimen reference frame (or sample reference frame)</td>
</tr>
<tr>
<td>SZ</td>
<td>stir zone</td>
</tr>
<tr>
<td>TEM</td>
<td>transmission electron microscopy</td>
</tr>
<tr>
<td>TKD</td>
<td>transmission Kikuchi diffraction</td>
</tr>
<tr>
<td>TKP</td>
<td>transmission Kikuchi pattern</td>
</tr>
<tr>
<td>TMAZ</td>
<td>thermo-mechanically affected zone</td>
</tr>
<tr>
<td>VFAW</td>
<td>vapor foil actuator welding</td>
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<tr>
<td>XEDS</td>
<td>energy-dispersive x-ray spectroscopy</td>
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Chapter 1: Introduction and Motivation

1.1 Motivation

Dissimilar metal welds can provide necessary physical and microstructural properties at different points of a product without the use of an additional fastener or chemical adhesive. These welds can produce joints where additional material or weight is eliminated but functional efficiency is maintained. Dissimilar joints have been utilized in gas pipelines where high-strength, low-cost steels are clad with Ni-base alloys to provide corrosion resistance where necessary. This combination allows for cost-efficiency, as Ni-base alloys are more expensive, while maintaining functionality, since carbon steels do not perform well in a corrosive environment. However, welds between dissimilar materials can often cause many issues, particularly at the region of composition mixing and diffusion. Intermetallic compounds, embrittlement, and diffusion of elements can cause poor physical properties of a weld joint. A solution to overcome or mitigate such challenges is to minimize the effect of the fusion weld thermal cycle by utilizing a solid-state weld method. These weld techniques commonly circumvent many weldability issues inherent to fusion welding, yet can create another unique set of problems often difficult to detect with current standards. Thus understanding the microstructure is necessary for these solid-state dissimilar metal welds, especially at high-spatial
resolutions due to the refined microstructure specific to these weld processes. This can provide insight into understanding how to eliminate or mitigate issues that occur through these material combinations and weld processes.

1.2 Introduction

Dissimilar metals welds have been used more often in industry, especially recently. The oil and gas industry has commonly used Ni-clad steel for pipe materials, and the transportation industry is increasingly pushing for lightweighting by way of joining aluminum to carbon steel. These material combinations, however, often cause many weldability issues due to the mismatch of physical properties exacerbated by the severe thermal temperatures of the weld process. Additionally, microstructural issues may ensue, such as the development of deleterious intermetallic compounds, diffusion and embrittlement, or unintended microstructural phase changes. A common solution is to utilize a solid-state joint for dissimilar material welds. This method creates a metallurgical bond without exceeding melting temperatures of the base material, circumventing many weldability issues such as solidification cracking, porosity, or embrittlement. It commonly provides a higher quality joint than most fusion welds, though they create a unique set of weldability and joint issues. Understanding the weld method and resultant weld microstructures will allow for the development of methods to eliminate such issues.

Weld techniques that are studied in this report are friction stir welding (FSW) and vapor foil actuator welding (VFAW). Both are solid-state welds that are
commonly used to join dissimilar metal welds. FSW joins butt and lap joints by plunging a rotating pin into material to induce plastic deformation and heating. VFAW creates a weld by impacting a flyer sheet onto a substrate material at high velocities. This method creates welds within microseconds and exhibits a small surrounding heat and deformation affected zone. These weld methods cause material and metallurgical reactions during the elevated thermal history that may be different than what is observed in a fusion weld. Analysis of the weld metallurgy is necessary to adequately understand the microstructural changes and weld process effects.

Investigation of these joints by various high resolution techniques is discussed in this report. Crystallographic analysis by transmission Kikuchi diffraction (TKD) and chemical analysis using energy dispersive X-ray spectroscopy (XEDS) is conducted in a scanning electron microscope (SEM) to examine regions micrometers away from the weld interfaces. Spatial resolutions down to 10 nm are necessary for solid-state welds because of the nature of the process that creates highly refined microstructures. The chemical and crystallographic information can be used to characterize these weld interfaces in the pursuit to create mechanically sound and defect-free welds between dissimilar material welds.

1.3 Objective

This study examines the localized interface of dissimilar material joints using electron microscopy techniques. Specifically, friction stir welds of carbon steel to Ni-
base alloys, carbon steel to aluminum alloys, and an impact weld between aluminum alloys to dual phase steel. TKD, XEDS, and nanoindentation are among the analyses that are utilized for these weld regions. The nature of nucleation and growth is explored through the morphology and orientation of the grains at the weld interface of each sample. Characterization of the FSW between Ni-base alloy to carbon steel is conducted, including SEM texture analysis and nanoindentation. In both welds between Al-alloy to steel, intermetallic compound formation is examined and ascertained using similar methods of analysis. Precipitates, grain size and morphology, crystal orientation and texture are analyzed and discussed through electron microscopy techniques as well as other supplementary analysis methods.
Chapter 2: Literature Review

2.1 Dissimilar Metal Welds

Dissimilar materials are often used in conjunction, and there is a variety of methods to join them, including mechanical fasteners, adhesives, and welding. Where fasteners and adhesives add a third component to affix two parts, welding joins two base parts and creates one seamless piece, typically by way of melting both pieces and re-solidifying as one piece. This can mitigate corrosion and fatigue more than the former methods, though it presents its own unique issues for creating a successful bond. All fusion welds, one that experiences melting of both base materials, will exhibit common regions within the weld, as shown in Figure 1.

![Figure 1. Regions of a Fusion Weld [1] © AWS](image)

There exists a fusion zone where the base material and possible filler metal fully melts and solidifies [2], and a heat-affected zone of the base material where the thermal cycle of the weld process can affect the metallurgical and mechanical
properties of the pre-welded base material. This often includes allotropic phase changes, recrystallization or annealing, and precipitation evolution. The fusion zone can be further divided into regions depending on the presence and type of filler metal used in the weld process as seen below in Figure 2.

![Diagram showing regions of a heterogeneous fusion weld](image)

*Figure 2. Modern Diagram Showing Regions of a Heterogeneous Fusion Weld*

If a heterogeneous filler metal, one dissimilar to the base materials, is applied during the weld process, there may be a transition region and an unmixed zone. The transition region will show mixing and diffusion between the filler and the base materials, and the unmixed zone will have fully melted base material that has not mixed with the filler material. In the heat-affected zone, a partially melted zone describes the base metal that is not fully melted adjacent to the fusion zone boundary.

In dissimilar metal welds, there will be some amount of diffusion between the base materials and a potential filler material. Figure 3 shows an example of a heterogeneous fusion butt weld joint configuration. Mixing in a dissimilar metal weld is inevitable, especially in fusion welds, where the high thermal cycles can
exacerbate the diffusion across the interface. Often, these effects are unaccounted for as the diffusion and inclusion of certain elements in the original base material matrix are not intended. This can cause an array of metallurgical issues, detailed in the following sections.

![Diagram of Dilution in a Butt Joint Configuration](image)

*Figure 3. Diagram of Dilution in a Butt Joint Configuration*

Aside from compositional mixing, dissimilar metal welds are susceptible to many issues due to a mismatch of material properties, enhanced by the elevated temperatures of the weld process. Disparities between thermal conductivities, melting points, thermal expansions, among other physical properties causes a multitude of mechanical problems. These range from intense residual stresses, uneven melting of base materials, and voids or porosity to severe differences in thermal contraction. Various weldability issues arise from different material combinations, though alloy selection and post-weld processing considerations may combat these issues.

### 2.1.1 Ni-base Alloys

Ni-based alloys exhibit high corrosion resistance, moderate to high strengths, and are used in high temperature service conditions up to 900°C. These alloys are
also expensive and are commonly considered high performance materials. They are often used for pressure vessels and pipes in marine environments for their high temperature service capabilities and corrosion resistance. The alloy composition has a significant nickel makeup, and alloying additions include chromium, iron, molybdenum, niobium, and titanium. The crystal lattice structure of Ni-base alloys is face-centered cubic, FCC, and austenitic. FCC structures have a closer packing factor of atoms than base-centered cubic (BCC) as seen in Figure 4 below, and can affect material properties in several ways. Compared to carbon steels which exhibit BCC crystal structure, Ni-base alloys have lower diffusivity of impurity elements, particularly at elevated temperatures.

\[\text{Figure 4. Crystal lattice structure of (a) BCC and (b) FCC. Adapted from Callister 6e [3].}\]

The number of slip planes in both of these crystal structures is relatively high when compared to hexagonally packed structures. They are generally ductile materials since dislocations can move freely in these structures. There are methods to strengthen alloys, namely Ni-base alloys that impede the movement of these
dislocations. The primary methods that utilize alloying elements are solid-solution strengthening and precipitation-hardening.

Understanding the properties of the two strengthening mechanism and how it affects the alloy during processing and heat treatments can determine the application and use of these two types of Ni-base alloys. This study incorporates the use of Ni-base alloy 625, a solid-solution strengthened alloy, so precipitation-hardened Ni-base alloys will not be discussed in depth.

Solid-solution strengthened Ni-base alloys contain alloying elements that impede dislocation as a substitutional or interstitial atom defect, commonly cobalt, chromium, molybdenum, iron, and tantalum. In Figure 5 below, both substitutional and interstitial elements can be seen in the lattice matrix. These help to impede the movement of dislocations and defects by adding strain to the crystal lattice. These alloys are typically chosen when moderate strength and excellent corrosion resistance are desired. These alloys also perform at high service temperatures, up to 800°C or in some cases, 1200°C.

![Figure 5. Substitutional and interstitial atom defects [4]. Left and middle show substitutional defects, right, interstitial solid solution.](image-url)
However, these solid-solution strengthened alloys often contain precipitates within the matrix. The precipitates present will depend on the composition as well as the processing of the alloy, and can often strengthen the alloy further. Table 9 in Appendix A shows possible precipitates and phases in Ni-base alloy 625.

