Effects of processing parameters on the embrittlement of self reacting friction stir welds

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EFFECTS OF PROCESSING PARAMETERS ON THE EMBRITTLEMENT OF SELF REACTING FRICTION STIR WELDS

A Thesis
Submitted to the Graduate Faculty of the Louisiana State University and Agricultural and Mechanical College in partial fulfillment of the requirements for the degree of Master of Science

in

The Department of Mechanical Engineering

by
David Eric Taylor
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Abstract

Friction Stir Welding (FSW) has been adopted as one of the major welding processes for joining Aluminum. Many aerospace and marine structures are welded through this novel FSW processes currently. The Lockheed Martin Space Systems (LMSS), Michoud Operations, in New Orleans is continuously pursuing Friction Stir Welding technologies in its efforts to advance fabrication of the external tanks of the space shuttle. Recently, a reduction in mechanical strength (embrittlement) has been observed especially in self-reacting (SR) friction stir welds. This strength reduction was attributed to Residual Oxide Defects (ROD) but the exact reasons for this type of behavior needed to be investigated. NASA-Lockheed Martin provided the FSW samples of Aluminum 2195 and 2219 and is interested to find out the existence and consequences of ROD from these samples. The existence of ROD could compromise the structural integrity of the external tanks and could result catastrophic brittle failures. It is also found that certain FSW processing parameters would yield these reduced mechanical properties. The strength of FSW Aluminum panels generally decreases with increasing tool travel-rate, decreasing rotation speed, and offset of the weld seam to the retreating side of the FSW tool. The microstructure of welds exhibiting these strength reductions as well as welds that behaved as expected are examined to determine microstructural effects of processing parameters. Both SEM and TEM works have been conducted on these self-reacting FSW specimens provided by NASA. Scanning Electron Microscopy (SEM) shows that these weld conditions are accompanied by large
precipitates along the grain boundary for both Al-2219 and Al-2195. Transmission Electron Microscopy (TEM) also shows the precipitates to be θ-particles (Al₂Cu), and intermetallics (Al₇Cu₂Fe) in the Al-2219, and T₁ (Al₂CuLi) and T₈ (Al₇Cu₄Li) particles in the Al-2195. The large size and heavy non-linear distribution of these precipitates, especially on the advancing side of the weld-seam may influence these properties. There appears to be no signs of Residual Oxide Defects in the micrograph samples analyzed in this study. A more complete understanding of these phenomena is necessary to ensure consistent and predictable weld properties.
Chapter 1

Introduction

1.1 Reasons and Motivation for this Research

Performance demands on today’s materials are greater than ever. Aerospace applications require two very important aspects: high strength and low weight, while also dealing with extreme environmental factors including corrosion, impact, and extreme temperatures. Due to their high strength, low weight and ductility, aluminum alloys have found favor with the aerospace industry. The external tank of the space shuttle, in particular, relies extensively on aluminum to minimize the weight of the largest and heaviest (when loaded) component of the space shuttle system. The external tank has gone through many changes from the original tank which weighed approximately 76,000 lbs dry. [NASA, 2005] The lightweight tank was slightly redesigned to reduce weight to approximately 66,000 lbs. The use of aluminum 2195 helped to reduce the weight of the super light weight tank even further approximately 7000 lbs. The AL- 2195 is roughly 5% lighter and 30% to 40% stronger than the AL- 2219 it largely replaces. Because these alloys are difficult to arc-fusion weld due to inherent porosity problem during arc welding, Friction Stir welding is used instead. [NASA, 2001]

Conventional methods of welding aluminum have proven difficult due to aluminum’s relatively low melting point as well as the lack of a warning sign before melting temperatures are reached (aluminum does not glow red before it
melts such as ferrous alloys). Although Gas Metal Arc Welding (GMAW or MIG) was originally developed to weld aluminum, the welds produced by this method are still susceptible to porosity and dross. Gas Tungsten Arc Welding (GTAW or TIG) can also be a suitable method for welding aluminum but works best on thin sections of aluminum. GTAW is more complex and much slower than GMAW and is prone to many of the same defects as GMAW.

Friction stir welding is a solid state process invented at The Welding Institute (TWI, Cambridge, United Kingdom) in 1991. [Thomas et al, 1991] In the simplest manifestation of this process a rotating tool is driven along a joint line. Frictional heat is generated and material flow occurs to create the weld.

A schematic diagram illustrating the process is shown in Figure 1-1.

Figure 1-1: Diagram of Friction Stir Welding Process [ Wahab and Painter, 2007]
The two key features of the friction stir welding tool are:

(a) **The Shoulder.**

This is the primary means for generating heat during the process which is produced through a combination of material deformation and frictional slip. The shoulder also prevents expulsion of the material and assists the movement of material around the tool.

(b) **The Pin.**

The pin’s primary function is to deform the bulk material at the joint line and its secondary function is to generate heat. Usually the tool is inclined 2-3 degrees toward the direction of travel, although some later tool designs allow the tool to be positioned normal to the surface. This angle is called the tilt angle.

The material is consolidated behind the tool and a solid joint is formed from the high temperatures, pressures and material deformation. A successful weld is produced when the correct tool design and operating parameters are used for a given material. The main operating parameters of interest are the linear welding speed and tool rotational speed.

1.2 **Heat Flow during the Friction Stir Welding Process**

A diagram describing the sources of heat generation and the potential losses is shown in Figure 1-2.
There are three sources of heat generation identified, namely,

- Heat generated under the shoulder.
- Heat generated on the pin surface.
- Heat generated by shearing within the material.

Many authors have assumed that all the heat is generated at the shoulder, and have ignored the heat generated by the pin and that from shearing of the material in the weld nugget.

A reduction in mechanical properties was observed in Friction Stir Welded Aluminum panels which were observed, initially, by Lockheed-Martin at their New Orleans Lab. The representative samples were provided for this research by Lockheed-Martin. This reduction in strength has initially been attributed to Residual Oxide Defects (ROD). When the Aluminum panel is exposed to air, prior to FS welding, an oxide layer on the surface could form;
and if these oxide layers are not broken down sufficiently during welding, then the oxide layer acts as a weak point in the specimen which is basically locked up within the specimen. This specific problem may be attributed to self-reacting FSW procedure. In the self-reacting FSW, the back pressure is introduced while the rotating pin-tool moves along the direction of welding, thereby producing a uniform FSW welding but there may be possibility of oxide layer may not have been broken down to a level that will make the specimen free of oxide layers.

Many of the problems associated with cooling from the liquid phase are avoided with friction stir welding because it is a solid state process. While this welding process typically produces welds which are as strong and ductile as the parent material, certain welding parameters have been found to produce a sharp decrease in strength and ductility in the welds. Particularly, it has been observed that rpm, feed rate, and tool offset are the main factors which produce weld embrittlement. This problem has been attributed to a retained or residual oxide defect, caused by incomplete breakup of the oxide layer during welding, but the exact cause are not known and needed to be determined. Although research has previously been conducted on the FSW behavior of aluminum alloys, very little work has been done on inhomogeneous welds. Studies involving self reacting pin tools and their effects on microstructure are also very limited in number due to the recent development of this technology. For these reasons the current research was designed with a number of objectives in mind.
Chapter 2

Objectives

Louisiana State University was tasked with studying the microstructure of friction stir welds provided by Lockheed Martin Space Systems to determine factors which may influence embrittlement attributed to Residual oxide defects. Through experimentation Lockheed Martin determined the processing parameters that most influenced the formation of the brittle welds. Samples representing the parameters most likely to cause embrittlement were provided to LSU for examination. For comparison purposes, samples were also provided which had been welded using parameters that would be most likely to cause good welds. In addition samples were provided of traditional butt and lap welds for comparison. Fractured brittle samples were provided which were welded using worst case parameters for fracture surface study.

Emphasis was given to the microstructure in the interfacial area of the samples. The lap and butt welded samples in particular were included for comparison purposes with the Self Reacting samples. We were also to identify the presence of any residual oxide layer that may be present.

It is expected we will identify a residual oxide layer as well as other microstructural effects of the brittle processing parameters. With this information we should be able to provide input to help reduce the likelihood of failures caused by this phenomenon.
2.1 Scope

Chapter 3 includes a review of literature pertaining to the current study. This includes literature involving the current state of Friction stir welding as well as studies relating to the aluminum alloys involved in this study. Particular attention was given to studies involving friction stir welding of AA 2219 and AA 2195. Chapter 4 contains the experimental procedure followed to conduct this research with particular attention to sample preparation for microstructural studies. Chapter 5 covers the results of the study as well as analysis and discussion of said results. Chapter 6 includes the conclusions drawn from this study as well as recommendations for future work.
Chapter 3

Literature Review

3.1 Development of Friction Stir Welding

Friction Stir Welding (FSW) was developed by The Welding Institute (TWI) Cambridge, UK for joining metals using a solid state process [Thomas et al, 1991]. A rotating welding tool is used to generate deformation and frictional heat to form a solid state joint between two workpieces. Because it is a solid state process melt related defects can be avoided as well as low distortion versus other joining techniques. Because of the lower temperatures involved and the lack of filler related defects, FSW can be used to weld metals that are otherwise difficult or impossible to weld. FSW yields high joint strength and low concentrations of hydrogen which is a benefit when joining metals susceptible to hydrogen cracking.

