Dissimilar Metal Joining in the Medical Device Industry

Thesis

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ABSTRACT

The medical device industry has stringent requirements for reliability, first time quality, biocompatibility, and process capability for its components and devices. The requirement of biocompatibility limits the materials available for use in long term implant applications and often requires the joining of exotic dissimilar metals. This provides a significant challenge when creating a robust weld schedule.

This thesis contains two case studies in dissimilar metal welding on the small scale. The first chapter is focused on characterization of cross wire platinum to niobium micro resistance spot welds. The chapter details how small changes in processing conditions and material can have a profound effect on microstructure, defects, and mechanical properties. Transmission electron microscopy, nanoindentation, and micro pillar compression experiments were used to optimize the properties and characterize the resulting structure, improving the robustness of the joint.

The second chapter utilized statistical methods to improve the microstructure and properties of a bulk metallic glass (Vitreloy 105) to titanium laser weld. A definitive screening design (DSD) experiment was performed to understand the effect of 11 different factors on the properties of a laser weld. The results showed pulse width, use of a zirconium filler metal, offset of the laser beam from the joint center, and pulse shape to be the prominent factors controlling the hardness and modulus of the weld.
Dedication

Dedicated to my son Brian
“We’ll enlighten you
death brings rebirth
soon you will bloom
waiting my turn again.”
(Todd Smith, 1998)
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Fields of Study

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INTRODUCTION

The objective of the research conducted for this thesis was to provide an overview of common dissimilar metal joining techniques utilized in the medical device industry and provide examples of experimental design and characterization techniques used. The uncommon material combinations which are often required to be joined in a robust and reliable manner seldom have published peer-reviewed literature that can be referred to; meaning the development of new joints must be started without the assistance of prior researcher’s work.

These constraints pose a problem as time to market and cost down are important metrics that must be met during the process and product development portion of the overall development timeline. For these reasons this thesis demonstrates the use of designed experiments that allow statistical models to be created for process optimization and how advanced characterization tools can be used as responses in these efficient experiments. Once the statistical analysis has been completed many of these advanced analytical techniques can be used to obtain information on the micro and nano scale that can be used to collect variables data and directly compare microstructure and properties between the optimized and original joints. Micro-pillar compression experiments and post mortem TEM can be used to analyze the weld zone response to a severe plastic deformation while micro-cantilever beam bending experiments are able to resolve the changes in local fracture properties. The results and conclusions of this thesis show that advanced characterization
and statistical methods can be used to accelerate the process development cycle and also increase reliability and quality of the resulting welds.

Dissimilar metal joining has been a challenge for engineers and scientists since the industrial revolution. Steels with differing amounts of carbon or other alloying elements, for example, require special understanding of phase transformations, kinetics, heat transfer, and creating large libraries of experimentally measured constitution diagrams (1-8).

Similarly, joining differing grades of nickel or nickel to steel alloys to each other requires special attention to heat input, dilution of solute elements, or dissolution of strengthening phases (9-12). Fusion welding processes such as arc, laser, and electron beam welding involve melting the work pieces and mixing them together. Arc welds often utilize filler metals that can be crucial for controlling weld zone chemistry and resulting microstructure. The use of filler material in laser and electron beam welding is not as common, and the fast cooling rates observed in these welds often result in fusion zones with inhomogeneous mixing of solute elements and non-uniform microstructures (13, 14). Many of the challenges associated with dissimilar metal joining can be mitigated by using solid state joining techniques such as friction stir, upset, ultrasonic, or in some cases, resistance welding (15).

Process development and manufacturing in the medical device industry with the push for miniaturization requires dissimilar materials with small geometrical features be reliably joined. The small part sizes and their delicate nature make friction stir and upset welding impractical. Ultrasonic micro welding is used to join thin foils to wires or films but as with friction stir and upset welding, ultrasonic joining is limited by part geometry and is best suited for soft materials such as copper, aluminum, and gold (16). Micro resistance spot
welding provides the flexibility of welding materials with different geometries, hardness values, and dissimilar metals (17).

In the medical device industry, where miniaturization is driven by the need for less invasive therapies and implantable devices that are highly reliable, there is a need for sophisticated micro joining techniques and methods to characterize them during process development. Furthermore, the requirement that materials are biocompatible and biostable for long term implant applications significantly reduce the number of metals and alloys available for use. Examples of metallic materials commonly utilized are platinum-iridium, gold, titanium and its alloys, MP35N (Co-35Ni-20Cr-10Mo), niobium, and tantalum. An assembled medical device may contain many of these materials in various components that must be joined together for an electrical connection, a mechanically loaded interface, or a hermetic seal. These requirements mandate the joining of oftentimes wildly dissimilar materials.

This body of work contains two chapters, each investigating a different dissimilar material system used in the medical device industry and different joining techniques. The first chapter investigates platinum to niobium cross wire micro-resistance weld. The material combination while unconventional was carefully selected. The base materials and joint are used in an environment that requires corrosion resistance (for body and possible drug contact), biocompatibility, low electrical resistivity, and extreme temperature stability for brazing processing of lower level components.

The use of advanced characterization techniques such as analytical scanning transmission electron microscopy, high resolution imaging, and nano mechanical testing, and finite element modeling can be used in conjunction with designed experiments (DOEs) to
optimize challenging welds and characterize the effects of noise variables not often characterized such as material surface roughness.

The second chapter reports the results of an 11 factor, three level definitive screening design (DSD) experiment to optimize a Vitreloy 105 (LM105) bulk metallic glass to grade 2 commercial purity titanium laser seam weld. The Vitreloy 105 to grade 2 titanium joint, while not commercially used at this time, has many possible applications for medical devices. The attractive material properties of Vitreloy 105 make it an excellent candidate for use in medical devices, however the intrinsic limitations on part size require the material be joined in some fashion. In this case, to one of the most commonly used materials in the medical device industry, grade 2 titanium. The 11 process factors studied range from laser parameters such as peak power and pulse width to factors that may control the local chemistry or cooling rate of the fusion zone. Nanoindentation, field emission gun scanning electron microscopy/energy dispersive spectroscopy (FEG SEM/EDS), weld penetration measurements, and visual inspection were used as responses for analyzing the experiment, and the optimized weld schedule was investigated closer. The fracture properties of the joint were investigated on a local level using micro cantilever beam testing to gage the consistency of fracture properties within the weld zone. Correlation of local fracture toughness with microstructure and chemistry is discussed as a path to improve dissimilar metal laser welds by controlling chemistry and local structure.
CHAPTER 1: OPTIMIZATION AND CHARACTERIZATION OF A DISSIMILAR PLATINUM TO NIOBIUM MICRO RESISTANCE WELD

BACKGROUND

This chapter will discuss the microstructural evolution and resulting properties of niobium micro resistance welded to platinum as it relates to welding process parameters. Platinum and niobium are biocompatible, biostable, corrosion resistant in biomedical applications, electrically conductive, and microstructurally stable. While both materials are commonly used for implant applications, the available literature on platinum-niobium alloys, especially joining them is sparse; the most current being the experimentally measured equilibrium phase diagram, shown in Figure 1 indicating the presence of numerous intermetallic compounds when the materials are mixed. (18). To the author’s knowledge, no data on welding platinum to niobium exists in open literature.
Figure 1 - Platinum-niobium phase diagram [18] Reprinted with permission from Springer.

Despite this, the stringent requirements for materials used for implant applications limit the number of available metals and alloys that can be implanted in a high risk medical device application. For this reason, medical devices require dissimilar materials be joined routinely. The less commonly utilized metallic materials used in the medical device industry include MP35N, niobium, tantalum, tungsten, β-titanium alloys, platinum, iridium, and gold. The phase diagrams for many of these binary alloys contain large numbers of intermetallic compounds with low symmetry crystal systems making mechanically robust joints difficult to produce (19). These issues coupled with a lack of
peer reviewed scientific literature require a detailed characterization of the microstructure-properties-processing relationship for unique welds be performed during product development. This chapter will discuss the basics of micro resistance welding and a brief literature review of micro resistance welding with a focus on the medical device industry.

MICRO RESISTANCE WELDING

Resistance spot welding utilizes pressure and localized heating from the contact resistance between two work pieces to create a weld zone through local melting and fusion or a short range diffusion controlled solid state joint. A schematic image showing an opposed electrode micro resistance weld process is shown in Figure 2

![Schematic diagram of the opposed electrode resistance weld process](image)

Figure 2 - Schematic diagram of the opposed electrode resistance weld process [17].

The heat input to the work pieces (and electrodes) is described by Equation 1, $Q=I^2Rt$ where, $Q$ is the energy in Joules, $I$ is the current, $R$ is resistance of the system, and $t$ is welding time. The resistance value contains seven components: four material resistances (the top electrode, bottom electrode, top work piece, bottom work piece) and
the three interface contact resistance values (contact between top electrode and work piece, contact between bottom electrode and bottom work piece, and contact between the work pieces, also known as the faying interface) (16). If the appropriate electrode materials are selected and they have a low resistance, such as copper, the highest resistance of the seven will be the contact resistance at the faying surface. The contact resistance at this surface is controlled by cleanliness of the work pieces, their surface roughness, hardness, and the contact pressure. The joule heating occurring from the application of current over time and local resistance at the faying surface results in melting. The electrode force is maintained momentarily until the weld can solidify.

It is important, before continuing, to discuss the impact of each process input on the welding process. The welding current is arguably the most important as it scales the heat generated by $I^2$. The large effect of this process parameter requires welding current be controlled using a power supply that controls the root mean square (RMS) current. Too little current will result in no weld, while excessive current may result in large amounts of heating that cause expulsion and softening of the base materials leading to possible weld failures. For these reasons, the welding current process window should be optimized using carefully designed experiments.

