FEASIBILITY STUDY OF CONSOLIDATION BY DIRECT COMPACTION AND
FRICION STIR PROCESSING OF COMMERCIALY
PURE TITANIUM POWDER

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Commercially pure titanium can take up to six months to successfully manufacture a six-inch in diameter ingot in which can be shipped to be melted and shaped into other useful components. The applications to the corrosion-resistant, light weight, strong metal are endless, yet so is the manufacturing processing time. At a cost of around $80 per pound of certain grades of titanium powder, the everyday consumer cannot afford to use titanium in the many ways it is beneficial simply because the number of processing steps it takes to manufacture consumes too much time, energy, and labor. In this research, the steps it takes from the raw powder form to the final part are proposed to be reduced from 4-8 steps to only 2 steps utilizing a new technology that may even improve upon the titanium properties at the same time as it is reducing the number of steps of manufacture. The two-step procedure involves selecting a cylindrical or rectangular die and punch to compress a small amount of commercially pure titanium to a strong-enough compact for transportation to the friction stir welder to be consolidated. Friction stir welding invented in 1991 in the United Kingdom uses a tool, similar to a drill bit, to approach a sample and gradually plunge into the material at a certain rotation rate of between 100 to 2,100 RPM. In the second step, the friction stir welder is used to process the titanium powder held in a tight holder to consolidate into a harder titanium form. The resulting samples are cut to expose the cross section and then grind, polished, and cleaned to be observed and tested using scanning electron microscopy (SEM), electron dispersive spectroscopy (EDS), and a Vickers microhardness tester. The results were that the thicker the sample, the harder the resulting consolidated sample peaking at 2 to 3 times harder than that of the original commercially pure titanium in solid form at a peak value of
435.9 hardness and overall average of 251.13 hardness. The combined results of the SEM and EDS have shown that the mixing of the sample holder material, titanium, and tool material were not of a large amount and therefore proves the feasibility of this study. This study should be continued to lessen the labor, energy, and cost of the production of titanium to therefore allow titanium to be improved upon and be more efficient for many applications across many industries.
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CHAPTER 1
INTRODUCTION

1.1 Titanium

Titanium (Ti) is a strong, corrosion-resistant, biocompatible, relatively light in weight, and high temperature-resistant applicable metal that comes with a high cost for its highly regarded properties for many industries [1] [2] [3]. Despite titanium being within the top ten most ample elements on Earth, the main factor for this useful metal not living up to its full potential is the expensive and time-consuming processes that are needed to separate this metal from its raw form of principally rutile and ilmenite [4]. The entire pure titanium break-down can take up to six months of processing to make a six-inch diameter ingot of commercially-pure quality due to its severe reactivity to oxygen and nitrogen [4]. Titanium can also be alloyed with many elements, but the most common alloying elements are aluminum (Al), vanadium (V), and tin (Sn) to create titanium-6-aluminum-4-vanadium (Ti-6Al-4V) and titanium-5-aluminum-2.5-tin (Ti-5Al-2.5Sn). The titanium market in the United States uses the Ti-6Al-4V alloy in more than 50% of the 6 million kilograms per year supply as recorded in 1956 [5].

1.2 Properties and Applications

For a metal, titanium has very extreme physical, thermal, and mechanical properties, a few shown in Table 1, that have proven useful in a variety of industries including but not limited to the aerospace, automotive, biomedical, marine, paper and dyes, processing, sporting goods, and transit industries [5].
### Table 1: Properties of commercially pure (CP) grade 1 titanium

<table>
<thead>
<tr>
<th>Properties</th>
<th>Value</th>
<th>Unit</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Purity</td>
<td>98.635-99.5</td>
<td>wt.%</td>
<td>[5]</td>
</tr>
<tr>
<td>Density</td>
<td>4.51</td>
<td>g/cm³</td>
<td>[6], [7]</td>
</tr>
<tr>
<td>Melting Point</td>
<td>1,665</td>
<td>°C</td>
<td>[6]</td>
</tr>
<tr>
<td>Hardness, Vickers</td>
<td>3,030</td>
<td>°F</td>
<td></td>
</tr>
<tr>
<td>Ultimate Tensile Strength</td>
<td>145</td>
<td>--</td>
<td></td>
</tr>
<tr>
<td>Yield Strength (Tensile)</td>
<td>344</td>
<td>MPa</td>
<td>[6], [7]</td>
</tr>
<tr>
<td>Modulus of Elasticity</td>
<td>275-410</td>
<td>MPa</td>
<td></td>
</tr>
<tr>
<td>Shear Modulus</td>
<td>105</td>
<td>GPa</td>
<td></td>
</tr>
</tbody>
</table>

Of all the metals, titanium has the best in strength at an ultimate tensile strength of 344 MPa for its low density of 4.51 g/cm³ [6]. Having the desirable strength comparable to average alloyed steel yet approximately 50% the density grants titanium to serve in place of some steel applications and shave off weight to achieve better fuel economy for transportation vehicles [1] [4]. The melting point of titanium is 1,665°C (3,030°F) which provides exceptional creep properties authorizing the metal to be useful under intense heating conditions [6]. The resistance to fatigue, corrosion, and fracture combined with the previously stated strength allows titanium to be adequate under extreme fluctuating temperatures in a caustic, high-stress induced environment for a multitude of cycles. These durable properties establish titanium as an excellent contender for the aerospace industries’ need for engine blades (Figure 1), and fasteners, for the automotive industries’ need for engine parts such as a connecting rod (Figure 2), and for the marine industries’ need for ship hulls and propeller blades [1] [5].
Figure 1: “Forged Ti-6Al-4V jet engine fan disks. Courtesy of Wyman-Gordon company.” [5]

Figure 2: “Forged alloy connecting rod for a racing engineer. Component, courtesy of Jet Engineering Inc.; photograph by R. T. Kiepura, ASM INTERNATIONAL.” [5]

Another example of titanium in use for its corrosive-resistant characteristic is in the chemical processing industry as pipe components shown in Figure 3. [5]
Figure 3: “Chemical processing industry. (a) valve body. (b) pump body. Courtesy of Oregon Metallurgical Corporation.” [5]

Many consumer products such as tools (Figure 4), jewelry, firearms, and tennis rackets are also made from titanium and titanium alloys [1] [5].

Figure 4: “Lightweight forged titanium alloy wrenches. Courtesy of Jet Engineering Inc.” [5]
Since titanium is also biocompatible, many surgical blades, accompanying surgical equipment as well as implants, and prosthetics have proven effective in the biomedical industry [1][5]. The nontoxicity to the human body has also brought about the realization that titanium can be useful before the pure metal stage in the form of titanium dioxide (TiO$_2$) shown in Figure 5 [1].

Figure 5: “Titanium dioxide is the most commonly used compound of titanium.” [1]

TiO$_2$ is used for many everyday products such as pigments, used to brighten paper, plastics, paint, and sunscreen [1]. The word ‘titanates’ refer to other titanium compounds such as titanium tetrachloride (TiCl$_4$) and titanium nitride (TiN) with common uses such as coatings for increased hardness for drill bits (Figure 6), heightened refractive appearance on jewels, and smokescreens [1].

Figure 6: "TiN-coated drill bit" [1]
1.2.1 Microscopic Properties

On a microscopic level, titanium alloys exist in two crystal orientations: hexagonal closed packed (HCP) at room temperature, and body-centered closed packed (BCC) at 883°C (1,621°F) or alpha (α) and beta (β) phases respectively [3]. When alloyed and heat treated, titanium can exist or be transformed into different phases called alpha (α), beta (β), and alpha + beta (α+β) phases that each have defining characteristics and properties.

1.2.1.1 Alpha (α) Alloys

The term alpha alloy is defined as a titanium alloy that is completely 100% HCP α-alloy and reached a chemical equilibrium at room temperature. Aluminum (Al), and tin (Sn) are usually responsible for α alloys or for HCP stabilizing, which tend to defy creep behavior, hold relatively good strength and toughness making it an acceptable choice to use in a welding process if needed [3] [5]. Alpha alloys skip a transitional stage known as the “ductile-to-brittle” stage which makes it easy to implement for both high- and extremely low-temperature purposes [3] [8]. Some negative characteristics of the α alloy include low workability, and little benefit in sintering which naturally leads to imperfections yet can be improved upon by repetitious working [3].

1.2.1.2 Beta (β) Alloys

The term beta alloy is defined as a titanium alloy that has completely 100% transformed into a BCC β-alloy and reached a chemical equilibrium at room temperature. Vanadium (V) as well as molybdenum (Mo) are popular BCC β-stabilizing alloying elements transforming titanium to the β alloy which is notable for its ability to be forged, hardened, cold-worked, and treated by heat for a resulting more dense but higher strength properties than that of the α alloy [3].
downfalls include the ability to undergo the transformative phase of “ductile to brittle” stage making them not a good choice for extremely low-temperature applications [8].

1.2.1.3 Alpha + Beta (α + β) Alloys

The term alpha + beta alloy is defined as a titanium alloy that is neither a 100% α alloy nor a 100% transformed β-alloy but has reached a chemical equilibrium at room temperature. The alpha + beta alloy share characteristics of both the alpha and beta phases such as the ability to be forged, the ability to be improved by heat treatments, as well as possess high strength at room to slightly higher temperatures all of which vary at the amount of β-stabilizing elements present [5]. The α + β alloys are not considered to be weldable at a percentage of greater than or equal to 20% of the β phase present [8].

1.3 Production

Starting from the raw minerals to aeronautical grade titanium consumes many steps and processes that can take up to six months [4]. Most of titanium is found in the largest quantities of titanium ore in beach sand minerals named rutile, ilmenite, and leucoxene [9] [10]. Not each of these minerals is extracted, purified, and refined the same way but they are similarly treated to obtain the same chemical makeup of titanium in the end. Once extracted and separated via a wet mill, the titanium ore is transported for purification. Various ways can separate the ore to leave titanium sponge powder. One way to remove most of compounded metals such as iron, magnesium, silicon, vanadium, zirconium is to place the ore into heated distillation tanks then moved in a chlorine liquid compound state to a reactor that pumps in argon (to avoid impure reactions with oxygen and nitrogen) while heating the chlorine liquid with added magnesium to force magnesium and chlorine to react and leave titanium with a few impurities; this is known as the Kroll Process
This remaining titanium is treated with water and hydrochloric acid and vacuumed to remove the collected magnesium and chlorine impurities from previous steps. This leaves the titanium pure enough in a sponge powder form to continue with the ingot production process and purity tests [11]. The sponge powder is then made into smaller pieces and quality tested using chemical and software examination. The powder is now compressed, welded, and then transported to a multi-iteration melting in a vacuum-equipped furnace to turn into an ingot of usable quality [12]-[32]. Inspection occurs for customers and other manufacturers for special needs and orders before shipped to be converted to other useable forms [9] [10]. Titanium sheets, for example, are made from rods which are heated to approximately two-thirds of the melting point then mechanically pressed for several hours. Then cutting, and polishing then the titanium sheet is ready to be manufactured again into the desired application [10] [33]-[35]. For this thesis, the steps of production are focused on from the titanium powder stage to the final part.