As a joining technique, FSW is quite robust. It can be used to weld dissimilar metals and has successfully been used to weld steels, aluminum alloys, titanium, copper, and magnesium alloys. Different variations of joints can be performed using FSW such as butt, lap, and spot welds among others. Butt welding aluminum alloys has received much of the focus. Butt welding materials of different thickness as well as tapered sections can also be performed. FSW can be used in some applications to reduce weight by replacing fasteners and reducing part count, which can actually reduce costs.
This is particularly important in the aerospace industry where weight is of critical importance.

It is necessary first to discuss the convention used for referring to locations within friction stir welds. Since friction stir welds are asymmetrical, it is necessary to accurately convey which side is intended when referring to specific locations within a weld with respect to the tool rotation and feed directions. The following convention will be used in the discussions to follow. The side of the welding tool where the motion of the surface of the welding tool is in the same direction as the feed direction is referred to as the \textit{advancing side}. The opposite side, where the motion of the surface of the welding tool opposes the feed direction, is referred to as the \textit{retreating side}. A terminology convention that is also used refers to the advancing and retreating sides as the shear side and the flow side, but since this convention makes assumptions about the material flow, the more generic terminology will be used here. Another convention used here is to refer to tool movement in indicating the feed direction, as opposed to workpiece movement. Figure A shows schematically the layout of a typical butt weld along with the advancing and retreating sides of the weld. This is the convention used by Colligan [Colligan, 1999] and others.

Other terms that are used to describe friction stir welds include joint profile, which refers to the outermost boundary between the welded area and the base metal. Also when talking about standard butt joints face, root and toe are often used to describe sections of the weld as depicted in figure 3-1. Overmatching and undermatching refer to welds that are stronger or weaker, respectively, to the base material. This can also be related to the joint
efficiency, which is defined as the ultimate tensile strength of the joint over the ultimate tensile strength of the base metal. Penetration ligament is used with butt welds to describe the distance from the tip of the pin to the bottom of the work piece also called the root tip or lack of penetration [Ding and Oelgoetz, 1999]. Sometimes the tool is angled slightly which causes the shoulder to penetrate the workpiece commonly referred to as shoulder plunge [Cederqvist and Reynolds, 2000].

![Figure 3-1: Root, Toe and Face of Friction Stir Weld](image)

3.2 Types of Friction Stir Welding Joints

3.2.1 Butt Joints

Butt welding involves two workpieces clamped to a backing plate with the mating edges in contact. A tool with a shoulder and pin is rotated and plunged into the mating line (figure 3-2). It is then fed along the mating line at a specific feed rate (or travel rate). On most occasions the tool is angled slightly with respect to the workpieces. Material is moved from in front of the tool
(leading edge) to behind it (training edge). This type of weld is susceptible to a lack of penetration defect due to distance between the bottom of the pin tool and the backing plate. This is potentially a site for corrosion or failure. Kissing bonds can also occur in butt joints. Oosterkamp defines a kissing bond as “two surfaces lying extremely close together, but not close enough for the majority of the original surface asperities to have deformed sufficiently to contact for atomic bonds to be created.” They concluded kissing bonds occur in friction stir welds when “aluminum in the shear zone is sliding over the pin surface.” [Oosterkamp et al. 2000]

Figure 3-2: Schematic Showing Key Concepts of Friction Stir Welding

Deqing et al. studied the relationship between the pin diameter to shoulder diameter ratio and the quality of welds. They found best welds were performed when the pin to shoulder ratio was about 1:3. They also found a strong correlation between travel rate and weld quality with best welds produced
when the rotation to travel rate ratio was between 14 and 23:1. [Deqing et al., 2004]

3.2.2 Lap Joints

In a lap joint, overlapping workpieces are welded together by the pin tool penetrating the top workpiece completely and partially penetrating the bottom workpiece. Lap welds require the stirring action to be more out of plane than butt welds. Many tools specifically designed for lap welding have a second shoulder that is located at the interface between the two workpieces being welded as described by Brooker et al [Brooker et al., 2000]. The interface lines on either side of the weld are potential sites for corrosion and failure.

3.3 Joint Profile

The Welding Institute (TWI) proposes a generalized joint profile shown in figure 3-3. The area farthest from the central nugget is the unaffected base material. The area depicted in part B in Figure 3-3 is the heat affected zone (HAZ). The HAZ does not experience any plastic deformation but is affected by the heat generated during welding. The microstructure in the HAZ is affected by this heat. The area depicted in part C is the thermo mechanically affected zone (TMAZ). The material within the TMAZ is mechanically affected by the weld tool as well as the heat generated during welding. The final area represented in part D in Figure 3-3 is the weld nugget or dynamically recrystallized zone (DXZ) [TWI, 2008].
Figure 3-3: Key metallographic areas of conventional butt FSW including unaffected material (A), Heat affected Zone (B), Thermo mechanically affected zone (C), and weld nugget or dynamic recrystallized zone [TWI, 2008]

3.4 Welding Parameters

The most widely disclosed parameters in friction stir welding are rotational speed, travel speed, normal force. Other parameters mentioned can include tool attitude (tilt angle), shoulder plunge, and tool offset. Slower travel speeds and rotation speeds are generally used for harder alloys or very thick sections. Increasing rotational speeds and decreasing travel speeds results in increased welding temperatures. Increasing travel speeds also has the effect of decreasing the time required to perform the weld and as such finding the fastest travel speed that will result in an acceptable weld is often desirable. All of the processing parameters should be optimized for the particular welding conditions including material type, material thickness, and required joint strength [Colligan, 1999].
3.5 **Friction Stir Weld Tool Design**

Simple one-piece steel tools were used in the beginning of FSW. These included a pin shaped as a simple cylinder that limited material flow and mixing and required slow welding speeds. As tool design progressed, threaded pins were used to increase mixing and increase welding speed as well as produce better quality welds. Scrolled shoulders were developed to reduce the need for tilting the tool enabling welding around corners. TWI introduced the frustum shaped pin and the use of grooves to improve joint quality in thick sections [TWI, 2008]. The self-reacting tool was developed to remove lack of penetration defects in butt welds as well as increase mixing [Colligan et al., 2003]. Some of the different tool designs are represented schematically in figure 3-4.

![Figure 3-4: Rough Drawing of Different Tool Designs](image)

Other types of welds and tools are also used in Friction stir welding. Spot welds can be performed and involve no travel of the pin tool. Because of the need to execute circumferential welds, work has been done to eliminate the
keyhole left during standard friction stir welding. Welding tapered sections and complex shapes have also lead to the development of advanced tools for FSW. Two tools to address these issues have been developed by NASA and others, the self reacting pin tool and the retractable pin tool (RPT).[Ding and Oelgoetz, 1999][Colligan et al., 2003]

NASA developed the RPT to automatically retract the pin at the end of the welding pass so they could close the keyhole. This tool allowed them to execute circumferential welds and butt welds on tapered sections [Ding and Oelgoetz, 1999]. The concept for the self reacting tool was introduced in the original TWI patent but was first demonstrated by Boeing. The Self reacting tool consists of two pieces, one on each side of the work piece. The pieces are rigidly connected and rotate in the same direction applying a clamping force on the work piece. [Colligan et al., 2003]

3.6 Material Flow in Friction Stir Welding

Colligan did a study to document the movement of material during FSW to model the deformation process. Colligan's experiments used 6061 and 7075 aluminum in a butt-welded configuration. Two methods of visualization are presented in the paper. The two methods depend on where in the weld the material originates. The two methods are simple extrusion and chaotic mixing. Two techniques were used to help visualize the flow. Small steel balls were used as tracers embedded at different positions. The weld was interrupted during the weld to show the distribution of the weld effectively showing the path
of the material. Radiography was used to reveal the path of the tracer around the weld tool. Tracers were embedded by machining a small groove into the side of the workpieces. The second technique used to view the flow of material was to stop the welding process while simultaneously raising the pin tool out of the workpiece leaving the material within the threads of the tool. The material was then sectioned and studied to see the flow of material immediately within the threads of the tool. 7075 required higher welding forces than the 6061, which caused problems with the stop motion method. The results of the tracer study showed increased scatter of the tracer material when the tool was positioned so that the tracer was on the advancing side of the tool. It also showed that not only did the tracer material travel more in the lateral plane of the work piece but also in the vertical direction. Colligan determined that when the tracer was deposited in a roughly continuous line behind the pin tool the material was simply extruded around the pin tool without mixing. Adjacent elements of material are deformed but remain adjacent to each other. The butting surfaces of the two pieces form a heat transfer barrier because of incomplete contact [Carter, 2003].