The process time, also an important factor and directly proportional to heat input according to Eq. 1, must be understood for the development of an effective and robust weld schedule. The contact resistance at the work piece interface and base materials rapidly changes as the weld occurs. As time increases the contact resistance at the faying interface rapidly decreases and the resistance of the work pieces gradually increases. For this reason, the time process window should remain shorter in the region where contact resistance
dominates. Increasing welding times may not be an effective method of improving weld quality as heating of the work pieces will increase, which may not result in a joint if the current is too low.

Lastly the electrode force is one of the most important process factors for creation of a robust weld schedule. As the electrodes deform the workpieces local asperities are pushed together and the native oxide is broken up, decreasing the contact resistance. However, large amounts of force create more contact area at the faying interface, thus decreasing the current density at the interface. If the welding force is at high values, contact resistance at the faying surface will decrease and require increased welding current and time to form a joint. If the welding force is set at a low enough level, the contact resistance may reach high enough levels to result in blown welds from the near instantaneous heat generation. This relationship is shown in Figure 3 below.

![Figure 3 - Schematic diagram showing effect of welding time on contact and bulk resistance values (20).](image)

Micro resistance welding is a specialized joining technique used mostly in the medical device and micro electronics industries. For this reason, literatures related to the microstructure-properties-processing relationships of micro resistance welds are sparse.
Small amounts of published literature on the subject are available that relate to more commonly used materials such as stainless steel, nickel, platinum-iridium alloys, gold coated nickel, beta titanium, and platinum.

Fukumoto et al. (21) studied the process-microstructure-properties relationship for thin austenitic (300 series) stainless steel sheet resistance welds. The results of his study showed a fairly narrow weld lobe diagram with a process window of approximately 200 amps. Figure 4 shows the evolution of weld zone microstructure as welding current and time were increased. As the heat generation at the faying surface increased, the amount of local melting increased resulting in a larger fusion zone and local deformation accompanied by increasing joint shear strength (21).

Figure 4 - Evolution of fusion zone nugget size as welding current and time were increased in a 304 stainless steel sheet micro resistance weld (21) reprinted with permission from Elsiver.
Another commonly used material and joint configuration in the medical device industry are beta titanium alloys in an overlap wire condition. Iijima et al. studied the microstructure and properties of fine wire β-Ti alloy resistance welds as welding electrode size was varied. In this study, the authors altered process parameters (current and force) as the weld electrode surface area increased. The welding time and temporal profiles of the weld were reported in this study. The authors used micro x-ray diffraction, metallography, and SEM analysis to characterize the evolution of microstructure and properties (22). Unfortunately, no phase evolution was resolved with x-ray diffraction, poor metallographic sample preparation and etching made resolving microstructural differences difficult (seen in Figure 5), and the pull strength data showed large amounts of spread with no concrete hypothesis as to why. The unclear results and conclusions of this study are a strong reminder that there is no replacement for a thorough transmission electron microscopy (TEM) analysis.

Figure 5 - Overlap β-titanium micro resistance welds showing effect of electrode force, weld time, and weld current on resulting joint [22]. A shows a lower current/force weld while B shows the result of excessive force. Reprinted with permission from Wiley Periodicals, Incorporated.
Fukumoto et al. also investigated the effect of weld process (current, force, time) on the strength and microstructure of crossed wire nickel resistance welds. The results of the investigation were consistent with the theory of micro resistance welding discussed in the background section above. Welding time and current increases only improve weld properties until the heating becomes high enough to create a large heat affected zone and eventually weaken the joint and surrounding HAZ (23). Similarly, increasing the weld force improves the joint strength until there is too little contact resistance to result in adequate heat input and the joint fails at low loads (23). These results are summarized in Figure 6. This study also experimented by altering one factor at a time (OFAT) and did not account for interactions between process variables or how the local microstructure is influenced at the edges of the process window versus the optimized weld parameters presented.

![Figure 6 - Process characterization results for nickel cross wire micro resistance welds (23). Figures reprinted with permission from Taylor & Francis.](image)

Further work on gold plated nickel wire resistance welds published by Fukumoto et al. investigated the effect of a 4 µm gold plating on nickel wire resistance weld parameters (current ranging from 250 A to 800 A, welding times of 1 ms or 80 ms, and a welding force...
held constant at 4 kgf) and the respective joints. The results of this study showed the weld zone microstructure evolved from a brazed interface caused by local melting of the gold coating, to a fusion joint where both workpieces melted and formed a homogenous weld (24). As with previous studies, this joint strength reached a maximum as weld current increased followed by a sharp decrease in strength with welding currents over 600 A (24). Interestingly, the expulsion of gold/nickel liquid on many of the joints created a fillet that filled in notches that were found to be stress risers in welds between bare nickel wires (24). The evolution of the joint microstructure as heat input was increased is shown in Figure 7.
Figure 7 - Evolution of gold plated nickel wire micro resistance weld microstructures as weld parameters are increased, increasing the energy input (24). Figure reprinted with permission from Springer.

The literature review of resistance micro welding of thin wires and sheets concludes with separate articles focused on joining platinum wire to 316 stainless steel. The first study by Chen et al. investigated a pure platinum wire to 316L stainless steel block using an
undefined electrode configuration. Similar to the previously mentioned studies, welding current and time were plotted against fracture load (25). The weld interfaces were also characterized using SEM-EDS to better quantify the chemistry. However, the weld zones were in many cases, sub-micron in width, seen in Figure 8, which is much smaller than the interaction volume and therefore, resolution of the EDS measurements reported.

Figure 8 - Embedment and fusion zone evolution in a dissimilar platinum-316 stainless steel cross wire resistance weld as input energy is increased (25). Figure reprinted with permission from Springer.
The final paper by Huang et al. investigated Pt-10Ir to 316 LVM cross wire resistance welds with a focus of increasing the robustness of the dissimilar metal joint by the addition of another welding pulse. The second pulse was optimized for welding current and electrode force (26). Figure 9 shows the effect of the welding current and pressure in the second welding pulse with respect to joint breaking force (JBF).

Figure 9 - Characterization results showing effect of second pulse parameters on joint breaking force (JBF) (26). Figure reprinted with permission from Taylor & Francis.

The currently available literature related to micro resistance welding reveals gaps in experimental techniques, design, and rigor that are required for the development of robust and reliable micro resistance welds used in life saving and life sustaining medical devices. The papers reviewed above all use a one factor at a time approach to experimental design. This is an inefficient method of experimentation that will not yield information on
interactions (the interaction of time and current for example) which are often critical for a complete characterization of the process inputs and responses.

The microstructural evaluations were insufficient to determine a microstructure-properties relationship for the small scale resistance welds analyzed. In most cases cross sections and metallographic analysis were utilized to gage joint quality. In some cases, no information was yielded ($\beta$-Ti wire welds), and other times the resolution of optical and standalone scanning electron microscopy was not sufficient to characterize the fusion zone. In cases where micro x-ray diffraction was used, the spot size was not specified but the weld zone size and fine microstructures formed would likely make identification of new phases difficult.

The final gap in the existing micro resistance welding literature is related to the testing of mechanical properties and performance. Pull, tear, or tension testing are useful tools for process monitoring during production, but as the process windows are tight in many dissimilar metal joints and most welds have reasonably high strength (but low ductility) at time zero or immediately following a weld cycle, pull testing only tests the heat affected zone of the base material where in many cases local annealing/softening has occurred and information on the weld zone is lost. A deeper understanding of process-properties-microstructure relationships is critical during the process and product development cycle for these reasons.

This chapter will show how designed experiments, advanced analytical electron microscopy, and nanomechanical testing can be utilized to characterize micro resistance welds used in the medical device industry and can improve process capability, yield, product reliability, and performance.
The objective of this study is to improve the platinum-niobium resistance weld by decreasing weld defects such as porosity, increasing the uniformity of the interfacial region and the microstructure and phases present in the weld zone.
EXPERIMENTAL PROCEDURE

MATERIALS AND WELDING PROCESS

Niobium and platinum wire samples were obtained and the wires were conditioned using subsequent manufacturing processes to create their higher-level components so they experience the same processing conditions as a real part (this includes parts cleaning processes, brazing, crimping, welding, etc.). This includes general handling, high temperatures, polymer coating, and cleaning processes. Table 1 lists the samples used for this study and their sample designations. The chemical composition for each sample were measured using inductively coupled plasma – optical emission spectroscopy (ICP-OES) in our lab and listed in Table 1 and material properties are listed in Table 2.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Nb</th>
<th>Pt</th>
<th>Hf</th>
<th>C</th>
<th>H</th>
<th>N</th>
<th>O</th>
<th>Ru</th>
</tr>
</thead>
<tbody>
<tr>
<td>Niobium Wire A</td>
<td>Balanc e</td>
<td>N/A</td>
<td>0.01 1</td>
<td>0.0083</td>
<td>0.0001</td>
<td>0.003 7</td>
<td>0.0093</td>
<td>N/A</td>
</tr>
<tr>
<td>Niobium Wire B</td>
<td>Balanc e</td>
<td>N/A</td>
<td>0.01 1</td>
<td>0.0011</td>
<td>0.0004</td>
<td>0.004 2</td>
<td>0.0086</td>
<td>N/A</td>
</tr>
<tr>
<td>Platinum Wire</td>
<td>N/A</td>
<td>Bal.</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
<td>0.007 4</td>
</tr>
</tbody>
</table>

Table 1 - Elemental composition of Platinum and Niobium samples. Values are wt. %.
Table 2 - Selected material properties of the platinum and niobium wires used in this study. Mechanical properties measured using tension testing and nanoindentation, resistivity and melting temperature from (27).