1.4 Powder Metallurgy

    Powders are classified by their shape, size, and quality by different methods. Shape is usually broken down into spherical, rounded, cylindrical, spongy, acicular, flakey, cubic, and aggregated; size is determined by what sizes of designated sizes of meshes it can fall through; quality is determined by porosity, flow characteristics, and chemical composition. Chemical composition “grades” are based on the purity of the powder, for example, commercially pure titanium is between 98.635-99.5% by weight. Meshe is used to a certain size of powders and can be classified by one number or by two numbers to indicate a range or a mix of sizes. For example, “230 through 200” means the powder being tested can fit through a mesh count of 200 open windows per inch but not a mesh defined as 230 open windows per inch. Shape is usually determined by the type of process used to gain the powder: for example two different gas
atomization methods might bring about two different shapes of powders based on what cooling medium used to make the powder from molten metal [11] [20] [36] [37]. Examples of specific powder-forming techniques are [8] [11].

- Centrifugal Gas Atomization
- Hydride-Dehydride
- Rotating-Electrode Process
- Melting Extraction Process
- Water Atomization

Powder metallurgy is the study of processes used to transform powders to make near-net shapes (NNS). Some common forms of powder metallurgy processes are [8] [11] [22] [38] [39] [40]-[50]:

- Hot Isostatic Pressing (HIP)
- Vacuum Hot Pressing (VHP)
- Cold Isostatic Pressing (CIP)
- Powder Injection Molding (PIM)

All of the process listed above for either making a powder into the desired particle shape or making the powder into a densified component all require compressing using a pressurized gas or liquid to deform the powder at an angle (for making a powder particle shape) and in a mold (for a solid part) [8] [11]. Sometimes a part mid-processed will be called “green” referring to the part as not finished. When talking about “green strength”, this refers to a mid-processed product’s strength [11] [51]-[55]. Demand for more intricate components have increased and therefore
titanium powder has come to be useful with powder metallurgy techniques to bring about certain components with strict specification requirements, for the aerospace industry for example. Some PM parts made for the aerospace industry are shown in Figure 7 [22] [38] [39] [40]-[50] [57].

Figure 7: “Parts produced from prealloyed powder using powder metallurgy
(a) nacelle frame for F14A, Ti-6Al-6V-2Sn; (b) radial impeller for F107 cruise missile engine, Ti-6Al-4V; (c) complex airframe component for the stealth bomber, Ti-6Al-4V; and (d) engine mount support, Ti-6Al-4V. (Courtesy Crucible Materials Corporation.)” [57]
1.5 Proposal of Production

The goal of this thesis is to prove that titanium powder can be cold compressed then taken to a new technology called a Friction Stir Welder (FSW) to be friction stir processed (FSP) and consolidated.

1.5.1 Friction Stir Welding and Processing

FSW is a relatively new technology invented in the United Kingdom in 1991 that without using a sacrificial material or shielding gas can process or weld certain materials by a solid-state mixing process. The term “solid-state” refers to a process that operates under the melting point of a material to improve upon the mechanical properties. The FSW process can be thought of as an automated spinning drill approaching the surface of a material and driving (term used is ‘plunge’) into it slowly until the material nearly melts and produce a weld of a joint or through the body of a material to process the properties. In Figure 8A, the probe/pin is shown as the plunging object before entering the material to be welded, then in Figure 8B the tool has travelled or traversed down the two materials to join them in a weld [58] [59] [61] [62].

Figure 8: Friction stir welding. A: before tool plunging. B: traversing down the weld line. [58]
The term “weld” is defined as the joining of two materials together whether it be two materials from the same base material (BM) like two titanium alloys (known as similar welding) or two different materials such as aluminum and magnesium (known as dissimilar welding). The term “process” is defined as the solid-state welding process in one material with the goal to improve upon the microstructural and mechanical properties of that one material. “Processing” is also referred when referencing the action of consolidating a powder on top of a solid base material of the same type [58] [59]-[64].

The parameters of the FSW machine are crucial for a successful weld or processing effect and include:

- Selecting the correct side in regards to the tool rotation direction to achieve the optimal weld (advancing side or retreating side)
- Tool rotation direction (clockwise or counterclockwise)
- Tool rotation speed (revolutions per minute)
- Tool plunging speed (how fast into the material)
- Tool plunging depth (into the material)
- Tool traversing/welding speed (moving along the workpiece)
- Dwelling time (not plunging or traversing)
- Tool tilt (0° referring to normal of the working surface)
- Tool offset (distance to the right or left of the welding line)
- Tool features (such as a scrolled shoulder, or conical pin/probe)

The sides referred to as advancing side (AS) and retreating sides (RS) are each side of the welding or processing center line. The AS refers to the side that the tool enters first looking at it
from above at the lowest point; the RS is the remaining side. The side definitions correlate to the clockwise or counterclockwise direction of tool rotation and traversing direction. Therefore looking from above, if the tool is rotating clockwise, traversing upwards, the first material it will enter will be the left side which is now defined as the AS. The tool plunging speed and plunging depth refers to the speed the tool dives into the material and how deep it will dive for the remainder of the weld. If the tool is not traversing (or moving along the weld line), and only spinning at a constant plunge depth, the tool is “dwelling” which can be used to build up heat in a material. Tool tilt is the tool’s angle relative to the normal of the work surface; 90° from every axis to the work piece is considered 0° and the tilt is described for the machine used is in the x-direction i.e. the length of the work piece. Tool offset is a strategic movement (in the y–direction) of the center of the tool to one side or the other of the work piece to prevent melting of one material or the other, commonly used for dissimilar welding. The tool shoulder is the face from which the pin/probe protrudes; the shoulder is primarily responsible for the “friction” because it is making parallel contact with the work piece while the pin/probe is responsible for the “stir” in the name “friction stir welding”. Tool features are related to the dimensions as well as the physical features on the pin/probe and shoulder; pin height, pin diameter, pin shape, pin features like threaded, fluted, or not, shoulder diameter, shoulder features like scrolled, concave, or not can all be customizable to fit the need [63] [64]. Figure 9 shows a few of the pin customizations [8] [9].
1.5.2 The Feasibility Concept

Based on sources [9] and [10], a flow chart shown as Figure 10, has been summarized for the processes to make sponge titanium powder into a usable material.

The proposed method involves only titanium powder being pressed mechanically then friction stir processed to consolidation; the new proposal concept is shown in Figure 11.
The proposed method of manufacture will greatly cut down on cost, energy usage from cutting 4-8 steps into only 2 steps. Chapter 2 will describe the materials and equipment used in the experimental approach. Chapter 3 will explain the designs, methods, and results of attempting to accomplish the first step of the 2-step process: compression. Chapter 4 will explain the designs, methods, and results of attempting to accomplish the second step of the 2-step process: consolidation by friction stir processing. Chapter 5 will explain the overall conclusions of the thesis as well as future work continuation methods and ideas.
CHAPTER 2
EXPERIMENTAL APPROACH

2.1 Introduction

Chapter 2 will describe the powders and equipment used for compression, friction stir processing, and mechanical testing analysis. Each section will be dedicated to a powder, or a specific machine with its specifications, and the parameters used.

2.2 Powders

Three powders were used for experiments; one aluminum (Al) powder and two commercially pure (CP) titanium powders were available for use. From Alpha Chemicals, this aluminum powder is >99.5% aluminum at 30 μm. The aluminum powder’s purpose was for training on the manual hydraulic press only and is shown in Figure 12. The first titanium powder was ordered from Puris, LLC © and was classified as 99.835 wt.% CP titanium, spherical powder grade 1, +250 μm (-60 mesh), shown in Figure 13 with the compositional breakdown shown in Table 2. The second titanium powder was received from Materials and Electrochemical Research Corporation (MER Corp.) and was classified as 99.5682% hydride/dehydride titanium sponge powder, -20+100 mesh, shown in Figure 14 with the compositional breakdown shown in Table 3. Both titanium powders were used for comparison throughout the experimental processes: compression and friction stir processing.

The three powders can be distinguished by knowing the aluminum is a fine light-gray reflective substance, where the spherical titanium powder has mixed sizes of larger, reflective, light gray beads, where the sponge titanium powder is a dark gray, fine substance.
Figure 12: Alpha Chemicals produced >99.5% aluminum powder, 30 μm. Used for training purposes on the manual hydraulic press.

Figure 13: Puris, LLC © produced 99.835 wt.% CP titanium grade 1 spherical powder, +250 μm. used for compression and friction stir processing.
Table 2: Composition of Puris, LLC © produced 99.835 wt.% CP Titanium Grade 1 Spherical Powder, +250 μm

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Fe</th>
<th>Na</th>
<th>Si</th>
<th>Y</th>
<th>Cu</th>
<th>Sn</th>
<th>O</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt.%</td>
<td>0.013</td>
<td>0.036</td>
<td>0.0005</td>
<td>0.0062</td>
<td>&lt;0.0005</td>
<td>0.0083</td>
<td>0.002</td>
<td>0.083</td>
<td>&lt;0.005</td>
</tr>
</tbody>
</table>

Figure 14: Materials and Electrochemical Research Corporation (MER Corp.) produced 99.5682% hydride/dehydride titanium sponge powder, -20+100 mesh. Used for compression and friction stir processing.
Table 3: Composition of Materials and Electrochemical Research Corporation (MER Corp.) produced 99.5682% hydride/dehydride titanium sponge powder, -20+100 mesh

<table>
<thead>
<tr>
<th>Element</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>&lt;0.005</td>
</tr>
<tr>
<td>C</td>
<td>0.0210</td>
</tr>
<tr>
<td>Cl</td>
<td>0.0690</td>
</tr>
<tr>
<td>Cr</td>
<td>0.0260</td>
</tr>
<tr>
<td>Fe</td>
<td>0.1300</td>
</tr>
<tr>
<td>H</td>
<td>0.0061</td>
</tr>
<tr>
<td>Mg</td>
<td>0.0310</td>
</tr>
<tr>
<td>Mn</td>
<td>&lt;0.0050</td>
</tr>
<tr>
<td>Mo</td>
<td>&lt;0.0050</td>
</tr>
<tr>
<td>N</td>
<td>&lt;0.0035</td>
</tr>
<tr>
<td>Na</td>
<td>0.0010</td>
</tr>
<tr>
<td>Ni</td>
<td>0.0240</td>
</tr>
<tr>
<td>O</td>
<td>0.0690</td>
</tr>
<tr>
<td>P</td>
<td>&lt;0.0100</td>
</tr>
<tr>
<td>Pb &amp; Cd</td>
<td>&lt;0.0002</td>
</tr>
<tr>
<td>S</td>
<td>&lt;0.0050</td>
</tr>
<tr>
<td>Si</td>
<td>&lt;0.0100</td>
</tr>
<tr>
<td>Sn</td>
<td>&lt;0.0050</td>
</tr>
<tr>
<td>V</td>
<td>&lt;0.0050</td>
</tr>
<tr>
<td>Zr</td>
<td>&lt;0.0050</td>
</tr>
</tbody>
</table>

2.3 Compression Equipment

2.3.1 Manual Hydraulic Press

The Carver® manual hydraulic press shown in Figure 15 and Figure 16 is available in the Material Science and Engineering department and was used throughout the experimental stages. The maximum compression capacity pressure is 24,000 pounds or 11 metric tons using a needle gauge. The aluminum powder was used as training specimen on this press, while both titanium powders benefited from the press for both cylindrical and rectangular pressing shapes.
Figure 15: Carver® manual laboratory model C hydraulic press specifications tag.