Seidel and Reynolds also used the marker insert technique to help visualize material flow in Friction Stir welds of AA 2195. Using markers placed at various depths on both the advancing and retreating side they found the material flow was not symmetrical between the advancing and retreating sides of the welds. Further, they found that markers placed on the advancing side of the weld ended up in a position behind its original position. Also, no material
was moved farther than one pin diameter back from its original position. [Seidel and Reynolds, 2001]

Donath et al. used titanium powder embedded in friction stir welds to study flow during welding. Computed microtomography and stop action technique were combined to visualize material flow with different positions and tool geometry. They found this method to be ideal for investigating material flow. [Beckman et al., 2004]

3.7 Joint Microstructures

The temperature generated during welding is a critical factor in determining microstructure of the weld. Most data available about temperatures in friction stir welding are gained from computer models. Many models compromise on the assumption that the temperature profile is symmetrical on the advancing and retreating side. The advancing and retreating sides of the weld can be quite different. Maeda et al. found that neither side is necessarily hotter all of the time, but rather it depends on the other welding conditions. The general tendency is for temperatures to increase with increasing rotational speed [Maeda et al., 2005]. Chao et al studied heat transfer in friction stir welded AA 2195 panels and found that temperature increases with decreasing travel speed as well as decreasing work piece thickness [Chao et al., 2003].

The evolution of the grain structure during FSW has been studied using stop action technique. This involves suddenly stopping the tool mid weld and observing the material around the tool. Pragnell and Heason found that high
angle grain boundary spacing is reduced by the geometric effect of strain and the grain refinement process is driven by grain subdivision. They found no evidence of continuous dynamic recrystallization by subgrain rotation. High temperature grain boundary migration was found to closely resemble geometric dynamic recrystallization. [Pragnell and Heason, 2005]

The effects of welding parameters on the microstructure of friction stir welds has previously been studied by Babu et al. They studied friction stir welds of AA 6082. They found a direct relationship between the travel speed and the tensile strength of the weld. They used rotation speeds between 460 rpm and 1700 rpm. They combined these rotation speeds with travel speeds between 115 mm/min and 585 mm/min. The high rotational speed and travel rate caused a tunnel defect that they found could be avoided by optimizing rotation and travel speed at 1230 rpm and 115-170 mm/min. [Babu et al., 2008]

Sato et al. studied oxide defects in FSW Al 5052-O. Welds were examined using Focused Ion Beam (FIB) and TEM. They were able to clearly identify oxide particles. [Sato et al., 2002]

3.8 Friction Stir Welding of Aluminum Alloys

3.8.1 FSW of Aluminum Alloy 2195

Colligan et al. investigated the relationship between the operating speeds and mechanical properties. Tool rotation was varied between 200 and 230 rpm. Travel speed varied from 1.2 and 3.7 in./min. The authors found the yield strength increased with travel speed regardless of rotation rate. They also
reported that most samples fractured on the retreating side of the weld. The exceptions were samples welded at the lowest and highest travel rates. Samples welded at the extreme travel rates with tool rotation at 200rpm failed along the weld face. They also found that a hardness gradient exists across the weld profile and across the depth of the joint. [Colligan et al., 2001]

Schneider and Nunes studied material flow with 2195 plate 0.323 in. thick. Welds were performed at 200rpm and 6 in./min in the rolling direction. Schneider and Nunes found the primary strengthening phase T₁ precipitates to be larger in the weld nugget zone than in the parent material. They also found that strengthening precipitates in the parent material over aged in the TMAZ and HAZ [Scneider and Nunes, 2004].

Studies were performed by Litwinski to examine the effect of travel speed on the tensile properties of FSW samples. Samples were naturally and artificially aged for various times from one hour to over two and a half years. Artificial aging was found to increase strength while sacrificing elongation. The longest natural aging at 2.5 years resulted in higher strength and elongation than the natural aging for shorter times. Increasing travel speed was also found to increase ultimate and yield strengths in all types of aging. [Litwinski, 2005]

Oertelt et al. have studied microstructure and hardness distribution on FSW 2195. Using a travel speed of 3.75 in/min. and rotation speed of 200 – 250 rpm, they found that some precipitation took place during FSW. The DXZ displayed fine equiaxed grains with supersaturation and the TMAZ showed elongated grains [Oertelt et al., 2001].
Due to the extensive use of AA 2195 in the external tank of the space shuttle, extensive knowledge of the tensile properties and welding properties are necessary. For this reason Chaturvedi et. Al. studied the effect of specimen orientation on Fracture and fatigue properties of AA 2195 when welded using Gas Tungsten Arc Welding (GTAW). They identified the primary strengthening precipitates as T₁ (Al₂CuLi), which was, replaced post weld with T_B (Al₇Cu₄Li). They found a brass type texture in the T8 base alloy with primary strengthening by T₁ precipitates. AA 4043 was used as a filler material for the welds. Properties of the materials were generally reduced after welding with welding at 45 degrees to the rolling direction yielding the greatest reduction. SEM was used to examine the fracture surfaces. The post weld Fusion zone contained primarily T₁ phase with the HAZ containing T_B phase.

Aluminum 2195 was of particular interest in this study because of its potential use in the next generation of space shuttle. The study also aimed to fill holes in current knowledge of the alloy, particularly in the areas of fracture and fatigue behavior. Gas Tungsten Arc Welding was used with AA 4043 filler. SEM, TEM, and light microscope were used to examine microstructures among other methods. Specimens ranging from 0 to 90 degrees in 15 degree increments with respect to the rolling direction were examined. Samples were polished with 600 grit sand paper and examined with SEM after fatigue testing.

The fusion zone consisted mostly of T₁ phase particles while the heat-affected zone saw the T₁ phase dissolve and be replaced by T_B phase. Micro cracks also formed along the grain boundaries. The dissolution of the T₁ phase
is likely due to the high peak temperatures which lead to liqation and possibly the micro cracking. The samples were post weld heat-treated which resulted in spheroidization of the T₁ phase and the dissolution of T₈ with no affect on micro cracking.

The samples were found to have the lowest fatigue strength when welding was performed at 45 degrees with respect to the rolling direction. The highest yield strength occurred when welding followed the rolling direction. Shear steps were evident in the fracture surfaces upon SEM examination. Cleavage cracking was exhibited in the welded materials while micro void coalescence was present in the post weld heat-treated samples. The base T₈ alloy exhibited fatigue cracks that initiated at the specimen surface and showed fatigue striations. These were absent in the welded samples both PWHT and otherwise. Welded samples showed crack initiation at the defects with cleavage crack propagation.

The fracture surface examinations done by Chatavurdi et al. are particularly useful in the present research. The identification of fracture properties of AA 2195 is of particular interest. There was however no examination of friction stir welding or of multiple aluminum alloys which this research examines [Chaturvedi and Chen, 2004].