<table>
<thead>
<tr>
<th>Material</th>
<th>Yield Stress (MPa)</th>
<th>Tensile Stress (MPa)</th>
<th>Modulus of Elasticity (GPa)</th>
<th>Resistivity (nΩ·m)</th>
<th>Melting Temperature (K)</th>
<th>Diameter (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Niobium</td>
<td>153</td>
<td>279</td>
<td>105</td>
<td>152</td>
<td>2750</td>
<td>382</td>
</tr>
<tr>
<td>Platinum</td>
<td>168</td>
<td>197</td>
<td>168</td>
<td>105</td>
<td>2041</td>
<td>304</td>
</tr>
</tbody>
</table>

The four welds selected for comparison in this study are representative of a welding process before and after a design of experiments (DOE) study. Conditions A and B represent the legacy weld schedule. After a carefully planned design of experiments (DOE) to optimize the width of the interface and minimize interfacial porosity, the optimal weld settings were then tested and are represented in conditions C and D. The initial DOE cannot be discussed in detail for confidentiality reasons but used weld zone interface width, pull strength, and a qualitative porosity score taken from FIB cross sections as responses. The welding parameters used for each sample condition can be found in Table 3. The DOE was a full factorial design and included electrode force, welding current, and time. The electrode polarity was kept constant with the platinum wire on the negative electrode. All welds were performed using a Miyachi Unitek Model 1-284-01 resistance welder with class II copper electrodes in the opposed electrode configuration. Surface roughness values were measured using a Veeco optical profilometer in 5 locations on each wire and the results averaged.
<table>
<thead>
<tr>
<th>Condition</th>
<th>Electrode Polarity</th>
<th>Current (kA)</th>
<th>Electrode Force (N)</th>
<th>Average Niobium Surface Roughness (Ra, nm)</th>
<th>Weld Duration Upslope/Hold/Downslope (ms)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Pt on (-) electrode</td>
<td>0.500</td>
<td>13.34</td>
<td>938.0 (Wire A)</td>
<td>25/22/2</td>
</tr>
<tr>
<td>B</td>
<td>Pt on (+) electrode</td>
<td>0.500</td>
<td>13.34</td>
<td>938.0 (Wire A)</td>
<td>25/22/2</td>
</tr>
<tr>
<td>C</td>
<td>Pt on (+) electrode</td>
<td>0.430</td>
<td>14.01</td>
<td>938.0 (Wire A)</td>
<td>3/2/0</td>
</tr>
<tr>
<td>D</td>
<td>Pt on (+) electrode</td>
<td>0.430</td>
<td>14.01</td>
<td>357.6 (Wire B)</td>
<td>3/2/0</td>
</tr>
</tbody>
</table>

Table 3 - Welding Parameters Used For This Study.

**Metallographic Sample Preparation**

All samples were mounted for metallographic preparation with the niobium wire in the longitudinal direction and the platinum wire in the transverse direction. All samples were mounted in ProbeMet conductive mounting media, ground to the center of the niobium wire, and prepared using standard metallographic techniques (28) using a Buehler Ecomet/Automet 250 automatic grinder-polisher followed by a vibratory polish to remove residual surface deformation. All cross sectioned joints were imaged with a Zeiss Axiovert Observer A1M metallographic microscope to visualize the morphology of the welds in the as polished condition. No suitable etchants that revealed the microstructure of both materials and weld zone could be found.

**Transmission Electron Microscopy**

Samples for transmission electron microscopy and micro pillar compression experiments were fabricated on FEI Versa and Scios DualBeam instruments equipped with an FEI EasyLift system. Films 15 µm wide for transmission electron microscopy were lifted out from the center of the weld zone, rotated 90°, and thinned with 30 kV Ga⁺ ions to a
thickness of 100 nm, and final thinned to electron transparency with an ion beam accelerating voltage of 5 kV.

Conventional and analytical TEM was performed using an FEI Tecnai G² F30 Schottky field-emission gun (FEG) TEM operating at 300 kV, equipped with an EDAX x-ray detector and an FEI Titan G2 60-300 probe corrected FEG scanning transmission electron microscopy (STEM) equipped with an X-FEG high brightness electron source, ChemiSTEM EDS detectors, and Gatan Enfinium electron energy loss spectroscopy (EELS) spectrometer. The semi-quantitative EDS measurements recorded during this study while repeatable from day to day and sample to sample, were obtained from samples with unknown chemistries and no available platinum-niobium alloy standard to calibrate to. Furthermore, the chemistries measured in each region of the weld zone often times were found to be in different phase fields on the platinum-niobium equilibrium phase diagram (18). Even though the non-equilibrium nature of these micro-resistance weld processes is recognized, a second analytical method to characterize the phases was required to verify the EDS results. The sensitivity of low loss EELS to crystal and electronic structure changes (29) made it an appropriate method to qualitatively confirm the regions of similar chemistry and morphology also had the same crystal structure.

NANOINDENTATION AND MICROPILLAR COMPRESSION TESTING

Nanoindentation testing was performed using a Hysitron TI-950 Triboindenter in displacement controlled mode with a 90° cube corner probe. The area function was carefully calculated prior to testing. At the start and end of each day testing occurred, the hardness and reduced modulus of the fused quartz standard was verified at the displacement depth tested (for this study, 50 nm) to ensure the tip geometry from the previously
calculated area function had not changed during testing. All analysis was performed using
a load function with a 5 second load to peak displacement, a 2 second hold, and 5 second
unloading segment.
Lastly, micro pillar samples of the weld zone and base materials were fabricated using
annular milling techniques at 30 kV using a 15 nA probe current for rough cutting and
progressively smaller probe currents down to 50 pA for final polishing. Pillar diameters
were approximately 5 µm with a taper angel of less than 5°.
All pillars were fabricated from metallographic cross sectioned parts using an FEI Versa
or Scios dual beam. The compression experiments were performed using a conical probe
polished to a 10 µm flat punch on a Hysitron TI 980 Triboindenter fitted with a Multirange
Nanoprobe. All compression testing was performed using a loading rate of 20 nm/sec and
a displacement to approximately 10% strain. Following the compression experiments, the
samples were prepared for TEM analysis using the methods listed above.
RESULTS

MACRO AND MICROSTRUCTURE ANALYSIS

Following the welding process, all joints maintained a shiny metallic silver color, only small amounts of expulsion could be observed, and the platinum wire embedded into the niobium wire which can be seen in Figure 10. The lack of visual indicators related to weld quality on the finished weld requires the joint to be investigated further.

Figure 10 - Optical micrograph of a niobium to platinum cross wire micro resistance weld.
Careful cross sections of each weld condition gave more insight into the joint quality. Representative metallographic cross sections seen in Figure 11 show varying levels of expulsion and platinum wire embedment that are expected to change as the welding conditions are modified. The cross sections revealed the wires were bonded, but no evidence of material mixing or local recrystallization was resolved using light microscopy.

Figure 11 - Optical micrographs of cross sectioned niobium to platinum cross wire resistance welds showing varying amounts of expulsion and embedment for (a) Condition A, (b) Condition B, (c) Condition C, and (d) Condition D joints. The platinum wire is seen in the transverse direction at the top of each image while the longitudinal niobium wire is found at the bottom.

Conventional bright field (BF) TEM imaging of at least three separate welds fabricated from each of the four processing conditions showed an array of microstructures and morphologies in the interfacial region that varied between welding conditions. Samples
from Condition A were found to have the thinnest boundary layer on the order of 300 nm and contain large amounts of porosity at the platinum-weld interface. Randomly distributed niobium rich regions, seen as small, globular, and dark regions in BF-TEM images, were also observed in parts joined using “A” conditions. Furthermore, Condition A welds had the least uniform structure in terms of weld zone thickness and morphology that includes large niobium rich globules and a lack of phases or weld regions observed in other conditions. Parts welded using Condition B had a similar microstructure to Condition A parts with the notable differences of a wider fusion zone, which sometimes reached 1 µm, contained less porosity, and had larger areas with niobium rich globules. Condition C samples were found to have a much more uniform microstructure in terms of phases present and thickness of each phase. No porosity was observed and only small spherical niobium rich particles were observed in the weld zone. The two regions closest to the niobium base material consisted of small grains with diameters on the order of 50-100 nm. The phase in the center was found to have a grain size of approximately 200 nm and was confirmed by high resolution (HR) TEM imaging to be heavily faulted. The two phases closest to the platinum base material also contained stacking faults, but these phases had smaller grain sizes ranging from 20-50 nm in thickness and morphology with a higher aspect ratio. Lastly, Condition D welds were found to have the most uniform microstructure, weld zone thickness, phase morphology, and lack of porosity. The morphology and average grain size was found to be similar to the weld zone of Condition C joints. A BF-STEM image, shown in Figure 12 displays a representative resistance weld interface structure with each phase labeled 1 through 5. Representative BF-TEM images of each welding condition are shown in Figure 13.
Figure 12 - BF-STEM image showing the distinct layers of a fusion zone found in a Condition D platinum-niobium resistance weld.
Figure 13 - Bright field TEM images showing the microstructure and morphology of weld Conditions A-D in figures a-d respectively.

High angle annular dark field (HAADF)-STEM imaging was performed to qualitatively image using atomic number contrast. The HAADF images, shown in Figure 14, reveal a gradual change in contrast as the weld zone moves from the niobium base material to the platinum base material. Careful HAADF imaging also revealed all phases nearest the niobium base material contained globules decreasing in concentration as the Pt base
material is approached. High resolution STEM imaging at the interface between regions 2 and 3 revealed the globules to be approximately 3 nm in diameter, niobium rich, and randomly dispersed in the weld zone. The particles were not observed to cross phase boundaries in any weld condition investigated in this study.