Figure 16: Carver® manual laboratory model C hydraulic press using a needle gauge, maximum compression pressure is 24,000 lb or 11 metric tons.
2.3.2 Automated Compression Machine

The Shimadzu Corporation © automated compression/tensile testing autograph machine shown in Figure 17 and Figure 18 is available in the Mechanical and Energy Engineering department and was used for the rectangular die and punch discussed in a subsequent section. The maximum compression capacity load is 250 kN using compatible software, digital readout, and customizable static or cyclic loads. The aluminum powder was not used on this machine, while the spherical titanium powders benefited from this machine for only the rectangular shapes due to the requested limitation of the Material Science and Engineering department on the cylindrical die and punch.

Figure 17: Shimadzu Corporation © automated compression/tensile testing autograph machine tag, maximum load capacity is 250 kN.
Figure 18: Shimadzu Corporation © automated compression/tensile testing autograph machine, maximum load capacity is 250 kN.

This machine cannot compress then compensate for any slippage in the powder as the hydraulic manual press can with manual checking over a period of time, although the automated compression machine can be set up for a custom cyclic process, that is, if programmed correctly can essentially do the same thing as the manual press regarding slippage.
2.4 Friction Stir Processing Equipment

2.4.1 Friction Stir Welder

The Manufacturing Technologies, Incorporated © (MTI) Friction Stir Welder, shown in Figure 19 and Figure 20 is available in the Material Science and Engineering department and was used for friction stir processing of both titanium powders. The aluminum powder was not used on this machine, while the two titanium powders benefited from processing. The maximum downward capacity of force is 67 kN, with a maximum tool rotational speed of 3,000 RPM using compatible software, and digital readout. The specifications of the University of North Texas (UNT) friction stir welding machine are shown in Table 4.

<table>
<thead>
<tr>
<th>Component</th>
<th>Specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spindle</td>
<td>Speed Range: 100-3,000 RPM Torque: 300Nm at 50 to 766 RPM</td>
</tr>
<tr>
<td>X-Axis</td>
<td>Stroke: 700 mm (27.5 in) Maximum Velocity: 3,000 mm/min (118 ipm) Maximum Force: 22.5 kN</td>
</tr>
<tr>
<td>Y-Axis</td>
<td>Stroke: 305 mm (12 in) Maximum Velocity: 3,000 mm/min (118 ipm) Maximum Force: 22.5 kN</td>
</tr>
<tr>
<td>Z-Axis</td>
<td>Stroke: 305 mm (12 in) Maximum Velocity: 3,000 mm/min (118 ipm) Maximum Force: 22.5 kN</td>
</tr>
<tr>
<td>A-Axis</td>
<td>± 6 degree-minute (hardstop-to-hardstop) ± 5 degree-minute (softstop-to-softstop/working range) ± 5 degree-minute (range requirement)</td>
</tr>
<tr>
<td>Service Requirements</td>
<td>Air: 80 psi (5.5 Bar) 3.5 scfm Argon: 60 ft³/h (28 l/min) Supply Voltage: 480 VAC/60 Hz, 3 Phase, 100 A, 89.4 fla</td>
</tr>
</tbody>
</table>

Reference: courtesy of the MTI manual and the Center for Friction Stir Processing at UNT.
Figure 19: Manufacturing Technologies, Incorporated © (MTI) friction stir welder model RM-1.

Left: computer aided design (CAD), right: friction stir welder at UNT.

Figure 20: Manufacturing Technologies, Incorporated © (MTI) friction stir welder tool and bench close-up at UNT.
2.5 Mechanical Testing Analysis Equipment

2.5.1 Scanning Electron Microscope and Energy Dispersive Spectroscopy

The scanning electron microscope (SEM) shown in Figure 21 is a high-powered electron beam microscope. It uses the projected focused electron beam (up to 30.0 kV) to produce an image of high quality at a macroscopic and microscopic level. There are many adjustments to the kind of image produced based on the desired behavior of the electron beam. An SEM can be used to analyze texture, distances, depth, and characterization for many materials of various sample sizes.

Figure 21: Scanning electron microscope (SEM) with electron dispersive spectroscopy (EDS) setup at the University of North Texas.

Energy dispersive spectroscopy, (EDS), is a secondary function connected to the SEM that allows for a compositional analysis of a sample at any given part. Many elements as well as their compounds and phases are included in a digital database that the EDAX program will recognize.
by a series of backscattered electrons; EDS displays the data collected as peaks on a graph to allow
for a count of the elements detected scattered across an SEM image. EDS also is able to collect
colored map images for where the detected elements lay within a captured SEM image. Certain
elements like carbon for example are not able to be analyzed due to the machine’s limitations, yet
for this experimental results, all metals are distinguished.

2.5.2 Optical Microscope

A Zeiss optical microscope (OM) with camera attachment was used to observe compacted
powder. The OM is used in place of scanning electron microscope (SEM) and possesses the
magnification ability much lower than that of the SEM but has the ability of producing an image
in room temperature atmosphere. The OM used for powder observation is shown in Figure 22.
2.5.3 Vickers Microhardness

The term ‘hardness’ refers to the material’s resistivity to indentation or deformation. A micro-hardness tester indents a material with a harder material and can then calculate the hardness of the sample by certain measurement procedures for the indented shape. Shown in Figure 23, this Vickers (type of hardness scale) microhardness tester features a diamond tip and square pyramid-shaped indenter, with specifications shown in Table 5.

Figure 23: Wilson® Vickers microhardness testing machine.
Table 5: Wilson® Vickers microhardness testing machine specifications courtesy of UNT Center of Friction Stir Processing laboratory

<table>
<thead>
<tr>
<th>Specification</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test Load (gf)</td>
<td>10, 25, 50, 100, 200, 300, 500, 1,000 (2,000)</td>
</tr>
<tr>
<td>Standards Compliant</td>
<td>EN-ISO 6507, ASTM E384 &amp; E92, And JIS</td>
</tr>
<tr>
<td>Test Cycle</td>
<td>Automatic (loading/ dwell/ unloading)</td>
</tr>
<tr>
<td>Dwell Time</td>
<td>5 - 99 seconds</td>
</tr>
<tr>
<td>Automatic Turret</td>
<td>3 position: 1 indenter, max. 2 objectives</td>
</tr>
<tr>
<td>Turret Speed</td>
<td>120° per second</td>
</tr>
<tr>
<td>Statistics</td>
<td>Number, Mean, Standard Deviation, Minimum, Maximum and Range</td>
</tr>
<tr>
<td>Digital Encoder Resolution</td>
<td>0.1μm</td>
</tr>
<tr>
<td>Total Magnification</td>
<td>100x, 500x</td>
</tr>
<tr>
<td>Measuring Range</td>
<td>200 μm at 10x</td>
</tr>
<tr>
<td>Display</td>
<td>5.7 in (144.8 mm) SMART-UI touch screen display with tab interface</td>
</tr>
<tr>
<td>Diagonal Length</td>
<td>4-digit (d1, d2)</td>
</tr>
<tr>
<td>Data Output</td>
<td>USB memory stick (csv format), RS232</td>
</tr>
<tr>
<td>Conversions</td>
<td>Brinell, Vickers, Rockwell and Tensile Strength according to ASTM E140, ISO 18625 and GB</td>
</tr>
<tr>
<td>Depth from Center line</td>
<td>130 mm (5.1 in)</td>
</tr>
<tr>
<td>Operating Temperature Range</td>
<td>10°C to 38°C (50°F to 100°F)</td>
</tr>
</tbody>
</table>
CHAPTER 3

COMPRESSION RESULTS

3.1 Cylindrical Die and Punch

3.1.1 Introduction

The stainless steel cylindrical die and punch, shown assembled in Figure 24 and separated in Figure 25, that was used in these experiments was available courtesy of the Material Science and Engineering department; this department requested that this die and punch not be used on the automated compression machine as well as if used on the manual hydraulic press that the pressure shall not exceed 5,000 lb. All three powders were pressed into green compacts using this ~1 cm-diameter samples. Originally the sample size was thought to be of too small of a size to retrieve mechanical data from it, yet had greater success than the rectangular die and punch designs.

Figure 24: Left: assembled cylindrical die and punch, right: aluminum sample retriever spacer.
3.1.2 Operation and Assembly

If one were to number the pieces from left to right in Figure 25, for assembly, the 4th piece would be placed on top of the 5th piece followed by the 3rd piece on top of both and aligned. At this point the measured powder would be released into the die body (3rd piece) and then using a fine-pointed cue-tip, one would dust the fallen particles into the chamber as well as the particles clinging to the inside walls of the chamber. Next the 2nd piece (punch), would be inserted followed by the 1st piece (protective cover) and initially hand compressed to ready powder for hydraulic pressing. The valve at the bottom center of the manual hydraulic press is now turned counterclockwise to release the grip pressure and open the gap between the grips. Once space
opens enough to hold the cylindrical die and punch assembly, the valve can be tightened and the assembly can be inserted. One can use the hydraulic lever to bring the die and punch to the upper clamp, align the protective top cap with the most centered area of the top clamp. Slow the pumping to align the die and punch properly then continue pumping at a slow and even rate. Once the pressure gauge reaches the desired load, one shall wait for slippage and the rearrangement of molecules to settle under that certain pressure. After 5 minutes, one should re-pump the pressure to the desired value. Evenly check the gage through a 30-minute period of time to continually correct the slippage.

After the 30-minute timed session, the valve can again be released until the die and punch assembly can be removed. Turn the assembly upside-down carefully to keep anything from slipping. The bottom piece will now be removed and replaced by the aluminum spacer. Once again replace the assembly back into the hydraulic manual press and pump until the punch can no longer moved through the die body. Release the assembly one last time to retrieve the sample from the assembly. Carefully clean the entirety of the assembly without water if possible. Repeat this process for every sample desired.