2.1.2 Steel to Ni-base Alloy Welds

The oil and gas industry commonly combines Ni-base alloy and carbon steels in dissimilar welds for cost-reduction and superior corrosion resistant material performance [5]. In welds of this material combination, there are many instances of carbon diffusion from the steel into the Ni-base alloy [6]. Carbon diffuses from the carbon steel and towards the Ni-base alloy FCC matrix. This results in a softened, carbon deficit region in the steel close to the interface. Because the diffusivity of carbon in FCC crystal structure is much lower than that in a BCC matrix, the carbon tends to pile up in the Ni-base alloy near the interface, increasing hardness, specifically during a post-weld heat treatment or subsequent weld passes. With enough carbon buildup during subsequent thermal cycles, martensite can form in the Ni-base alloy located adjacent to the softened steel region at the interface. This can be detrimental to joints, as the juxtaposition of the two regions may cause premature failure. This embrittlement phenomenon has been documented in many studies and even during industrial applications of subsea piping and oil extraction [7].
However, utilizing solid-state weld methods that avoid exceeding melting temperatures may mitigate excessive diffusion. Studies of solid-state welds between Ni-base alloys and steels are limited, although some have reported welds made with limited carbon diffusion and no martensite formation at the interface [8]. The lower thermal cycles of the solid-state weld process eliminates the need for a post-weld heat treatment and precludes the formation of martensite by limiting carbon diffusion.

2.1.4 Aluminum Alloys

Aluminum alloys are lightweight and typically more ductile than both steel and Ni-base alloys. These alloys are made primarily of aluminum and are commonly alloyed with copper, silicon, magnesium, manganese, zinc, among other elements. Alloys are categorized by primary alloying elements, and this study focuses on 6XXX series alloys, alloyed with silicon and magnesium. Aluminum alloys are generally weaker, but less dense and have greater corrosion resistance than most carbon steels. They are frequently used in the transportation industry, namely aerospace and increasingly in automotive, as well as packaging. Due to its formability, low cost, and moderate corrosion resistance, aluminum alloys are ideal for food packaging [9].

Aluminum alloys, like Ni-base alloys, solidify in an austenitic FCC structure matrix. They are strengthened by mechanisms similar to those discussed for the Ni-base alloys.
Strain hardening, or cold working, is a common strengthening mechanism in aluminum alloys. Strain hardening entails plastically deforming an alloy and introducing dislocations and defects that inhibit dislocation movement and increase yield strengths. With application of high temperature for extended times, these dislocations can undergo recovery and recrystallization which eliminates the hardening imparted by cold working. This is typically seen in most fusion weld HAZs from the high heat input of the process. Some solid-state techniques such as friction stir welding or impact welding can join materials without experiencing a loss in joint efficiency and may even enhance the strain hardening. These welds do not achieve temperatures causing melting and re-solidification, and the force required for these welds refines grain structures and commonly improves material tensile strengths due to the refined microstructure [10].

Aluminum alloys are also solid-solution strengthened in a similar manner as Ni-based alloys, explained in section 2.1.1 Ni-base Alloys. However, 6XXX series alloys employ magnesium and silicon as primary alloying elements, and these attribute primarily to precipitation-hardening.

Precipitation-hardening or age hardening in aluminum alloys can produce alloys of excellent yield strengths. In 6XXX series alloys, the precipitate that forms is Mg$_2$Si, visible in the phase diagram in Figure 6 below. However, this precipitate evolves through stages that have varying hardening effects, shown by Figure 7 – the peak hardness is usually the desired aging condition and further aging can cause over-aging, characterized by particle coarsening and a loss in strength. The silicon and magnesium elements in solution gather into embryo clusters [11] to Guinier-
Preston II $\beta''$ zones presenting as needle-shaped precipitates to metastable transition or intermediate $\beta'$ phase and finally to stable $\text{Mg}_2\text{Si}$ precipitates [12]. If precipitates reach the final stage of evolution, they are overaged, as peak hardness is typically observed in the metastable $\beta'$ precipitates.

![Figure 6. Phase diagram with stability curves of 6XXX series Al-Si-Mg alloy [12]](image)

![Figure 7. Precipitate evolution in 6XXX series Al-Si-Mg alloy [13]](image)
During welding of such alloys, evolution of precipitates occurs in the weld fusion zone as well as the HAZ. In the fusion zone, precipitates and elements are melted and solidified in solid solution. In the HAZ, precipitates adjacent to the composite zone will experience reversion and dissolve into the surrounding matrix, reducing the strength if the base metal was at peak aged condition. Further from the weld region, precipitates may overage, and cause a loss in strength. Joint efficiency in aluminum alloys can be a significant problem if not properly controlled. Using solid-state weld techniques that typically utilize lower heat inputs, there may be precipitate evolution to a lesser degree, where reversion and dissolution of precipitates may be avoided, through studies by Ahmed, Threadgill, Bush, and others [10, 14, 15, 16, 17]. There is however, a reduction in strength where smaller precipitates are dissolved and larger precipitates are coarsened in the HAZ, usually where peak temperatures reach approximately 350°C [17].

2.1.5 Steel to Aluminum Alloy Welds

The demand for Al-alloys and steel joints are increasing as the steel-dominated transportation industry is competing for lighter, more efficient vehicles. Due to its comparable formability, low cost, and low density of aluminum alloys, its popularity in application in automotive industry has risen significantly. However, joining these two materials presents a formidable metallurgical challenge: intermetallic compound formation.
Like Ni-steel welded joints, interdiffusion commonly presents metallurgical issue during the process. Intermetallic compounds (IMC) between the aluminum and the iron can form in the aluminum iron solution of the weld [18]. The phases and intermetallics can be seen in the Aluminum-Iron phase diagram in Figure 8, tabulated in Table 11 in Appendix A. These compounds have been identified by the phase diagram and further determined by work done on intermetallic compounds by Chumak, Corby, Black, and Villars. [19] [20] [21] [22] [23] [24] [25]. Some of the intermetallics that may form are stoichiometric, or have a fixed chemical composition, while others, non-stoichiometric compounds, may exist with a range of compositions. These IMCs are hard and brittle, and are generally deleterious to an alloy. They are not desired in most joints or welded structures, and can cause premature and catastrophic failures.
2.2 Solid-State Welding

Fusion welds commonly present problems resulting from the thermal cycle processing, especially for dissimilar metal welds as mentioned above. Solid-state welds can maintain peak temperatures under material melting temperatures and thus circumvent many weldability issues, including solidification cracking, porosity, and embrittlement caused by element diffusion and deleterious phase formation in
the weld regions. These weld processes utilize forging, plastic deformation or elevated temperature processing of solid metal to induce material flow and metallurgical bonding. In practice, softer metals with low melting temperatures have been more promising to join this way due to ease of plastic deformation and ability to reach elevated temperatures that allow for solid-state bonding. Additionally, the energies required for creating these welds with higher yield strengths are considerably higher, and also result in higher tool wear when applicable. Thus, low-temperature melting, soft materials such as aluminum and magnesium are more commonly joined in this manner.

Studies in SS welding of nickel alloys, show stir zone microstructure to contain a face-centered cubic (FCC) austenitic nickel matrix and various precipitates. Segregation and re-precipitation occurs in fast, low thermal cycle SS welds like linear friction welding [26] [27], while distribution of precipitates is more commonly observed in slower SS joining methods like Friction Stir Welding (FSW) [28] [29]. In both studies, intense recrystallization occurred throughout the weld process due to the low stacking fault energy of the FCC crystal lattice. Peak temperatures of FSW within these alloys reach approximately 800°C, and in solid-solution strengthened nickel alloys, MC carbides such as niobium and titanium carbides are dispersed within the stir zone. Ni$_3$Nb ($\gamma''$) and M$_6$C carbides are observed in the stir zone of Ni-base alloy 625 when post-processing is applied after FSW about 20-80 nm large [29].

In steels, SS welding can provide many advantages with respect to metallurgical structure and thermal processing. Some steels can undergo phase
transformations to brittle martensite if cooled too fast from the austenitization temperature, usually around 700-800°C. Carbon in steels increases the formation of γ-austenite phase at elevated temperatures and swift cooling prevents diffusion of the alloying element throughout the material, causing diffusionless, “displacive” or “military” transformation of the crystal lattice to the brittle martensite often seen in stir zones of higher carbon steels [30, 31, 32]. Since SS weld methods produce much lower peak temperatures, martensite in these materials may be prevented, and post-weld heat treatments of such joints can be bypassed. Current literature on SS welds of carbon steels show improved mechanical properties at weld areas when compared to base metal in both mild carbon steels and high-carbon steels [33] [34].

In dissimilar bonds, welds in the solid state will decrease the opportunity for mixing and diffusion comparatively due to the lack of melting and liquid mixing between the two base materials. Dissimilar SS bonds between Ni-based alloys and steels should exhibit much less embrittlement compared to their fusion welded counterparts, because of the reduced susceptibility to martensitic phase transformation [8].

### 2.2.1 Friction Stir Welding

Friction Stir Welding is a solid-state welding method developed by TWI in 1991 [16] that has been deployed in various manufacturing industries today. Used heavily in the aerospace industry, it is now being implemented in automotive production to join dissimilar welds between aluminum alloys and steels for subframes, seatbacks, and body paneling [35, 36, 37]. FSW can produce high quality
joints that do not require any post processing, but also imparts heavy tool wear and necessitates frequent tool changes when welding harder alloys. FSW often results in greater thermomechanical properties since temperatures are kept below melting – melting and re-solidification is avoided, and the thermomechanical processing refines grain size and increases toughness [27] [28].

FSW is used to create a metallurgical bond between two materials, usually in a butt or lap joint configuration, exemplified in Figure 9.

![Diagram of friction stir weld regions](image)

**Figure 9. Diagram of friction stir weld regions [38]**

A threaded pin rotates and plunges into the solid joint interface, generating frictional and deformational heating through deformation, extrusion and forging. A shoulder piece is attached to the pin and rotates on the surface of the plate and prevents material from being extruded out of the plate. Base plates are clamped to ensure quality joint fit up in the presence of the intense stresses produced in the process. The cross section of the weld is similar to that of a fusion weld, where a stir
zone or weld nugget is akin to the fusion zone, and a heat affected zone exists in both. The thermo-mechanically affected zone is the area where material flow has been induced to form the metallurgical bond between base metals. Because the tool is rotating in one direction, the resulting weld nugget, or “stir zone,” of the thermo-mechanically affected is asymmetric, described by an advancing and retreating side. The advancing side undergoes more shearing and mixing as the pin rotates in the direction opposing weld travel direction. The retreating side rotates in the same direction as weld travel, and consequently sees a lesser degree of grain refinement and material mixing.

Although this process may circumvent many physical and material weldability issues related to fusion weld methods, there is a unique set of defects that may present when making FSW. Wormholes, or voids in the thermo-mechanically affected zone, lack of penetration, lack of fusion may be formed where there exists some incomplete bonding or consolidation of the base materials. Additionally, with incorrect process parameters there may also be aesthetic defects such as excess flash, indentation or root flow. These are common defects that may be avoided with careful optimization of weld parameters such as tool RPM, weld travel speed, pin tool offset, and axial, or downward, force of the pin and shoulder tool. Many individuals have conducted optimization of FSW on various materials and joint configurations in order to produce welds that are mechanically and metallurgically sound which require no post-weld processing [39, 40, 41].