Figure 14 - HAADF STEM images showing the microstructure and morphology of weld conditions A-D in figures a-d respectively. Platinum base material is shown as brighter and niobium base material as darker contrast.
Figure 15 shows side by side FIB cross sections and bright field TEM images of Condition A welds where large amounts of porosity are visible. Figure 16 shows a HAADF-STEM overview image of weld Condition D and the corresponding atomic resolution HAADF-STEM image of the interface between regions 2 and three where the niobium rich particles are observed to be randomly dispersed in the matrix materials.

Figure 15 - (a) FIB cross section and (b) BF-TEM images showing porosity at the platinum-weld zone interface in Condition A welds.

Figure 16 - Condition D weld shown in Figure 12 with superimposed square showing approximate location of (b) atomic resolution HAADF-STEM image showing the interface between regions 2 and 3 and small niobium rich globules 2-3 nm in diameter.
CHEMICAL ANALYSIS

Characterization of the chemistry across the platinum-niobium resistance weld interface is critical for an understanding of the effects of weld process parameters on the microstructure and mechanical properties of the resulting joint. In this study, STEM-EDS scans were acquired from each of the distinct interfacial zones found in each of the samples joined with the four different processing conditions. The semi-quantitative EDS results, shown together in Figure 17 and tabulated in Table 4, show the chemistry in each layer to be consistent between all processing conditions. However, Condition D was found to have statistically lower niobium/higher platinum concentrations in region 1 and higher niobium/lower platinum concentrations in region 3. The chemical analysis of all resistance weld conditions showed one major departure from the general five-layer structure observed in Conditions B-D. The second phase closest to the niobium base material was absent in all samples of Condition A analyzed. This observation will be revisited later in the discussion.
Figure 17 - Semi-quantitative STEM-EDS results for each phase in the niobium-platinum resistance welds investigated. The boxplots show the niobium concentration in each phase region starting with region 1 (a) nearest the niobium base material and ending at region 5 (e) nearest the platinum base material.

To further characterize the local chemistry and structure EELS spectrum imaging experiments, plotted in Figure 18 were performed. Comparison of individual low loss EEL
spectra within the different weld zone regions and base material reveal distinct plasmon loss shifts from the niobium base material, across the weld, into the platinum base material. Similar to the results of the EDS analysis, EELS spectrum image line scans were not able to resolve the distinct region 2 phase in weld condition A which was observed in all other weld processing conditions. Representative plasmon loss energy spectra, taken from weld condition C, revealed fine structure peak energy shifts and gradual shape changes as the ratio of platinum to niobium changed across the fusion zone. Comparison between the fine structures analyzed in all weld conditions showed remarkable agreement both in peak energy and structure.

![Figure 18 - Overlaid EEL spectra from each of the five distinct phases in Condition C showing energy shifts in the plasmon loss portion of the spectrum from the niobium base material through the weld zone, into the platinum base material.](image-url)
MECHANICAL PERFORMANCE

Results of the nanoindentation study are presented as composite hardness and indentation modulus. The small grain size, inconsistent weld zone width, and proximity of many of the weld zone phases to the base material required indents were performed in the center of the interface and assume the plastic zone interacts with most of the phases across the weld zone. For the purposes of this work all hardness and indentation modulus values are considered a composite hardness of the weld instead of being reported as phase specific. The results of the nanoindentation experiments are summarized as box-whisker plots shown in Figure 20. The results show consistent hardness values in the weld zone of Conditions A-C with average values of approximately 11 GPa. The indent locations where high hardness data points (~17 GPa outliers) were collected in Conditions A and B corresponded to indentations in niobium rich phases as seen in the HAADF-STEM image above and confirmed with backscatter SEM imaging and EDS in. A representative set of indents in a condition C weld and a HAADF-STEM image showing the structure of a lamellar phase with a backscatter SEM image inset showing the location of a Berkovich indent on the niobium rich phase shown in Figure 19.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Region 1</th>
<th>Region 2</th>
<th>Region 3</th>
<th>Region 4</th>
<th>Region 5</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Nb$<em>{78}$Pt$</em>{22}$</td>
<td>Not Present</td>
<td>Nb$<em>{49}$Pt$</em>{51}$</td>
<td>Nb$<em>{36}$Pt$</em>{70}$</td>
<td>Nb$<em>{25}$Pt$</em>{75}$</td>
</tr>
<tr>
<td>B</td>
<td>Nb$<em>{30}$Pt$</em>{80}$</td>
<td>Nb$<em>{68}$Pt$</em>{32}$</td>
<td>Nb$<em>{52}$Pt$</em>{48}$</td>
<td>Nb$<em>{36}$Pt$</em>{48}$</td>
<td>Nb$<em>{21}$Pt$</em>{79}$</td>
</tr>
<tr>
<td>C</td>
<td>Nb$<em>{52}$Pt$</em>{18}$</td>
<td>Nb$<em>{64}$Pt$</em>{36}$</td>
<td>Nb$<em>{52}$Pt$</em>{36}$</td>
<td>Nb$<em>{27}$Pt$</em>{73}$</td>
<td>Nb$<em>{21}$Pt$</em>{79}$</td>
</tr>
<tr>
<td>D</td>
<td>Nb$<em>{78}$Pt$</em>{25}$</td>
<td>Nb$<em>{64}$Pt$</em>{36}$</td>
<td>Nb$<em>{57}$Pt$</em>{43}$</td>
<td>Nb$<em>{32}$Pt$</em>{68}$</td>
<td>Nb$<em>{25}$Pt$</em>{75}$</td>
</tr>
</tbody>
</table>

Table 4 - Compositions in atomic percent of each apparent phase of Nb-Pt resistance weld. Values are the average of EDS line scan data for each sample and apparent phase region.
The average hardness value in Condition C was comparable to Conditions A and B but the more uniform microstructure and slightly wider weld zone in Condition C resulted in a lower standard deviation in the data set.

Figure 20 - (a) nanoindentation hardness and (b) indentation modulus values for each of the weld conditions investigated.
The general microstructures observed in Figures 13 and 14 showed all four processing conditions yielded welds with slightly different morphologies but the same layered microstructure. Condition D was found to have hardness values nearly 3 GPa lower than Conditions A-C. The indentation modulus values in all welding conditions ranged from 170 GPa to 250 GPa, in all cases the weld zone indentation modulus was found to be higher than the platinum base material.

The small dimensions and cross wire geometry of the weld make testing the actual mechanical properties of the joint difficult. Furthermore, the base materials are fully annealed because of previous processing steps making all attempts at tension or pull testing fail in the base material. While this analysis demonstrates, the weld is stronger than the base materials in uniaxial tension, little information on the properties and performance of the actual weld itself are gained.

To better characterize the local response of each weld zone to a severe plastic deformation micro pillar samples were fabricated that included both base materials and the weld zone. FIB/SEM analysis of pillars following compression experiments revealed large amounts of the deformation had been accommodated by slip of the much softer base materials as shown in Figure 21b. FIB cross sections of compressed pillars show severe deformation of the intermetallic layers that did not result in apparent fracture but instead followed the flow patterns of the base material as seen in Figure 21c.
Figure 21 - Niobium-platinum resistance weld micro pillar joined using Condition A settings (a) in the as milled condition with red dotted lines showing the weld zone location (b) compressed to 10% strain where the red lines highlight slip in the base materials and (c) FIB cross sectioned showing deformation of intermetallic weld zone.
Following FIB cross section analysis, the compressed samples were lifted out and prepared for TEM analysis. Again, the microstructures of the compressed samples showed much of the deformation was accommodated by the base materials based on large dislocation pile ups observed at the base material-weld zone interfaces. The microstructure of Condition A welds following a severe plastic strain showed only signs of heavily faulted intermetallic phases. Few instances of cracking were observed and were only found in areas of heavy deformation as seen in Figure 22 a and c where the weld zone had been bent nearly 90° during the compression testing or a crack nucleated from preexisting porosity in the weld. Condition C welds responded similarly to a large compressive strain. Figure 22 d through e shows a low magnification micrograph of a Condition C weld following a 10% compressive strain. Only a single intergranular crack was found. As with the deformed welds in Condition A, many of the phases in Condition C weld zones were found to accommodate the strain by forming stacking faults. Interestingly, the intermetallic phase labeled as “2” in Figures 12 and 13 was found to contain dislocation loops and sub-grain boundaries. In one location, shown in Figure 22e, the line of dislocation loops were observed to have caused an adjacent phase to shear as the dislocations passed through the phase boundary.
Figure 22 - BF-TEM images of deformed micro pillars of niobium-platinum resistance welds (a-c) Condition A and (d-f) Condition C.
DISCUSSION

The lack of indicators related to material mixing/fusion in the welded wires using visual inspection or careful cross sectioning and light microscopy can often be an issue when process control is critical and the use conditions of the joint requires high reliability. For these reasons, advanced characterization and finite element modeling are required for a complete understanding of the process-microstructure-properties relationship. These tools were used during process development in conjunction with designed experiments to give confidence the weld process parameters resulted in repeatable and reliable joints with well characterized microstructures.

The first improvement resulting from issues revealed by FIB/SEM and TEM characterization was the removal of porosity observed in Condition A welds by changing the material in contact with the positive welding electrode. At this time the mechanism of porosity formation in this weld is not completely understood, but hypotheses include Kirkendall voids accelerated by the application of current or evolution of gasses from surface contamination. Porosity at the base material weld interface was observed to be prevalent and a source of crack initiation when the joint was heavily strained as shown in Figure 11a and c.