3.1.3 Calculations

If an appropriate desired height is defined for the compacts, the powder mass can be calculated for this desired chosen height. An appropriate sample height was chosen based on the following considerations:

- Must not exceed the height of the die body or the compressing region
- Must not exceed the clearance height between the top of the retrieval aluminum spacer and sample spacer height when inserted in the die body
• Must be within a reasonable height to compensate for desired evenly-distributed stress distribution between layers of powder within a potential compact

• Must have enough green compact strength for transport to the friction stir welder stage

• Must be large enough to serve its purpose as representing a model of a titanium part such as a dental implant

An appropriate goal height was determined to be approximately 0.25 in (6.35 mm) based on all the limitations and limited amount of powder available. One can calculate the amount the mass of the powder needed to compress to be approximately the desired height of 0.25 in by a simple tweaking of the basic cylinder volume formula and basic density formula shown as Equation 1 and Equation 2 respectively.

\[ V = \pi r^2 h \]  
Equation 1

\[ \rho = \frac{m}{V} \]  
Equation 2

Using the pure titanium grade 1 density of 4.51 g/cm\(^3\) [58], and 50% assumed compression of the powder, the measured diameter of the die hole of 0.994 cm, and the assumed conversion of 1 in is approximately 25.4 mm, one may modify Equation 1, and Equation 2 to look like Equation 3 and then solve for mass.

\[
50\% \times 4.51 \frac{g}{cm^3} = \frac{\text{mass}}{\pi \left(\frac{0.994 \text{ cm}}{2}\right)^2 \times 6.35 \text{ mm}}
\]  
Equation 3
The resulting goal mass of powder with the above assumptions for a cylindrical body of 0.25 in height is 1.111 g. The final height after compression at different pressures is compensated for in the friction stir processing step later.

3.1.4 Results

The cylindrical die and punch is successful at compressing each one of the powders by being comprised of one solid piece: the die body. The spacer in between the bottom die component and the sample is crucial for avoiding leaks of powder through the bottom. The clearance between the punch and the die body hole diameters is very tight thus making the two surfaces able to slide passed each other due to extensive polishing to prevent powder leakage through the top of the die. Without corners, circular cross-sectional areas have the advantage of not having weaker stress points seen in shapes with corners.

3.1.4.1 Aluminum Samples

Aluminum compression samples were made using the calculation for titanium powder mass but with pure aluminum density value and was utilized for training on the manual hydraulic press shown in Figure 26. Each sample was weighed, emptied into the die, manually pushed down with a pointed cue tip and then compressed using the manual hydraulic Carver press to 5,000 lb, held for one minute, then released, and manually was pumped out by the Carver press. No further tests or measurements were taken.
3.1.4.2 Titanium Samples

Many titanium samples were made using the same method as the aluminum powder compressions with an added timed component. Titanium’s density is around twice that of aluminum’s therefore the account for slippage and relaxation is taken into account for both titanium powders. The larger-particle spherical titanium powder was compacted at 5,000, 4,000, 3,000, and 2,000 lb₉ with continual loss of height management and green compact strength as compaction pressure decreased particularly between the 3,000 and 2,000 lb₉ stage. The finer-particle sponge titanium powder was compacted at 5,000, and 4,000 lb₉ with less slippage and relaxation time as the spherical powder. The variances in pressure was to observe if the compression stage was a vital stage to the consolidation feasibility and if so how much energy could be saved in the compression stage and still gain the same results in the FSP stage.

Once pumped to the designated pressure, a time was set for every 5 minutes in a 30-minute time period for checking and pumping back to the designated pressure to compensate for slippage and relaxation of the titanium powder. Figure 27 shows the resulting compacts compressed at 5,000 lb₉ and adjusted every 5 minutes in a 30-minute timed session.
Figure 27: Left: larger particle spherical titanium compacted at 5,000 lb using the manual hydraulic press, right: finer grained sponge titanium compacted at 5,000 lb using the manual hydraulic press.

A cylindrical sample at 2,000 lb is shown in Figure 28 for comparison, the remaining various load results are similar to Figure 27 with a slightly variation of height and rigidness.
3.2 Rectangular Die and Punch Designing Process

Since the samples that can be produced by the cylindrical die and punch are limited in a small diameter, a rectangular die and punch experienced the design process comparable to the cylindrical design but has a larger compact overall surface area for a feasibility of a larger part. All of the calculations regarding the mass of powder needed for compressing to a certain height were of the same nature as the calculations for the cylindrical mass calculation; the volume of the predicted compact was the only component that changed. The height value of 0.25 in or approximately 6.35 mm was kept for consistency across the two die and punches.

Since it is known that a successful cylindrical compact can be made at 5,000 lbf, the rectangular compact force can be calculated with the basic equation for stress shown in Equation 4 and solved shown in Equation 5.

\[ \frac{F_{\text{rectangular}}}{A_{\perp \text{rectangular}}} = \sigma_{\text{cylindrical, rectangular}} = \frac{F_{\text{cylindrical}}}{A_{\perp \text{cylindrical}}} \]

\[ \sigma = \text{stress} \]

\[ F = \text{force} \]

Equation 4
\[
\frac{F_{\text{rectangular}}}{A_{\perp \text{rectangular}}} = 64.432 \frac{lb_f}{mm^2} = \frac{5,000 lb_f}{\pi r^2 (9.94 \text{ mm})^2}
\]
\[A_{\perp} = \text{cross-sectional area} \quad \text{Equation 5}\]

Solving for force in Equation 5, the force required to create the same stress as the cylindrical compact in the rectangular compact is determined by the chosen length and width dimensions of the sample.

3.2.1 First Design

Originally, a cubical hole of the dimensions 1 in x 1 in x 1 in with 0.5 in outer walls and 0.5 in thick base was discussed as one solid body die piece similar to the cylindrical die and bottom part but not separable. This poses the issue that once compressed, the die would need to be destroyed in order to retrieve the compact; this is where a hollow design with a scrap metal base was discussed. This elementary hollow design would propose an exit for the newly compacted bar as well as a way to reuse the die and punch. The UNT Mechanical Technologies department machine shop was the source for heavy cutting and manufacturing of this die and punch and once notified of the limitations of this machine shop, the die and punch design was split into four separate wall pieces for milling out a shape with corners out of a solid bar was out of the range of resources at this machine shop. With four separate pieces, a cheap solution to removable compression to keep these four walls together was a requirement and was solved by ordering four small iron C-clamps for easy fitting, removing, and compression. Specifications of the clamps are listed in Appendix A: Table 18.

A point was brought up that in a shape with corners, a cubical compact will not be able to withstand its own weight and unevenly distributed stress levels well without mass amounts of compression. To avoid hitting the maximum compression strength of a cheap die and punch material and to avoid the maximum capacity of the machines available, a rectangular compact
shape was decided with dimensions of 2 in long, 0.5 in wide, and 0.25 in thick (for comparable results with the cylindrical die and punch compacts thickness). The rectangular die and punch were to be constructed of a stainless steel T-316/316L, specifications shown in Table 6, to closely match the qualities of the cylindrical die and punch.

Table 6: Rectangular die and punch material: stainless steel T-316/316L composition approximations, meets ASTM A276 standards [65].

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Cr</th>
<th>Fe</th>
<th>Mn</th>
<th>Mo</th>
<th>Ni</th>
<th>P</th>
<th>S</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percentage</td>
<td>0.08</td>
<td>18 max</td>
<td>82</td>
<td>2</td>
<td>3 max</td>
<td>14 max</td>
<td>0.045</td>
<td>0.03</td>
<td>1</td>
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</tbody>
</table>

Once material was received, milling was performed for flattening of the original material to prevent any defects in the alignment and shape of the die hole. The height in which the original material was shipped was kept if possible; then desired widths of the four walls were matched and then the inside faces were polished. The punch was constructed last to be approximately 0.5 in taller than the die body as a precaution for compacts that may get stuck in the die, the punch may still be recovered. The punch was polished on all faces to allow the powder to be against a polished punch and allow the die body and punch glide past each other in the compression process. The bottom piece was a scrap aluminum plate used for keeping the compression off of any other surface that might be of concern. The first finished rectangular die and punch design is shown in Figure 29.
Figure 29: Rectangular die and punch: design 1

(Die dimensions: two 75.79 mm long by 4.14 mm wide by 50.10 mm tall rectangular sides and two 4.14 mm long by 4.14 mm wide by 50.10 mm tall rectangular short sides. Punch dimensions: 50.84 mm long by 12.60 mm wide by 63.88 mm tall.)
Note that the lateral walls are longer in length than necessary, but were cut long to avoid inaccurate punch alignment; the increased lateral wall length doesn’t affect the compressing process. The procedure taken to compress the titanium spherical powder was the same as the cylindrical die and punch procedure with varying compression loads with the hydraulic manual press. Without an aluminum spacer to retrieve the compact, the die and punch were not flipped over, and the compact was revealed by the unclamping and physical separation of the die walls. The spherical titanium powder was the sole powder composition to be compressed in this die for limited quantity of the sponge titanium powder was available, therefore the spherical powder was used for trial and error until this compression stage was perfected.

3.2.1.1 First Design Results

The compact quality and geometrical integrity of the die and punch were considered for the feasibility of the die designs. At a load of 3,000 lb, the spherical titanium powder would not show any signs of compression. At the load of 5,500 lb, groups of particles started to show compaction in the form of small shiny groups as shown in Figure 30.
Figure 30: Rectangular compression failure at 5,500 lb$_f$ and 9.059 g, shiny groupings shown on right are the only evidence of partial compression. Left: bottom layer fallen out of the die, right: remaining powder originally compacted in die, then pressed out of die.

At loads of 6,000 lb$_f$, 6,500 lb$_f$, and 7,000 lb$_f$, significantly more compression occurred with an evident rectangular-like shape shown in Figure 31 and Figure 32.
Figure 31: Rectangular compact failure at 6,000 lbf. Top: top view of semi-compact sample, bottom: side view of semi-compact sample.

Figure 32: Rectangular compact failure at 7,000 lbf. Left: top view into die body with compact sample with punch removed, right top: after extraction, powder leakage through bottom, right bottom: after extraction, orthogonal view of semi-compact sample.
Without a sample spacer and custom-fit base plate, significant leakage of uncompressed powder slippage between the defects of the aluminum base plate and became the sample spacer for the remaining layers to compact. Leakage was caused by the unsteadiness of transportation of the die from the hydraulic manual press to the lab table for extraction. At the second step of extraction, where unclamping full capacity compression also induced die movement on base plate which moved the bottom layer of powder. The next step involves separating the die walls from the compact, which sometimes could be lifted straight up, but other times it was required to tip the walls away from the compressing region to free the compact; this wall movement either by the compact falling or by creating uneven stress concentrations caused the outer faces and vertices to crumble upon extraction. At this point, none of the resulting samples possessed the green strength to be transported.