J. Rodriguez and A. J. Ramirez studied the optimization of FSW between Ni-based alloy 625 and ASTM A516 Steel [39]. Optimal welds were made using a W-
Re/PCBN (Tungsten-Rhenium Polycrystalline Cubic Boron Nitride) composite tool. The tool had a shoulder diameter of 26.0 mm and pin length of 6.1 mm and diameters of 3.9 and 10.0 mm at the smallest and largest widths of pin threading. They found the optimal weld speed of 100 mm/min, rotational speed of 300 rev/min, and a tool offset of 1.63 mm at 30 kN of downward force.

Studies on FSW of Ni-based alloys or joints of Ni-base alloys to steels are less common due to the relative difficulty of creating such welds – greater thermal cycles, forces and tool change frequencies are required. For FSW of Ni-base alloy and A516, previous studies have been conducted to evaluate microstructure throughout the weld, including the stir zone, TMAZ, and HAZ.

A second study by J. Rodriguez shows that peak temperatures within the stir zone exceed 1100 °C. Recovery mechanisms common to low carbon steels in the austenitic temperature region likely experience discontinuous dynamic recrystallation because of the low stacking fault energy of the alloy and the predicted thermomechanical history during the weld process. Coarse allotriomorphic and Widmanstätten ferrite were observed in the A516 stir zone, shown below in Figure 10 [42]. In the base material and the HAZ, a similar microstructure seen in HAZ of fusion welds of steels is observed: ferrite and pearlite colonies are present with evidence of heating up to the $A_1$ and $A_3$ temperatures.
Carbides in the Ni-625 base material were identified as MC and M$_{23}$C$_7$ within the austenite matrix. M$_{23}$C$_7$ was observed only at the grain boundaries. The base material contained twins caused by annealing and an average grain size of 12 µm. The MC precipitates were characterized as (Ti, Nb)C and NbC. Rodriguez states that the MC and M$_{23}$C$_7$ carbides are also found in the regions of the FSW, including the stir zone and interface, fractured and dragged from the SZ and base material.

Song and Nakata similarly studied FSW of Inconel 625 Alloy [40]. Grain refinement and improved mechanical properties by over 20% were observed during mechanical testing. MC precipitates (Nb and Nb/Ti rich) shown in Figure 11 were observed in both the base material and stir zone. Grain sizes were reduced from 10.3 µm in the base material down to 2.1µm on average in the stir zone.

*Figure 10. Microstructure of A516 stir zone in a FSW between A516 and Ni625: allotriomorphic and Widmanstätten ferrite can be observed. [42]*
Knowledge on FSW for aluminum and steel is much more prevalent. Aluminum has been an ideal material for this weld process given its low yield strength and melt temperature. Moreover, the drive for light-weighting in the transportation industry has fueled the pursuit of sound, intermetallic-free Al-steel joints to incorporate the low density alloy in a steel dominated industry. Because the formation of intermetallic compounds requires temperatures near melting, FSW and other solid-state processes have been studied to create intermetallic-free Al-steel weldments.

E.A.T. Lopez and A.J. Ramirez conducted studies on FSW between aluminum alloy 6063-T5 and AISI1020 steel to find weld parameters for a defect-free and
intermetallic compound-free joint. Equiaxed micrograins ranging between 0.5 and 2.0 µm are observed in the stir zone, caused by growth of the $\alpha_{Al}$ grains by the thermal cycles experienced. There is also deformation and severe rotation of grains in the thermomechanically affected zone as well as recovery and dynamic recrystallization in the weld stir zone. Mg$_2$Si precipitates, observed in the base material, are not observed in and near to the stir zone of the welded joint, suggesting that the thermomechanical effects of the weld process cause dissolution of such precipitates. Within the steel region of these FSW joints, grains are observed to experience no ferrite growth because of the very low thermal cycle of the FSW processing. Grain sizes in the steel stir zone are observed to be approximately 2.0 µm large.

Sato et al. also conducted studies of FSW on Al-6063 and observed microstructural changes after welding. The recrystallized grain size of the weld increased exponentially with increasing maximum temperature. TEM analysis allowed observation of a distribution of the strengthening precipitates intragranularly and a precipitation-free zone near the grain boundaries in all the welds.

Studies have investigated FSW joints of the same process and material alloy combination more comprehensively, but have limited datasets at nanometer resolutions. Optical and electron microscopy throughout the SZ, TMAZ, HAZ and BM have been conducted for preliminary microstructural characterization [42, 43, 44, 45]. Using advanced microscopy such as TKD in the SEM, phases and precipitates
within larger areas at high magnifications immediately adjacent to solid-state dissimilar material welds can be revealed.

2.2.2 Vaporized Foil Actuator Welding

Vaporized foil actuator welding (VFAW) is an impact weld method that joins solid metal alloys by a high-velocity collision of two plates [44]. A weld is created when a flyer plate is launched towards a substrate by an explosion, and a metallurgical bond is created between the flyer and substrate, illustrated in the diagram in Figure 12. Aluminum foil is vaporized as a capacitor bank discharges to launch the flyer plate. There is minimal heat input, but the interface may see temperatures up to 1500°C upon impact. Energy input ranges from 6-12 kJ and welds are created within several microseconds, faster than most magnetic impulse weld methods. This method has recently been developed by Anupam Vivek and Glenn Daehn in the Impulse Manufacturing Laboratory at the Ohio State University [45, 46].

![Diagram of VFAW Flyer and Substrate](image)

*Figure 12. Diagram of VFAW Flyer and Substrate [47]*

Studies on the nature of the impact on the wavy interface observed in many impact welds have been conducted. One study by A. Nassiri and B. Kinsey shows a simulated impact using smoothed particle hydrodynamics that explains the
formation and nature of the wavy interface formation [48]. The shearing that occurs at the material interfaces causes the wavy feature during the impact of the weld process.

Like FSW, there are still defects that may be present in the weld. Most common flaws are lack of fusion at the interface or melting of the base materials, due to too low or too high energies of the controlled vaporization. There may also be cracking or fracture of the base materials upon impact if the toughness and ductility of these plates are too low. These are easily optimized because, like FSW, the parameters are easily replicable and adjusted in subsequent welds.

Impact welds between aluminum and steel have been studied, mostly with magnetic pulse weld methods. A wavy morphology is nearly always observed at the interface of collision welds, and Nassiri and Kinsey modelled the collision weld with smoothed particle hydrodynamics and arbitrary Lagrangian–Eulerian methods [49, 48]. Lee et al. looked at the strength of Al-steel lap joints by magnetic pressure seam welding. Successful lap joint welds were attained in several microseconds with no observable temperature increase at the weld interface. Mechanical testing produced no failures at the weld interface. Further characterization of the microstructures using SEM and TEM methods revealed aluminum grains reduced to 100 nm with even finer intermetallic particles at the weld interface, which may prove to be inconsequential in Al-steel weld due to the minute size. In these collision welds, changes in the microstructure are limited to approximately 50 µm from the material interface, as seen in Figure 13. This study will focus on the localized areas adjacent
to the material interface of the high-velocity impact VFAW as these are the areas that exhibit microstructural change from the impact. Within 50 µm of the interface using resolutions down to several nanometers, more information can be taken from the interfaces of high-velocity impact welds.

Figure 13. Optical micrographs of the region close to the weld interface: (a) SPCC steel (b) Al-6111.

2.3 Transmission Kikuchi Diffraction

A scanning electron microscope has the capability to image and characterize samples at high resolutions by scanning a surface with a focused beam of electrons. The resolution can reach features less than a nanometer, much more powerful than
even the best optical microscopes that have resolutions down to 200 nanometers [50]. Additionally, more characteristics of an alloy can be extracted such as composition, microstructure, grain size, grain orientation, among many other useful metrics.

Transmission Kikuchi diffraction was used to analyze sample weld interfaces. A field emission gun SEM is used in this study, and transmission Kikuchi diffraction (TKD) was used extensively for the samples examined.

Transmission Kikuchi diffraction is comparable to electron backscatter diffraction (EBSD) in the SEM. TKD utilizes a thin foil sample in the SEM rather than the bulk sample of EBSD, and the same diffraction mechanisms create the diffraction patterns for TKD commonly seen in EBSD techniques. EBSD has been utilized for materials and metallurgical research extensively since the creation of automated indexing for diffraction patterns. Grain orientations, crystal lattice parameters, grain boundaries, and material identification can be understood using this method.

As seen in Figure 14, the main difference between the two methods is that TKD utilizes thin foil samples to obtain diffraction patterns via electron transmission, where EBSD collects diffraction patterns formed by electron backscattering. This modification allows TKD to resolve data at much higher spatial resolutions due to significantly reduced electron scattering shown in Figure 15. Studies have shown that the smallest effective step size using TKD is 10 nm and the optimal tilt angles are between 10-30° [51].
This scattering due to the incident electron diffraction energies and angles on materials with specific crystal lattice parameters, crystal structures, and grain orientations creates the Kikuchi diffraction patterns that are observed in the TKD and EBSD methods. These diffraction patterns are produced when the Bragg condition is satisfied by the crystal planes in the tilted sample, and coherent electrons are diffracted in specific orientations at that Bragg angle. Thus, wide opposing ‘cones’ of coherent electron signal originating from the interaction volume of the sample, called Kossel cones, create the near-linear patterns observed in a...
transmission Kikuchi pattern (TKP), demonstrated in Figure 16 [53]. In this manner, TKD provides crystallographic information, which can discern lattice spacing of certain groups, crystal systems, and orientation of individual crystals among other things.

Diffraction patterns originate from the bottom 20 nm of the sample foil. This allows transmission of patterns from foil thicknesses ranging from 5 nm to upwards of 3 microns thick [55], but general success has been seen in the range of 200-500 nm of foil thickness. However, the diffraction and quality of the pattern is dependent on the material as well as the foil thickness. Materials that exhibit greater electron scattering will see lower pattern quality at the same foil thickness. Figure 17 below are Monte Carlo simulations created with CASINO software for the sample weld materials used in this study. The electron scattering and beam spreading of electrons are displayed, and the interaction size of the electron beam during TKD of a thin foil is approximated.
Figure 17. Scattering of electron beam during TKD of various samples at thin foil thicknesses created with Monte Carlo simulations (CASINO software) for weld samples.
The electron beam in the aluminum alloy encounters much less scattering and beam spreading, and will exhibit greater patter quality even at foil thicknesses greater than 300 nm. The steel and Ni-base alloy however, cause greater beam spreading and thinner foils approximately 200 nm thick are necessary for optimal pattern quality in TKD.

Texture of a material indicates anisotropy; specifically texture describes the “totality of crystallite orientations” of a material sample [56]. More often than not, processed materials exhibit varying levels of anisotropy. Materials have common textures dependent on the thermo-mechanical processing history, crystal lattice, and composition. With texture, various physical properties can change with an anisotropic material. Tensile strength is a well-known property that changes in a given anisotropic material when tensile tested in different directions relative to its orientation or texture.

Shearing texture is commonly seen in materials undergoing FSW or near the interfaces of impact and collision welds. The simple shearing texture is centrosymmetric and can be observed in materials using TKD and EBSD orientation data. Studies regarding textures observed in FSW materials exist for alloys such as steels and stainless steels, Al-alloys, pure iron, and copper [57, 15, 58, 59, 60, 61, 54, 62, 63]. Shearing in materials during the FSW depends on the crystal structure of the materials and, based on previous reports, the stacking fault energy of various alloys.
Chapter 3: Experimental Methods

3.1 Materials and Weld Parameters

Three specific solid-state dissimilar material weld samples were provided for this study. These welds were created by J. Rodriguez [42], E. A. Torres [43], and A. Nassiri, as shown in the table below and examined in the as-welded condition. The final microstructure of the three weld samples of two material combinations and two weld methods were examined in this study. Weld parameters are discussed as well as the characterization techniques that were used to analyze the samples.

Table 1. Weld sample names and parameters

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Base Metal Alloys</th>
<th>Weld Process</th>
<th>Provided by</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni-steel FSW</td>
<td>Ni-base alloy 625 to ASTM A516 steel</td>
<td>Friction Stir Weld</td>
<td>J. Rodriguez [42]</td>
</tr>
<tr>
<td>Al-steel FSW</td>
<td>Al-6063 to AISI 1020 steel</td>
<td>Friction Stir Weld</td>
<td>E. A. Torres [43]</td>
</tr>
<tr>
<td>Al-steel VFAW</td>
<td>Al-6111-T4 to dual-phase JSC980 steel</td>
<td>Vaporized Foil Actuator Weld</td>
<td>A. Nassiri</td>
</tr>
</tbody>
</table>

Table 1 shows each weld sample in this study, the materials used and the weld process. These sample names will be used to refer to each sample throughout this document.
3.1.1 Friction Stir Welding of Ni-based Alloy 625 to ASTM A516 Carbon Steel

Friction stir welds were made by J. Rodriguez [42] [39] between 500 mm × 90 mm × 6.6 mm thick plates of Ni-based alloy 625 and plain carbon steel ASTM A516 in a butt joint configuration. Chemical compositions of plate alloys can be seen in Table 2 below [14]. Determination of weld parameters are described elsewhere [39].

Table 2. Chemical Compositions (wt%) of ASTM A516 Gr 60 Steel and Ni-based Alloy 625.

<table>
<thead>
<tr>
<th>Element</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Nb</th>
<th>Fe</th>
<th>Si</th>
<th>Ti</th>
<th>Al</th>
<th>Mn</th>
<th>Co</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>A516*</td>
<td>0.01</td>
<td>0.03</td>
<td>-</td>
<td>-</td>
<td>Bal</td>
<td>0.25</td>
<td>-</td>
<td>0.29</td>
<td>0.95</td>
<td>-</td>
<td>0.15</td>
</tr>
<tr>
<td>A625*</td>
<td>60.72</td>
<td>22.59</td>
<td>9.39</td>
<td>3.50</td>
<td>3.00</td>
<td>0.22</td>
<td>0.24</td>
<td>0.18</td>
<td>0.09</td>
<td>0.05</td>
<td>0.02</td>
</tr>
</tbody>
</table>

* Chemical composition provided by the manufacturer.

The FSW was created with a W-Re/PCBN (tungsten-rhenium polycrystalline cubic boron nitride) composite tool with a shoulder diameter 26.0 mm and pin dimensions 6.1 mm length. Threaded diameters were of 3.9 and 10.0 mm at the smallest and largest. The weld was made at a weld speed of 100 mm/min, rotational speed of 300 rev/min, a tool axial offset $O_a$ describing the distance from the tool axis to the joint line, of 1.63 mm toward the steel plate, and 30 kN axial force as seen in Figure 18 below [14]. Tool penetration is not described here, as the process was force controlled.
3.1.2 Friction Stir Welding of Aluminum Alloy 6063 to AISI Steel 1020

The chemical composition of the welded base plate alloys are listed in Table 3 [43]. The FSW was created with a rotational speed of 300 rpm, velocity of 150 mm/min, a tangential displacement, describing the distance between the pin tangent and the joint interface towards the steel, of 1.5 mm. The FSW process for this sample was depth-controlled; tool penetration was maintained at 1.65 mm into the between 500 mm × 85 mm ×2.0 mm thick plate.
Figure 19. Schematic of Al-Fe FSW parameters (a) joint assembly adapted from Yasui [64] and (b) diagram of the \( D_T \) and \( D_R \).

Table 3. Composition of Al-alloy 6063 and AISI Steel 1020 [43]

<table>
<thead>
<tr>
<th>6063-T5 (wt%)</th>
<th></th>
<th></th>
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<tr>
<td></td>
<td>Al</td>
<td>Si</td>
<td>Mg</td>
<td>Fe</td>
<td>Cr</td>
<td>Mn</td>
<td>Cu</td>
<td>Ti</td>
</tr>
<tr>
<td>Bal.</td>
<td>0.46</td>
<td>0.39</td>
<td>0.13</td>
<td>0.04</td>
<td>0.03</td>
<td>0.02</td>
<td>0.01</td>
<td></td>
</tr>
<tr>
<td>SAE 1020 (wt%)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Fe</td>
<td>C</td>
<td>Mn</td>
<td>P</td>
<td>S</td>
<td>Si</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Bal.</td>
<td>0.17-0.23</td>
<td>0.30-0.60</td>
<td>0.035</td>
<td>0.040</td>
<td>0.15-0.40</td>
<td>--</td>
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<td></td>
</tr>
</tbody>
</table>

The friction stir weld pin used to create this is a WC-14Co tungsten carbide metal-matrix tool with ceramic enforcement. The pin diameter is 5.7 mm in diameter and 1.35 in length. The shoulder has a diameter of 25 mm.

3.1.3 Vaporized Foil Actuator Welding of Aluminum Alloy 6111 to DP Steel JSC980

The VFAW weld between Al-alloy 6111-T6 plate to dual-phase steel JSC980 was created at the Ohio State University Impulse Manufacturing Laboratory by Ali
Nassiri working with both Glenn Daehn and Anupam Vivek. The weld process parameters that created the characterized sample weld include a 727 m/s impact velocity, an impact angle of 13° and an energy discharge of 10 kJ of the capacitor bank. The impact angle varies across the width of the weld, as seen in the cross section view of Figure 20. At the center of the weld in Figure 20b, the impact angle is 0° because this is where the vaporized aluminum foil (seen in Figure 20a) starts to launch the flyer plate toward the substrate during welding. As the flyer plate collides into the substrate, the weld is created from the center of the width of the weld out towards the sides. The combination of the location of the vaporizing aluminum foil and the standoff distance between the plates causes impact angle to increase moving from the center towards the edge of the width of the weld. Optimal impact angle variables have been reported to be between 5° and 20° [65]. The flyer in this experiment was 1 mm thick Al-alloy 6111-T6 in the artificially aged condition, and the base plate was 1.5 mm thick dual-phase JSC980 steel.

Figure 20. (a) Cross section schematic of VFW weld set up and (b) diagram of cross section of welded joint from A. Vivek [65].
Table 4. Composition of Al-alloy 6111 and dual-phase JSC980 steel [43]

<table>
<thead>
<tr>
<th></th>
<th>Al</th>
<th>Si</th>
<th>Mg</th>
<th>Fe</th>
<th>C</th>
<th>Mn</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al 6111</td>
<td>98.05</td>
<td>0.9</td>
<td>0.75</td>
<td>--</td>
<td>--</td>
<td>0.3</td>
</tr>
<tr>
<td>JSC 980</td>
<td>--</td>
<td>0.6</td>
<td>--</td>
<td>99.25</td>
<td>0.15</td>
<td>0.27</td>
</tr>
</tbody>
</table>
3.2 Sample Preparation

A cross section of each weld was cut and mounted in 1.25” Bakelite or cold-mounted in epoxy. It was ground with sandpaper up to 1200 grit and further polished using diamond suspension or colloidal silica down to 0.5 μm. At this stage, nanoindentation of samples could be conducted.

The mounted sample was then etched according to the material. Because each sample contained carbon steel, all three samples were etched with 2% Nital solution for 5-10 s in order to obtain light etching of the steel for visibility of the weld interface. To etch the Ni-base alloy of the Ni-Steel FSW, an electrolytic etch was applied in 10% chromic acid at 5 V, 2 A, and 25-45 s following the Nital etch. After etching, optical and electron microscopy was done, and thin foils from the interface were taken.

A Helios focused ion beam (FIB) was used for thin foil extraction at the interface of each sample, as seen in Figure 32. Foil extraction, thinning, and polishing a dissimilar metal weld interface posed a significant challenge in preparing a quality foil essential to the quality of TKD analyses. A 35 μm × 3 μm × 2 μm platinum bar was deposited and trenches were created in the area of interest perpendicular to the material interface. Steel proved to be more difficult to create a trench due to re-deposition of materials in the trench. The foil was cut approximately 25-30 μm × 7-10 μm before thinning. Thinning and polishing the foils was the most difficult of the preparation process. Foil thinning was conducted at 30 kV accelerating voltage for all foil materials while stepping down beam currents.
Several passes were made on each side at approximately 2° from parallel. Once adequate thickness was achieved, accelerating voltage was adjusted to 5 kV and beam currents to 1-2 nA or lower. Some success was observed when thinning in sections of 10 µm wide at 4-7° from parallel. Aluminum proved to be the easiest to thin and polish for satisfactory TKD results. Thinning steels often resulted in curtaining of foils and damage to the sample if it is thinned until ‘bright’. Interfaces between the aluminum and steel were also troublesome, as preferential thinning in the aluminum or at the interface (within nanometers) caused damage and sample loss, as shown in Figure 21.

*Figure 21. Excessive foil thinning resulting in damage to foil (a) before thinning and (b) after thinning. This sample is the Al-steel VFAW sample Foil 1.*
Thinning at 5 kV for more frequent passes was gentler and, albeit time-intensive, less damaging overall on the foil samples. After polishing on a focused-ion beam, the sample was further polished in a low-energy ion mill. Additionally, if carbon build-up was excessive, a simple plasma cleaning process eliminated carbon buildup from electron microscopy.

3.3 Characterization and Analysis

Preliminary optical and SEM imaging characterization was conducted on the bulk cross section in order to locate the optimal location for foil extraction. SEM imaging, TKD and XEDS analyses were run in an XL-30 ESEM. S. Romo and I worked to design and fabricate a graphite TKD sample holder to be used for simultaneous TKD and XEDS mapping, as seen in Figure 22. TKD and XEDS analyses were all conducted on a field emission gun SEM with electron beam accelerating voltage of 30 kV, spot size of 6, and aperture at 100 μm. A working distance of 4-10 mm and 20° sample tilt was maintained for these SEM methods. Step sizes down to 6 nm were used for mapping.
Figure 22. Graphite TKD Holder Prototype (a) side view and (b) top view designed by G. Lee and S. R. Arango.

Figure 23. CAD designs for TKD holder (a) 4 part design and (b) 2 part design

Additional design iterations are being developed, shown in Figure 23. Figure 23a is made of 4 parts – the top and bottom holder, and 2 smaller tips that can be created of a lightweight, more durable material, such as beryllium. The design in Figure 23b is comprised of only 2 parts, a top and bottom portion of the holder that will be held together with a spring-load mechanism.
3.4 TKD (EBSD) Post-Processing

TKD data was analyzed for grain size, orientation, shape, and texture. The orientations for the three samples were rotated to align the local deformation shear frame to the electron microscope frame. Data cleanup was also conducted using data analysis software for the TKD and XEDS map data. Neighboring orientation correlation cleanup was applied first where each point with 6 neighbors of the same orientation is changed to match that orientation. Cleanup level of 0, minimum confidence index of 0.1, and grain tolerance angle of 5 was used. Grain dilation was also applied, changing only points that do not belong to any grain as defined to the orientation and confidence index of the neighboring point with the highest confidence index. Single iteration with grain tolerance angle 5 and minimum grain size 3 was used for cleanup parameters.

The crystallographic data was rotated based on the location and orientation of the foil in the friction stir weld cross section to obtain accurate representation of the crystal orientation and texture of the TKD analyses. These rotations are shown and explained below in Figure 24-Figure 30. Rotation of friction stir weld data has been discussed in studies by Abbasi, Nelson, and Sorenson [57]; by Fonda and Knipling [66]; Abbasi et al. [67]; and Ahmed et al. [15].

The coordinate system of the friction stir weld, the “specimen reference frame” (SRF) also referred to as a “sample reference frame”, is shown in Figure 24. This is typically defined by the weld travel direction, the rolling direction of the base material, and/or the through-thickness direction of the plate.
Figure 24. Specimen Reference Frame of FSW

Figure 25. "Local shear deformation frame" of a FSW shown in (a) top view (b) front view (c) dimetric view and (d) dimetric view of FIB foil with shear direction (note that SPN and SD are not orthogonal to cross sectional cut plane).

The “local shear deformation frame” (LSDF), defined by the shear direction (SD) and shear plane normal (SPN) direction of the weld at the sample foil location, is shown
in Figure 25. The shear plane will coincide with the material interface as well as the shear direction. SPN will be orthogonal to the shear plane and thus the SD. It should be noted that the LSDF has no relation to the SRF defined by the weld plate due to the intense and random deformation and mixing of material during the FSW process that may not align with and reflect the weld travel direction.

The thin foil should always be cut and extracted perpendicular to the interface for the simplest rotation method of the collected dataset, as shown in Figure 25d. The LSDF will be observed in the foil at the material interface. This reference frame will be used to align the data orientation to provide accurate and relevant texture information.

*Figure 26. Representative foil created in CAD software (bottom) to reflect thin foil samples in study.*
The reference frame or orientation of the TKD data taken by the collection software projected onto the sample foil also known as the “electron microscope reference frame” (EMRF), is shown in Figure 27.
Figure 27. Reference frame of electron microscope in OIM software (a) used in EBSD analysis (b) used in EBSD mode with thin foil (c) “electron microscope reference frame” used in TKD mode with thin foil. Foils with EMRF axes are shown at the left. Adapted from EDAX OIMA manual [68].
The EMRF can be changed in the software. Here, a common orientation or the EMRF used in EBSD is shown in Figure 27a above. Figure 27b shows the same EMRF with a thin foil sample rather than an EBSD bulk sample and the corresponding EMRF axes imposed on a representative foil as seen in the SEM during TKD analyses. In the data collection software, there is an option to select “transmission EBSD” (or TKD) mode. This selection will rotate the commonly used EMRF orientation by -90° about Axis 2, taking sample tilt into account. The final EMRF used in this study is shown in Figure 27c, with the same projected axes shown on a representative foil at the right. This reference frame is important to understand relative to the sample foil being analyzed, as it will be rotated to align with the LSDF observed in the foil. Figure 28-Figure 30 show three interface orientations commonly seen in a thin foil extracted at the interface.

Foil with three different interface orientations are shown in Figure 28-Figure 30 with both the EMRF and LSDF labelled. The rotations necessary for texture analysis will bring the EMRF to align with the LSDF of the sample foil. Axis 1 shall be aligned to SPN, and Axis 2 shall be aligned with SD [66]. An ideal foil orientation, common foil orientation, and non-ideal foil orientation of LSDF will be discussed.

Figure 28 shows the top, front, and isometric view of a thin foil with an ideal LSDF orientation as well as the EMRF used in this study. Here, only one rotation is necessary to align the axes of the EMRF with the correct directions of the LSDF. The
SD goes along the material interface, and SPN is normal to the shear plane that coincides with the interface.

Figure 28. Ideal orientation of dissimilar metal weld interface in a thin foil
Figure 29 shows the top, front, and isometric view of a foil with a typical LSDF orientation. Here, the material interface is offset by an angle $\alpha$. This rotation is also relatively simple and only requires one rotation to align the EMRF and LSDF correctly.
**Figure 29. Common foil orientation of dissimilar metal weld interface**

a) Top View (common foil)

b) Front View (common foil)

c) Isometric View (common foil)

Goal:
Axis 1 = SPN
Axis 2 = SD

Rotate:
Axis 1 = +0°
Axis 2 = +0°
Axis 3 = +90° - α
Figure 30 is the non-ideal orientation of the LSDF on a thin foil made at a FSW interface. This will require two rotations and the textures are less accurate, experienced in this study with several sample foils. This orientation will usually be avoided when foils are made perpendicular to the local dissimilar material interface.
at the stir zone of a FSW. This material interface and shear plane exhibits two directions offset from orthogonal, α and β. Rotations should be made about Axis 3 first at 90°- α, then about Axis 2 at -90°+ β.

The data collection software used in this study, EDAX OIMAnalysis, did not have the capability to differentiate EBSD and TKD datasets. This resulted in a 90° discrepancy of analyzed textures and actual textures. Thus, the data must be rotated again by 90° about Axis 2 before conducting any orientation or texture analysis.

3.4 Nanoindentation

Nanoindentation hardness tests were conducted on all weld samples using a standard Berkovich indentor tip and a depth-control indentation method. Indentation depths were kept at 700 nm to maintain indentation areas of approximately 0.5 µm² with 14 µm spacing between indents. For testing both the steel and Ni-base alloy, the Poisson’s ratio was ν=0.3 while tests on the aluminum alloy used Poisson’s ration of ν=0.35. Hardness and modulus values were given in GPa. Calibration of each batch test was run on fused silica before starting.
Chapter 4: Results and Discussion

4.1 FSW Ni-Based Alloy 625 to Carbon Steel

SEM analysis and nanoindentation was conducted on the sample weld shown in Figure 31. Additional post-processing was done to the data, discussed below.

![Image of macroscopic cross section](image)

*Figure 31. Macroscopic cross section of Ni-Steel FSW indicating locations of FIB foil extraction and nanoindentation map.*

4.1.1 SEM Analysis

Using a field emission gun SEM, metallographic analysis was conducted in addition to crystallographic and chemical characterization. Upon examination of microstructural phases, the steel and Ni-base alloy microstructures correspond to observations of A516 and Ni-625 FSW studies done by J. Rodriguez[14] and K. H.
Song [28]. Pearlite and ferrite is observed in the steel stir zone and refined grains and can be seen in the Ni-base alloy stir zone [14].

![Image](image.png)

Figure 32. TKD thin foil of Ni-steel FSW Interface (a) Foil 1 and (b) Foil 2

TKD analysis of the foil shows refined grains along the material interface of both alloys in the stir zone. Figure 33 below shows maps of TKD and XEDS analysis of a stir zone region. Figure 33(a) shows the phase map of the region identified by TKD. The FCC austenite of the Ni-base region is red, adjacent to the blue ferrite of the steel. Precipitates of MC and M23C6 are also observed dispersed throughout the red austenitic regions both inter- and intra-granularly. Figure 33(b) depicts a map obtained from the TKD analysis that presents the inverse pole figure (IPF) data representing the grain orientation of the region normal to the Z (out-of-page) direction. In Figure 33(c), XEDS compositional data can be observed, with an analysis for iron, nickel, chromium, molybdenum, and niobium. At the right are transmission Kikuchi patterns (TKP) for each phase that is identified in Figure 33(a). A pattern can be seen for austenite (Ni), ferrite (steel), MC, and M23C6.
Figure 33. Maps of (a) phase, (b) IPF and IQ greyscale overlay, (c) XEDS chemical analysis of Ni-steel FSW Foil 1. Kikuchi patterns observed during TKD analysis are shown at the right.

The Ni-based alloy matrix consists of austenite grains and a nickel-chromium solution. The carbon steel side is identified by the ferrite crystal phase and the iron-
based matrix. Approaching the material interface, grain sizes down to 200 nm are achieved. During the FSW process, dynamic recrystallization from the shearing and plastic deformation from the rotating pool creates fine grain sizes [28]. Figure 34 below shows XEDS of the same region, separated by matrix elements and precipitate elements. Figure 34(a) contains iron, nickel, and chromium measurements of the steel and Ni-base alloy. Figure 34(b) shows only molybdenum and niobium measurements, which are present within precipitates within the Ni-base alloy stir zone matrix.

Figure 34. XEDS composition maps of the same Ni-steel FSW weld interface Foil 1 area shown in Figure 33. (a) shows only matrix elements of both materials, and (b) shows
elements found in particles within the austenite (Ni) region, rich in molybdenum and niobium.

With TKD, crystallographic analysis of the stir weld shows identification of the niobium-rich precipitates as MC (NbC), the same found within the base material. However, the Mo-rich precipitates are recognized as $\text{M}_{23}\text{C}_6$ (Cr$_{23}$C$_6$) or $\text{M}_6\text{C}$ (Mo$_6$C). $\text{M}_{23}\text{C}_6$ and $\text{M}_6\text{C}$ are both cubic structured phases and have exhibited lattice parameters between 1.08-1.12 nm [69]. Figure 34 also shows that the areas of the Mo-rich precipitate may also exhibit some chromium composition. Based on other literature, this study is $\text{M}_{23}\text{C}_6$ because these carbides are present in Ni-base alloy 625 base material. $\text{M}_{23}\text{C}_6$ are commonly Cr-rich, though some carbides may be a mixed carbide rich in both chromium and molybdenum. TEM characterization is necessary to definitively confirm the crystalline structure.

![TEM images showing niobium and molybdenum-rich precipitates](image)

*Figure 35. Maps of Ni-base alloy Foil 2: stir zone at shoulder of Ni-steel FSW joint (a) XEDS chemical analysis of (b) IPF color map + IQ grayscale.*

### 4.1.3 Texture

Orientation of the FSW interface was evaluated. Texture analysis and the resulting pole figures (PF) of Figure 33 and Figure 34 are shown in Figure 37 and Figure 36.
below. Studies of friction stir processed (FSP) and FSW alloys of FCC and BCC crystal structures have determined that the typical shear components of FSW BCC materials are in the $D_2 (1\bar{1}2)[111]$ texture component [62] in pure iron, and $D_1 (\bar{1}12)[111]$, $D_2 (1\bar{1}2)[111]$, and $F (110)[001]$ in steel [57]. Figure 36 below shows the PF of the ferrite in the Ni-steel FSW sample. Maximum intensity is 15.66 times random. There is strong F shear texture component observed in the ferrite phase of the local microstructure. The PF in Figure 37 shows a strong B texture component with maximum intensity at 11.43 times random. The FCC shear components reported in literature include those for aluminum alloys, NiAl bronze alloy, copper, and stainless steels. Except in aluminum alloys, textures reported in FSW are generally weak, an observation thought to be attributed to the relatively low stacking fault energy (SFE) of the alloys compared with other FCC metals, such as nickel and copper. FCC aluminum alloys have reportedly exhibited $B/\bar{B}$ and some C texture components [15] [59] [70] [58] [71] [60] [72]. Other FCC alloys, including pure copper, superaustenitic stainless steels, and NiAl bronze alloys report A and B textures.
Figure 36. PF of Ferrite (Fe) in Foil 1 Ni-steel FSW and ideal shear textures observed in FSW BCC materials [66] overlaid.
Figure 37. PF of Austenite (Ni) in Foil 1 Ni-steel FSW and ideal shear textures observed in FSW FCC materials [66] overlaid.

4.1.4 TEM

Analysis in the transmission electron microscopy was conducted to determine the composition and crystallographic structure of the Mo-rich precipitate. Figure 38 shows a bright field image and the SAD pattern of the precipitate.
Using the SAD pattern, this precipitate was determined to be a $M_{23}C_7$ precipitate, which corresponds to the chemical composition as well as other literature reporting the presence of $M_{23}C_7$ carbides in Ni-base alloy 625.

4.1.5 Nanoindentation

Nanoindentation tests were conducted along the interface of the weld at the regions marked by arrows in Figure 31 near the regions from where the foils were extracted. The results provide hardness and modulus values of the interface area. Additionally, measurements in the base metal were taken to evaluate grain refinement or cold working from the FSW process. The base metal values of each alloy are presented in Table 5 below. Reported values of steels and nickel alloys are additionally tabulated in Table 6.
Table 5. Nanoindentation measurements of Ni625 and A516 base metal in the joint away from the Ni-steel FSW interface. Hardness and modulus, measured in GPa, are provided below.

<table>
<thead>
<tr>
<th>Base Metal</th>
<th>Hardness (GPa)</th>
<th>Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni-625</td>
<td>4.66</td>
<td>219.93</td>
</tr>
<tr>
<td>Steel A516</td>
<td>3.09</td>
<td>224.61</td>
</tr>
</tbody>
</table>

Table 6. Reported nanoindentation values for steel, nickel, and nickel based alloys

<table>
<thead>
<tr>
<th>Material</th>
<th>Load or Depth Controlled</th>
<th>Max Load or Depth</th>
<th>Measured Hardness (GPa)</th>
<th>Measured Modulus (GPa)</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low carbon Steel</td>
<td>Load</td>
<td>5.0 mN</td>
<td>2.0-3.6</td>
<td>150-270</td>
<td>[73]</td>
</tr>
<tr>
<td>Intergranular Ferrite</td>
<td>n/a</td>
<td>n/a</td>
<td>2.676-2.800</td>
<td>n/a</td>
<td>[74]</td>
</tr>
<tr>
<td>Welded SM490</td>
<td>Load</td>
<td>150 mN</td>
<td>2.78-2.86</td>
<td>210.4-213.4</td>
<td>[75]</td>
</tr>
<tr>
<td>DP980 Steel Ferrite</td>
<td>Depth</td>
<td>100-400 nm</td>
<td>4.5</td>
<td>n/a</td>
<td>[76, 77]</td>
</tr>
<tr>
<td>Low carbon Bainitic Steel</td>
<td>Load</td>
<td>1.0-4.0 mN</td>
<td>3.3-4.5</td>
<td>n/a</td>
<td>[78]</td>
</tr>
<tr>
<td>Fusion Boundary of A533 and Alloy 152</td>
<td>Depth</td>
<td>1000 nm</td>
<td>4.0-5.0</td>
<td>n/a</td>
<td>[79]</td>
</tr>
<tr>
<td>Nickel 690</td>
<td>Depth</td>
<td>150 nm</td>
<td>2.7-3.2</td>
<td>n/a</td>
<td>[80]</td>
</tr>
<tr>
<td>Microcrystalline Nickel</td>
<td>Depth</td>
<td>2000 nm</td>
<td>2.3</td>
<td>235</td>
<td>[81]</td>
</tr>
<tr>
<td>Cold Worked Nickel</td>
<td>load</td>
<td>50 mN</td>
<td>2.5-2.7</td>
<td>210-250</td>
<td>[82]</td>
</tr>
</tbody>
</table>
The measured hardness values of the base material approximate the hardness values reported for steels, specifically those with large volume fractions of ferrite. Nickel hardness values are slightly higher than reported, though that may be due to the difference in previous mechanical or metallurgical processing of the base material compared with that in literature.

The following figures present the nanoindentation map and the associated data at several areas of the joint. The hardness map of Nanoindentation Test 1 of Figure 31 conducted at the FSW interface is presented in Figure 39. There is a clear variation in hardness values between the two alloys. However, some error may be observed due to the inherent nature of FSW cross sections and the nanoindentation test. FSW experience severe plastic deformation and interfaces viewed at a cross sectional cut may not continue into the weld at an orthogonal direction. Thus, nanoindentation of areas near to a material interface may reflect values of both material hardness values and moduli. Because the nanoindentation collects data from a projected volume around the indent, the data collected within several microns of the interfaces may present values averaging both nickel and steel properties. Figure 40 presents the data points on a frequency plot for both modulus and hardness.
Two peaks in Figure 40a can be seen, corresponding to the hardness value of A516 and Ni625 of 3.2 GPa and 5.2 GPa respectively. These values are equal to or greater than that of the base materials. The Ni-base alloy hardness at the interface has increased by 0.6 GPa on average, while the steel hardness at the stir zone is similar to the parent material. The severe plastic deformation and grain refinement in of the FSW process contributes to this increase, as explained by the Hall-Petch relationship [10] [40] [83]. Additionally, the FSW process has been reported to break apart and drag precipitates within the base metal into the stir zone. These finer precipitates can improve the strength of the alloy, explained by the Ashby-Orowan model [84] [42] [85]. The Ni625 stir zone has precipitates, most below 0.5 μm in diameter, which can contribute to the jump in hardness due to the smaller size compared with those in the base material [8]. The modulus frequency plot in Figure 40b has one peak because the moduli of low carbon steels and nickel-based alloys are very similar.
Figure 40. (a) Hardness and (b) modulus values in GPa by frequency for values in the stir zone region of the Ni-steel FSW joint seen in Figure 39.

Figure 41 is the hardness map of Nanoindent Test 2 at the interface shown in Figure 31; this is taken at the shoulder of the weld joint rather than the stir zone. Figure 42 below shows the frequency plot for the hardness and modulus data.
The hardness values of the materials at the interface near the shoulder of the FSW joint exhibit two distinct hardness values at 5.0 and 5.8 GPa corresponding to the Ni-base alloy and A516 steel, respectively. The Ni-base alloy does not vary much when compared to the values in the previous test near the center of the stir zone. However, the steel sees a more significant increase in hardness – an increase to 5.8 GPa compared to the 3.2 GPa in the center of the weld. This may be due to greater thermal cycles from increased frictional and plastic deformation heating from the shoulder tooling. With increased heat, microstructural phase changes or growth may occur. This microstructure can result in a significant hardness increase. The
modulus, like the previous test, does not show two distinct peaks as the modulus of steel and Ni-based alloys are similar.

Figure 42. (a) Hardness and (b) modulus values in GPa by frequency for values at the interface of the shoulder region of the Ni-steel FSW joint in Figure 41.
4.2 FSW Al to Steel

The FSW between aluminum alloy 6063 to AISI 1020 was examined using SEM methods to ascertain the presence of intermetallic compounds.

![Al-6063 RS to AISI-1020 AS FSW cross section by optical microscopy](image)

*Figure 43. Al-steel FSW cross section by optical microscopy*

The figure above is an optical micrograph of the Al-alloy 6063 to AISI 1020 carbon steel FSW. The joint interface and pin displacement can be observed in this image. Characterization of this weld cross section is discussed in the following sections.

Several foil extractions were created at the interface of weld joint where diffusion or mixing of the alloys was apparent, including the area presented in Figure 44 below. The foils extracted, shown in Figure 45, were analyzed using TKD and XEDS. Grain sizes and morphologies were characterized while examining for intermetallic compound presence.
Figure 44. SEM image of Al-steel FSW joint (a) with SE detector and (b) BSE detector where mixing was observed.

Figure 45. (a) Foil 1 and (b) Foil 2 created at the Al-steel FSW joint interface with SEM analyses marked.

The analyses highlighted in discussed in Figure 45 are discussed this report in the following figures. Figure 46 below shows the orientation and chemical
composition of the Al-steel interface of Analysis 1 on Foil 1 from two separate scans stitched together. Fine grains can be observed in the carbon steel between 100-500 nm large. The aluminum grains are much larger -- between 1-3 µm large in Figure 46a. Additionally, the chemical composition of the region can be seen in Figure 46b. The interface of the XEDS map is relatively sharp and implies that there is limited inter-diffusion at this area, which may preclude intermetallic nucleation and growth.
Figure 46. TKD analysis 1 of Foil 1 in Al-steel FSW joint (a) color IPF and IQ greyscale overlay and (b) XEDS chemical composition analysis.
Figure 47. XEDS map of Analysis 2 of the Al-steel FSW interface Foil 1 showing (a) aluminum (b) iron and (c) both aluminum and iron counts.

Figure 47 shows the XEDS map of Analysis 2 of Foil 1. There is very limited diffusion or mixing of aluminum and steel in this ‘vein’ of aluminum within a steel matrix. The lack of mixing may suggest lower thermal cycles of the weld that result in less mixing, and may also play a role in inhibiting the formation of intermetallic compounds.
Figure 48. TKD map of Analysis 2 of Al-steel interface Foil 1 showing IPF orientation and grayscale overlay of (a) confidence interval and (b) image quality.

Figure 48 above shows the IPF map with either the CI (a) or IQ (b) greyscale overlay in area of Analysis 2 of Foil 1. The aluminum-rich vein present exhibits low image quality and confidence interval and very scarce data points of indexed or resolved patterns.

Figure 49 shows the TKD and XEDS analysis results from the area of Analysis 3 on Foil 2 of the Al-steel FSW interface.
There is more alloy diffusion observed in this region in the XEDS analysis. However, no intermetallic compounds were observed in the TKD map. At the interface of the two materials, there exists a region where the patterns are not resolved due to exposure, gain, and background calibration. Using point analysis at the interface of the area in Figure 49, no transmission Kikuchi patterns were identified as that of potential intermetallic compounds. Each point sampled in the unresolved area was matched with the ferrite steel crystal lattice or the FCC aluminum lattice.
Figure 50. Transmission Kikuchi patterns of the ferrite and FCC austenite in the unresolved region of Analysis 3 in Foil 1 of the Al-steel FSW interface in Figure 49.

4.3 VFAW Al to Steel

The analysis approach for this Al-steel VFAW sample was similar to that of the Al-steel friction stir weld discussed in Section 4.2 FSW Al to Steel. Optical microscopy and scanning electron microscopy techniques were used to analyze the sample interface. Figure 51 below shows a sample interface exhibiting the wavy interface feature characteristic of impact welds.

Figure 51. Optical micrograph of Al-steel VFAW weld interface
In Figure 51, both materials can be seen. The dual-phase steel is etched in this image, and there is an intermediate shade apparent at the interface of the weld. The largest area exhibiting this feature is at the peak of the wavy feature. This region at the interface is the area of interest, and further investigation is conducted, discussed in the following sections.

4.3.1 SEM Analysis

SEM images of the interface using SE detector were taken prior to extracting a thin foil, as seen in Figure 52 below.

Figure 52. SEM image taken with SE detector of wavy interface region of intermediate shade in Al-steel VFAW weld.

Figure 52 shows the peak of the wavy region exhibiting an intermediate shade when compared to that of the two base materials. This may indicate mixing or diffusion of the aluminum and iron materials during the weld process, and may lead to intermetallic compound formation. Foils were taken from regions at the crest and the trough of the wavy feature of the interface.
Figure 53. (a) Foil 1 before final polish (b) Foil 1 during analysis and (c) Foil 2 taken from Al-steel VFAW sample interface as seen in Figure 52.
Figure 54 and Figure 55 are TKD maps of the Foil 1 using TKD. The steel region in Figure 54 shows indications of martensite and ferrite. Ferrite is identified by the transmission Kikuchi pattern, but the lath-structure observed is typical for martensitic microstructures. The body centered tetragonal (BCT) martensite structure can often be identified as ferrite by TKD because of the similarities in crystal lattice parameters and structure. Both fine grains (100 nm) and large grains (2 μm) are observed in the steel region of this weld. Figure 55 shows aluminum grains down to 200 nm as well as some larger than 3 μm, exhibiting some texture by the shading of the inverse pole figure orientation.

![Figure 54. TKD map of IPF and CI overlay of steel region of Al-steel VFAW interface Foil 1 with lath structure marked with white arrows. IPF orientation of scan is the same as the Al region scan in Figure 55.](image)
Figure 55. TKD map of IPF and CI overlay of aluminum and intermediate region of Al-steel VFAW interface Foil 1. IPF orientation is the same as that of the steel scan in Figure 54.

The intermediate region of Figure 55 has just a fraction of the confidence interval that the other two regions in the foil exhibit, but some grains of uniform orientation are faintly visible. This indicates crystallinity and grain structures in this region, so it is not amorphous. Figure 56 is a TKD map of the IPF and confidence interval greyscale overlay of a foil stitched together from several smaller maps across the interface.
Figure 56. TKD map of Foil 2 in Al-steel VFAW interface stitched from several maps. Aluminum alloy is at the left and steel is at the right. Unresolved black region in the middle is the intermediate region and a circle indicates where a point analysis technique is discussed in below.

The steel is on the right and exhibits grains approximately 100 μm from the impact weld process, smaller than the observed aluminum grain size. On the left, the aluminum alloy can be seen and grains are between 100 nm to 2 μm large. The intermediate region spans over 1 μm wide between the two base materials. XEDS line scans as well as point analysis was conducted along this unresolved region.
Figure 57. XEDS line across interface region of Foil 2 of Al-steel FSW

Figure 58. XEDS line across interface region of Foil 2 of Al-steel FSW. Diffusion of aluminum and iron can be observed.
Across the joint interface, the chemical composition of the interface region was investigated using XEDS. Both aluminum and iron can be observed within and immediate adjacent to the IMC region. This can be indicative of mixing as well as intermetallics, though it does not provide confirmation of the presence of one over the other.

Using point analysis at this unresolved region, transmission Kikuchi patterns of intermetallic compounds are observed, exemplified in Figure 59. A red circle marks the region were point analysis was conducted. A transmission Kikuchi pattern was obtained and the automated indexing software provided three solutions for the diffraction pattern shown in Figure 59 below. The three solutions are FeAl, FeAl$_2$, and ferrite.

![Figure 59. Transmission Kikuchi pattern from point analysis of Foil 2 at Al-steel VFAW interface shown in Figure 56 at the left with possible indexed solutions at the right.](image)

The composition of this point was also measured with XEDS and the measured chemical compositions are shown below in Table 7. The chemical compositions of the three solutions from TKD analysis of Figure 59 above are tabulated in Table 8.
Table 7. XEDS point analysis data from the red spot marked in Foil 2 in Al-steel VFAW interface shown in Figure 56.

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt %</th>
<th>At%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>53.04</td>
<td>70.04</td>
</tr>
<tr>
<td>Fe</td>
<td>46.96</td>
<td>29.96</td>
</tr>
</tbody>
</table>

Table 8. Composition of possible compounds or phases shown in Figure 59 determined from the OIM analysis software with identification of FeAl₂ based on chemical data.

<table>
<thead>
<tr>
<th>IMC</th>
<th>wt% Al</th>
<th>at% Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ferrite</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>FeAl</td>
<td>25</td>
<td>45</td>
</tr>
<tr>
<td>FeAl₂</td>
<td>48</td>
<td>65</td>
</tr>
</tbody>
</table>

It should be noted that analysis by XEDS on a thin foil has a markedly larger margin of error compared with XEDS analysis composition of bulk samples. Of all possible intermetallic compounds between aluminum and iron, compositions of FeAl₂, Fe₂Al₅, and FeAl₃ only (tabulated in Table 11) correspond with the XEDS measurements of the point analysis in Table 7. Table 8 includes only the possible solutions determined using TKD in Figure 59; the single compound that exhibits
corresponding chemical composition with the point of interest is FeAl₂. Thus it can be concluded that FeAl₂ exists within the interface region.

4.3.2 Texture

From the TKD data analysis of the VFAW interface, texture analysis can be conducted for the weld. The local shear deformation frame was aligned with the electron microscope frame using appropriate rotations, similar to the method used for the FSW samples. However for this weld process, the shear plane is coincident with the surface plane of the VFAW weld where the aluminum flyer plate is bonded to the steel substrate. The shear direction is described by the vector originating at centerline of the weld moving towards the edge parallel with the interface plane [49, 86]. Figure 60 shows the shearing nature described and confirmed through various studies. The shear plane lies on the plane of the material interface and the shear direction goes in the direction of the impact weld, from the center to the outsides.

![Diagram of shearing nature](image)

*Figure 60. Nature of shearing and wave formation at interface of impact welds [87, 88]*.
Pole figures for the ferrite and FCC austenite were created, seen in Figure 61 and Figure 62. The austenite of the aluminum region in Figure 61 shows a maximum intensity of 11.2 times random and exhibits shear texture component of B/-B {112}<110> strongly, which is commonly reported in simple shear processes of aluminum alloys. There are also weaker A {111}<112> shear components present, also reported in the literature [59] [58] [71] [60] [72] [89].

Figure 61. Pole figure of FCC austenite (Al) at Al-steel VFAW interface
The ferrite pole figures from the steel region shown in Figure 62 exhibit a simple shear texture, D \{112\}<111> shear component, which is typically observed in steels. The pole figures have a maximum intensity of 10.4 times random.

4.3.3 Nanoindentation

Nanoindentation tests were taken of the bulk samples at the interface. Hardness traverses and maps were utilized to measure hardness and modulus
values of the base material, the affected regions adjacent to the interface, and potentially the visible intermediate shaded region. Figure 63 shows three hardness traverses across the interface, and a map of the interface region stitched together from several micrographs. Figure 65 and Figure 64 are the data analyses of the nanoindentation values. A frequency plot of the nanoindentation map taken in Figure 63b is displayed in Figure 65 below.

Figure 63. Nanoindentation (a) traverses and (b) map across the Al-steel VFAW interface.
There is a clear difference in both hardness and modulus values on either side of the interface and no indications of fluctuation as the distance from the interface changes. The values in this diagram also correspond with the hardness values expected based on previous nanoindentation studies [76].

Figure 64. Nanoindentation traverse values at the VFAW interface for (a) modulus and (b) hardness.
Near the interface however, there exists several points that are intermediate values between the two expected base materials. This is likely due to the presence of both materials in the affected area of the nanoindentation, resulting in value between both material values.

![Graph](image)

Figure 65. Frequency plot of (a) hardness and (b) modulus values of the nanoindentation values taken at the interface of an Al-steel VFAW interface.
Two distributions can be observed in both hardness and modulus frequency diagrams. The data clusters correspond to the measured values of the JSC980 steel and aluminum alloy 6063. The hardness and modulus of the steel is approximately 4.8 GPa and 275 GPa where the hardness and modulus values for the aluminum alloy average 1.8 GPa and 125 GPa, respectively. There are no other values observed outside of the two large clusters corresponding with the reported values for both dual-phase steel and 6XXX series aluminum alloy. This may be due to a combination of calibration error of the nanoindentation test, the location of intermetallic compounds relative to the affected indentation volume, or the interaction between the intermetallic phases and the weld metal matrix, among other possible factors. The three traverses in Figure 63 are displayed in Figure 64 as a graph against the distance from the interface.
Chapter 5: Conclusion

Dissimilar material solid-state welds were studied using high-resolution methods to isolate data and properties to regions within micrometers of the interface. Understanding the microstructure and features present within these welds, particularly at regions of the interface, may allow for better understanding of failure mechanisms and thus better design of systems that benefit from dissimilar material joints. Friction stir welds of Ni-base alloy to carbon steel, Al-alloy to carbon steel, and vaporized foil actuator welds between Al-alloy to steel were examined using various techniques.

The Ni-steel FSW was examined using SEM techniques and nanoindentation. TKD and XEDS were used to characterize the weld material at the interface within the stir zone. Refined grains of both FCC austenitic Ni-base alloy and BCC ferrite in the steel was observed, with lower image quality, or pattern quality, in the fine steel grains. Additionally, precipitates in the Ni-base alloy regions adjacent to the interface were observed. Using orientation and crystallographic information along with chemical composition analysis, the precipitates were identified as MC (NbC) carbides as well as $M_{23}C_6$ or $M_6C$ carbides. With additional investigation on the TEM, the precipitates were confirmed as $M_{23}C_7$ (Cr- and Mo-rich) carbides. Texture analysis was conducted on the foils, and simple shear textures, corresponding to
current studies, was observed at the grains near the interface of the FSW. The austenite of the Ni-base alloys presented A and B shear texture components, corresponding to research of FSW nickel and other non-aluminum alloy FSW textures. The BCC ferrite of the steel showed strong texture (about 21 times random) in the F shear component, also confirmed by literature. Texture information for friction stir welds have not been reported at such localized areas. Literature has suggested the simple shear texture of FSW material joint interfaces, however, these tests were conducted using EBSD [15] [59] [70] [58] [71] [60] [72]. The analyses capture texture data further from the interface as well and do not isolate the texture that is indicative of the shear deformation occurring, as the analyses in this study shows. Nanoindentation of the joint interface was conducted as well. Base metal nanoindentation values were measured as well as the values at the stir zone and shoulder at the stir zone and shoulder region. The values at the interface were slightly higher than that of the base material for the Ni-base alloy, and the steel hardness values were about 2.5 GPa greater at the shoulder region of the stir zone compared to the base material.

The Al-alloy 6063 to carbon steel AISI 1020 FSW was analyzed using SEM methods for intermetallic compounds. Using TKD, the interface of the weld joint was analyzed down to 10 nm resolution. The weld process refined the aluminum alloy grains to between 1 and 3 μm. In the steel region, grains were as small as 100 nm. Some regions at the interface were not resolved in the TKD maps presented, but with point analysis, no transmission Kikuchi patterns were observed that did not correspond to austenitic aluminum or ferrite diffraction patterns. Several areas of
the FSW interface showed more evident diffusion of iron and aluminum. However, no crystallographic data supported the nucleation or growth of the deleterious intermetallic compounds commonly seen in Al-steel welds.

The VFAW impact weld between Al-alloy 6111-T6 and dual-phase steel JSC980 was also examined for intermetallic compound formation. This weld contained an intermediate shaded region between the two base materials at the interface which suggested the presence of potential intermetallic compounds. Upon further inspection, TKD and XEDS positively confirmed the presence of FeAl$_2$ and Fe$_3$Al$_5$. Texture analysis of the data shows simple shearing of the base materials within micrometers of the metallurgically bonded interface, supporting previous work that suggests that a shearing action causes the wavy feature at the interface of many impact welds. Nanoindentation tests were conducted at the weld interface, and the data confirm the hardness and modulus values of both the steel and aluminum alloy material.
Chapter 6: Future Work

Further analysis of these weld interfaces would provide a deeper understanding of the microstructure at the interface and throughout the weld regions.

More TEM work regarding the precipitates within the Ni-base alloy FSW to steel may provide information on the nature of their formation. Orientation relationships between the precipitates and the nickel matrix and a thorough understanding of the thermal cycle experienced by various weld regions can support this work. Additionally, thorough characterization of the precipitates closer to the shoulder region was not conducted, though there may be different microstructures at this region that can be confirmed using high-resolution techniques. For example, confirmation of the same precipitates observed by J. Rodriguez [8] in other areas beside the stir zone can be examined to understand the mechanism for varying nanohardness values across the joint.

Work for the Al-steel FSW should be done to create more foils and samples of the interface. Additionally, TEM analysis for these intermetallic compounds will be necessary. The thermal history, crystallographic information of the interface regions can provide insight on how these brittle, deleterious compounds form. Additionally, understanding the temperature and time effects on intermetallic compound
formation between these materials can provide understanding of how to avoid these intermetallics.

In the VFAW between aluminum alloy and carbon steel, optimizing the parameters can significantly reduce the intermetallic compound formation [90, 46, 44]. As reported by finite element and smoothed particle simulations, temperatures at the weld interface reach several times the melting point of aluminum, which may form the intermediate shade. However, work can also be done to understand the role of other alloying elements in the formation of intermetallic compounds, such as silicon or magnesium. Studies [21, 90] have investigated potential evidence that the presence of these alloys of the weld may increase susceptibility of intermetallic formation.
References


2012.


Appendix A: Material Phases and Composition

Figure 66. Phase Diagram of Ni-Based Alloy 625 [91]
Table 9. Phases and Precipitates Observed in Ni-Based Alloy 625 and ASTM A516 [20] [21]

<table>
<thead>
<tr>
<th>Phase</th>
<th>Structure</th>
<th>Lattice Parameter (nm)</th>
<th>Composition</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>γ</td>
<td>Cubic (FCC)</td>
<td>0.357-0.3607</td>
<td>Solid Solution</td>
<td>[92]</td>
</tr>
<tr>
<td>MC</td>
<td>Cubic</td>
<td>0.43-0.4449</td>
<td>(Nb) C</td>
<td>[93]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.43-0.4427</td>
<td>(Nb, Ti) C</td>
<td>[94]</td>
</tr>
<tr>
<td>M6C</td>
<td>Cubic</td>
<td>1.13</td>
<td>(Cr, Mo, Ni)₆C</td>
<td>[94]</td>
</tr>
<tr>
<td>M23C6</td>
<td>Cubic</td>
<td>1.08</td>
<td>Cr₂₃C₆</td>
<td>[93]</td>
</tr>
<tr>
<td>Laves</td>
<td>Hexagonal</td>
<td>0.47</td>
<td>(Cr, Ni)₂(Nb, Mo)</td>
<td>[93]</td>
</tr>
<tr>
<td>γ&quot;</td>
<td>Ordered Tetragonal</td>
<td>0.36</td>
<td>Ni₃(Nb, Ti, Al)</td>
<td>[93]</td>
</tr>
<tr>
<td>δ</td>
<td>Orthogonal</td>
<td>0.51</td>
<td>Ni₃Nb</td>
<td>[93]</td>
</tr>
<tr>
<td>(Cr, Nb)₂N</td>
<td>Tetragonal</td>
<td>0.3</td>
<td>(Cr, Nb)₂N</td>
<td>[93]</td>
</tr>
</tbody>
</table>

Table 10. XEDS Energy Peaks for Elements of Interest [95]

<table>
<thead>
<tr>
<th>Element</th>
<th>Symbol</th>
<th>Kα</th>
<th>Lα</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron</td>
<td>Fe</td>
<td>6.398</td>
<td>0.705</td>
</tr>
<tr>
<td>Nickel</td>
<td>Ni</td>
<td>7.471</td>
<td>0.851</td>
</tr>
<tr>
<td>Chromium</td>
<td>Cr</td>
<td>5.411</td>
<td>0.573</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>Mo</td>
<td>17.441</td>
<td>2.293</td>
</tr>
<tr>
<td>Niobium</td>
<td>Nb</td>
<td>16.581</td>
<td>2.166</td>
</tr>
<tr>
<td>Aluminum (TKD Holder)</td>
<td>Al</td>
<td>--</td>
<td>0.277</td>
</tr>
<tr>
<td>Carbon (TKD Holder)</td>
<td>C</td>
<td>--</td>
<td>1.486</td>
</tr>
</tbody>
</table>
Table 11. Al-Fe Intermetallic Compounds

<table>
<thead>
<tr>
<th>IMC</th>
<th>wt% Al</th>
<th>at% Al</th>
<th>Crystal structure</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe$_3$Al</td>
<td>17</td>
<td>25</td>
<td>Fm-3m; cubic</td>
<td>[20]</td>
</tr>
<tr>
<td>FeAl</td>
<td>25</td>
<td>45</td>
<td>Pm-3m (221); cubic</td>
<td>[21] [23]</td>
</tr>
<tr>
<td>FeAl$_2$</td>
<td>48</td>
<td>65</td>
<td>P-1; triclinic</td>
<td>[96] [22]</td>
</tr>
<tr>
<td>Fe$_2$Al$_3$</td>
<td>54</td>
<td>70</td>
<td>Cmcm (63); orthorhombic</td>
<td>[21] [25]</td>
</tr>
<tr>
<td>FeAl$_3$</td>
<td>60</td>
<td>73</td>
<td>C2/m (12); monoclinic</td>
<td>[21] [24]</td>
</tr>
</tbody>
</table>