The issue of weld porosity was addressed by changing the current path through the work pieces (platinum on the positive electrode). While the weld zone thickness and number of distinguishable phases changed as a result, the inhomogeneous structure and large niobium
rich globules observed in Condition A welds remained. The islands of pure niobium, confirmed by STEM EDS and EELS suggest that the weld is not solid state but fusion and the niobium base material reached the melting temperature at the interface of the work pieces. The nanoindentation data from Conditions A and B had a large spread in values and high value outliers which were found to be indents taken in a region of high niobium concentration. Regions of fusion welds with localized high hardness may act as stress risers or crack initiation sites and should be avoided if possible (13, 14).

The follow up experiment was designed with the intent to lower the weld temperature and obtain more repeatable microstructures and therefore properties. By decreasing the welding time, peak welding current and increasing the force (decreasing the contact resistance) the total energy input to the weld was decreased as shown in Eq. 1.

The resulting weld, Condition C, had a marked qualitative increase in weld width, no porosity, and a much more uniform microstructure. While the local heating was decreased using Condition C weld parameters, reducing the contact resistance further was of further interest. As the weld parameters had been optimized from the designed experiment, a common noise variable that is not traditionally controlled but important nonetheless, was investigated: the surface roughness of the work pieces. Similar to electrode force, the smaller number of contact spots at the welding interface decreases the contact resistance (30). Condition D welds were performed using the same parameters as Condition C however; the surface roughness of the niobium wire was nearly a factor of 3 less than those used for Conditions A-C. Wires with low surface roughness resulted in a joint with similar morphology to Condition C.
The chemical analysis of each joint using semi-quantitative EDS revealed each region of Conditions A-C had niobium and platinum concentrations that were statistically similar. Condition D, while having morphology nearly identical to Condition C welds, had statistically significantly lower concentrations of niobium in region 1 and 5 but slightly higher niobium concentrations in region 3 compared to the other three weld conditions. These variations in chemistry shift the above mentioned regions to different phase fields on the equilibrium phase diagram (18) when compared to the same regions in Conditions A-C.

The changes in energy input reveal niobium rich particles of varying sizes that decreased as energy input was lowered suggest the amount of niobium base material melting is highest in Conditions A and B. The larger (50-100 nm) sized globules, randomly distributed within welds processed using Conditions A and B suggest upon solidification, the solubility limit of niobium in the phases that form in the weld zone decreases. Excess solute is expelled from the crystal and agglomerates into niobium rich regions. STEM imaging shows the niobium rich phases to have a nano-grained lamellar structure (Figure 19b), which could partially explain the high hardness values recorded at these locations. As the energy input was decreased the number and size of niobium rich globules decreased dramatically. Condition C and D welds contained very few, although representative examples are seen in Figure 14(c-d) as white globular features 25-50 nm in diameter. As mentioned in the results section, all conditions contain 3-5 nm diameter niobium rich particles. These particles have high concentrations in the niobium rich regions of the welds decreasing as phases nearest the platinum base material are approached
The first method of probing the mechanical properties of the weld zone using nanoindentation was unable to resolve phase specific mechanical properties but nonetheless gave insight into the mechanical and elastic properties of the weld zone as a composite. The hardness values of Conditions A-C were found to be a factor of five larger than the base materials. This result is not surprising considering the discussion above related to microstructure, morphology, and chemistry of the welds. All joints are multiphase, nano-grained, and contain niobium rich precipitates which separately could be responsible for incremental strengthening of the weld zone compared to the base material. Together, these proposed strengthening mechanisms result in the high hardness values recorded in the weld zones. The hardness values measured in the weld zone of Condition D joints were between the base materials and weld zones in Conditions A-C. This could
be an artifact of the more uniform and slightly wider weld zone in Condition D welds or possibly related to the local heating, melting, and solidification that resulted from lower contact resistance at the weld interface due to lower niobium surface roughness.

The composite indentation modulus values did not follow the same trend as hardness. The indentation modulus values of Condition A and D were statistically similar while Conditions B and C had similar indentation modulus values. Inspection of the samples following indentation experiments provided evidence that the two distributions of indentation modulus recorded for this work were a result of the location of the indent in each weld, the phases that elastically deformed, and if the indent was centered on an individual phase or phase interfaces. The spread in indentation modulus values from ex-situ testing are not avoidable at this time. Further experiments using in-situ techniques should be used in the future to measure phase specific hardness and elastic properties.

Arguably the most important output of the weld process is the mechanical performance of the newly formed fusion zone. As mentioned above, the traditional tensile pull and peel tests of this system result in fracture in the base materials and not the stronger weld zone. This signifies the phases formed in the weld zone have a higher tensile or shear strength than the base materials in uniaxial tension or in peel tests but other important pieces of information pertaining to properties or performance are neglected including susceptibility of the weld zone to cracking, weld zone uniformity, and the presence of weld defects and their effects. The results of micropillar compression testing demonstrated the robustness of these joints under extreme deformation. Post mortem BF-TEM imaging showed the extensive plastic deformation in the weld zone of a Condition A pillar. Large dislocation pile ups were observed at the base material-weld zone interfaces. Plasticity within the weld
zone appears to be accommodated by the formation of a heavily faulted structure in the platinum rich regions of the joint. The cracking that was observed appears to have nucleated from porosity at the weld zone-base material interfaces and was intergranular in nature as shown in Figure 22 a and c. This observation was another strong indication that minimizing the porosity created in Condition A welds, especially if the use condition of the weld is mechanically loaded or fatigued, improves performance. Condition C welds showed similar instances of intergranular cracking but with no observed porosity in the weld zone. The amount of cracking overall was not possible to discern as only one TEM sample from each set of deformed pillars was thinned. The most interesting observation in Condition C pillar compression experiments was the dislocation plasticity seen in the phase referred to as region 2. The line of dislocation loops can be seen across a grain and shearing a neighboring phase (Figure 22f). This finding has important implications for the reliability of the joint with optimized properties and shows dislocation plasticity in the intermetallic phases instead of catastrophic brittle fracture.
SUMMARY

In conclusion, we showed robust and reliable resistance welds can be created from platinum and niobium fine wires in spite of their melting temperature differences. The key conclusions from this body of work are as follows:

1) Lowering the applied current and weld duration to niobium-platinum cross wire resistance welds results in joints with five separate phase regions across the width of the weld zone, no porosity, and uniform structure/morphology.

2) The chemistry of these phases was confirmed to be consistent between the weld conditions investigated in this study using STEM EDS and EELS.

3) Factors in the process often not reported or considered to be noise variables such as electrode polarity and wire surface roughness were shown to have an effect on weld repeatability, defect formation, and mechanical performance.

4) The hardness of the weld zone, probed using instrumented nanoindentation was found to be between 2.5 and 5 times harder than the base materials. The indentation modulus was found to range from 195 to 230 GPa, higher than the base materials (160 GPa for platinum and 85 GPa for niobium).

5) Micropillar compression testing to 10% strain showed much of the deformation to be accommodated by the soft base materials. In some cases extreme deformation occurred in the weld zone and small intergranular cracks were observed but appeared to be localized. The Condition C weld investigated was found to have dislocation plasticity in the niobium
rich phase labeled region 2 signifying at least small amounts of plasticity can be accommodated in the weld zone itself.
CHAPTER 2: OPTIMIZATION OF A VITRELOY 105 TO TITANIUM LASER WELD

BACKGROUND

The constant goal of more reliable, miniaturized, and manufacturable medical devices requires the use of advanced materials and processes. The requirements of strength, corrosion resistance, biocompatibility, and manufacturability make the material selection process difficult. The high strength, corrosion resistance, low elastic modulus, low density, high electrical resistivity, and high elastic limit \((31, 32)\) makes metallic glasses ideal candidates for many medical devices that require permanent implant. One of the most unique properties of bulk metallic glasses is their ability to be thermoplastically formed into near net shape components \((33, 34)\). While thermoplastic forming of metallic glass components is an area of active research \((35, 36)\), critical part sizes exist based on the glass forming ability of the material being formed, viscosity, critical cooling rate, and part geometry \((37)\). The inherent size limitations of thermoplastically formed bulk metallic glass parts require they be joined to themselves or dissimilar materials to become more widely used in advanced engineering applications. Many authors have successfully demonstrated homogeneous bulk metallic glass welds using laser \((38-41)\), electron beam \((42)\), resistance \((43)\), and friction \((42)\) techniques. Joining metallic glasses can be challenging as once the work pieces are melted they must cool at a sufficiently fast rate to avoid crystallization. Oftentimes, crystallization can also occur in the heat affected zone of the weld if the heat input is not carefully controlled. The process development to join
metallic glasses to themselves is important, however large scale adoption of metallic glass parts and components is unlikely until robust joining methods to existing crystalline engineering materials are developed. In the medical device industry, titanium and titanium alloys are some of the most common materials used for implant applications and would be excellent candidates for joining to bulk metallic glasses. Furthermore, the prevalence of laser welding, marking, ablation, and cutting processes utilized in the medical device industry makes fusion welding using a laser the most cost effective, available, and flexible joining technique available. A number of studies have been published that pertain to fusion welding bulk metallic glasses to crystalline materials. Results of electron beam welding to zirconium (42, 44), titanium (45), stainless steel (46), and nickel (47) and laser welding to zirconium (48) have been reported. In all cases zirconium has been found to make robust joints with Zr_{41}Ti_{14}Cu_{12}Ni_{10}Be_{23} bulk metallic glasses (42, 44), but titanium fusion welds have had inconsistent results. Some researchers have reported brittle joints (44), others reporting joint strengths exceeding the titanium base material (45). The previously mentioned studies did not report how the processing parameters were chosen, did not attempt to optimize the weld microstructure, and mainly focused on characterization of the resulting weld using the reported processing parameters. This work will focus on characterization of the weld but also contain a significant focus on the relationship between twelve distinct weld processing parameters and the microstructure, properties, and performance of the dissimilar metal laser weld. The first experiment, an eleven factor, three level designed experiment was performed in 26 runs using an experimental set up known as a definitive screening design (DSD) (49). DSD studies combine screening and optimization design of experiments while requiring fewer runs than comparable full
factorial designed experiments. The objective of this study was using designed experiments to produce a Vitreloy 105 to grade 2 titanium laser weld with a weld zone with uniform and minimized hardness and reduced modulus, maximum weld penetration, and no cracking or similar weld defects.
EXPERIMENTAL

EXPERIMENTAL DESIGN

To study the eleven factors at three levels with traditional fractional factorial designs 128 runs would be required to not convolute second order interactions, an important output of designed experiments. To optimize the weld further, more experiments would be required to optimize the results with center points. This was time and cost prohibitive. This issue was resolved with the use of definitive screening design experiments. Table 5 and 5 shown below contain the chemical composition of the materials used in this study and the factors and levels of the designed experiment.