To draw conclusions about the geometrical integrity of the first die design, some powder was compressed in the first design with industry clamps under 40,000 lbf for a time period of 30 minutes. The results were similar to Figure 31 and Figure 32; the die walls were bowing outward under the immense forces and the clamping side forces by clamps were not enough to compensate for the powder resisting compression and forcing the die walls outwards.

Another method was introduced to conclude how to approach the second design. Referring back to Equation 5, the force calculated to achieve the same stress in the rectangular compact as the cylindrical compact had at 5,000 lbf, a force of 41,569 lbf must be achieved. A machine shop, industrial-used vice and manual compression machine was used two times to overcome the resisting powder forces, and experienced 40,000 lbf and 60,000 lbf. The resulting compactions are extracted similarly to how the aluminum spacer is utilized in the cylindrical die design but with
scrap steel to sustain the extraction forces instead of a hollow cylinder. The resulting compactions are shown in Figure 33 and Figure 34.

Figure 33: Rectangular compact success at 40,000 lbf. Top: top view, bottom: orthogonal view.
Figure 33 fell apart at the touch, and could therefore be easily broken by hand, Figure 34 however, didn’t fall apart at the touch but with some muster could be broken by hand as well as seen by the crack in the image.

Even though the first design achieved being the cheaper approach, the clamping was time consuming to assemble, disassemble, and reassemble with a steady hand, within an efficient time frame, and with preciseness; also the vice was very heavy and inconvenient to move and use.

Overall, the first design took a step toward the cheaper approach while with added side clamping force the design wasn’t as effective as the solid-body-piece cylindrical die still therefore design steps were taken towards the existing cylindrical die and punch design.
3.2.2 Second Design

The second design’s purpose was to overcome the extreme side forces without sacrificing the die body’s height for convenience; the attempted solution is welding the walls together from the outside, (which eliminates the need for clamps or a vice) and creating a custom bottom piece from 1018 carbon steel (see Table 7 for composition breakdown) to prevent sliding in all directions. The inside (where the punch is inserted) must be carefully avoided when welding the walls together, because if any major defects appear then the die isn’t any good and must start over. At this point since high forces around 40,000 lb$\text{f}$ are needed to be achieved for a successful rectangular compact, the automated compression machine must be used for the needed compressive force needed is outside the range of the hydraulic manual press capabilities. The next step is to add in the two steel bars (composed of the same 1018 carbon steel, see Table 7) from earlier experience with the vice to extract the compactions with the least effort. The Computer-Aided Drawing for the second design is shown in Figure 35 where the second design die and punch and accessories are shown in Figure 36.

Table 7: Rectangular die and punch custom base plate and retrieval bars material: using cold drawn 1018 carbon steel composition approximations [66]

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Fe</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>0.14-0.20</td>
<td>98.81-99.26</td>
<td>0.60-0.90</td>
<td>&lt;0.040</td>
<td>&lt;0.050</td>
</tr>
</tbody>
</table>
Figure 35: Top two: computer-aided drawing (CAD) of second rectangular die and punch design. Bottom two: actual die and punch, left two: ready for compression, right two: ready for/already completed compact sample extraction.

(Die dimensions: two 75.79 mm long by 4.14 mm wide by 50.10 mm tall rectangular sides and two 4.14 mm long by 4.14 mm wide by 50.10 mm tall rectangular short sides. Punch dimensions: 50.84 mm long by 12.60 mm wide by 63.88 mm tall. Sample retrieval bar dimensions: 87.05 mm long by 12.66 mm wide by 12.60 mm tall. Base plate dimensions: 130.70 mm long by 100.82
mm wide by 24.94 mm tall with a notch located 31.78 mm from edge with dimensions of 130.70 mm long by 37.16 mm wide and 12.7 mm deep.)

Figure 36: Second die and punch design continued. Left: with punch being removed, right: custom base plate for die and punch.

Forces from 45,000 lb\textsubscript{f} up to 49,000 lb\textsubscript{f} (2,000 lb\textsubscript{f} lower than the automated compression machine’s force capacity) were tested with this design and multiple observations took place.

3.2.2.1 Second Design Results

The second design was successful at what it was designed to overcome, but didn’t exactly achieve what was expected of it. The custom base plate was successful in the elimination of the bowing/bending of the walls, as well as keeping the die and punch relatively motionless for transportation. The welded structure was successful in not separating and/or not bowing nor bending under the powder’s resistive force. The retrieval bars were as successful as the aluminum spacer is for the cylindrical die and punch. The automated compression machine’s cyclic programming must be utilized at this step, for mentioned previously, the machine needs to
compensate for slippage in the powder. All the samples undergone six 5-minute cycles where one cycle consisted of achieving the maximum compression force and holding it for 5 minutes then cooling down to the minimum force for 0 seconds for an approximate procedure performed for the manual checking for slippage on the manual press.

Table 8 shows the successful compaction data with this design.

Table 8: Second rectangular design compact successes

<table>
<thead>
<tr>
<th>Trial Number</th>
<th>Mass (g)</th>
<th>Maximum Force in Cycle (lb$_f$)</th>
<th>Minimum Force in Cycle (lb$_f$)</th>
<th>Resulting Observation</th>
<th>Corresponding Image</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9.061</td>
<td>45,000</td>
<td>43,000</td>
<td>Crack down the side, unavoidable</td>
<td>Figure 37</td>
</tr>
<tr>
<td>2</td>
<td>9.070</td>
<td>46,000</td>
<td>44,000</td>
<td>Accidentally cracked down the length into 2+ pieces by extracting too fast</td>
<td>Figure 38</td>
</tr>
<tr>
<td>3</td>
<td>9.070</td>
<td>47,000</td>
<td>45,000</td>
<td>Tested bending: cracked down the width</td>
<td>None; same as trial 1</td>
</tr>
<tr>
<td>4</td>
<td>9.069</td>
<td>48,000</td>
<td>46,000</td>
<td>Crack down side; same as trial 1</td>
<td>None; same as trial 1</td>
</tr>
<tr>
<td>5</td>
<td>9.071</td>
<td>49,000</td>
<td>47,000</td>
<td>Crack down side; same as trial 1</td>
<td>None; same as trial 1</td>
</tr>
</tbody>
</table>
Figure 37: Rectangular sample using automated compression machine, trial 1 at 45,000 lb.

Crack down the side. Left: after extraction, before collection, orthogonal view, right: top view.
Compaction trials 3 through 5 were not shown because they observed the same defect as trial 1. After observing the newly welded die and punch, there were a few defects observed that may have been the source of the issue. The bottom face of the die body had a few minor chips missing out of it near the sample compression cavity. Post-welding the punch didn’t seem to slide as tightly and as accurately as before the welding, therefore this could cause uneven forces within the compression cavity and cause a split in the compacts. Since the compaction process with the rectangular die and punch was not perfected, the sponge titanium powder was not used for compaction in the rectangular die and punch.
3.3 Conclusion

The cylindrical die and punch was an easy way to achieve a titanium pellet and the obvious choice over the rectangular die designs. The second die design had many benefits to it over the first design yet wasn’t flawless. The first design was cheaper, had more steps for one compression, would deform under high pressure, and would leak powder due to a scrap metal base plate without features. The second design was more time efficient, more convenient, but had some chips and some internal flaws that damaged the compacts. Overall, the second design was able to withstand higher pressures without deforming as well as extract in a convenient and controlled rate environment. The third design is planning on being a defect-free, smaller cross-sectional area die and punch without any additional modifications.

The cylindrical pellets will be taken to the FSP step as well as the last few rectangular samples, despite the defects, to see the outcome.

The goal of compressing a compact at this stage was to achieve enough green strength to transport the compact to the FSP stage which was achieved.
4.1. Introduction

At the friction stir processing (FSP) stage, several methods were tried for successful welds for both the rectangular compacts as well as the cylindrical compacts. A sample holder was made of 1018 carbon steel block (see Table 7 for composition) and various-dimensioned volumes were milled out throughout this stage to try new methods of sample holding.

4.2. Design 1 with Rectangular Compact

The theory behind this method is to plunge straight down on one end of the compact with enough force to make it mix and accept the FSW tool’s generated heat and consolidate. As the FSW tool runs down the length of the compact, the compact should consolidate with the traverse movement.

4.2.1. Equipment Use

Rectangular compacts were tried first for the size of the compacts was more ideal for the goal of consolidating powder to part at an applicable size. In order to FSP on powder compacts, a sample holder block was made by taking a block of 1018 carbon steel (see Table 7 for composition) of dimensions 118.43 mm long by 152.61 mm wide by 25.03 mm high (to span the width of the FSP working table for convenience) and milling out a 52.35 mm long by 13.48 mm wide by 7.17 mm deep to tightly fit the rectangular samples but have a bit of clearance as too reduce powder leakage, shown in Figure 39.
The first FSP tool that was obtained was a D2M, tungsten alloy (see Table 9 under “Original, as Given” entry for composition and dimension details) made for high-temperature applications and with a hardness higher than that of titanium; this tool was hypothesized as the correct tool to prove the overall goal concept. This tool was modified right away from a shoulder diameter of 16.00 mm to 13.40 mm so the tool could fit into the groove made in the sample holder, the image and dimensions are recorded in Table 9 as “First Modification” entry. Compacted samples 5 and 1 (refer to Table 8) were stacked on top of each other to be closer to the surface of the sample holder. The first attempt at FSP with this method was carried out at 600 RPM, 1 mm/min plunging speed, at a plunge of 7.1 mm, and a traverse speed of 37 mm/min. The second attempt was conducted with the same FSP parameters, with samples 3 and 4, but with another modification to the D2M tool shown in Table 9 as the “Second Modification” entry.
Table 9: D2M (W-Ni-Mo-Fe) tool modifications

<table>
<thead>
<tr>
<th>Shoulder</th>
<th>Pin</th>
<th>Figure Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Diameter (mm)</td>
<td>Height (mm)</td>
</tr>
<tr>
<td>Original, as Given</td>
<td>16.00</td>
<td>15.49</td>
</tr>
<tr>
<td>1st Modification</td>
<td>13.40</td>
<td>15.49</td>
</tr>
<tr>
<td>2nd Modification</td>
<td>13.36</td>
<td>9.48</td>
</tr>
<tr>
<td>3rd Modification</td>
<td>11.08</td>
<td>9.54</td>
</tr>
</tbody>
</table>
4.2.2. Results

The rectangular compacts were not successfully consolidated due to compact not staying put in the sample holder. Figure 40 shows the result from the FSP run at the previously mentioned parameters.