**3.8.2 FSW of Aluminum Alloy 2219**

Cao and Kou used AA 2219 to study the possibility of liqation in FSW. Previous studies had shown that temperatures could possibly reach the lower
bounds of melting temperatures for alloys such as 6061, 7030 and 7075. They proposed the θ particles in AA 2219 would act as “in-situ micro sensors” for liquation. The 548°C eutectic temperature was suitable for the study and friction stir welded samples were compared against gas metal arc welded samples to provide a benchmark (figure 3-5). They found no evidence of liquation but did observe significant agglomeration in the samples as shown in figure 3-6. In AA 2219 liquation can occur under both equilibrium and non-equilibrium conditions which means it is independent of heating rate. Cao and Kou further found that θ particles could reach sizes of 100 µm during friction stir welding [Cao and Kou, 2005].
Figure 3-5 Shows the GMAW Sample, Which Displays Liqueation. [Cao and Kou, 2005]
Figure 3-6 The Friction Stir Welded Sample That Shows No Evidence of Liquation, but Shows Significant Agglomeration. [Cao and Kou, 2005]
Kostrivas and Lippold also studied melting in aluminum alloys including AA 2219 and AA 2195 when welded with conventional methods. They reported the solidus temperatures at 543 °C and 540 °C respectively for the two alloys. Liquidus temperatures were reported at 643 °C for AA 2219 and 640 °C for AA 2195 [Kostrivas and Lippold, 2004]

3.8.3 FSW of Dissimilar Aluminum Alloys

Larsson et al. studied the microstructure of friction stir welded dissimilar aluminum alloys common to the shipping industry. AA 5083 is heavily used in shipbuilding for its resistance to corrosion in seawater. AA 6082 is used for extrusions and is chosen for its hardenability. MIG welding is typically used to join the metals but presents two distinct disadvantages. The base metal has a tendency to deform and the heat affected zone experiences a decrease in strength. These are similar weaknesses to those of TIG welding and other fusion welding techniques. Multiple orientations were used with some with the AA5083 on the advancing side and some with AA 6082 on the advancing side. The authors also tested the use of a consumable strip in between the two metals which they referred to as a strip-joint. The authors found that the onion ring patterns consisted of alternating bands of the two alloys and no intermediate compositions. They concluded that the material with the lower hot strength should be placed on the advancing side and should be used as the consumable strip. They also found that higher travel speeds produced better welds than lower travel speeds [Larrson et al., 2000]. Others have performed similar
studies such as Vural et al. who studied welding of EN AW 2024 and EN AW 5754. They found the welds of the two aluminum alloys to be particularly sensitive to welding parameters. [Vural et al., 2007]

Studies have also been performed to study the possibility of welding Aluminum with other metals and alloys. Jiang and Kovacevic studied welding Aluminum 6061 with AISI 1018 steel. Their welds were performed with the aluminum on the advancing side and steel on the retreating side. They found that acceptable welds could be performed but that melting occurred in the aluminum. [Jiang and Kovacevic, 2004]
Chapter 4
Experimental Procedures

4.1 Introduction

This chapter deals with the materials involved in this study as well as the procedures necessary to duplicate the results obtained here. The tool design and the exact welding parameters used to complete the friction stir welds were considered proprietary information and as such were not disclosed to LSU. In these cases the information that was provided was used to give approximate values or present a general example to illustrate the ideas. Approximate welding parameters for self reacting welds is presented as well as an example of a left hand right hand self reacting tool which is similar to the one used by Lockheed Martin in the preparation of the samples. The sample preparation procedure followed in this study is also presented including polishing and etching. Finally procedures and equipment used for SEM and TEM examination are also presented.

4.2 Materials

Aluminum Alloy 2219 and 2195 was supplied by Lockheed Martin Space Systems in welded plates of 0.584 cm (0.23 in) with typical composition shown in Table 4-1. AA 2219 has been in use on the space shuttle since its inception in the early 1980’s [NASA, 2005]. In an effort to reduce weight, AA 2195 was developed to replace much of the AA 2219 in use on the space shuttle.
[NASA, 2001] Because both alloys are still used, welds were provided with multiple combinations of the two alloys welded together.

Table 4-1: Typical Weight % Composition of AA 2219 and 2195

<table>
<thead>
<tr>
<th></th>
<th>AA 2219</th>
<th>AA 2195</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>92.57</td>
<td>93.9</td>
</tr>
<tr>
<td>Cu</td>
<td>6.3</td>
<td>4</td>
</tr>
<tr>
<td>Li</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>Si</td>
<td>0.2</td>
<td>0.03</td>
</tr>
<tr>
<td>Fe</td>
<td>0.3</td>
<td>0.05</td>
</tr>
<tr>
<td>Mn</td>
<td>0.3</td>
<td>0.05</td>
</tr>
<tr>
<td>Mg</td>
<td>0.02</td>
<td>0.4</td>
</tr>
<tr>
<td>Zn</td>
<td>0.1</td>
<td>0.01</td>
</tr>
<tr>
<td>Ti</td>
<td>0.06</td>
<td>0.02</td>
</tr>
<tr>
<td>Zr</td>
<td>0.15</td>
<td>0.14</td>
</tr>
<tr>
<td>Ag</td>
<td></td>
<td>0.4</td>
</tr>
</tbody>
</table>

4.3 Joining Procedures

Various types of joints are encountered in the design of the external tank of the space shuttle. Because of the unique nature of the joints, different FSW procedures are used. Lap welding and conventional butt welding have been in use for over ten years and are fairly well understood. Self reacting welds are fairly new and not well understood in terms of heat transfer, and material flow as well as microstructural evolution. Because of the lack of research on self reacting welds, lap and conventional butt welds were provided for comparison purposes.
4.3.1 Lap Welding

Lap welding involves welding pieces that overlap as shown in figure 4-1. This type of welding is primarily used by Lockheed Martin to weld parts or bracing onto the main structure of the external tank. The main components necessary for Lap welding are the shoulder and pin which are similar to those used in conventional butt joint friction stir welding. The key differences from the tools used for butt joints are the length and shape of the pin. For lap welds the pin must be long enough to penetrate the top workpiece completely and penetrate deep enough into the bottom workpiece to allow sufficient material movement. Similarly the pin tool used for lap welding has to be designed to provide significant vertical movement of material to achieve adequate mixing of the two pieces. Lap welds for this study were provided by Lockheed Martin space systems and were provided for comparison purposes to the Embrittled welds. All Lap welds provided were welded with the AA2219 on top, which is the side the shoulder passed over. Welding parameters and tool details were not provided by Lockheed Martin.
4.3.2 Butt Welding

Butt welding involves two workpieces joined end to end. The Welding tool passes along the interface between the two workpieces as shown in figure 4-2. This type of welding lends itself to a lack of penetration defect which is often the source of failures and corrosion. This occurs due to the pin tool not completely penetrating the workpiece. Situations where two surfaces need to be joined together end to end are candidates for conventional butt welding provided the materials are close to the same thickness. The need for a backing plate as well as other issues make welding cylindrical shapes with this process difficult. Limitations have caused this process to largely be replaced by self reacting welds on the external tank of the space shuttle. Butt welds for this study were provided by Lockheed Martin space systems and were provided for comparison.
purposes to the Embrittled welds. All butt welds provided were welded with the AA2219 on the advancing side and the AA2195 on the retreating side.

![Figure 4-2: Diagram of Butt Weld Performed with FSW](image)

4.3.3 Self Reacting Friction Stir Welding

Self Reacting welds are similar to conventional butt welds since the workpieces lie end to end when they are welded. Unlike butt welds however the use of a backing anvil is not necessary. Instead there is a shoulder on both sides of the workpieces and the pin passes completely through the pieces between the shoulders as shown in figure 4-3. These two shoulders provide a clamping force on the workpieces. The key advantages of this process are the ability to weld complex shapes and the improved material flow created by the second shoulder.
Figure 4-3: Close up of Self Reacting Friction Stir Welding Tool [31]

All samples were welded by Lockheed Martin Space Systems Michoud. As part of a project to identify causes of “Residual Oxide Defect” failures Lockheed identified certain parameters which increased the likelihood of ROD type failures. Samples were provided to LSU for metallographic examination. Samples provided were welded using parameters that were found to create good welds and samples welding using ROD parameters. The tool used to perform the weld was a left hand/right hand pin tool shown in picture 4-4. It should be noted that the tool shown in picture 4-4 is simply a representative tool as LSU was not given access to the actual tool used to perform the welds.
Zach Loftis of Lockheed Martin used a Design of Experiments approach to determine the factors that affect the presence of ROD type failure. He found that Tool Offset was the most important factor in the presence of ROD. Tool offset is illustrated in Figure 4-2. Other major factors influencing the presence of ROD were Travel rate and RPM. In general ROD failure was more likely with increasing travel and decreasing RPM.

With this knowledge Lockheed Martin provided LSU with samples welded with approximate conditions shown in Table 4-2. Tool clamping load was 3000 to 4000 pounds.
4.4 Characterization

4.4.1 Optical Sample Preparation

Aluminum samples were sectioned and polished using SiC paper ranging from 240 to 800 grit. 1-µm deagglomerated alpha alumina. The exact sequence of polishing is presented in Table 4-3

Table 4-3: Grinding and Polishing Sequence

<table>
<thead>
<tr>
<th>Abrasive and size</th>
<th>Brittle samples</th>
<th>Ductile Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC 240 grit</td>
<td>0.125</td>
<td>-0.125</td>
</tr>
<tr>
<td>SiC 320 grit</td>
<td>14 ipm</td>
<td>10 ipm</td>
</tr>
<tr>
<td>SiC 400 grit</td>
<td>180</td>
<td>160</td>
</tr>
<tr>
<td>SiC 600 grit</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SiC 800 grit</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1 µm alumina</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

All samples were ground with water as the lubricant. Keller’s reagent (table 4-4) was used to etch the specimens and reveal the grain structure.

Table 4-4: Keller’s Reagent

<table>
<thead>
<tr>
<th>Keller’s</th>
</tr>
</thead>
</table>
4.4.2 SEM

Samples for SEM analysis were sectioned and polished following the sequence outlined in table 4-3. Microstructures were examined using a Hitachi S-3600N extra-large chamber variable pressure Scanning Electron Microscope (VP-SEM). SEM micrographs were taken of all samples including fracture surfaces of “brittle” samples.

Figure 4-6: Hitachi S-3600N Extra-Large Chamber VP-SEM

4.4.3 TEM
Thin sections were taken from the welded samples and polished until a thin foil could be produced. Foils for transmission electron microscopy were prepared using a Gatan Model 656 Dimple Grinder (Figure 4-7) and Gatan Model 691 Precision Ion Polishing System (PIPS) (Figure 4-8).

Figure 4-7: Gatan Model 656 Dimple Grinder

Figure 4-8: Gatan Model 691 Precision Ion Polishing System (PIPS)
The foils were examined using a JEOL 2010 High-resolution Transmission Electron Microscope (HRTEM). Bright Field and Dark Field microscopy was used to characterize the microstructure of the alloys and to identify precipitates in the samples.

Figure 4-9: JEOL 2010 High-Resolution Transmission Electron Microscope

The experimental steps to prepare ion-milled TEM sample:

1. Punch a 3 mm diameter disc from the thin foil sample in the area to be examined.
2. Place Pyrex with a small piece of low melting wax on the top on a hot plate. When the wax melts, place the sample disc onto the Pyrex where the wax is located, remove the Pyrex from the hot plate. The disc is mounted on the Pyrex after the wax is cured.
3. Place the Pyrex in a Disc Grinder to carry out mechanical polishing on sand paper or diamond papers with different grain size. A mirror-finished surface has to be obtained after the final stage of polishing.
4. Remove the disc from the Pyrex by melting wax on the hot plate and clean the sample in acetone to remove the wax.
5. Mount the polished side of the disc down onto the Pyrex using low melting point wax and mechanically polish the sample on 600 grit sand paper to reduce the thickness of samples to about 100 µm. A flat surface should be produced at this stage.
6. Place the Pyrex on the precision dimple grinder and polish the sample using Cu wheel and fine diamond paste until the sample thins to about 15 µm.
7. Polish the sample using Felt polishing ring and alumina polishing suspension to obtain a mirror finish surface.
8. Remove the sample from the Pyrex.
9. Ion-mil the sample (PIPS, 4.5 kV, 4-5°).
Chapter 5
Results and Discussion

5.1 Introduction

A reduction in mechanical properties was observed in Friction Stir Welded (FSW) Aluminum panels welded by Lockheed Martin Space Systems. This reduction in strength was attributed to Residual Oxide Defect (ROD). It was also found that certain processing parameters would yield these reduced mechanical properties. The probability of brittle or weak FSW panels generally increases with increasing tool travel rate, decreasing rotation speed, and offset of the weld seam to the retreating side of the FSW tool. The microstructure of welds exhibiting these strength reductions as well as welds that behaved as expected were examined to determine microstructural effects of processing parameters. For comparison purposes samples were also examined which had been welded using conventional butt-welding and Lap welding. Scanning Electron Microscopy (SEM) shows that these weld conditions are accompanied by large precipitates along the grain boundary for both Al 2219 and Al 2195. Transmission Electron Microscopy (TEM) was used to identify the precipitates as θ (AlCu) and intermetallics (AlCuFe) in the Al 2219 and T1 (AlCuLi) and TB (AlCuLi) particles in the Al 2195. SEM and TEM examination showed no significant residual oxide layer. Comparisons to conventional butt welded samples and lap welded samples showed the same precipitates as found in the self reacting samples but without the large sizes obtained in the self reacting samples.
5.2 Butt Welded 2219/2195

Scanning Electron Microscopy was used to examine traditional butt-welded samples first to offer a baseline with a well understood traditional friction stir weld. SEM observations on cross sections showed a rather dispersed interface represented in figure 5-1. The post weld interface was fairly easy to identify in the Butt-welded samples with heavy precipitates on the 2219 side of the interface.

Fig. 5-1: (a) Schematic Representation of the Cross Section of the Butt FSW 2219/2195. (b) SEM Micrograph Showing an Interface Region
Since the conventional butt welds were provided for comparison to the self-reacting samples, SEM micrographs were taken to determine the size of the precipitates. The size of intermetallic particles varied along the material interface with some particles in the size range of 10 µm (Figure 5-2).

Figure 5-2: Close Up SEM Image of Intermetallic Particle Taken near Interface of Butt Welded AA2219/AA2195 FSW Sample.
The SEM images showed a clear post weld interface and a large number of precipitates on the AA 2219 side of the interface. This corresponds with the advancing side of the weld. Transmission Electron Microscopy was used to identify the precipitates. The composition of the particulates is determined with Energy Dispersive Spectrometry (EDS), which is particularly effective with TEM samples due to the thin specimens. The post weld interface was clearly visible again in the TEM images. In agreement with the SEM observations, TEM analysis revealed a heavier precipitate/particle presence in the 2219 side of the interface as shown in Fig. 5-3.

Figure 5-3: Bright Field TEM at the 2219/2195 Interface Showing Heavier Precipitate/Particle Presence in the 2219 Side.
TEM examination shows large particles on the 2219 side of the interface, Fig. 5-4. These large particles were identified as intermetallics (Al$_7$Cu$_2$Fe) by using EDS spectra and Selected Area Electron Diffraction (SAED sometimes referred to as Selected Area Diffraction SAD). SAD is used to generate a Selected Area Diffraction Pattern (SADP) that is used to determine crystalline structure. By obtaining the lattice parameters the crystalline structure can be determined. The intermetallic particles were identified to have a tetragonal space group P4/mnc structure with $a=6.33$ Å and $c=14.81$ Å.

![EDS spectrum and SAD pattern](image)

**Figure 5-4:** Bright Field TEM at the 2219 Side of the Interface Showing Large Intermetallic Particles. EDS Spectra and SAD Patterns Show Particle Composition and Structure, Respectively.
Once the large particles were identified and confirmed by examining a few examples, TEM was further used to determine the composition of the smaller precipitates. EDS was used to identify the smaller precipitates as $\theta$ (Al$_2$Cu) particles (figure 5-5). Bright Field ED pattern analysis of the $\theta$ particles showed they have a body-centered tetragonal structure with $a=6.05\text{Å}$ and $c=4.86\text{Å}$. This clearly identified precipitates on the 2219 side as intermetallics or $\theta$ particles.

Figure 5-5: Particles Identified as $\theta$ (Al$_2$Cu) along with Corresponding EDS Spectra and ED Pattern.
5.3 Lap Welded 2219/2195

SEM examination of Lap welded samples showed results similar to the previous observations of the butt welded samples. Figure 5-6 (a) shows an overview of the post weld interface and the general. The interface was clearly visible and marked by heavier precipitation in the 2219 side as shown in Figure 5-6 (b).

Figure 5-6: (a) Schematic Representation of the Cross Section of Lap FSW 2219/2195 and (b) SEM Micrograph Showing Interface.
Close examination of the post-weld interface using SEM shows fairly small precipitate particles (figure 5-7). These precipitates are on the order of 5μm, which is significantly smaller than those found in the conventional butt welded samples.

Figure 5-7: SEM Image of Lap Welded FSW AA2219/AA2195 Showing Clear Interface Between the Two Alloys.

It is unclear if the smaller precipitate size is a result of the inherently weaker mixing that occurs in lap friction stir welds. Without much information on the parameters used to create the welds the cause of the different precipitate size cannot reasonably be determined. TEM examination was not performed on the lap-welded samples.
5.4 Self Reacting Friction Stir Welds

5.4.1 Ductile Sample

SEM observations were made on SRFSW cross sections. The interface was visible but it was dispersed as shown in the schematic of Fig. 5-8. Fracture occurred on the advancing side of the weld in the 2219 material as represented by the dotted line in figure 5-8. The dispersed interface indicates significant mixing occurred at the interface of the two alloys.

![Fig. 5-8 Schematic Representation of the SRFSW Interface Observed in the Panels with Negative Offset. Dotted Line Indicates Location of Fracture.](image)

A photograph of the fractured ductile weld is shown in Figure 5-9. The weld fractured on the advancing side of the weld in the Heat affected zone and showed slight necking around the fracture location.
SEM observations showed a dispersed 2219/2195 interface with a lower density of Cu-rich particles, Fig. 5-10 compared to that of the embrittled FSW. Particle size for the “ductile” weld was on the order of 5µm which is similar to the Lap welded samples. Also of note is the lack of a very distinct line separating the two alloys. This is indicative of a good weld with significant mixing between the two alloys.

![Figure 5-9 Photograph of Fractured Ductile Sample](image)

The “ductile” weld was welded with the pre-weld interface on the advancing side of the tool. The speed of the shoulder is higher on the advancing side of the weld because of the direction of rotation and the forward motion of the weld tool as it moves along the workpiece. The difference in

![Fig. 5-10: SEM Micrograph Showing Dispersed 2219/2195 Interface and Lower Density of Particles. (a) An Intrusion of 2219 in the 2195 Side and (b) High Magnification of 2195 Side Showing Lower Concentration of Large Cu-rich Particles](image)
linear speed relative to the workpiece is twice the feed rate. In other words if the feed rate is 14 in/min then the advancing side of the weld is moving 28 in/min faster than the retreating side of the tool (See appendix B for more calculations relating to tool speed). The weld conditions used to produce the ductile welds (table 5-1) were at first thought to be sufficient to break up any residual oxide layers and prevent brittle fracture. Instead it appears from the SEM images that the mixing conditions instead acted to prevent the precipitates from growing and agglomerating into large particles which could affect weld strength.

Table 5-1: Welding Parameters for Ductile Samples

<table>
<thead>
<tr>
<th>Tool Offset (in.)</th>
<th>-0.125</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Rate</td>
<td>10 ipm</td>
</tr>
<tr>
<td>RPM</td>
<td>160</td>
</tr>
</tbody>
</table>

5.4.2 Brittle Sample

SEM observations were made on SRFSW cross sections. A sharp interface was discernible as shown in the schematic of Fig. 5-11. Fractured samples showed the fracture line closely followed the post weld interface.

Fig. 5-11: Schematic Representation of the SRFSW Interface Observed in the Panels with Positive Offset.
Observations show a high contrast in appearance between the 2219 and 2195 sides, Figure 5-12. The 2219 side exhibits high density of large Cu-rich particles and Cu-rich precipitates along grain boundaries, Figs. 5-12(b) and (c).

Fig. 5-12: Interface of SRFSW AA 2219/2195 (a) low magnification, (b) high magnification showing large (10-20 µm) Cu-rich particles and smaller precipitates along grain boundaries and (c) an area in the AA 2219 side close to the interface showing high concentration of size Cu-rich particles.

SEM observations were also made to determine the different particle sizes between different sections of the welded samples. The micrographs shown in Figure 5-13 were taken in at the same magnification along different sections of the welded sample. The precipitates proved to be larger as you move closer to the center of the weld interface. The difference between particle size between the unaffected material and the thermo mechanically affected zone is very distinct. The precipitates in the TMAZ of the brittle sample are larger than those in any of the previously mentioned examples and may be a contributing factor to the reduced mechanical properties.
TEM was once again used to examine the brittle SRFSW sample to confirm the presence of the same precipitates as those seen in the conventional butt welded samples. The TEM images showed a clear interface between the two alloys after welding much like the observations of the conventional butt welded samples. TEM observations made on the brittle SRFSW interface showed a distinct difference in precipitate density between the 2219 side and 2195 side, Fig. 5-14. Observations in the 2195 side showed absence of precipitates. These observations are consistent with the SEM observations.

Figure 5-13: Precipitate Size Discrepancy Between Microstructural Zones.
Fig. 5-14: TEM Micrograph at the 2219/2195 Interface Showing High Density of Precipitates in the 2219 Side and Absence of Precipitates in the 2195 Side.

While examining the samples special attention was also paid to particles that could possibly be residual oxides. No oxides were found and all precipitates examined were determined to be either intermetallics or $\theta$ particles. Figure 5-15 shows some particularly interesting intermetallics.
Diffraction Pattern analysis from the large particles as the one shown in Figure 5-16 particle 1. These particles were confirmed to be $\text{Al}_7\text{Cu}_2\text{Fe}$ intermetallics, Fig. 5-16. Selected area diffraction also confirmed a tetragonal P4/mnc structure. This matches the findings for the conventional butt welded samples. The diffraction patterns for the intermetallics are displayed in figure 5-17. Figure 5-18 shows a detailed electron diffraction pattern confirming the presence of $\theta$ phase.
Figure 5-16: Identification of Particles on the 2219 Side of the Brittle Self Reacting Friction Stir Weld.

Figure 5-17: SAD Patterns for Large Intermetallic Particle 1.
The brittle samples discussed here and in section 5.4.3 were welded with conditions given in table 5-2. These welding conditions are significantly slower.
on the retreating side of the weld close to the pin tool. This appears to have the effect of allowing the precipitates to agglomerate and grow to large sizes which may reduce the strength of the welds.

Table 5-2: Weld Parameters for Brittle Samples

<table>
<thead>
<tr>
<th>Tool Offset (in)</th>
<th>0.125</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Rate</td>
<td>14 ipm</td>
</tr>
<tr>
<td>RPM</td>
<td>180</td>
</tr>
</tbody>
</table>

5.4.3 Fractured Brittle Sample

Fracture Surface analysis showed the presence of two modes of fracture. Relatively flat, low-energy (brittle) fracture was present in regions close to the specimen edge, Fig. 5-19(b). Ductile fracture was due to micro void formation and coalescence. Micro-voids were associated with the presence of precipitates, Fig. 5-19(c). Thus, the fracture can be described as overall brittle but locally ductile since all plastic deformation was concentrated in the sharp interface.

Fig. 5-19: Fracture Surface Appearance of SRFSW AA 2219/2195 (a) overall appearance, (b) fractography shows two modes of fracture, low-energy, brittle fracture and fracture from micro-ductility and (c) micro-void formation associated with presence of precipitates.

The fractured samples shown in figure 5-20 showed failure on the retreating side of the weld. It is notable that the tool was placed so that the pre-weld interface was on the retreating side of the tool. Examination of unfractured
samples shows that the post weld interface is also on the retreating side of the weld.

Figure 5-20: Picture of Fracture Surface.

5.4.4 Self Reacting 2219/2219

SEM analysis shows low density of precipitates in the advancing side, Fig. 5-21(a) and high density of large \( \theta \) particles and precipitates in the retreating side, Figs. 5-21(b) and (c). Examination of micrographs, Figs. 5-21(b) and (c), shows continuous precipitation of large size precipitates along the grain boundaries and evidence of melting and agglomeration of \( \theta \) phase forming extremely large particles (~10 \( \mu \)m).
Figure 5-21: SEM Micrographs of (a) Advancing Side and Retreating Side (b and c).

For the AA2219/AA2219 sample there is no distinct interface between the two workpieces since they are the same material. Because of this TEM images were taken at various points from the advancing side to the retreating side of the weld where the post weld interface is believed to be. TEM observations in the advancing side of SRFSW 2219/2219 showed a θ population composed of a few extremely large particles, Fig. 5-22(a), and a fine distribution of small precipitates, Fig. 5-22(b). A few intermetallics were also present, Fig. 5-22(a).
TEM samples were also prepared from the Dynamic Recrystallized Zone (DXZ) or weld nugget. The TEM observations in the nugget show more intermetallics, Fig. 5-23 (a) and coarser $\theta$ precipitates, Figs. 5-23(a) and (b), from those on the advancing side of the weld. Figure 5-23 (c) shows EDS spectrum identifying precipitates in figure 5-23 (a) as intermetallics. Figure 5-23 (d) shows EDS spectrum identifying precipitates from Figure 5-23 (b) as $\theta$ particles.

Figure 5-22: TEM Image of Weld with EDS Spectra
a) TEM image of advancing side of weld showing $\theta$ and intermetallic particles. b) TEM image showing distribution of small precipitates. c) EDS spectra of intermetallics d) EDS spectra of $\theta$ particles.
TEM observations on the retreating side (close to the interface where fracture occurred) showing higher density of intermetallics and extremely large \( \theta \) particles, Fig. 5-24(a), along with coarse \( \theta \) precipitates, Fig. 5-24(b), compared to the rest of the weld zones.
5.4.5 Self Reacting 2195/2195

SEM examinations of cross sections showed heavy precipitate activity close to the retreating side where fracture occurred, Fig. 5-25. TEM observations of the nugget showed no precipitation. The zone in the retreating side closer to the fracture interface start showing coarse and fine precipitates, Fig. 5-26. Precipitates were identified as $T_1$ and $T_B$. 

Figure 5-24: (a) and (b) TEM Micrographs and (c) and (d) EDS Spectra from Particles in the Retreating Side of SRFSW 2219/2219 Close to the Interface Where Fracture Occurred.
Figure 5-25: High Magnification of 2195 Interface Showing Heavy Precipitates.

Figure 5-26: (a) and (b) Low Magnification TEM Images Showing Particles Distributed in the Grains and at Grain Boundaries in a Region Approaching the Fracture Interface.
TEM observations near the fracture interface revealed $T_1$ precipitates and heavy presence of coarse $T_B$ particles, Fig. 5-27(a). The ED pattern confirms the presence of $T_1$ precipitates.

![Figure 5-27: Bright-field TEM Image and ED Pattern](image)

(a) Bright-field TEM image and (b) (110) electron diffraction pattern showing presence of $T_1$ phase. Coarse and medium size $T_B$ particles are also shown in (a).

Figure 5-28 shows TEM images of the different particles found in the AA 2195/2195 welds with associated EDS spectra. EDS observations confirm presence of $T_1$ and $T_B$ particles.
Observation showed heavier precipitation in terms of $T_1$ and $T_B$. Coarse $T_B$ particles were found at the grain boundaries.

### 5.5 Known ROD Specimen

A sample was presented to us as a specimen known to have a defect that was believed to be residual oxide. The specimen showed a point of interest when viewed through an optical microscope. This turned out to be a crack or void which did not show significant residual oxide upon EDAX analysis (figure 5-29).
Figure 5-29: SEM Image of “Known ROD Specimen” Showing a Crack or Void

The higher magnification (x1600) of the known ROD samples shown below in Fig. 5-30. This figure shows only small void in the sample rather than qualitative indication of ROD.

Figure 5-30: SEM Image of “Known ROD Specimen” Showing Small Void.
EDAX analysis was performed both inside and outside the void to determine any differences in composition as well as detect the presence of oxides. Oxygen was present in the area around the void (Figure 5-31) as well as inside the void (figure 5-32). Oxygen content was slightly higher inside the void than outside. There was not, however, significant oxide found. Aluminum oxide (Al₂O₃) would have a much higher atomic percentage than those found in this sample.

![EDAX Composition Analysis of Material Surrounding the Void or Crack of “Known ROD Specimen”](image)

Figure 5-31: EDAX Composition Analysis of Material Surrounding the Void or Crack of “Known ROD Specimen”
Figure 5-32: EDAX Analysis of “Known ROD Specimen” Taken Inside the Void

<table>
<thead>
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<th>Element</th>
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<th>Atomic %</th>
<th>Intensity</th>
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<td>97.15</td>
<td>11640.1</td>
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<tr>
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<td>43.9</td>
</tr>
<tr>
<td>Cu</td>
<td>3.50</td>
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Chapter 6
Conclusions and Recommendations

1. Friction stir welds of AA 2219 and AA 2195 were provided by Lockheed Martin Space systems and studied at Louisiana State University using SEM and TEM methods. Of particular interest were self reacting welds which displayed reduced mechanical properties attributed to residual oxide defect. No oxide was detected on these samples.

Figure 6-1: Basic Illustration of the FSW Process. The self reacting setup has a shoulder on both sides of the work piece. Figure B shows the interface between the 2219 and 2195 of the brittle (bad) weld. Figure C is a close up of the same interface showing the large theta particles in the 2219. Figure D is an SEM image of the fracture surface. Figure E is the TEM image of the 2219 side of a brittle weld showing large theta particles and intermetallics. Figure F shows the interface between 2219 and 2195 on a ductile (good) weld.
2. Although the brittle failure exhibited by the “bad” welds was attributed to residual oxide defect, no oxides were detected at the post weld interface. A clear discrepancy was observed between precipitate size in ductile samples and brittle samples as shown in figure 6-2. It is believed this is caused by the difference in tool velocity close to the FSW pin. It is believed the velocity was insufficient near the pin tool when combined with the offset of the brittle welds and caused the brittle condition through larger precipitates.

Figure 6-2: Overview of Differences between Ductile and Brittle Samples. Figures a, b, and c correspond with a brittle fracture. Figure a shows the interface between the 2219 and 2195 and figure b shows a close up of the same interface. Figure c shows the particle size in the thermo mechanically affected zone. Figures D, E and F correspond to a ductile fracture. Figure D shows the much more dispersed interface seen on the brittle samples and Figure E shows a magnified view of the same. Figure F shows the TMAZ of the ductile sample for comparison purposes with the brittle sample.
3. Ductile and Brittle samples displayed significantly different modes of failure with the ductile sample showing a ductile fracture on the advancing side of the weld and the Brittle sample experiencing brittle fracture on the retreating side of the weld. These different modes of fracture were accompanied by drastically different particle sizes between the two samples.

Figure 6-3: Comparison of Ductile and Brittle Samples. Figures a, b, and c correspond with a brittle fracture shown in figure d. The fracture occurs in the Thermo-Mechanically affected zone shown in figure c. Figure e shows the TMAZ of a ductile sample which is shown in figure f. It can also be noted that the ductile sample breaks in the Heat affected zone rather than the TMAZ.

Recommendations for welds to avoid brittle conditions

1. Avoid situations with positive offset (joint positioned on retreating side of FSW tool).

2. Keep feed rate closer to 12 ipm when a positive offset must be used.
Recommendations for future work

1. Position of the pre-weld interface was one of the key factors in determining if the weld would be brittle or ductile. Using embedded thermocouples to determine the heat transfer effects of the interface when offset from the tool would give a meaningful glimpse into what causes the large precipitates to form. While this has been done with conventional welds very little has been done with self reacting welds. Also very little if any work has been done examining the effect of tool offset on the thermal profile of the FSW process.

2. The conditions required to produce a brittle weld resulted in very poor mixing conditions on the retreating side of the tool. Using a tracer material along with stop action could give insight into what is going on along the interface on these bad welds. It is possible that the material along the interface is simply being extruded around the pin tool with very little mixing as described by Colligan [Colligan et al, 2001]. This would result in a sort of kissing bond and could further explain the brittle fracture phenomenon.
References


11. http://www.twi.co.uk/content/fswwqual.html


http://ntrs.nasa.gov/archive/nasa/casi.ntrs.nasa.gov/20080015864_2007032184.pdf
Appendix A

First AAM Conference Paper
Analysis of Embrittled Friction-Stir-Welds

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Abstract

Aluminum alloys 2219 and 2195 have found considerable importance in the construction of the external tank of the space shuttles. Because of this fact, knowledge of the welding properties of the two metals is critical and was studied in this research. A reduction in mechanical properties was observed in Friction-Stir-Welded (FSW) Aluminum panels. This reduction in strength has been attributed to Residual Oxide Defect (ROD). It was also found that certain processing parameters would yield these reduced mechanical properties. The strength of FSW Aluminum panels generally decreases with increasing tool travel rate, decreasing rotation speed, and offset of the weld-seam to the retreating side of the FSW tool. The microstructure of welds exhibiting these strength reduction as well as welds that behaved as expected were examined to determine microstructural effects of processing parameters. Scanning Electron Microscopy (SEM) shows that these weld conditions are accompanied by large precipitates along the grain boundary for both Al 2219 and Al 2195. Transmission Electron Microscopy also shows the precipitates to be θ particles (Al₂Cu), and intermetallics in the 2219, and T1(Al₂CuLi) and TB (Al₇Cu₄Li) particles in the Al 2195. The large size and heavy distribution of these
precipitates, especially on the advancing side of the weld seam may influence these properties. There seem to be no presence of ROD in the samples analyzed in this research. SEM examination was also performed on fracture surfaces of samples exhibiting reduced properties and is also discussed.

Keywords: Friction-Stir-Welding (FSW), Aluminum Alloys 2219 and 2195, Microstructure

Introduction

Demands on today’s materials are greater than ever. Aerospace applications require high strength and low weight while dealing with extreme environmental factors including corrosion, impact, and extreme temperatures. Due to their high strength, low weight and ductility, aluminum alloys have found favor with the aerospace industry. The external tank of the space shuttle in particular, relies extensively on aluminum to minimize the weight of the largest and heaviest (when loaded) component of the space shuttle system. The external tank has gone through many changes from the original tank which weighed approximately 76,000 lbs dry [1]. The lightweight tank was slightly redesigned to reduce weight to approximately 66,000 lbs. The use of aluminum 2195 helped to reduce the weight of the super light weight tank even further approximately 7000 lbs. The Al- 2195 is roughly 5% lighter and 30% to 40% stronger than the Al- 2219 it largely replaces. Because these alloys are difficult to fusion weld Friction- Stir- welding is used instead [2].

Conventional methods of welding aluminum have proven difficult due to its relatively low melting point as well as the lack of a warning sign before
melting temperatures are reached (aluminum does not glow red before it melts such as ferrous alloys). Although Gas- Metal- Arc- Welding (GMAW or MIG) was originally developed to weld aluminum the welds produced by this method are still susceptible to porosity and dross. Gas- Tungsten- Arc- Welding (GTAW or TIG) can also be a suitable method for welding aluminum but works best on thin sections of aluminum. GTAW is more complex and much slower than GMAW and is prone to many of the same defects as GMAW.

Friction- stir- welding is a solid-state process invented at the Welding Institute (Cambridge, United Kingdom) in 1991 [3]. Many of the problems associated with cooling from the liquid phase are avoided with friction- stir- welding because it is a solid-state process. While this welding process typically produces welds, which are as strong and ductile as the parent material, certain welding parameters have been found to produce a sharp decrease in strength and ductility in the welds. Particularly, it has been observed that rpm, feed rate, and tool- offset are the main factors, which produce weld embrittlement. This problem has generally been attributed to a retained or residual oxide defect, caused by incomplete breakup of the oxide layer during welding, but the exact cause needed to be determined. Although research has previously been conducted on the FSW behavior of aluminum alloys, very little work has been done on inhomogeneous welds and this particular inhomogeneity could cause catastrophic failures in the structures. Studies involving self- reacting pin-tools and their effects on microstructure are also few in numbers due to the recent development of this technology. For these reasons the current research was
designed with a number of objectives in mind and to increase an understanding on the residual oxide defects.

**Material and Experimental procedure**

Aluminum plates of alloys 2219 and 2195, with compositions approximately matching those in Table 1 shown below, were welded in various combinations with different self-reacting friction-stir-welding (SRFSW) procedures by Lockheed Martin in New Orleans, U.S.A. The samples were prepared by polishing with silicon carbide polishing wheels of 80, 120, 240, 600, and 1000 grit in that order. Samples are Rough polished with 6-micron aluminum oxide and final polished with 1.0 micron and 0.3 micron alumina. Kellers etch was used to reveal structural detail. Microstructures were characterized with Scanning electron microscope and Transmission electron microscopes (TEM). Tensile tests were performed using MTS universal testing machine and tested samples were examined with SEM.

<table>
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<tr>
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<th>AA 2195</th>
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<tbody>
<tr>
<td>Al</td>
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<tr>
<td>Cu</td>
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<tr>
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</table>

Table 1. Composition of the alloys
**Results**

SEM observations were made on SRFSW cross sections. The interface was visible but it was dispersed as shown in the schematic of Fig. 1. Fracture was found to be in the leading 2219 side.

![Fig. 1 Schematic representation of the SRFSW interface observed in the panels with negative offset. Dotted line indicates location of fracture.](image)

SEM observations showed a dispersed 2219/2195 interface with a lower density of Cu-rich particles, Fig. 2 compared to that of the embrittled FSW.

![Fig. 2: SEM micrograph showing dispersed 2219/2195 interface and lower density of particles. (a) An intrusion of 2219 in the 2195 side and (b) high magnification of 2219 side showing lower concentration of large Cu-rich particles.](image)
Self Reacting Brittle Sample

SEM observations were made on SRFSW cross sections. A sharp interface was discernible as shown in the schematic of Fig. 3. Fracture was found to follow that interface.

![Fig. 3 Schematic representation of the SRFSW interface observed in the panels with positive offset.](image)

Observations show a high contrast in appearance between the 2219 and 2195 sides as shown in Fig. 4(a). The 2219 side exhibits high density of large Cu-rich particles and Cu-rich precipitates along grain boundaries, Figs. 4(b) and (c).

![Fig. 4 Interface of SRFSW AA 2219/2195 (a) low magnification, (b) high magnification showing large (10-20 µm) Cu-rich particles and smaller precipitates along grain boundaries and (c) an area in the AA 2219 side close to the interface showing high concentration of size Cu-rich particles.](image)
TEM observations made on the embrittled SRFSW interface showed a distinct difference in precipitate density between the 2219 side and 2195 side, Fig. 4. Observations in the 2195 side showed absence of precipitates. These observations are consistent with the SEM observations.

Fig. 4: TEM micrograph at the 2219/2195 interface showing high density of precipitates in the 2219 side and absence of precipitates in the 2195 side.

**Self Reacting Fractured Brittle Samples**

Fracture Surface analysis showed the presence of two modes of fracture. Relatively flat, low-energy (brittle) fracture was present in regions close to the specimen edge, Fig. 5(b). Ductile fracture was due to micro void formation and coalescence. Micro-voids were associated with the presence of precipitates, Fig. 5(c). Thus, the fracture can be described as overall brittle but locally ductile since all plastic deformation was concentrated in the sharp interface.
Self Reacting 2219/2195

SEM analysis shows low density of precipitates in the advancing side, Fig. 6(a) and high density of large \( \theta \)-particles and precipitates in the retreating side, Figs. 6(b) and (c). Examination of micrographs, Figs. 6(b) and (c), shows continuous precipitation of large size precipitates along the grain boundaries and evidence of melting and agglomeration of \( \theta \)-phase forming extremely large particles (~10 \( \mu \)m).

Fig. 6: SEM micrographs of (a) advancing side, (b) and (c) retreating side.

Self Reacting 2195/2195

SEM examinations of cross sections showed heavy precipitate activity close to the retreating side where fracture occurred, Fig. 7.
Fig. 7: Heavy precipitates near retreating side of self-reacting 2195/2195 weld. TEM observations of the nugget showed no precipitation. The zone in the retreating side closer to the fracture interface start showing coarse and fine precipitates, Fig. 8. Precipitates were identified as T1 and TB.

Fig. 8 (a) and (b): Low magnification TEM images showing particles distributed in the grains and at grain boundaries in a region approaching the fracture interface.
Conclusions

1. Brittle samples showed heavy precipitate presence along grain boundaries near weld interface.

2. Fracture followed the weld interface very closely with the brittle samples.

3. Precipitates were identified as $\theta$–particles ($\text{Al}_2\text{Cu}$), and intermetallics in the 2219, and $\text{T}1(\text{Al}_2\text{CuLi})$ and $\text{TB} (\text{Al}_7\text{Cu}_4\text{Li})$ particles in the Al 2195.

4. No residual oxide was found in any of the samples examined in this study.

Acknowledgement:

The authors gratefully acknowledge the financial support received from the grant NCAM/UNO-Account # 127406181 and assistance from Lockheed Martin facilities in New Orleans for providing self-reacting Friction-Stir Weld samples.

References


2. National Aeronautics and Space Administration “Space Shuttle Technology Summary: Friction Stir Welding” Publication 8-1263, FS-2001-03-60-MSFC


Alloys”, 7th International Conference on Aluminum Alloys, Trans Tech Publications Ltd.

Appendix B

Velocity Calculations

To calculate the velocity at a single point of the FSW tool we use equation 1.

\[ V = \omega r \quad \text{equation 1} \]

For the brittle sample we have parameters given in table B-1.

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<td>14</td>
<td>180</td>
<td>0.5</td>
<td>1</td>
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</tbody>
</table>

Table B-1 parameters for brittle weld

Using the parameters given we can find the x and y components of velocity with y representing the direction of tool travel. These results are given in Table B-2.

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<th>Angle</th>
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</tr>
</tbody>
</table>

Table B-2 results for brittle parameters. All velocities are given in inches per minute.

Similarly we can find the velocity components of the ductile welds with parameters given in table B-3.

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</table>

Table B-3 Weld parameters for ductile welds

The velocity results for the ductile welds are given in table B-4.

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Figure B-1 shows a plot a point on the outside shoulder for a 1 inch shoulder and the weld parameters matching the brittle welds. The travel per rotation of 0.078 inches displayed in the plot is very close to the distance between ridges seen along the path of Friction stir welds.

![Figure B-1 Plot of Shoulder travel for brittle welds.](image)

Figure B-2 shows a plot of the tool travel for the ductile welding conditions. Travel per rotation is 0.0625 inches.
Figure B-2 Plot of Shoulder travel for Ductile welds.
Vita

David Eric Taylor was born in State College, Pennsylvania in May of 1981 to Rodney and Marilyn Taylor. After completing his secondary education at Ouachita Parish High School in Monroe, Louisiana, he went to Louisiana Tech University in Ruston, Louisiana. There he earned his Bachelor of Science in Mechanical Engineering in May of 2003. After graduation he worked for Standridge Color Corporation before continuing his education at Louisiana State University. David has presented his work in self-reacting friction stir welding for NASA on multiple occasions as well as presenting his work at the 2008 First American Academy of Mechanics conference. He currently works for Wink Companies LLC as an engineer and is a candidate for the degree of Master of Science in Mechanical Engineering in the spring semester of 2009.