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<th>Ti</th>
<th>Cu</th>
<th>Ni</th>
<th>Al</th>
<th>C</th>
<th>N</th>
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Table 5 - Elemental composition of materials used in this study. Values are in wt. %. A composition of N/A signifies the element was not present. A value of NR signifies the element was not tested for. All data collected for this study using ICP-OES, carbon/sulfur analysis, and gas metals analysis.
The factors and levels of the designed experiment were carefully selected to include process specific factors such as peak power, pulse width, frequency, cover gas, and cover gas flow rate. Factors that may influence the chemical composition or cooling rate of the weld were also investigated and include the addition of filler metal, offset of the laser beam from the weld centerline, and pulse shape. The final factors chosen for this study include the process fiber size which influences the energy density of the laser and the surface finish of the metallic glass base material (shiny or dull), which may play a role in coupling the laser beam to material. The complete experimental design can be found in Table 7.
Table 6 - Factors and Levels used for DSD Experiment 1. *Temporal profiles of the pulse shapes can be found in Figure 22 below. N/A signifies no center point or third level analyzed.

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<th>Freq. (Hz)</th>
<th>Table Speed (mm/min)</th>
<th>Cover Gas Flow Rate (SCFH)</th>
<th>Pulse Shape</th>
<th>Filler Metal</th>
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Figure 24 - Pulse shapes used in definitive screening design study.
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<th>Freq (Hz)</th>
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<td>800</td>
<td>0.5</td>
<td>15</td>
<td>1.5</td>
<td>40</td>
<td>Anneal</td>
<td>Tin</td>
<td>100 µm into Vit 105</td>
<td>Argon</td>
<td>400 µm</td>
<td>Dull</td>
</tr>
<tr>
<td>24</td>
<td>800</td>
<td>0.5</td>
<td>5</td>
<td>2</td>
<td>50</td>
<td>Anneal</td>
<td>Tin</td>
<td>100 µm into Vit 105</td>
<td>Argon</td>
<td>200 µm</td>
<td>Dull</td>
</tr>
<tr>
<td>25</td>
<td>800</td>
<td>1</td>
<td>15</td>
<td>1</td>
<td>50</td>
<td>Peak</td>
<td>Tin</td>
<td>100 µm into Vit 105</td>
<td>Argon</td>
<td>200 µm</td>
<td>Shiny</td>
</tr>
<tr>
<td>26</td>
<td>950</td>
<td>0.5</td>
<td>5</td>
<td>1</td>
<td>50</td>
<td>Square</td>
<td>None</td>
<td>100 µm into Vit 105</td>
<td>Argon</td>
<td>400 µm</td>
<td>Dull</td>
</tr>
</tbody>
</table>

Table 7 - Experimental Design for the definitive screening design.
LASER WELDING

Laser welding was performed using a custom four-axis CNC system integrated by Innovative Laser Technologies (Minneapolis, Minnesota). The system was equipped with a Lasag SLS 200 CL16 pulsed Nd:YAG solid-state laser operating at an emission wavelength of 1064 nm paired with a processing head with on-axis assist gas delivery. The Lasag SLS 200 CL16 laser was selected based on its ability to deliver high peak power (up to 7.0 kW), which is known to be required for welding highly reflective materials such as the LM105 bulk metallic glass (50). Laser average power values were collected using an Ophir power sensor and meter.

Laser spot size was adjusted by switching between 200 and 400 µm process fibers, both of which were confirmed via a Spiricon SP620U beam profiler to deliver a beam with a Gaussian distribution. Results of the beam profiling are reported as the D4σ values shown in Table 8 and Figure 25. Pulse shapes were verified using an Ophir FPS1 photodetector, Thorlabs PDA200C photodiode amplifier, and Tektronix TDS 410A oscilloscope.

<table>
<thead>
<tr>
<th>Fiber Diameter (µm)</th>
<th>D4σX (µm)</th>
<th>D4σY (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>217.7</td>
<td>221.6</td>
</tr>
<tr>
<td>400</td>
<td>455.8</td>
<td>456.2</td>
</tr>
</tbody>
</table>

Table 8 - Beam diameter measurements.
A stainless steel fixture was used to hold the weld samples in a butt-joint configuration with intimate contact during welding. The fixture was designed to provide cover gas to the underside of the welding sample in addition to the topside assist gas delivered by the Lasag welding head. Gas flow rates were controlled by an Aalborg mass flow controller. The samples used in this work were machined to 10 mm x 10 mm x 0.9 mm thick coupons.
METALLOGRAPHIC SAMPLE PREPARATION

All samples were mounted transversely in EpoHeat mounting media, ground to the center of the test plate, and prepared using standard metallographic techniques (51) using a Buehler Ecomet/Automet 250 automatic grinder-polisher followed by a vibratory polish to remove residual surface deformation using a Buehler Vibromet. All samples were imaged with a Zeiss Axiovert metallographic microscope for weld penetration measurements.

NANOINDENTATION

Nanoindentation testing was performed using a Hysitron TI950 Triboindenter with a Berkovich tip and a carefully calculated area function. The validity of the area function was confirmed before and after each sample was mapped using the fused quartz standard. All indents were performed in load controlled mode to a peak load of 3000 µN. The load function was a typical 5 second upslope, 2 second hold, and 5 second unloading segment. The indentation parameters were chosen to yield an indentation depth of approximately 200 nm into the test samples.

SCANNING ELECTRON MICROSCOPY AND ENERGY DISPERSIVE SPECTROSCOPY

Scanning electron microscopy and energy dispersive spectroscopy (EDS) experiments were performed using an FEI Versa 3D FIB/FEGSEM. Imaging was performed with accelerating voltages ranging from 2kV to 20kV using backscattered electrons. EDS measurements were recorded using an Oxford Instruments EDS detector with an 80 mm² window. All data was collected and analyzed using Oxford Aztec software.
TRANSMISSION ELECTRON MICROSCOPY

Samples for transmission electron microscopy and micro beam bending experiments were fabricated on FEI Versa and Scios DualBeam instruments equipped with an FEI EasyLift system. Films 15 µm wide for transmission electron microscopy were lifted out from the site specific locations of the weld zone and thinned with 30 kV Ga\(^+\) ions to a thickness of 100 nm and final thinned to electron transparency with an ion beam accelerating voltage of 5 kV.

Conventional and analytical TEM was performed using an FEI Tecnai G\(^2\) F30 Schottky FEG TEM operating at 300 kV, equipped with an EDAX x-ray detector and Gatan electron energy loss spectrometer.

MICRO CANTILEVER BEAM TESTING

Micro cantilever beams of the weld zone and base materials were prepared on a small weld cross section that was polished using an Allied MultiPrep and fabricated using milling techniques at 30 kV using a 50-65 nA probe current for rough cutting and progressively smaller probe currents down to 300 pA for final polishing. Beam dimensions were approximately 7 µm. All beams were polished using a 2° overtilt to minimize taper. The beam geometries were pentagonal and fabricated using a similar method as Di Maio et al. (52) and Zhao et al. (53). The compression experiments were performed using a conical diamond indenter tip with a 2 µm radius on a Hysitron PI 85 PicoIndenter. All micro cantilever testing was performed using a loading rate of 20 nm*sec\(^{-1}\) until fracture occurred or a maximum displacement of 5000 nm was reached. An image of the labeled pillars showing their locations in the weld zone can be seen in Figure 26.
Figure 26 - Scanning electron micrograph showing locations of micro cantilever beams in the weld zone of an optimized Vitreloy 105 to titanium laser weld.
RESULTS

DEFINITIVE SCREENING DESIGN

The results of the first designed experiment showed only four of eleven factors to be significant: pulse width, pulse shape, filler metal, and beam offset. The conclusions of the DOE were interesting because the results showed, aside from pulse width which is often the process parameter used to control weld penetration, the mechanical properties (from both nanoindentation and microcantilever beam testing) were found to be more a factor of local chemistry than the laser parameters studied (energy input). This result makes filler metal and beam offset reasonable factors when working to optimize the mechanical properties of a dissimilar metal laser weld. In order to create a model that took all responses into account (visual, weld penetration, average hardness, standard deviation of hardness, indentation modulus, and standard deviation of indentation modulus), the responses were given a “Z score” defined by equation 2 where $\bar{x}_i$ is the average value (hardness, indentation modulus) within a sample, $\bar{x}$ is the average of data from all samples, and $\sigma$ is the standard deviation of the data from all samples. Z scores are used to normalize large data sets for use in Z aggregate analysis discussed below.

$$Z \text{ Score} = \frac{\bar{x}_i - x_i}{\sigma}$$  (2)

Following the creation of a Z score for each response, an aggregate Z score or $Z_{agg}$ was calculated. This aggregate score considered the preferred sign, positive or negative, of the
individual Z scores (for example, in this study the goal was to minimize weld hardness and hardness variation in the weld zone would make hardness a negative number). The aggregate Z score is defined in equation 3 below.

\[ Z_{agg} = \text{Visual}^* (Z_{Pen} - Z_{Er \text{ Mean}} - Z_{Er \text{ Stdev}} - Z_{H \text{ Mean}} - Z_{H \text{ Stdev}}) \]  

(3)

The main effect plots for the responses measured revealed the most important factors to be pulse width, pulse shape, filler metal, and offset of beam and are shown in Figure 24. The results of the aggregate Z analysis are shown in Figure 25 and include the main effects, interactions and regression analysis. The regression analysis showed nearly 78% of the variability of the model was accounted for in the factors pulse width, offset of beam, table speed and the two-way interactions of offset of beam/table speed and offset of beam and pulse width.
Figure 27 - Main effects plots for each experimental response from the definitive screening design experiment.
Figure 28 - Main effects plot, interaction plot, and regression analysis for the Z\textsubscript{agg} analysis.

Results of the designed experiment are shown below in Figures 30-35 and include optical micrographs of cross sectioned welds, measured penetration values, hardness, and indentation modulus for each run.
The cross sectioned welds show tremendous amounts of variability between runs and qualitatively show the pronounced effects of some factors. Filler metal, for example, in the runs where tin was used showed the foil vaporized in all weld conditions but one and resulted in severe cracking, weld spatter, and low penetration values. This is demonstrated in Figures 29-31 (a-c, g, n, p, w). The effects of energy input are also clear when comparing weld penetration. High peak power and pulse width drive a deeper keyhole and create nearly full penetration welds as seen in Figures 29-31 (b, k, l, v). Conversely low values of energy per pulse result in low penetration values, and in extreme cases nearly no coupling causing samples to fracture from handling as seen in Figures 29-31 (q, s, x). For comparison purposes, homogenous welds of both Vitreloy 105 and grade 2 titanium are shown in Figure 32.
Figure 29 - Optical micrographs of (a)-(i) runs 1-9 of the definitive screening design experiment showing the large amounts of variability introduced to the process for each run using the parameters found in Table 6. Scale bars correspond to 200 microns.
Figure 30 - Optical micrographs of (j)-(r) runs 10-18 of the definitive screening design experiment showing the large amounts of variability introduced to the process for each run using the parameters found in Table 6. Scale bars correspond to 200 microns.
Figure 31 - Optical micrographs of (s)-(z) runs 19-26 of the definitive screening design experiment showing the large amounts of variability introduced to the process for each run using the parameters found in Table 6. Scale bars correspond to 200 microns.
Figure 32 - Optical micrographs showing homogenous laser welds of (a) Vitreloy 105 and (b) grade 2 titanium. Scale bars correspond to 200 microns.

Figure 33 - Individual value plot showing weld penetration values for each run of the DOE.
Figure 34 - (a) hardness and (b) indentation modulus interval plots showing the distributions for each run in the definitive screening design experiment.
OPTIMIZED WELD SCHEDULE

The optimized weld parameters from the definitive screening design analysis are shown in Table 9. All data presented in section 8.2 were collected from welds joined using the optimized parameters.

<table>
<thead>
<tr>
<th>Peak Power (W)</th>
<th>Pulse Width (ms)</th>
<th>Frequency (Hz)</th>
<th>Table Speed (in/min)</th>
<th>Cover Gas Flow (SCFH)</th>
<th>Pulse Shape</th>
<th>Filler Metal</th>
<th>Offset of Beam (µm)</th>
<th>Cover Gas</th>
<th>Spot Size (µm)</th>
<th>Surface Finish</th>
</tr>
</thead>
<tbody>
<tr>
<td>950</td>
<td>1</td>
<td>10</td>
<td>2</td>
<td>45</td>
<td>Square</td>
<td>Zirconium</td>
<td>100 µm into Titanium</td>
<td>Argon</td>
<td>400 µm</td>
<td>Shiny</td>
</tr>
</tbody>
</table>

Table 9 – Optimized weld parameters from the DSD study.

MECHANICAL PROPERTIES

The mechanical properties of the optimized joints were studied using three-point bend testing on the weld coupons, nanoindentation maps across the fusion zone, and micro cantilever beam testing to probe size specific mechanical properties of the weld zone.

Nanoindentation maps were collected across the optimized weld zones to gage the consistency of hardness and indentation modulus as a function of location in the fusion zone. The results for the optimized weld seen in Figure 35, show the hardness and indentation modulus to be reasonably consistent except for small “hot” spots that were locally much harder than the rest of the weld.
Figure 35 - Contour maps showing hardness and indentation modulus values in the optimized joint weld zone. Titanium is pictured on the left, Vitreloy 105 on the right.

EDS mapping showed chemical inhomogeneities in the weld zone that agreed with backscatter electron imaging. Areas with locally high concentrations of titanium were observed randomly throughout the weld zone shown in the EDS maps in Figure 36. These
results correlate well with the small variations observed in hardness and indentation modulus as changes in local chemistry may be related to material property variation. Variations like this are expected with a keyhole mode laser weld process due to weld pool convection, material mixing, and the non-equilibrium conditions resulting from the fast cooling rates intrinsic to the laser weld process (54).

![EDS elemental maps showing some chemical inhomogeneity in the weld zone.](image)

EDS line scans were performed across the weld zone to better quantify local changes in chemical composition as a function of distance. The linescans reveal fairly large local fluctuations in composition that, as will be discussed later, have a profound effect on mechanical performance. The semi quantitative linescans show titanium concentrations can fluctuate 50 at% locally across the weld while zirconium concentrations fluctuated
approximately 30 at%. Nickel, copper, and aluminum concentrations stayed consistent with only a few at% variation across the weld zone for each.

![Figure 37 - EDS linescans across the optimized weld zone showing variation in chemistry in different locations (a) near the top of the weld and (b) near the weld root.](image)
MICROSTRUCTURAL CHARACTERIZATION

A scanning electron microscope investigation of the optimized joints showed significant microstructural variation throughout the fusion zone. Higher magnification backscatter electron (BSE) images show swirl patterns of varying contrast that again displays the non-homogenous mixing observed directly using EDS or indirectly using nanoindentation. Figure 38 shows overview BSE micrographs where macro segregation is clearly present.
Figure 38 - Overview BSE images of the microstructure in the optimized weld fusion zone (a) Top surface of the weld zone showing macro-segregation of titanium (regions with dark contrast) and a swirl pattern consistent with large amounts of weld pool convection and (b) similar swirl patterns observed near the weld root but with much less variation in contrast suggesting more uniform chemistry in the weld root region.
Closer inspection of the base material-fusion zone interfaces revealed a thin region of planar solidification that turned into layers of dendritic solidification and finally a cellular dendritic structure. This observation is clearly shown on the titanium side of the weld. The metallic glass-fusion zone interface shows a faint discolored region right before dendrites form. A closer inspection of the weld interface using TEM may be required to definitively characterize the solidification mode. Figure 39 shows the titanium-fusion zone and LM105-fusion zone interfaces and the evolution of the solidification microstructure nucleating at the base material interfaces.
Figure 39 - Representative backscatter SEM images showing the solidification fronts on both sides of the base material to fusion zone interface. Planar solidification is observed on both sides of the weld zone but in a short distance dendrites are formed as solute atoms are expelled into the weld pool upon cooling.
Micro Cantilever Beam Testing

Three point bend testing on weld coupons is a useful characterization technique to quantify and compare the strength and ductility of welds. Analysis of the fracture surfaces revealed areas of brittle, glass like fracture, while areas containing localized ductile tearing were evident. These observations lead to the creation of micro-cantilever beams in various locations across the fusion zone to study the microstructure-chemistry-fracture property relationship. For this study, 15 micro-cantilevers were FIB milled from joints fabricated using the optimized weld schedule and deflected to fracture or a displacement of 3000-4500 nm. All load-displacement curves are plotted together in Figure 41.

Figure 40 - Load displacement curves for all beams tested in this study.
Prior to fabrication of the notch and testing of the beams, backscatter SEM imaging and EDS line scans were performed along the length of each cantilever beam to characterize the local chemistry and structure of each beam tested. Beam 1 had the highest average titanium and lowest zirconium concentrations of all beams analyzed in this study. The aluminum, nickel, and copper concentrations were consistently at or below 5 at%. The microstructure consists of dendrites that become small globular features with no apparent network connecting them. Beam 7 was found to have the lowest titanium concentration and a microstructure with a continuous network of cellular dendrites. Beam 14 contained large chemistry fluctuations along the length of the beam. The microstructure was also inconsistent with regions of continuous cellular dendrites when the concentration of titanium was between 40 and 55 at%. When the concentration of titanium was above or below this range, the microstructure appeared to be single phase as no structure could be observed with the resolution of a FEGSEM. Representative line scan data is shown in Figure 42 below.
Figure 41 - Backscattered electron micrographs and corresponding semi-quantitative EDS line scans showing representative elemental compositions along the length of beams in the optimized LM105-Ti fusion zone.

The beams shown in Figure 42 are representative of three distinct mechanical responses and fracture morphologies observed during the in-situ testing. Column (a) shows a local region in the weld where large amounts of plasticity, ductile tearing, and shear banding were observed. Close examination of the deformed beam reveal small globular phases, rich in zirconium and copper as shown by EDS, that had blunted or deflected shear bands, effectively toughening the beams. The beam in column (b) had a much different response to the deflection by failing in a highly brittle manner. The fracture surfaces revealed the presence of Wallner lines (Figure 44), a sign of glassy fracture.

The beam in column (c) was observed to have behavior that was a mixture of those observed in column (a) and (b). The fracture surface had regions where mixed mode fracture could be observed. Small areas of the fracture contained micro voids that are
correlated with ductile rupture while other locations had a flat, featureless surface consistent with a brittle fracture.

Figure 42 - Montage of representative beam fractures modes observed in the current study. Fracture types range from ductile tearing (column (a)), brittle cleavage (column (b)), and mixed mode failure (column (c)).
Figure 43 - Wallner lines observed on the fracture surface of beam three.

The load displacement curves and corresponding post-mortem bright field transmission electron micrographs reveal an interesting microstructure-property relationship. The beams that yielded without catastrophic failure all had titanium concentrations above 60 at%. These beams appear to have accommodated the applied bending strains by a mixture of deformation twinning and the interaction of shear bands with small, globular phases rich in zirconium. This phenomenon is shown in Figure 44 (a) represented by Beam 1. Figure 44 (b) shows the load-displacement curve and corresponding microstructure of Beam 7 where brittle cleavage fracture was observed. The TEM image reveals a continuous network of a zirconium rich intermetallic compound in a cellular structure. Lastly, a subset of beams fractured after small amounts of plastic deformation and stable
crack growth. These samples were found to have microstructures with the zirconium rich cellular structure but it was not continuous, allowing for small amounts of plasticity before the catastrophic brittle fracture.

Figure 44 - Representative load – displacement curves for micro cantilever beams and their corresponding post mortem BF-TEM image at different locations in the fusion zone (a) Beam 1 showing the bending strain was accommodated by shear banding and twinning. (b) Beam 7 showing a continuous network of zirconium rich intermetallic that corresponded to a linear elastic response during bending. (c) Beam 14 showing small amounts of plasticity following an initial yield event. The microstructure shows the dark zirconium rich regions but they are not in a continuous network. The light contrast titanium rich regions contain evidence of dislocation plasticity.
DISCUSSION

The results of the definitive screening design experiment show the hardness of the welds can be decreased nearly 1 GPa with the addition of thin sheets of zirconium filler metal and offsetting the laser beam into the titanium base material compared to a LM105-Ti laser weld with no filler metal and a centered beam, shown in Figure 36a. While this work improved the joint properties, and resulted in a weld with less variability across the weld zone, the results require further discussion. Hardness of the weld zone is a critical output parameter that is simple to measure and helpful for gaging the approximate yield stress of a joint. However, in many cases the hardness does not tell the complete story. During this designed experiment the offset values, beam diameters, and the inclusion of a zirconium foil resulted in low penetration values in DOE runs where the beam was offset into the metallic glass base material as seen in Figure 45. These low penetration values skewed the results of the DOE analysis as the visual grade and weld penetration were weighted slightly more heavily than hardness and modulus. The high reflectivity of the metallic glass requires higher peak laser power values than a weld with the beam positioned in the center of the joint or offset into the titanium base material. This phenomenon coupled with samples run using the 200 µm process fiber being offset into the metallic glass base material 100 µm resulted in difficulty coupling the two coupons in some cases.
A closer inspection of the interfacial microstructure in the welds with artificially low penetration values shows a weld zone to titanium interface that is approximately 3 microns wide as seen in Figure 46. The average hardness across the weld zone, while high at the interface between the titanium base material and weld, was nearly 1 GPa lower than the weld schedule determined to be optimal after analyzing the data from the designed experiment. These findings require further experimentation to be performed with smaller beam offsets to remove the low penetration artifact and re-optimize the weld schedule.
One of the more interesting observations of this study was from the micro cantilever beam fracture toughness testing. The ability to analyze fracture properties locally in various regions of the fusion zone revealed small areas where in-situ composites, or small crystalline phases contained in a glassy matrix, (55-59) were formed, locations of low toughness, and areas with small amounts of plasticity. While these findings will not improve the fracture performance or toughness of the existing weld, it provides a path for improving the performance of the weld by controlling the structure and chemistry. The beams that performed best had titanium concentrations above 60 at% resulting in a titanium rich matrix containing small, spherical, zirconium rich particles. These small particles approximately 100 nm in diameter toughened the beams by halting shear bands that formed...
from propagating further. In some cases, the shear banding deflected around the particles as seen in Figure 47.

Figure 47 - Backscatter SEM images showing interaction of zirconium rich particles with shear bands in a micro-cantilever with high toughness.

The load displacement curves for the micro cantilever bending experiments were separated by failure mode as shown in Figure 48 with their representative corresponding failed profile image. As discussed above, the beams that failed in a ductile manner (or didn’t fail at all) contained a titanium rich matrix with spherical zirconium rich particles that blunt or deflect shear bands or cracks. A mixed mode failure, where small amounts of plasticity and stable crack growth was observed prior to catastrophic failure showed a network of zirconium rich phases that were not continuous. At times, they were interrupted by zirconium rich particles with a spherical morphology. Close inspection of the fractured samples shows evidence of deformation and secondary cracking that occurred before final fracture. It was determined that the samples that failed primarily through brittle fracture had a flat, relatively featureless fracture surface, no signs of plasticity, and no secondary cracking. In agreement with the transmission electron microscope images, the samples showed the
network of zirconium rich phase to be continuous and the apparent source of low ductility catastrophic fracture. Further understanding of these phases, their chemistry, structure, and how to control their formation may not only improve metallic glass to titanium welds, but may also help in the development of *in-situ* metallic glass composite materials.

Figure 48 - Load-displacement curves and representative fractured beam profile images showing (a) ductile bending failure (b) mixed mode fracture and (c) brittle failure of micro-cantilever beams fabricated from the fusion zone of a Vitreloy 105 to titanium laser weld.
SUMMARY

The results of this study demonstrated the feasibility of laser welding Vitreloy 105 to commercial purity titanium. The data collected and analyzed using definitive screening design experiments allowed many factors at different levels to be analyzed in a small number of runs, which is a powerful and efficient way of determining main effects for a given process. Of the 11 factors investigated, only three were determined to be significant when optimizing hardness, indentation modulus, weld penetration, and visual performance (discoloration, weld spatter, cracking). The major factors were pulse width, filler metal, laser beam offset. To a lesser extent, pulse shape is also a factor. The following conclusions were made on a Vitreloy 105-titanium weld processed using optimized weld parameters:

- Definitive screening design experiments are powerful tools for efficiently understanding the relationship between processing parameters and resulting properties of welds. The ability to resolve the effect of process parameter interactions on process output is also critical for development of a robust process.
- Optimization of the Vitreloy 105 to titanium laser weld resulted in a joint with average hardness value nearly 1 GPa lower than a homogenous Vitreloy 105 laser weld or a Vitreloy 105 to titanium laser weld without filler metal.
- Offsetting the beam into the titanium base material helped to control the amount of metallic glass that melted and mixed into the weld zone. This factor aided the
uniformity of the weld zone compared to samples with no beam offset. This is important for long term process capability.

- The fracture surface of the optimized welds following three-point bend testing revealed mixed mode fracture. Some locations of the fracture surface were found to have features with characteristics of brittle fracture while other areas contained regions of apparent ductile rupture.

- Local fracture properties in the optimized weld zone were investigated using micro-cantilever beam bending experiments. These tests revealed a link between microstructure and chemistry on fracture toughness. This work found that robust and ductile structure rich in titanium with small zirconium rich particles can be formed.
CONCLUSIONS

The preceding studies have shown that both niobium-platinum and Vitreloy 105-grade 2 titanium welds are possible. With carefully planned experiments and advanced characterization, these joints can be made more reliable, capable, and more robust to noise variables that may arise. The use of designed experiments, advanced characterization techniques, and test methods can be used to understand the microstructure-process-properties relationships in the above studied welds and others.
FURTHER WORK

Further studies are suggested and include:

- Ongoing finite element modeling work using Abaqus, which in conjunction with the detailed experimental work, may be used to further streamline the development of similar joints in the future. The currently proposed coupled thermal-structural-electrical model is being used to predict temperature profiles in the weld zone as a function of electrode force, weld current, electrode spacing, and welding time/temporal profile (upslope, hold, downslope time). The current model uses both experimentally measured inputs either measured on the actual materials being modeled (mechanical properties) and thermo-physical properties taken from (27). The current model contains two steps application of the electrode force, in this case 3 lbf to model condition A and B weld settings and an applied current of 500 A. The application of electrode force step shown in Figure 49a results in a deformation and Von Mises stress in the platinum wire comparable with experimental observations. The application of current in step 2 of the model proved to be more challenging and has not converged when the experimental current density of 500 A/mm² that is applied. At this time the model converges only when the applied current density is 0.1 A/mm² or lower as pictured in Figure 49b. Work on this model is ongoing and will be reported in later publications.
Figure 49 - Results of preliminary thermal-mechanical-electrical model of the Condition A resistance weld showing (a) the Von Mises stress (in MPa) following the application of electrode force and (b) temperature contours (in K) following the application of a A/mm$^2$ body current for a weld duration of 49 ms. The part orientation is the same as seen in the cross sections found in Figure 11.

- Further investigations into the porosity observed in Condition A platinum-niobium resistance welds and more detailed studies related to mechanisms related to the
formation and removal of them as the direction of current through the joint is changed.

- Further DOE's need to be performed to optimize the microstructure of the Vitreloy 105 to titanium weld zone using the information gathered in the above results. These findings are important steps towards the creation and fabrication of robust bulk metallic glass dissimilar welds and *in-situ* bulk metallic glass composite materials.
REFERENCES