![Figure 40: FSP rectangular compact failure 1.](image-url)

The rectangular compact was believed to not have been consolidated because the pin was not long enough to effectively stir and produce enough heat to consolidate the titanium powder. Figure 41 shows the result from the FSP run at the previously mentioned modified parameters.
A similar result occurred for the second trial as the first trial which was the opposite expected result. This rectangular FSP design was discontinued, while the cylindrical FSP designs were seen through to look for differing results.

4.3. Design 2 with Cylindrical Samples

To avoid damage to the tool as much as possible, a similar theory to the rectangular FSP design was developed. The thinking was to shorten the height as well as the diameter of the pin of the D2M tool and mill a hole in the sample holder barely wider (than the diameter of the compressed cylindrical samples and plunge into the deep hole and consolidate this way. The pin height and diameter are kept in mind when choosing a plunge depth for the goal is to consolidate as much powder as possible and if the volume of the pin is too much, the powder may not consolidate.
4.3.1. Equipment Use

The retrieval method is simply milling a smaller hole through the sample-holding hole with a diameter of 12.60 mm and a depth of 8.03 mm through the block to make a hole for a 3.02 mm-diameter scrap titanium bar to fill the hole when processing and then another 25.60 mm long scrap titanium bar will be used to hammer out the predicted consolidated titanium from the bottom of the sample holder. The new proposed sample block is shown in Figure 42 with the scrap titanium bars.

![Figure 42: Left: zoomed in new cylindrical hole approach, middle: new sample holder block (note the hole on the right was a milling mistake), right: two scrap titanium bars used for planned sample retrieval.](image)

This approach is designed to give approximately 1.0 mm clearance of the tool diameter when plunging into this hole, as well as approximately a 2.0 mm climb for powder to escape,
hopefully minimizing powder leakage. Cylindrical samples compressed at 5,000 lb were used for this method of testing.

4.3.2. Results

The FSP parameters for the first day of attempting this method are listed in Table 10.

Table 10: Initial FSP parameters for the retrieval cylindrical hole approach.

<table>
<thead>
<tr>
<th>Trial</th>
<th>Rotational Speed (RPM)</th>
<th>Plunge Speed (mm/min)</th>
<th>Plunge Depth (mm)</th>
<th>Dwelling Time (sec)</th>
<th>Results</th>
<th>Figure Reference, Top: without disturbance, Bottom (if applicable): after investigational digging</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>800</td>
<td>1</td>
<td>6.0</td>
<td>120</td>
<td>no change</td>
<td><img src="https://via.placeholder.com/150" alt="Image" /></td>
</tr>
<tr>
<td>2</td>
<td>800</td>
<td>1</td>
<td>6.0</td>
<td>120</td>
<td>no change</td>
<td><img src="https://via.placeholder.com/150" alt="Image" /></td>
</tr>
<tr>
<td>3</td>
<td>1200</td>
<td>1</td>
<td>6.0</td>
<td>120</td>
<td>no change</td>
<td><img src="https://via.placeholder.com/150" alt="Image" /></td>
</tr>
</tbody>
</table>
Whilst observing Komarasamy’s report [67], the tool rotational speed and plunge depth and were too high and deep respectively. Therefore Table 11 shows the results of the applied lowered parameters.
Table 11: Parameters used for FSP according to Komarasamy [67].

<table>
<thead>
<tr>
<th>Trial</th>
<th>Rotational Speed (RPM)</th>
<th>Plunge Speed (mm/min)</th>
<th>Plunge Depth (mm)</th>
<th>Dwelling Time (sec)</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>600</td>
<td>0.5</td>
<td>2.0</td>
<td>120</td>
<td>no change</td>
</tr>
</tbody>
</table>

Unfortunately the lowered parameters did not work under the current design, and will be applied for the next design based off of Komarasamy’s report [67].

4.4. Design 3 with Cylindrical Sample

Using Komarasamy’s work [67] as guidance, the third FSP design attempt involves FSP into the sample holder block to completely cut off any leakage escape routes for the compacted powders. There will be leakage from the initial plunge due to volume displacement of the pin but the goal is to keep most of the powder in and force it to consolidate.

4.4.1. Equipment Use

This tight-fit approach involves milling out another hole with a diameter of approximately 10.5 mm and a depth of approximately 6.35 mm in the sample holder. The sample will be inserted into this hole and then FSP at a constant 600 RPM, and 0.5 mm/min plunge speed with varying plunge depths depending on the sample height and varying times depending on the results. A retrieval hole is not milled out to avoid any powder leakage; retrieval involves cutting up the carbon block. A tool steel tool is used to save the life of the D2M tool in case of extreme wear when the shoulder comes into contact with the carbon steel sample holder.
4.4.2. Results

Table 12 shows the plunge depth was varied and attempted a few times to succeed in consolidation for “hole 1”. Once hole 1 was successful, nine more holes were milled out of the exact dimensions to attempt to reproduce or improve upon the success of hole 1. Hole 1 was successful by consolidating a thin outer rim layer and compressing the remaining powder from movement indicating the process needed further plunging for a full solid weld. Results for hole 2 are shown in Table 12 under “hole 2” with the tool steel tool. Although hole 2 results were better than hole 1, the tool was changed to the D2M tungsten alloy tool for the wear and “stuck” titanium and steel on the tool steel tool was significant, shown in Figure 43, and to save time on mechanical cleaning, the tools were changed out and the results compared.

Figure 43: Significant tool wear and material “stickiness” on the tool steel tool.

With the resulting tool wear and stickiness of the titanium product in mind, the D2M tool’s wear and therefore new dimensions are recorded and used as the “new tool” for each run instead
of mechanically cleaning each time. The results of the trials with the D2M tungsten alloy tool and subsequent tool wear can be seen in Table 13 and Table 14 respectively.

At hole 4, the measuring for the calculation prediction of the correct plunge depth was carried out to see if the reproduction of hole 3 could be achieved by this method. Once the cylindrical sample was inserted into the milled hole, using a caliper, the clearance between the top of the sample and the sample holder surface was measured and recorded. When the new pin height was measured it was also recorded then added to the measured clearance to attempt to completely submerge the pin. To assure the shoulder contacted, the sum was raised 0.1 mm. This method proved effective for holes 3 through 5 and then was modified when the powders were switched out. For holes 8 through 10 cases, the second shoulder height was added into the previously used equation and was proved not perfect but better although the theory of FSP twice is also a likely factor to why these outcomes looked better the second time around.
Table 12: FSP reduced parameters using the tool steel tool, 600 RPM, and at a plunge speed of 0.5 mm/min were kept constant.

<table>
<thead>
<tr>
<th>Hole Number</th>
<th>Powder Type (Compressed at lbf)</th>
<th>Trial/Attempt Number</th>
<th>Plunge Depth (mm)</th>
<th>Dwelling Time (sec)</th>
<th>Results</th>
<th>Figure Reference (Left: before vacuum, Right: after vacuum)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Spherical (5,000)</td>
<td>1</td>
<td>2</td>
<td>120</td>
<td>no shoulder contact</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td>Spherical (5,000)</td>
<td>2</td>
<td>2.4</td>
<td>120</td>
<td>little shoulder contact-lot of initial force</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td>Spherical (5,000)</td>
<td>3</td>
<td>2.6</td>
<td>120</td>
<td>Partial success: thin layer consolidated on outside rim</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td>Sponge (5,000)</td>
<td>1</td>
<td>2.6</td>
<td>180</td>
<td>tiny bit consolidated</td>
<td>None</td>
</tr>
<tr>
<td>2</td>
<td>Sponge (5,000)</td>
<td>2</td>
<td>3.6</td>
<td>180</td>
<td>worked, achieved maximum 3,000 N, powder in layers though</td>
<td>None</td>
</tr>
</tbody>
</table>
Table 13: FSP reduced parameters using the D2M, tungsten alloy tool, 600 RPM, at a plunge speed of 0.5 mm/min and dwell time of 180 seconds were kept constant.

<table>
<thead>
<tr>
<th>Hole Number</th>
<th>Powder Type (Spherical or Sponge)</th>
<th>Trial/Attempt Number</th>
<th>Plunge Depth (mm)</th>
<th>Results</th>
<th>Figure Reference (Left: before vacuum, Right: after vacuum)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>Spherical (4,000)</td>
<td>1</td>
<td>4.0</td>
<td>Good looking weld</td>
<td><img src="image1" alt="Figure Reference" /></td>
</tr>
<tr>
<td>4</td>
<td>Spherical (4,000)</td>
<td>1</td>
<td>3.9</td>
<td>Good looking weld</td>
<td><img src="image2" alt="Figure Reference" /></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
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<td>---</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td></td>
<td></td>
<td>3.6</td>
<td>Good looking weld with a round crack defect</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Sponge (5,000 except for hole 10 which was 3,000)</td>
<td></td>
<td>4.3</td>
<td>Okay looking weld with powder layers</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td></td>
<td></td>
<td>3.9</td>
<td>Okay looking weld with powder layers</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>3.4</td>
<td>Failed</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-----</td>
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<td>-----</td>
<td>--------</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>1</td>
<td>3.9</td>
<td>Failed</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>4.4</td>
<td>Not the greatest looking, still powder build ups</td>
<td></td>
<td></td>
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<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>4.3</td>
<td>Not the greatest looking, still powder build ups</td>
<td></td>
<td></td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>1</td>
<td>2.5</td>
<td>Failed</td>
<td>None</td>
<td></td>
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<td>10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>4.0</td>
<td>Not the greatest looking, still powder build ups and separation of layers</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 14: Tool dimension changes due to tool wear for successes by hole number

<table>
<thead>
<tr>
<th>Material</th>
<th>Shoulder Diameter (mm)</th>
<th>Shoulder Height (mm)</th>
<th>Features</th>
<th>Base Diameter (mm)</th>
<th>End/Tip Diameter (mm)</th>
<th>Pin Base Height (mm)</th>
<th>At Shoulder Edge Height (mm)</th>
<th>Features</th>
<th>Figure Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>For Hole 1 &amp; 2</td>
<td>Tool Steel</td>
<td>12.00</td>
<td>11.30</td>
<td>Concave</td>
<td>5.25</td>
<td>4.04</td>
<td>1.81</td>
<td>1.85</td>
<td>Conical, threaded</td>
</tr>
<tr>
<td>For Hole 3</td>
<td>D2M Tool: W-Ni-Mo-Fe</td>
<td>11.08</td>
<td>9.54</td>
<td>none</td>
<td>4.05</td>
<td>4.05</td>
<td>2.05</td>
<td>2.05</td>
<td>none</td>
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<td>For Hole 4</td>
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<td>9.80</td>
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<td>7.88</td>
<td>4.10</td>
<td>2.22</td>
<td>2.22</td>
<td>conical for 1.21 mm height from shoulder, then 1.01 mm until end without features</td>
</tr>
<tr>
<td>Hole</td>
<td>Diameter</td>
<td>Shoulder</td>
<td>Tool Wear</td>
<td>Pin Diameter</td>
<td>Pin Height</td>
<td>Remarks</td>
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<tr>
<td>5</td>
<td>11.08</td>
<td>9.25</td>
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<td>3.53</td>
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<td>1.60</td>
<td>4.09</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>conical pin</td>
<td>from</td>
<td>shoulder face</td>
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<td>6</td>
<td>11.08</td>
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<td>11.08</td>
<td>4.10</td>
<td>2.21</td>
<td>2.21</td>
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<td></td>
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<td>conical</td>
<td>from</td>
<td>shoulder edge</td>
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<td></td>
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<td>to pin edge</td>
<td>shoulder edge</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>tool wear</td>
<td>induced</td>
<td>the</td>
<td>reduction of</td>
<td></td>
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<td></td>
<td>reduction of</td>
<td>part of</td>
<td>part of the</td>
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<td></td>
<td>the shoulder</td>
<td>shoulder to</td>
<td>shoulder to a</td>
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<td></td>
<td>to a diameter</td>
<td>a diameter</td>
<td>diameter of</td>
<td></td>
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<tr>
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<td></td>
<td></td>
<td></td>
<td>of 10.56 mm</td>
<td>of 10.56 mm</td>
<td>10.56 mm for</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>for a height</td>
<td>for a height</td>
<td>a height of</td>
<td></td>
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<td></td>
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<td></td>
<td>of 1.46 mm</td>
<td>of 1.46 mm</td>
<td>1.46 mm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>11.08</td>
<td>9.88 (both shoulders)</td>
<td>tool wear induced the reduction of part of the shoulder to a diameter of 10.56 mm for a height of 1.46 mm</td>
<td>10.56</td>
<td>3.95</td>
<td>1.90</td>
<td>1.90</td>
<td>none</td>
<td>No significant visible difference</td>
</tr>
<tr>
<td>8</td>
<td>11.08</td>
<td>10.01 (both shoulders)</td>
<td>tool wear induced the reduction of part of the shoulder to a diameter of 10.17 mm for a height of 1.16 mm</td>
<td>3.95</td>
<td>3.95</td>
<td>1.60</td>
<td>1.60</td>
<td>none</td>
<td></td>
</tr>
<tr>
<td>For 2nd Trial Hole 8</td>
<td>11.03</td>
<td>1.68 (between shoulders)</td>
<td>tool wear induced the reduction of part of the shoulder to a diameter of 10.51 mm for a height of 1.68 mm</td>
<td>3.39</td>
<td>3.39</td>
<td>1.32-1.65</td>
<td>1.32-1.65</td>
<td>uneven stickiness of titanium on the 2nd shoulder and pin induced a varying pin height</td>
<td>No significant visible change</td>
</tr>
<tr>
<td>---------------------</td>
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<td>-----------------------------</td>
</tr>
<tr>
<td>For Hole 9</td>
<td>11.03</td>
<td>1.69 (between shoulders)</td>
<td>2nd shoulder diameter is 10.05 mm</td>
<td>4.01</td>
<td>4.01</td>
<td>1.61-1.71</td>
<td>1.61-1.71</td>
<td>uneven stickiness of titanium on the 2nd shoulder and pin induced a varying pin height</td>
<td><img src="image1.jpg" alt="image" /></td>
</tr>
<tr>
<td>For 2nd Trial Hole 9</td>
<td>11.03</td>
<td>1.37 (between shoulders)</td>
<td>2nd shoulder diameter is 10.02 mm</td>
<td>3.95</td>
<td>3.95</td>
<td>1.54-1.74</td>
<td>1.54-1.74</td>
<td>uneven stickiness of titanium on the 2nd shoulder and pin induced a varying pin height</td>
<td>No significant visible change</td>
</tr>
<tr>
<td>For Hole 10 (both trials)</td>
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<td>1.43 (between shoulders)</td>
<td>2nd shoulder diameter is 9.58 mm</td>
<td>4.18</td>
<td>4.18</td>
<td>1.72-1.77</td>
<td>1.72-1.77</td>
<td>uneven stickiness of titanium on the 2nd shoulder and pin induced a varying pin height</td>
<td><img src="image2.jpg" alt="image" /></td>
</tr>
</tbody>
</table>
After the results were collected, SEM images were collected at differing magnifications, EDS composition analyses were accumulated as well as hardness mechanical testing data was collected.

4.5. Mechanical Testing

After all ten holes were filled of consolidated titanium samples, the carbon steel sample holder was sent to the shop to be milled down to be easily cut down and grinded from there. The three best looking results were chosen for analysis; the best results were holes 3 through 5. These samples were then cut to expose the cross-sectional area of each weld. These were then grinded with 600, 800, and 1200 silicon carbide metallurgical grinding paper with water then dried with pressurized air to avoid rusting, then chem-pol cloth was used with a solution of 0.05% colloidal silica. Each sample was then immersed in ethanol and put into an ultrasonic cleaner for 5 minutes, with the temperature set to 32°F, 5 power, and high frequency. The samples were each removed from the ultrasonic cleaner and ethanol and quickly dried under pressurized air. Each sample was again cleaned by hand, swab, ethanol, then dried by pressurized air at least three times. The cleaning process was repeated the day of SEM and EDS observation and analysis. Tensile testing was not collected for the cross-sectional areas were not of the proper thickness nor dimensions to have tensile testing samples obtained from them. Figure 44 shows the samples ready for hardness testing, SEM observation, and EDS analyses.
4.5.1. Hardness

Using the Vickers micro-hardness tester shown in Figure 23, 300 gr was forced onto each sample for ten seconds using a diamond indenter. Sixteen times the micro-hardness was collected across the consolidated region including the base material, 1018 carbon steel for hardness comparison. Each hardness test taken was estimated to be in the middle of the consolidated region, calculated and moved by hand to collect comparable results across the three samples. This number of hardness tests taken has exceeded the required number of five hardness tests taken for the ASTM Handbook for Validity of Mechanical Testing [68]. According to [11], nearly pure titanium should have a Brinell hardness of 200 which is between 210 and 220 Vickers hardness according to [69]. According to [6], multiple grades of commercially pure titanium should have a Vickers hardness of 145. Figure 45 shows the positions at which each hardness test was approximately taken.
Figure 45: Cross-sectional area sketch of hardness tests.

Table 15, Table 16, and Table 17, as well as Figure 46, Figure 47, and Figure 48 show the hardness results for sample 3, 4, and 5 respectively.
Table 15: Sample 3 hardness results

<table>
<thead>
<tr>
<th>Distance (mm)</th>
<th>Vickers Hardness (HV0.3)</th>
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<tbody>
<tr>
<td>-4.000</td>
<td>178.9</td>
</tr>
<tr>
<td>-3.000</td>
<td>164.3</td>
</tr>
<tr>
<td>-2.000</td>
<td>199.7</td>
</tr>
<tr>
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<td>8.000</td>
<td>174.6</td>
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<tr>
<td>9.000</td>
<td>212.8</td>
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<td>178.1</td>
</tr>
<tr>
<td>12.000</td>
<td>156.7</td>
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</table>

Figure 46: Sample 3 hardness results.
Table 16: Sample 4 hardness results

<table>
<thead>
<tr>
<th>Distance (mm)</th>
<th>Vickers Hardness (HV0.3)</th>
</tr>
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<tbody>
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<tr>
<td>9.000</td>
<td>153.0</td>
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<td>10.000</td>
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</tr>
<tr>
<td>11.000</td>
<td>158.5</td>
</tr>
<tr>
<td>12.000</td>
<td>149.0</td>
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</table>

Figure 47: Sample 4 hardness results.
Table 17: Sample 5 hardness results

<table>
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<tr>
<th>Distance (mm)</th>
<th>Vickers Hardness (HV0.3)</th>
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<tbody>
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<td>-2.000</td>
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<tr>
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<td>149.6</td>
</tr>
<tr>
<td>12.000</td>
<td>151.1</td>
</tr>
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</table>

Figure 48: Sample 5 hardness results.
The averages of the titanium region of each sample’s hardness is 245.3, 200.7, and 307.4 for sample 3, 4, and 5 respectively. The trend seems to be that in the thickest part of each sample, there is a significant spike of hardness that averages to be 418.4, not applicable, and 409.23 for sample 3, 4, and 5 respectively. Regardless, both sample 3 and 5 either met or exceeded the source [11], [69], and [6] hardness data of bulk titanium. This proves that the friction stir processing not only consolidated the titanium but also improved its hardness properties which makes it probable that other mechanical properties such as tensile strength have also improved.

4.5.2. Optical Microscope (OM) and Scanning Electron Microscope (SEM)

Optical microscopy was used to see the “before” images of the compressed spherical powder and scanning electron microscopy was used for the cross-sectional “after” images of the best-looking welds. Figure 49 and Figure 50 show the compacted spherical titanium powder at two magnifications.
Figure 49: Optical microscopy image of compacted spherical powder at 50 μm scale or 10x magnification.

Figure 50: Optical microscopy image of compacted spherical powder at 20 μm scale or 20x magnification.
The surface looks relatively flat while in the porous (blank areas) the spherical particles can be seen made out to be just floating indicating not a stable compression. Figure 51 shows regions in which the corresponding SEM images were taken from.

![Cross-Sectional Area Sketch with SEM Region Indications](image)

**Figure 51: SEM image regions.**

Region A is a macroscopic image taken between 40x and 45x magnification for a basic look at the cross section. Region B describes the area around the pin in which should indicate the most mixing and taken at 10,000x magnification. Region C and D are taken at 10,000x magnification and are interfaces of either side that can be further studies to understand how the materials are mixing and how to possible optimize the FSP without mixing of the carbon steel.
Figure 52: Sample 3 SEM results, top: A region (40x), bottom left to right (10,000x): C region, B region, and D region.
Figure 53: Sample 4 SEM results, Top: A region (45x), bottom left to right (10,000x): C region, B region, and D region.
Figure 54: Sample 5 SEM results, top: A region (40x), bottom left to right (10,000x): C region, B region, and D region.
In sample 3 (Figure 52), the consolidated powder section is approximately 1-2 mm deep and be distinguished by the dark uniform area between the white-streak area (FSP weld) and the disorganized clumpy area (unconsolidated powder area). In region B, the FSP weld edge can be seen at the top of the image while the consolidated titanium area consumes most of the image. In region C, the consolidated titanium can be distinguished as the darker gray material on the right side of the image while in region D, the opposite remains true. All regions B-D seem to define the mixing of the titanium very well and a clean interface.

In sample 4 (Figure 53), all regions are distinguished in relatively the same manner as sample 3. The consolidated powder section seen in region A is approximately less than 1 mm deep but is marginally shallower than sample 3 making it the most shallow sample out of all three, this may account for the hardness staying below 300. Region C and D showed relatively more mixing than sample 3 that left a less defined interface separation between the titanium and the carbon steel and the mixing of the steel was deposited into region B were a few particulates are show in the darker uniform area.

In sample 5 (Figure 54), all regions are distinguished in relatively the same manner as sample 3 and 4. The consolidated powder section seen in region A is approximately 2 mm deep making it the deepest consolidated region of all three samples correlating to the highest peak hardness values for more of the sample length than any of the samples. Although the D region defines a relatively clean interface between the carbon steel and the consolidated titanium, region C and region B show evidence of mixing.

Compared to the OM images, every sample benefited from being consolidated as well as FSP for improvement in microstructural properties.
4.5.3. Energy Dispersive Spectroscopy (EDS)

EDS is a separate functionality of the SEM that allows for compositional breakdown of an image in the 10,000x to 15,000x magnification range. Figure 55 shows where in the cross section of each sample the EDS function of the SEM was used.

Cross-Sectional Area Sketch with SEM and EDS Region Indications

Figure 55: SEM regions used for EDS.

All SEM images corresponding to EDS were taken at 10,000x magnification for analysis. Figure 56 through Figure 67 show the EDS results from samples 3 through 5 of region A through D with titanium shown as red, iron shown as green and tungsten shown as blue.
Figure 56: Sample 3, region A (10,000x), left to right, top to bottom: SEM image, titanium (Ti) content, peak chart.

Figure 57: Sample 4, region A (10,000x), left to right: SEM image, titanium (Ti) content, iron (Fe) content, peak chart.
Figure 58: Sample 5, region A (10,000x), left to right, top to bottom: SEM image, titanium (Ti) content, iron (Fe) content, peak chart.

Figure 59: Sample 3, region B (10,000x), left to right, top to bottom: SEM image, titanium (Ti) content, peak chart.
Figure 60: Sample 4, region B (10,000x), left to right, top to bottom: SEM image, titanium (Ti) content, iron (Fe) content, tungsten (W) content, peak chart.

Figure 61: Sample 5, region B (10,000x), left to right, top to bottom: SEM image, titanium (Ti) content, iron (Fe) content, tungsten (W) content, peak chart.
Figure 62: Sample 3, region C (10,000x), left to right, top to bottom: SEM image, titanium (Ti) content, iron (Fe) content, peak chart.

Figure 63: Sample 4, region C (10,000x), left to right, top to bottom: SEM image, titanium (Ti) content, iron (Fe) content, peak chart.
Figure 64: Sample 5, region C (10,000x), left to right, top to bottom: SEM image, titanium (Ti) content, iron (Fe) content, peak chart.

Figure 65: Sample 3, region D (10,000x), left to right, top to bottom: SEM image, titanium (Ti) content, iron (Fe) content, peak chart.
Figure 66: Sample 4, region D (10,000x), left to right, top to bottom: SEM image, titanium (Ti) content, iron (Fe) content, peak chart.

Figure 67: Sample 5, region D (10,000x), left to right, top to bottom: SEM image, titanium (Ti) content, iron (Fe) content, peak chart.
Figure 56 through Figure 58 show that the correlation between sample 3 through 5 in region A (or the middle of the FSP zone) is that the consolidated area is mostly titanium with sample 4 containing the most iron in this area.

Figure 59 through Figure 61 show that the correlation between sample 3 through 5 in region B (or the upper edge of the FSP zone) is that the consolidated area is mostly titanium and little iron in the mix with a little tungsten from the tool in the mix with sample 5 possessing the most contaminants. Because sample 5 had the most tungsten in the mix, one may assume that the high hardness values could be a result of this area with tungsten. The hardness tests were not taken in this area; however, the tungsten may have a little effect on the upper hardness of the weld.

Figure 62 through Figure 67 show that the correlation between sample 3 through 5 in region C and D (or the left and right interfaces respectively) is that the carbon steel and titanium mixed minimally at these points.

4.6. Conclusion

Not mechanical cleaning the tungsten alloy tool may have caused more tungsten to be deposited in sample 5. The thickness in sample 5 may be built up titanium from the other sample runs, not from the individual run itself, although sample 4 does not support the theory. Having a “new” tool and therefore new tool features after each run could have affected the next run as well. Rounding to the next tenth of a millimeter for the plunging calculation could have an effect on how much material mixed, and stuck. Knowing these errors, the FSP proved to be effective in consolidating as well as improving the hardness of the powder titanium.
CHAPTER 5
CONCLUSIONS AND FUTURE WORK

5.1 Conclusion

The titanium powder can be mechanically compressed into a cylindrical shape then friction stir processed in a tight holder to become solid and harder than the original hardness property of commercially pure titanium. The overall consolidated region of the best welds was between 1mm and 2mm deep while the trend remained: the thicker the processed/consolidated region, the longer the spiked hardness was throughout the region. The highest recorded hardness was 435.9 found on sample 5 which according to the source is 2 to 3 times higher than the recorded hardness for commercially pure titanium. The averages of the spiked regions of each sample were 418.4, not applicable, and 409.23 with the overall average from one interface to the interface on the opposite side being 245.3, 200.7, and 307.4 for sample 3, 4, and 5 respectively.

The overall SEM and EDS results show that at the surface of the FSP weld, tungsten is found mixed from tool deposits while in the middle of the consolidated zone, tungsten is not found and is relatively comprised of all titanium with a homogeneous microstructure with small deposits of iron. The interfaces on both sides show relatively clean separation between the carbon steel sample holder material and the consolidated titanium region.

The thicker processed region in sample 5 could have been from using the same tool without mechanical cleaning therefore titanium from previous runs could have stuck to the tool and deposited on the new sample run making sample 5 the thickest, hardest sample; this theory would also describe why sample 4 is very thin.
5.2 Future Work

The goal is to allow titanium to be used for more than just aircraft. Even though this thesis described the basis of the proposed titanium production steps, later production will involve complex molds for the FSW to make usable components, and with a few runs of the machine over the area, it is hoped that the mechanical properties of the consolidated titanium can be controlled and then further improved for many applications. The details of this overall future goal include but are not limited to trying:

- Variation of powder size and composition
- Tungsten and other tools with features and mechanical cleaning
- Titanium sample holder block
- Alloying the titanium before compaction
- Alloying the titanium by using a gradient method
- Different exothermic reactions during the FSP step to increase heat production
- Continuation of using the rectangular compact shape

The future work can be channeled towards many industries with many extended applications everything from the consumer’s products to air force industrial jets.
APPENDIX A

COMPACT IRON C-CLAMP SPECIFICATIONS [71]
<table>
<thead>
<tr>
<th>Feature</th>
<th>Value</th>
</tr>
</thead>
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</tr>
<tr>
<td>Opening Maximum (in)</td>
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</tr>
<tr>
<td>Holding Capacity (lb)</td>
<td>400</td>
</tr>
<tr>
<td>Material</td>
<td>Iron</td>
</tr>
</tbody>
</table>
APPENDIX B

FRICION STIR PROCESSING SUCCESSFUL CODE EXAMPLE
Major Notes: Hole 4, Spherical Titanium Powder, Tungsten Alloy Tool, 03.30.2016

[Code Start]

(UNIVERSITY OF NORTH TEXAS RUN OFF-REGRESSION TEST)

(MATERIAL - N/A)

(SHOULDER PART NUMBER: N/A)

(PIN PART NUMBER: )

(SET PIN LENGTH: 2.22 mm tungsten tool no features)

(TOOL LENGTH: 142.61 MM)

(HEAD TILT = 0)

(*****PROGRAM SETUP*****)

G60.6 A0 (SET MACHINE IN JOINT MODE)

G17 (XY PLANE)

G40 (CANCEL CUTTER COMPENSATION)

G90 (ABSOLUTE DIMENSION INPUT (M,L))

G94 (FEEDRATE IN IMP OR MMPM (M,L))

G54 (Coord System 1)
G26 (SPINDLE DETECT ON)

M48 (ENABLE OVERRIDES)

(M49 (DISABLE OVERRIDES)

M90.1 (FEEDRATE - FRAX - XYZ CONTROL SETTING)

(M90.2 (FEEDRATE - FRAX - UVW CONTROL SETTING)

(M90.3 (FEEDRATE - FRAX - XYZUVW CONTROL SETTING)

(M90.4 (FEEDRATE - FRAX - XYZABC CONTROL SETTING)

(M90.5 (FEEDRATE - FRAX - UVWABC CONTROL SETTING)

(M90.6 (FEEDRATE - FRAX - XYZABCUVW CONTROL SETTING)

G43.1H1 (CALL TOOL OFFSET 7)

(*****EXTRACT SETUP*****)

(G60.9 Define EXTRACT Features)

(   A[REL Depth1] I[Velocity1])

(   B[REL Depth2] J[Velocity2])

(   W[ABS F1pos] K[Velocity3])

(   R[OUT MASK] P[OUT Prev] Q[OUT Post])

(   ADD THE FOLLOWING TOGETHER TO GET DESIRED CONTROL)

(   1 = ARGON)

(   2 = PROCESS MONITOR ARGON)

(   4 = TOOL COOLANT)

(   8 = PROCESS MONITOR TOOL FLOW)

(   16 = ANVIL COOLANT)

(   32 = PROCESS MONITOR ANVIL COOLANT FLOW)
( 64 = PROCESS MONITOR MAX COOLANT TEMP)
( 128 = AIR BLAST)
( 256 = HOLD DOWN CYLINDERS)
( 512 = PROCESS MONITOR HOLD DOWN PRESSURE)

G60.9 A1 I30 B1 J500 W0 K1000 C-2.7 R0P0Q0 U70 V11.4 (Upper Head)

(M07  (TURN TOOL COOLANT ON)

(*****APPROACH PART*****)

G00 Z30  (RAPID MOVEMENT / Z START POSITION)

G60.3  (RESET FORGE F1-Axis)

M150  (RESET F1F LOAD CELLS)

G00 X0 Y0 (A0 (PLUNGE LOCATION 1)

M87  (Turn ON Datalog)

G01 Z10 F400  (FEED MOVE TO POSITION WITH FEEDRATE MM/MIN)

M4 S600

  (CCW @ RPM)

  (FEED MOVE TO POSITION WITH FEEDRATE MM/MIN)

G01 Z2 F25

G01 Z0 F1

(*****DRY PLUNGE*****)

G60.1 W-3.90 Z-4.00 V0.5

G25  (SPINDLE DETECT OFF)

(*****DWELLING*****)

100
G04 P180 (dwell for 3 minutes)

(*****EXTRACT*****)

G60.9

M88  (Turn OFF Datalog)

M88.1 (save data log)

(*****STOP SPINDLE*****)

M5  (STOP SPINDLE)

(M09  (TURN TOOL COOLANT OFF)

(*****MOVE CLEAR OF PART*****)

G00 Z30

M30

[Code End]
REFERENCES


[65] Online link:

[66] Online link:


