

A High Pressure Cell for Spark Plasma Sintering

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Abstract

Many nanostructured materials have been shown to have performance gains strongly dependent on the grain size in the material. Nanostructured thermoelectric materials for instance have found great performance increases through reduction of the grain sizes, due mostly to the scattering of phonons while retaining a good electrical conductivity. Other such examples abound where the grain size plays an important role in the performance of the material, including magnetic materials, proton fuel cell membranes, or simply improving the mechanical properties of a system through the Hall-Petch relationship.

A considerable amount of effort has been applied into reducing the grain size of an existing powder as well as retaining the small grain size during a sintering operation to create a bulk specimen. Frequently conventional sintering methods use temperatures and time scales that lead to deleterious grain growth. Spark Plasma Sintering (SPS) has been found to reduce the temperatures and times required to densify a sintered material by rapidly heating the sample compared to conventional “diffusive” sintering techniques. More recently the addition of high pressure to the SPS process has been shown to reduce the temperatures and sintering times even further, allowing for the retention of grain sizes as small as 10 nm.

In this work we design and test a new kind of pressure cell for SPS to deviate from the conventional graphite die arrangements common to the literature. The new swaged alumina core cell was designed for pressures of 1 GPa and temperatures in excess of 1,000 °C, with a sample diameter of 12.7 mm. It is hoped that this design will lead to improved cells that are fully reusable allowing for the economical production of sintered samples with grain sizes smaller than 50 nm.

The cell also tests the use of the NiCrAl alloy as an electrode material. This alloy, having been fabricated for neutron scattering studies, has properties that may make it useful for high temperature, high strength applications. In addition to the work with the SPS cell the fabrication and characterization of this alloy is discussed.

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Chapter 1: Introduction and Objective

Sintering is a term used to describe a process used to bond particles into a coherent body by application of heat and/or pressure. [1] The process is almost as old as civilization itself; ancient sintered ceramic structures were found in archeological excavations dating back nearly 26,000 years. The sintering of gold, silver, copper and bronze metals occurred around 3,000 BC in the Middle East, iron around 2,300 BC in Egypt (and again around AD 400 in India). Alloys of gold and platinum were sintered by the Inca in Ecuador and Columbia around 300 AD, and this process led to an explosion of interest in Europe (usually relying on toxic additives). The term “sintering” first came about in the 1700s to describe the thermal bonding of minerals in geology. However, despite the historical records of sintering the process was largely trial and error and only scrutinized by qualitative models after 1900, with quantitative and mathematical models appearing near the middle of the 20th century. [2]

Typically the primary goal of sintering is to achieve the highest density possible in the final processed material, although there are applications where an intermediate density is desirable (e.g. for some sintered titanium implants it may be more important to match the Young’s modulus of bone rather than have the modulus of fully dense titanium [3]). The sintered material will inevitably initially have grains of various shapes with “voids” or “pores” between them. If the particles have a theoretical density of ρ_t , and the volumetric mass of the sintered material is given by $\bar{\rho} = m/V$, then the porosity is given by $\theta = V_{pores}/V = 1 - \rho = 1 - \bar{\rho}/\rho_t$.

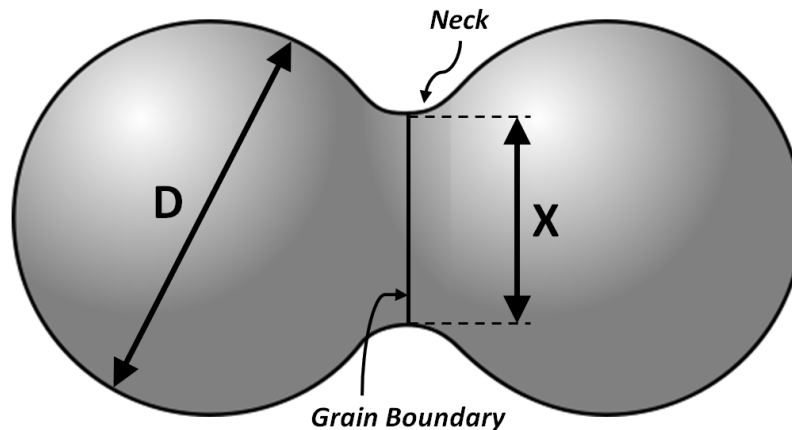


Figure 1 – Schematic representation of two spherical particles undergoing sintering.¹

¹ Derivative work adapted from public domain image “File:Frittag 2 grains.svg” created by author “CDang” on the Wikimedia Commons (commons.wikimedia.org).

Figure 1 shows a schematic representation of two spherical particles of diameter 'D' (and particle radius ' r_p ') undergoing sintering. The two particles merge to form a neck with a diameter 'X' (and neck radius ' r_n '). For the simple case of a spherical particle pressing against a flat plate, the neck radius conforms to the following law:

$$\left(\frac{r_n}{r_p}\right)^n = \frac{kt}{r_p^m} \quad \text{Eq. 1}$$

where ' t ' is time and ' k ' is constant. The constants ' n ' and ' m ' depend on the dominant mechanism of matter transport as shown in Table 1. [1]

Table 1 - Constants for use in Eq. 1. From ref. [1].

<i>Mechanism</i>	<i>n</i>	<i>m</i>
Viscous or plastic flow	2	1
Evaporation/condensation	3	2
Bulk diffusion	5	3
Surface diffusion	7	4

Evaporation and condensation occurs due to the effect of surface curvature on the vapor pressure of the material. Glass spheres and amorphous materials tend to sinter by viscous flow and crystalline metals and ceramics by bulk diffusion. Plastic flow refers to dislocation migration in a crystalline material. [1] Altering the density of a porous material through sintering affects the strength of the final material; in general, increasing density corresponds to a higher yield and ultimate strength, higher elastic modulus, and increased hardness and fracture toughness. The electrical and thermal conductivities also usually increase with increasing density.

Pressure is often used in conjunction with heating to aid in the sintering of a material. External pressure aids sintering through particle packing, sliding, fragmentation and deformation. While difficult to model theoretically, a semi-empirical relation was developed by Fryer [4] to relate the relative density ' ρ ' to pressure and temperature:

$$\left(\frac{\rho}{\theta}\right)^{2/3} = \frac{2}{3} \left(\frac{z}{l^2}\right) \frac{D\Omega Pt}{kT} + C \quad \text{Eq. 2}$$

where ' θ ' is again the fractional porosity, ' P ' is the applied pressure, ' Ω ' the vacancy volume, ' l ' the grain size, ' t ' time, ' k ' the Boltzmann's constant, ' T ' the absolute temperature and both ' z ' and ' C ' constants. A plot of $(\rho/\theta)^{2/3}$ versus ' t ' then gives a straight line. This relationship has been shown to work well for macroscopic particles of alumina. [1] The pressure compaction of nano powder

alumina however does not conform to conventional models since much of the energy is expended to break down agglomerates. [5]

The application of thermal energy to aid in sintering also leads to grain growth. Such growth is usually undesirable, since minimizing the grain size increases the mechanical strength of the final sintered material as shown by the Hall-Petch relationship

$$\sigma_y = \sigma_0 + \frac{k_y}{\sqrt{d}} \quad \text{Eq. 3}$$

where ' σ_y ' is the yield stress, ' σ_0 ' is the stress required for dislocation movement, ' k_y ' the strengthening coefficient, and ' d ' the average grain diameter. Both ' σ_0 ' and ' k_y ' are dependent on the material, for copper $\sigma_0 = 25 \text{ MPa}$ and $k_y = 0.11 \text{ MPa} \cdot \sqrt{m}$. [6] [7] More relevant to this work, grain growth is also often deleterious to the sintering of functional ceramics where smaller grain sizes (i.e. $< 50 \text{ nm}$) lead to drastic performance increases. For example, the ionic conductivity of CaF_2 and BaF_2 heterolayered films was shown to increase by orders of magnitude with a layer thickness decreasing to $\sim 50 \text{ nm}$. [8]

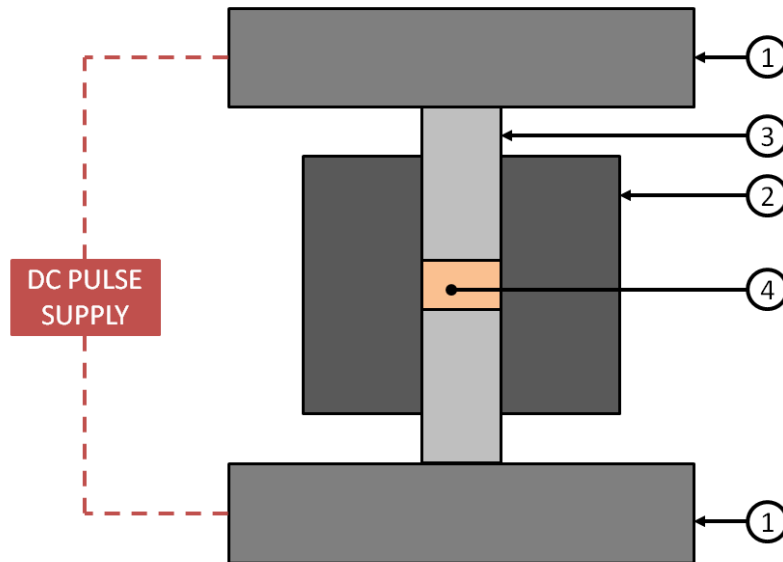


Figure 2 - Schematic of the SPS process. Labels: (1) Electrodes, (2) graphite die, (3) punch, (4) powder sample.

Spark Plasma Sintering (SPS) has emerged as a possible candidate to aid in the sintering of nanopowders. As shown in Figure 2, SPS passes a pulsed DC current from electrodes (1) through punches (3) in order to rapidly heat the graphite die (2). The powder sample (4) is heated much more rapidly than conventional sintering where the entire body is heated through external heating

elements. The electrodes are typically water cooled and simultaneously apply a pressure on the powder. The entire setup is enclosed in a water cooled vacuum chamber to prevent oxidation, or to introduce a specific atmosphere. [9]

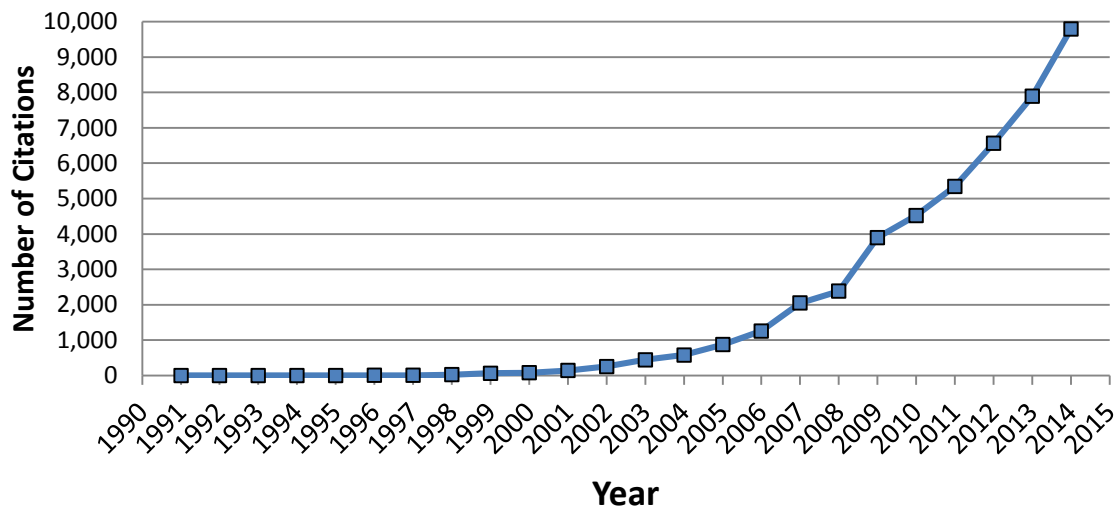


Figure 3 - The number of citations for "Spark Plasma Sintering" by year found using a Web of Science™ search.

Despite the resurgence of interest in SPS shown in Figure 3, the technique is hardly new. Nobel Prize winning Henri Moissan invented an electric furnace which he described in his 1897 book, and while he unsuccessfully attempted to create artificial diamond he instead used the furnace to create new compounds of tungsten and carbon. [10] [2] Sintering with electric current was first described in the US in 1906, and patents emerged in 1913 and 1922 describing sintering with electric current combined with pressure. In the 1960s Inoue in Japan patented the use of pulsed current calling the method “spark sintering”.² Much of his work was purchased by Lockheed Missile and Space Co. and given some commercial use, but was never too successful due to a lack of applications and being unable to justify the issues and costs associated with the equipment. [9] [11]

The exponential growth shown in Figure 3 is due to a new availability of commercial equipment in the 1990s, combined with a growing list of applications from on-going research. SPS has found applications in the sintering of materials with melting temperatures higher than 2000 °C (such as refractory metals and intermetallics), ultra-high-temperature ceramics (UHTCs, such as HfB₂),

² Throughout this report the process is referred to as “Spark Plasma Sintering” or SPS, although this name is now considered a misnomer due to the lack of plasma being involved in the process. [29] SPS is now also known more correctly as the “Field-Assisted Sintering Technique”, or FAST, though SPS is still used in literature.

transparent polycrystalline ceramics, functionally graded materials, non-equilibrium materials, and nanostructured materials. [9] The ability of SPS to densify nanopowders while minimizing grain growth is particularly useful. Conventional sintering methods, especially those without applied pressure usually fail to densify nanopowders with grain sizes below 50 nm. The task of densifying powders with grain sizes below 50 nm has proven to be very difficult, with the problem being compounded by a lack of theoretical understanding of the mechanisms behind the densification and sintering of extremely fine nanopowders. [12] SPS devices can typically achieve heating rates on the sample between 100 and 300 °C/min (with up to 1,000 °C/min reported) with currents well above 1 kA. The rapid heating leads to a remarkable performance increase over conventional sintering methods; for example a Yttria Stabilized Zirconia (YSZ) nanopowder can be fully densified with an applied pressure of 100 MPa in only five minutes with a temperature below 1,200 °C, compared to conventional sintering requiring hours at 1,400 °C. [13] [12]

As with conventional sintering, the sintering temperature and grain growth can be reduced in the SPS process through application of applied pressure. High Pressure Spark Plasma Sintering (HP-SPS) has been shown to allow for fully dense ceramics with a grain size of ~10 nm, and is therefore one of the most promising candidates for the fabrication of bulk functional nanomaterials. [12] [14] Despite these proven advantages, HP-SPS is a fairly new technique with the cells used not drastically deviating from conventional graphite die arrangements. Typical HP-SPS devices will take advantage of stronger die materials, but not new geometries or methods (e.g. using a silicon carbide or tungsten carbide punch). [14]

There were two objectives of the work detailed in this thesis: (1) to develop a new prototype HP-SPS cell allowing for repeatable operation at ~1 GPa applied pressure and temperatures in excess of 1,200 °C with an inner die diameter of ~0.5 inch (12.7 mm), and (2) develop and characterize a NiCrAl alloy believed to be a good candidate for the electrodes used in such a HP-SPS cell. The NiCrAl alloy will be described in the next section. It is hoped that the HP-SPS cell developed here will be used to aid current on-going research at UTK and ORNL towards the development of advanced proton fuel cell membranes based on nanostructured ceramic compounds.

Chapter 2: Fabrication and Characterization of the NiCrAl Alloy

The NiCrAl alloy, also known as the “Russian alloy” 40HNU-VI (40хНю-ВИ), is an alloy developed by the former Soviet Union largely for use in the high pressure community due to its very high strength. The composition of the alloy is approximately 39-41% (by weight) chromium, 3.3-3.8% aluminum, and balance nickel. The yield strength is remarkable with a room temperature yield strength of ~2.0 GPa attainable by high treatment (with a tensile strength of ~2.10 GPa), with approximately a 2% elongation (with ~ 60 HRC hardness). With heat treatment the ductility can be improved while sacrificing the yield strength; heat treated for a yield strength of ~1.6 GPa the elongation can be increased to ~8%. Some other noteworthy properties of the alloy include: [15] [16]

- Compared to other high strength alloys (i.e. maraging steels, cobalt containing nickel based superalloys, etc.) the alloy has better neutronics properties, allowing its use in high pressure neutron scattering cells – especially at low temperature and high magnetic fields. [15]
- A magnetic susceptibility of $4 \cdot 10^{-8} \text{ m}^3/\text{kg}$ at room temperature, and $1 \cdot 10^{-7} \text{ m}^3/\text{kg}$ at 4 K. [16]
- With an FCC crystal structure reported, the alloy has also been shown to have “good” properties at cryogenic temperatures. [17]
- The alloy strengthening process is accomplished by age hardening during heat treatment, but the alloy can also be cold worked (or work hardened) and subsequently age hardened to produce hardnesses of up to 64-67 HRC (though, with virtually no ductility or toughness). [17]
- The electrical resistivity of NiCrAl is approximately $8 \cdot 10^{-7} \Omega \cdot \text{m}$ at room temperature (comparable to the $\sim 6.9 \cdot 10^{-7} \Omega \cdot \text{m}$ of stainless steel) and $6.4 \cdot 10^{-7} \Omega \cdot \text{m}$ at 4.2 K. [17]

Despite the interesting properties of the alloy, it remained mostly unavailable outside the Russian Federation until Uwatoko et. al. managed to fabricate a version of the alloy that utilized 50 ppm B to “improve the forging process”. They describe fabrication of the alloy using high purity starting constituents – 99.9% Ni, 99% Cr, and 99.999% Al. The weight percentages used were 56.5% Ni, 40.0% Cr, 3.5% Al, with the aforementioned 50 ppm addition of boron. This was used to form an ingot of 7 kg, which was then hot worked to rods of “various sizes” at 1,200 °C corresponding to a reduction of area between 90-95% after hot working. [18]

In this thesis the fabrication of the alloy following the Japanese method above is described. The resulting alloy will be primarily used for low-temperature neutron scattering cells at the Spallation Neutron Source (SNS) and High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL). High temperature properties are not yet known for the alloy, but it is hoped that if the precipitates

are formed during age hardening are from the intermetallic nickel aluminide (i.e. NiAl, NiAl₃, or Ni₃Al), then the high temperature properties might be favorable as indicated by prior research on this intermetallic compound. For example, some nickel-aluminide alloys have been shown to have yield strengths actually increase up to temperatures of ~600 °C. [19] [20]

2.1 Fabrication

The alloy was fabricated by Sophisticated Alloys, Inc. (Butler, PA, USA) by vacuum induction melting 100 lbs (~45.6 kg) of material with the weight percentages and purities shown in Table 2.

Table 2 - Specified weight percentages and purities for the fabrication of the NiCrAl alloy.

<i>Element</i>	<i>Composition (Wt. %)</i>	<i>Purity</i>
Ni	Balance	99.99%
Cr	40.00	99.9%
Al	3.50	99.999%
B	0.005	99.5%

This material was cast into a rod with an outer diameter of 4.3 inches (~109.2 mm) and 25 inches long (635 mm). At this point a chemical analysis of the major elements was completed to verify the desired composition in Table 2. The results of this chemical analysis are shown in Table 3.

Table 3 - Metal chemistry results for the fabricated NiCrAl rod.

<i>Element</i>	<i>Composition (Wt. %)</i>	<i>Method, ASTM</i>
Al	3.47%	ASTM E1097-07/CTP 3005/ DCP
B	0.006%	ASTM E1097-07/CTP 3005/ DCP
Cr	40.25%	ASTM E1097-07/CTP 3005/ DCP

The resulting rod was then centerless ground to clean the outside surface, and open die forged to an outer diameter of 3 inches (76.2 mm) while maintaining a temperature of 1,200 °C. This was then rotary forged to an outer diameter of 0.930 inch (~23.6 mm), again while maintaining 1,200 °C. This rod was annealed at 1,200 °C for one hour then rapidly water quenched. Finally, the rod was aged for two hours at 700 °C in a vacuum then air cooled, as shown in Figure 4. The final rod was centerless ground to 0.860 inch (~21.8 mm).

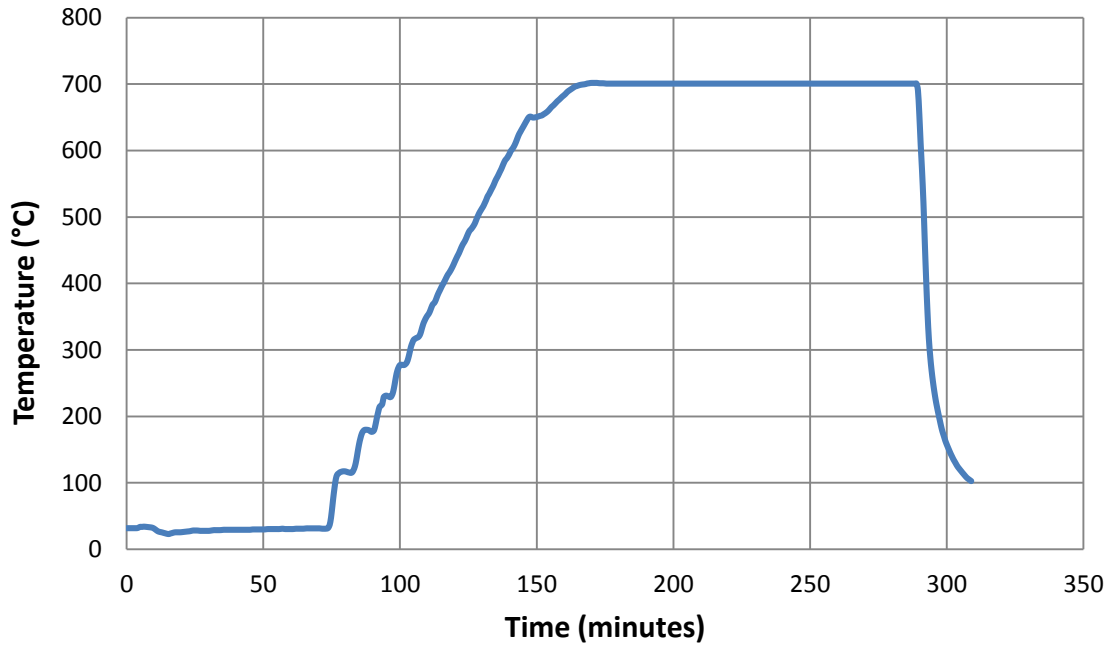


Figure 4 - Heat treatment report of the NiCrAl rod

Lastly, some preliminary hardness testing was completed on the rod prior to shipping. The approximate locations of the hardness testing and results are shown in Figure 5.

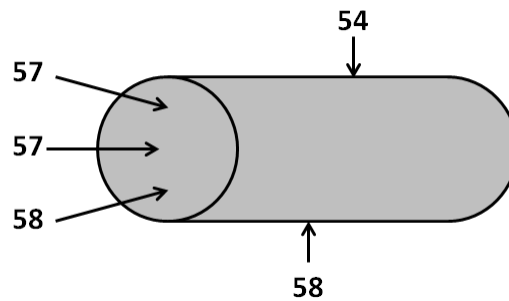


Figure 5 - Preliminary hardness testing on the final NiCrAl rod completed by Sophisticated Alloy, Inc. (hardness in HRC).

2.2 Hardness Testing

When the sample arrived at ORNL a section was removed for microhardness testing as shown in Figure 6. It was assumed that the forging operations resulted in a rod with approximate axisymmetry, so only one quadrant of the rod was inspected.

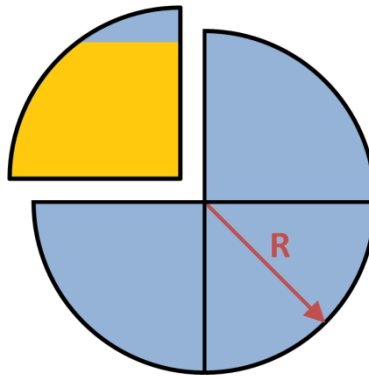


Figure 6 - The area (shown in yellow) where hardness testing was performed. The radius 'R' of the rod is ~11 mm (total rod diameter 0.86 inch).

The results of the microhardness testing performed at ORNL are shown in Figure 7. Towards the outside of the rod the hardness increases to a maximum value of ~60 HRC (~690 Vickers), and the center of the rod has the lowest hardness of about 56 HRC (~630 Vickers) – as would be expected with heat treatment. Both pores and precipitates appear to be visible.

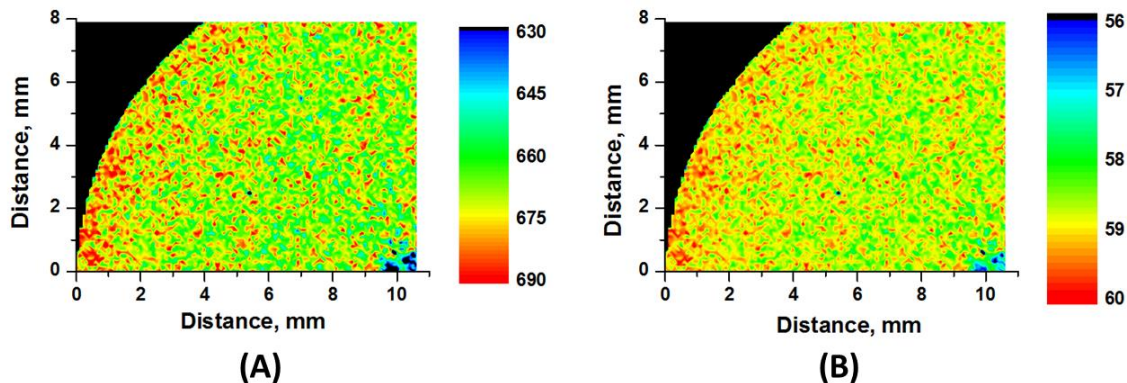


Figure 7 - Results of the microhardness testing. (A) the Vickers hardness map, and (B) the HRC hardness map.

Figure 8 shows metallographic images for the specimen, for both polished and etched conditions from magnifications from 20x to 1000x. The etched conditions clearly show significant porosity.

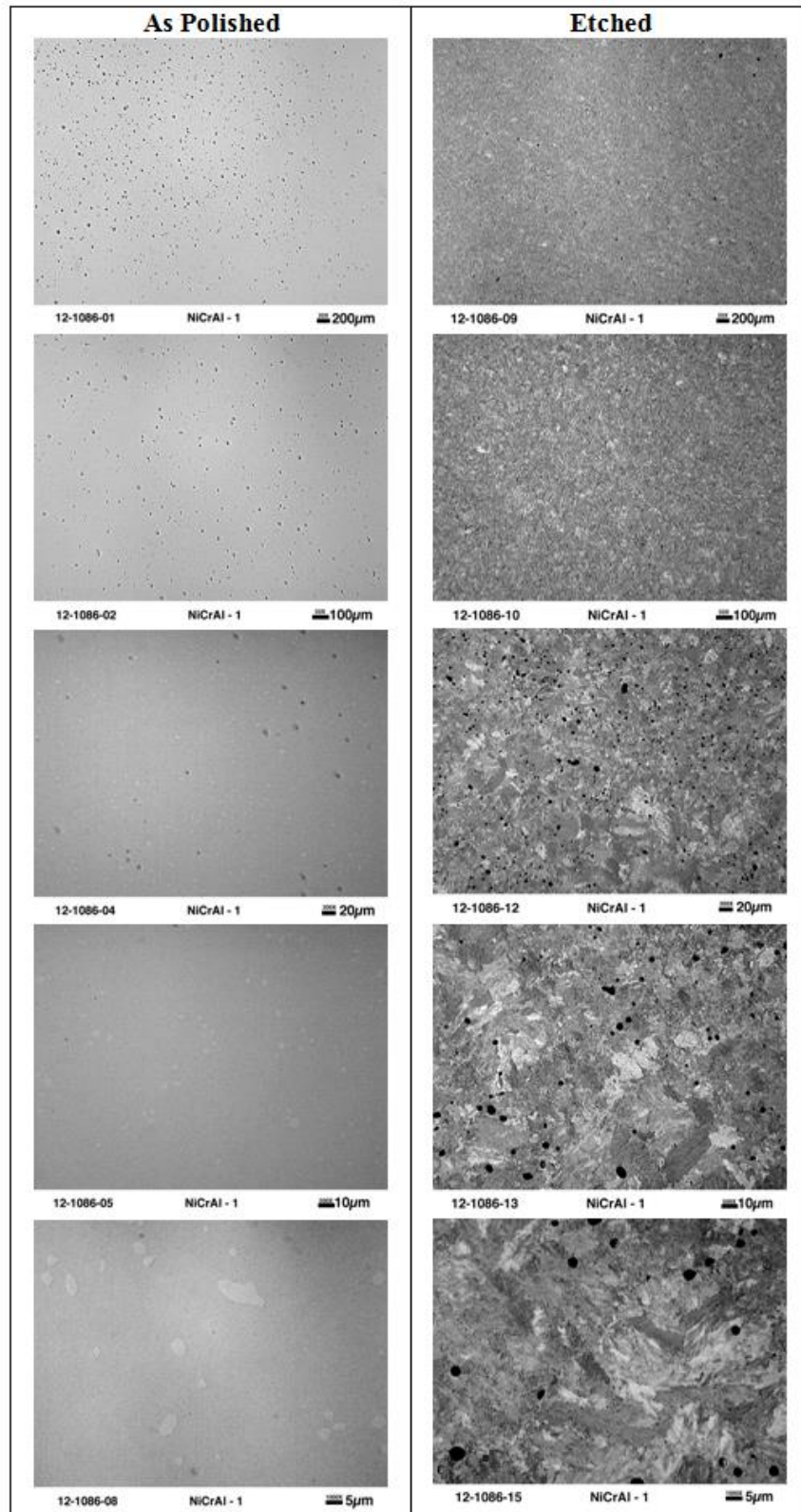


Figure 8 – Metallographic images for as-polished and etched conditions.

2.3 Tensile Testing

A non-standard specimen was designed to allow for four identical samples to be taken from the cross-section of the 0.86 inch (~21.8 mm) diameter rod. As shown in Figure 28 in “Appendix A” on page 37, the specimens have an outer diameter of 0.25 inch (6.35 mm), and a length of 2 inches (50.8 mm). The gauge length of the specimen is 0.75 inch (~19 mm) and the gauge diameter is 0.125 inch (~3.2 mm). These specimens were taken from the locations shown in Figure 9.

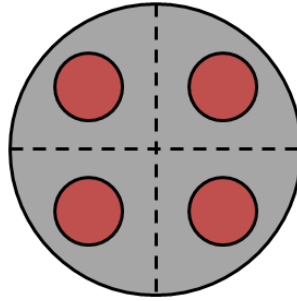


Figure 9 - Approximate locations of the tensile specimens (shown in red) with respect to the original rod.

Two specimens were evaluated at room temperature, and another two at 500 °C. A 0.5 inch (12.7 mm) gage extensometer was used to measure elongation, and all specimens were run at a nominal strain rate of 10^{-4} s^{-1} . The results of the tensile testing are shown in Figure 10.

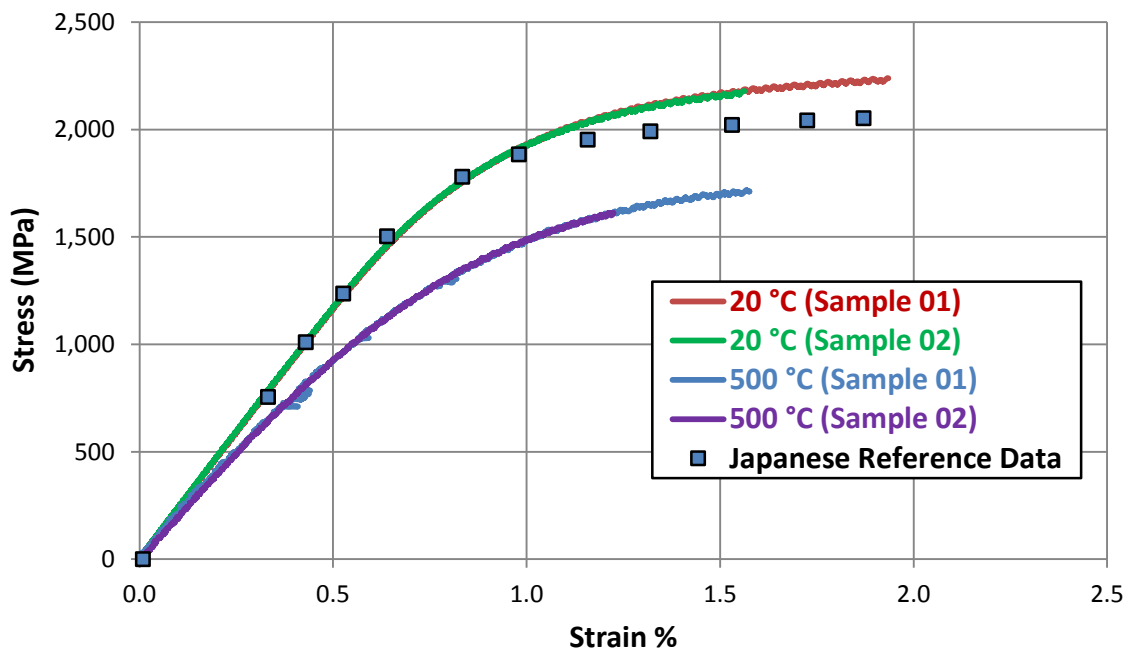


Figure 10 – Room temperature and 500 °C stress-strain curves for NiCrAl.

While the yield stresses appear to be very repeatable, the tensile strength values differ greatly with each specimen. This is likely due to the small cross-sectional area of the specimens combined with the varying hardness and porosities observed in both Figure 7 and Figure 8. Still, at room temperature the specimens indicated tensile strengths of 2,179 MPa and 2,239 MPa with total elongations of 1.56% and 1.93% respectively. At 500 °C the tensile strength decreased, with the two specimens indicating tensile strengths of 1,717 MPa and 1,612 MPa with total elongations of 1.57% and 1.22% respectively. Unpublished data from Japan (courtesy of Y. Uwatoko) for the room temperature testing of a sample aged at 700 °C agrees fairly well with the data, indicating the fabrication was successful.

2.4 Thermal Expansion

A small disk of the NiCrAl alloy was fabricated for thermal expansion measurements per ASTM E831-12 “Standard Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis”. [21] The sample used is shown in Figure 29 in “Appendix A” on page 38.

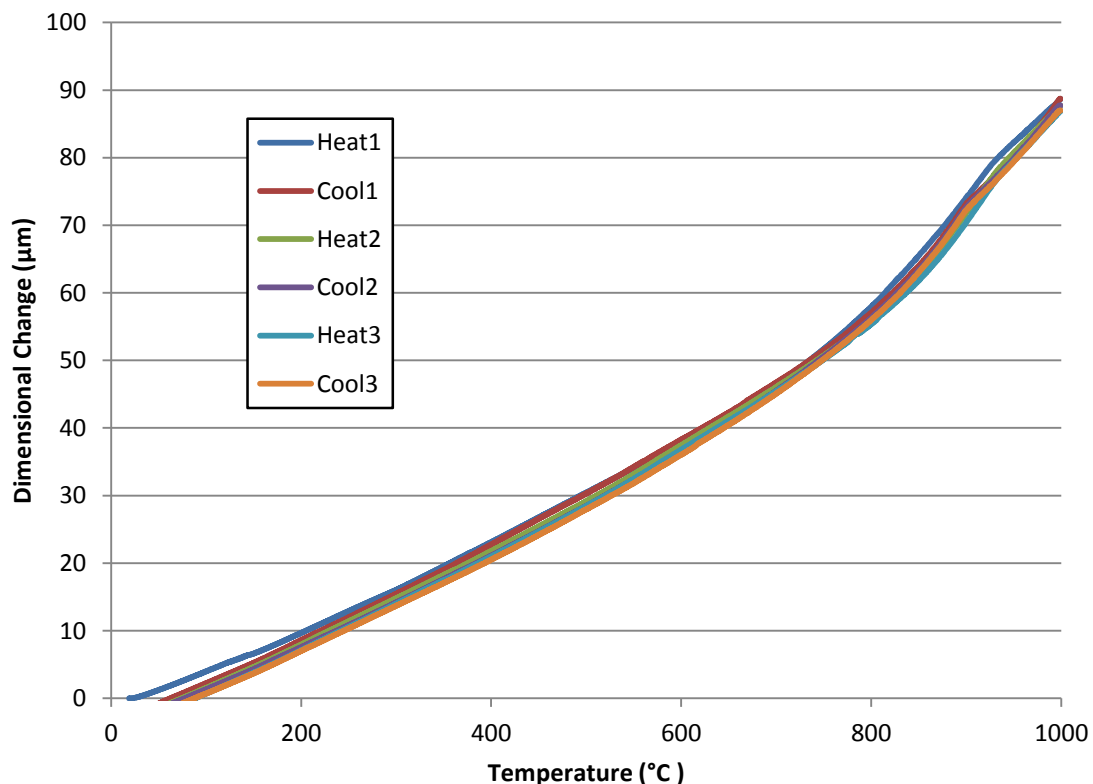


Figure 11 - The dimensional change in the specimen, with multiple heating and cooling cycles.

Figure 11 shows the dimensional changes in the specimen with increasing temperature. It appears to smoothly increase, although the slope does show a marked change at ~900 °C. The instantaneous coefficient of thermal expansion is calculated from the derivative

$$\alpha_L = \frac{1}{L} \frac{dL}{dT} \quad \text{Eq. 4}$$

where here ‘ L ’ is the original sample length, and ‘ dL/dT ’ is the rate of change.

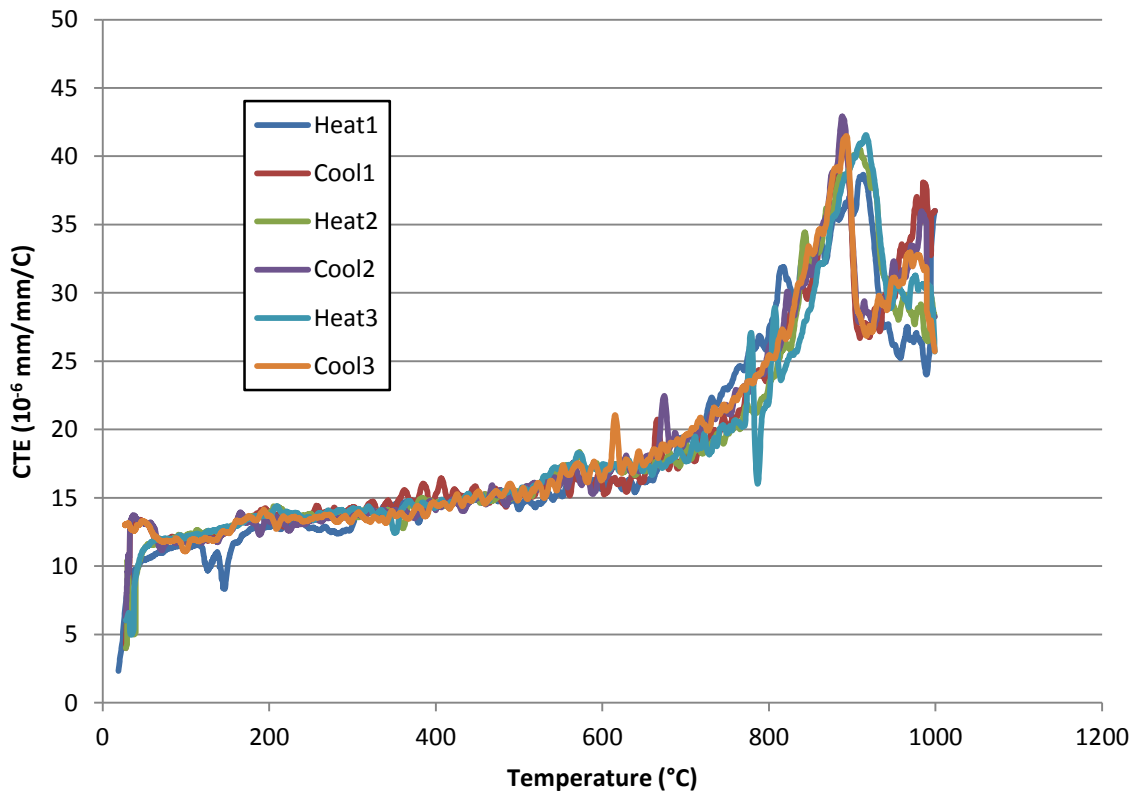


Figure 12 - The instantaneous CTE for the NiCrAl alloy.

The instantaneous CTE for the NiCrAl alloy is shown in Figure 12. The abrupt transition is clearer with the derivative, and could indicate an effect like oxidation or a phase change. Oxidation was ruled out by repeating the tests in an inert argon atmosphere (data not shown), so the jump is likely due to a phase change. It is often useful to designate a linear CTE for a range of temperatures so quick estimations can be made. For this case Eq. 4 can be approximated by $\alpha_L = \Delta L / (L \Delta T)$ where ‘ ΔL ’ is the total dimensional change across the range of interest and likewise ‘ ΔT ’ the total temperature change. Doing this with the data shown in

Figure 11 we find the CTE from 30 to 1,000 °C to be approximately 18.6×10^{-6} mm/mm/°C, the CTE from 30 to 900 °C to be approximately 17.1×10^{-6} mm/mm/°C, and the CTE from 30 to 800 °C to be approximately 15.2×10^{-6} mm/mm/°C.

2.5 Elastic Modulus Measurements

The elastic modulus of a material can be found using Resonant Ultrasound Spectroscopy (RUS). This technique measures the complete elastic tensor of a material by finding the vibrational resonance spectrum of the sample. The resonant spectrum will depend on the sample geometry, density, and elastic moduli. The former two properties are easily measured or controlled, with typical samples being precision machined and polished parallelepipeds (the sample used is shown in Figure 30 in “Appendix A” on page 38). The sample is then sandwiched between two transducers (one “drive” transducer and one “pickup” transducer) and a frequency sweep performed to determine the spectrum.

In the generalized Hooke’s law in tensor notation, there are 21 elastic constants for the general anisotropic case, as shown in Eq. 5. [6]

$$\begin{bmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{zz} \\ \sigma_{yz} \\ \sigma_{zx} \\ \sigma_{xy} \end{bmatrix} = \begin{bmatrix} C_{11} & C_{12} & C_{13} & C_{14} & C_{15} & C_{16} \\ C_{21} & C_{22} & C_{23} & C_{24} & C_{25} & C_{26} \\ C_{31} & C_{32} & C_{33} & C_{34} & C_{35} & C_{36} \\ C_{41} & C_{42} & C_{43} & C_{44} & C_{45} & C_{46} \\ C_{51} & C_{52} & C_{53} & C_{54} & C_{55} & C_{56} \\ C_{61} & C_{62} & C_{63} & C_{64} & C_{65} & C_{66} \end{bmatrix} \begin{bmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{zz} \\ \sigma_{yz} \\ \sigma_{zx} \\ \sigma_{xy} \end{bmatrix} \quad \text{Eq. 5}$$

However, as in the case of the NiCrAl alloy, if the material is completely isotropic the number of constants reduces to two. Here $C_{11} = C_{22} = C_{33}$, $C_{13} = C_{23} = C_{12}$, $C_{44} = C_{55} = C_{66}$, and $2C_{66} = (C_{11} - C_{12})$. [22] The RUS measurements provide C_{11} and C_{44} , allowing all other values to be calculated. The shear modulus μ is simply C_{44} , and the bulk modulus K can be found by

$$K = C_{11} - (4/3)\mu \quad \text{Eq. 6}$$

With both ‘ K ’ and ‘ μ ’ known, the Poisson’s ratio ‘ ν ’, Young’s modulus ‘ E ’, and Lamé parameter ‘ λ ’ can found using the relations shown in Table 4.

Table 4 – Relationships between different moduli and parameters. From ref. [23].

μ	K	λ	E	ν
$\frac{3(K - \lambda)}{2}$	$\lambda + \frac{2\mu}{3}$	$K - \frac{2\mu}{3}$	$\frac{9K\mu}{3K + \mu}$	$\frac{\lambda}{2(\lambda + \mu)}$
$\lambda \left(\frac{1 - 2\nu}{2\nu} \right)$	$\mu \left[\frac{2(1 + \nu)}{3(1 - 2\nu)} \right]$	$\frac{2\mu\nu}{1 - 2\nu}$	$2\mu(1 + \nu)$	$\frac{\lambda}{3K - \lambda}$
$3K \left(\frac{1 - 2\nu}{2 + 2\nu} \right)$	$\lambda \left(\frac{1 + \nu}{3\nu} \right)$	$3K \left(\frac{\nu}{1 + \nu} \right)$	$\mu \left(\frac{3\lambda + 2\mu}{\lambda + \mu} \right)$	$\frac{3K - 2\mu}{2(3K + \mu)}$
$\frac{E}{2(1 + \nu)}$	$\frac{E}{3(1 - 2\nu)}$	$\frac{E\nu}{3(1 + \nu)(1 - 2\nu)}$	$3K(1 - 2\nu)$	$\frac{3K - E}{6K}$

A plot of the Young's modulus from 300 K to 50 K is shown in Figure 13.

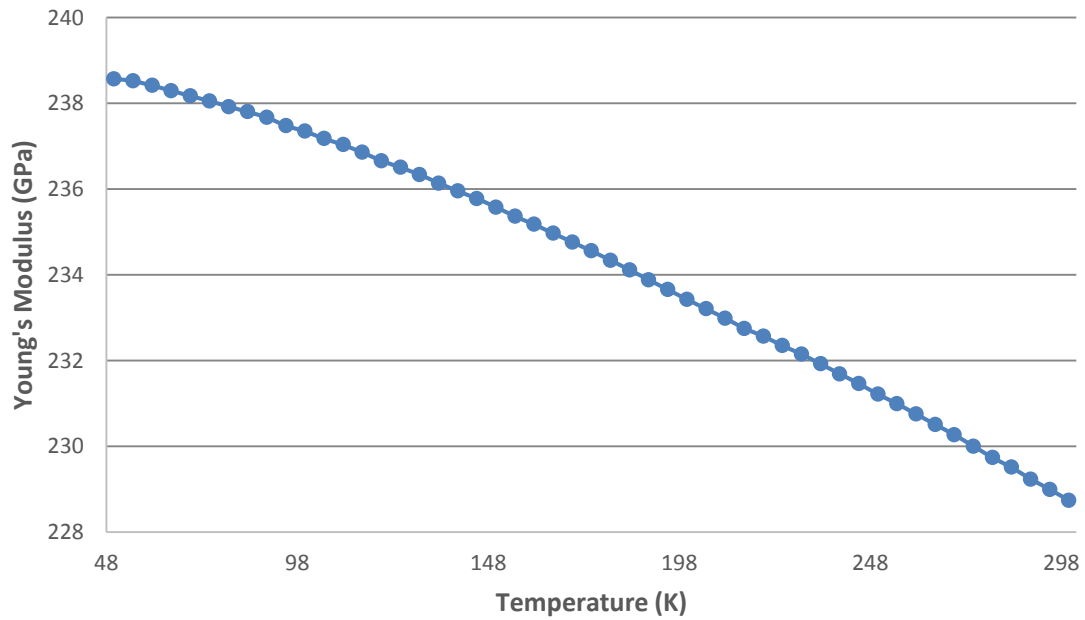


Figure 13 - The Young's modulus from 300 K to 50 K as determined from RUS measurements.

The values for the moduli and constants listed in Table 4 are given in Table 5 for a variety of temperatures.

Table 5 - Values for the moduli and constants from Table 4 for a variety of temperatures.

T (K)	μ (GPa)	K (GPa)	λ (GPa)	E (GPa)	ν
300	88.00	185.14	126.22	228.74	0.294
275	88.90	185.75	126.48	230.01	0.294
250	89.42	186.04	126.43	231.22	0.293
225	89.91	186.30	126.36	232.35	0.292
200	90.39	186.36	126.10	233.43	0.291
175	90.87	186.72	126.14	234.56	0.291
150	91.32	186.84	125.96	235.58	0.290
125	91.75	186.71	125.54	236.51	0.289
100	92.15	186.46	125.03	237.35	0.288
75	92.46	186.58	124.94	238.06	0.287
50	92.69	186.61	124.82	238.57	0.287

The Young's modulus of pure Ni is ~170 GPa, and Cr ~250 GPa. As shown in Table 5 the Young's modulus of NiCrAl alloy at room temperature is ~229 GPa, which is greater than most steels; for example alloy 304 stainless steel has a Young's modulus between 193-200 GPa and most steels do not generally exceed around 200 GPa (AISI S7 tool steel has a modulus of ~207 GPa). This combination of high elastic modulus and high yield strength is comparable to the some of the heat treated tool steels.

Chapter 3: The High Pressure SPS Cell

3.1 Design Strategy

By necessity, the inner wall of a pressure vessel designed for use with SPS must be nonconductive to prevent shorting of the electrodes. This is by far the most serious constraint to take into consideration since nonconductive materials are generally weak in tension (like ceramics) or unable to operate at the high temperatures required for SPS (as is the case with plastics). The ideal material would be one that is electrically non-conducting, low thermal conductivity, high tensile strength, ductile (for safety with high pressure), tough (resistance to cracking), thermal shock resistant, and capable of operating at high temperature. There are additional constraints with manufacturability and cost. Such constraints have typically limited SPS vessels to using graphite crucibles – a material that is not generally suited for use in pressure vessels.

Ceramic materials have poor ductility but in general possess higher compressive strengths than metals. A concept used in high pressure research is to take a ring of a ceramic material, such as alumina or tungsten carbide, and subject it to a high external pressure by means of an interference fit. This produces large compressive stresses in the ceramic ring, which the ring can easily withstand. Application of an internal pressure then counteracts the existing compressive stresses allowing the ring to withstand much higher pressures. The “belt” apparatus design by H. Tracy Hall uses this concept with multiple rings and generated pressures and temperatures high enough to create artificial diamonds. [24] [10]

The first design was planned to be a simple “proof of concept” pressure cell based on this method using an aluminum oxide “alumina” core and a Grade 5 (ASTM) Ti-6Al-4V titanium binding ring. Alumina was chosen for its high compressive strength of approximately 2,103 MPa (~305,000 psi), while also being cheap compared to other ceramics such as YTZP. The Grade 5 titanium binding ring was chosen for both its high strength and low thermal expansion. The low thermal expansion was chosen to prevent expansion from decreasing the press fit interference pressure as will be described in the next section. If the concept cell shows promise this constraint could be dropped in favor of more expensive water cooling channels machined into the cell.

Figure 14 shows a cross-sectional view of the prototype high pressure SPS cell. The alumina core (3) is shown swaged with the titanium binding ring (4). The NiCrAl electrodes (2) are threaded and joined to a stainless steel “electrode piston” (1) that is pushed against by the pistons of the SPS machine. Both the bottom and top electrodes are identical, though the bottom electrode has an intermediate spacer (5) to keep the sample centered in the alumina before

sintering. This cell was designed to be relatively inexpensive, with water cooling added to a future revision if the prototype design demonstrates use at higher pressure.

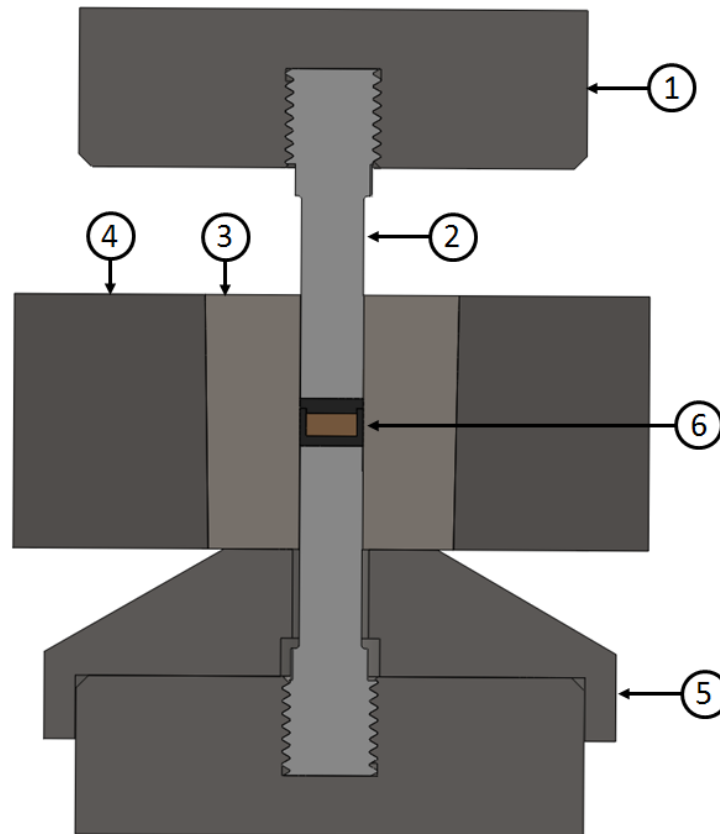


Figure 14 - The prototype high pressure SPS cell. Labels: (1) Electrode piston, (2) NiCrAl electrode, (3) alumina core, (4) titanium binding ring, (5) bottom spacer, (6) graphite sample assembly.

3.2 Stress Analysis

The binding ring assembly (items 3 and 4 in Figure 14) had to be designed carefully such that the interference pressure produced from swaging would produce high compressive stresses, but both parts would survive both the loading process and the SPS process. The design strategy here was to keep the inner radius of the titanium ring purely elastic, with the outside radius at a very low stress for safety reasons. The alumina material chosen had a compressive strength of 2,103 MPa (305) psi and a flexural strength of 352 MPa (51 ksi). For the alumina ring the goal was to place compressive stresses that do not exceed $\sim 1/2$ the compressive strength of the material (i.e. ~ 150 ksi), allowing for a

comfortable margin of safety. During the SPS process when pressure is exerted on the inside surface of the alumina ring the goal was to keep the tensile stresses below half the flexural strength. The analysis done here is a simple analytical approach that does not take into account the thermal stresses.

Figure 15 shows schematically the two regions of a press fit pressure vessel made from two materials. The inner (orange) cylinder terminates at the radius ' r_a ' and is made from a material with an elastic modulus of ' E_i ' and a Poisson's ratio of ' ν_i '. Likewise the outer cylinder (red) is made from a material with an elastic modulus ' E_o ' and a Poisson's ratio of ' ν_o '.

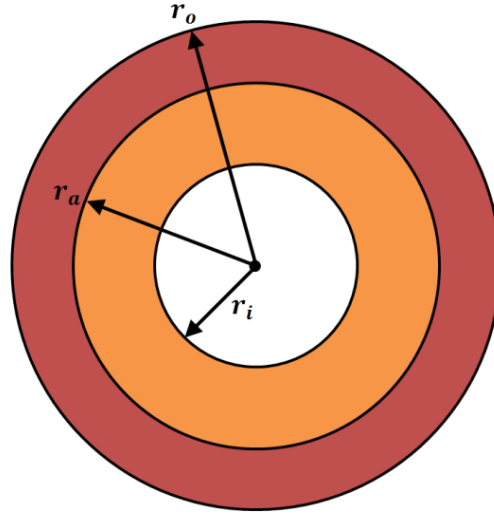


Figure 15 – Schematic showing the radii of the interference fits.

Given an interference ' δ ' between the two cylinders, formed either by a press fit or heat shrink, an interfacial pressure ' p_a ' will form pushing inward on the inner cylinder and outward on the outer cylinder. The interference is related to this interfacial pressure by [25]

$$\delta = \frac{r_a p_a}{E_o} \left(\frac{r_o^2 + r_a^2}{r_o^2 - r_a^2} + \nu_o \right) + \frac{r_a p_a}{E_i} \left(\frac{r_a^2 + r_i^2}{r_a^2 - r_i^2} - \nu_i \right) \quad \text{Eq. 7}$$

The interference pressure ' p_a ' is then given by

$$p_a = \frac{\delta}{r_a} \left[\frac{1}{E_o} \left(\frac{r_o^2 + r_a^2}{r_o^2 - r_a^2} + \nu_o \right) + \frac{1}{E_i} \left(\frac{r_a^2 + r_i^2}{r_a^2 - r_i^2} - \nu_i \right) \right]^{-1} \quad \text{Eq. 8}$$

If the two materials are the same such that $E = E_o = E_i$ and $\nu = \nu_o = \nu_i$ then this simplifies to [26]

$$p_a = \frac{E\delta}{r_a} \left[\frac{(r_o^2 - r_a^2)(r_a^2 - r_i^2)}{2r_a^2(r_o^2 - r_i^2)} \right] \quad \text{Eq. 9}$$

This interfacial pressure gives rise to residual stresses that can be found using the standard Lamé equations. The Lamé stresses are given by:

$$\sigma_\theta = \frac{p_i r_i^2 - p_o r_o^2 - r_i^2 r_o^2 (p_o - p_i) / r_i^2}{r_o^2 - r_i^2} \quad \text{Eq. 10}$$

$$\sigma_r = \frac{p_i r_i^2 - p_o r_o^2 + r_i^2 r_o^2 (p_o - p_i) / r_i^2}{r_o^2 - r_i^2} \quad \text{Eq. 11}$$

where ' σ_θ ' is the tangential stress, and ' σ_r ' is the radial stress. For the inner cylinder $p_i = 0$ and $p_o = p_a$. Similarly, for the outer cylinder $p_i = p_a$ and $p_o = 0$. The longitudinal stress ' σ_L ' is not being considered since it is assumed the pressure cell can be treated as an "open cylinder" where the force is taken in the pistons rather than in the wall material. In reality, there will be some longitudinal stresses from the press fit friction effects, but these are too difficult to approximate analytically and must be solved for using FEM. The longitudinal stresses are given by

$$\sigma_L = \frac{p_i r_i^2 - p_o r_o^2}{r_o^2 - r_i^2} \quad \text{Eq. 12}$$

For this analysis we will assume the longitudinal stresses are negligible compared to the radial and tangential stresses. Often it is useful to find the von Mises equivalent stresses given by

$$\sigma_v = \sqrt{\frac{(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_1 - \sigma_3)^2}{2}} \quad \text{Eq. 13}$$

where ' σ_1 ', ' σ_2 ', and ' σ_3 ' are the principal stresses corresponding to ' σ_θ ', ' σ_r ', and ' σ_L ' respectively. As mentioned the residual stresses are found by using the interference pressure ' p_i ' with the Lamé equations separately for each ring. The operating stresses are then found by ignoring the residual stresses and treating both the alumina ring and titanium ring as one complete ring subjected to a single pressure ' p_{op} ' on the inside radius of the alumina ring. Finally, the operating stresses are found by superimposing the operating Lamé stresses onto the residual stresses (i.e. $\sigma_\theta^{tot} = \sigma_\theta^R + \sigma_\theta^{op}$). The python code for calculating the residual and operating stresses can be found in Appendix B on page 39.

Figure 16 shows the calculated residual stresses in the alumina titanium press fit assembly for an interference of 0.008 inch (~0.20 mm). The analytical values are

compared to FEM values found using the ABAQUS FEM software package, both in good agreement. The residual tangential stress on the inner radius of the alumina core for this interference is found to be -136,886 psi (~943.8 MPa). This is still less than half the compressive strength for the grade of alumina used in the design of the prototype cell.

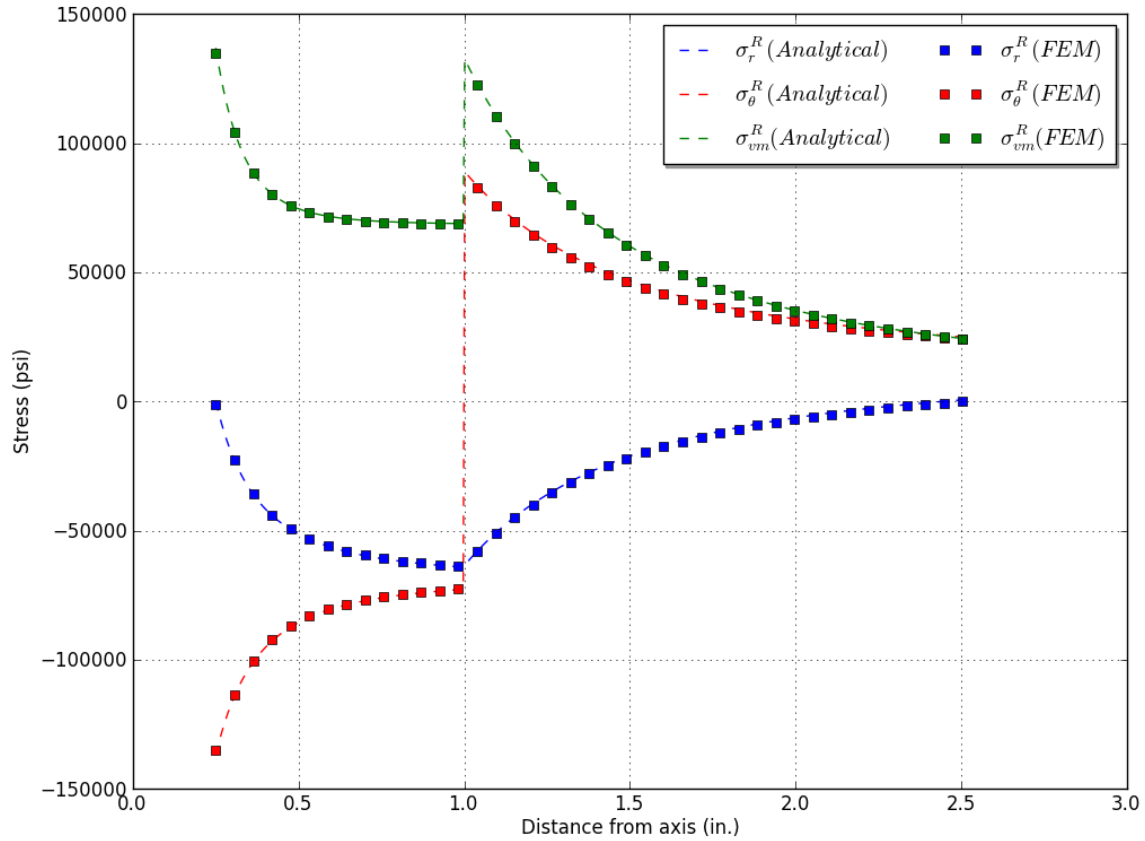


Figure 16 – The calculated residual stresses in the alumina and titanium press fit assembly. Analytical values are compared to FEM.

The total stresses found from combining the operating stresses to the residual stresses are shown in Figure 17. Here the maximum tangential stresses are found to be 11,160 psi (~77 MPa). Without the press fit the maximum tangential stresses are calculated to be 147,968 psi (~1,020 MPa). The press fit residual stresses clearly provide an advantage, with the tensile stresses kept below the flexural strength of alumina. The results are again in good agreement with FEM.

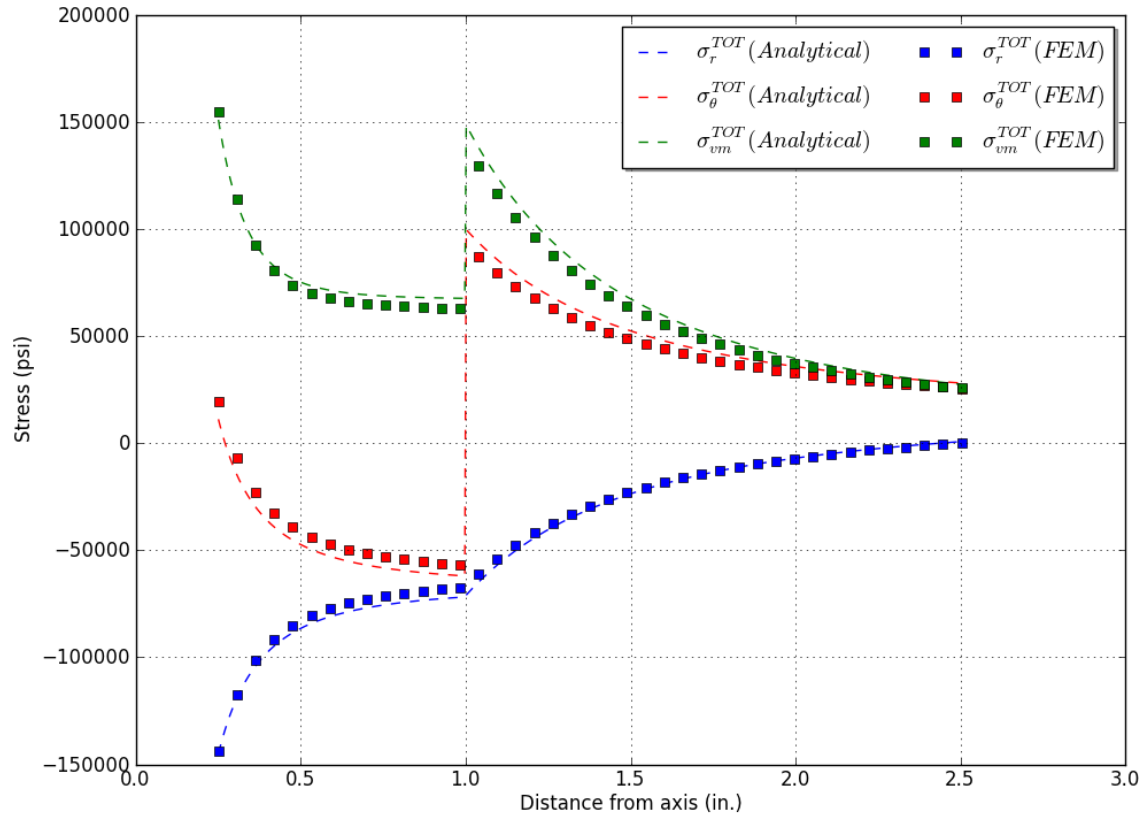


Figure 17 - The calculated operating stresses in the alumina and titanium press fit assembly. Analytical values are compared to FEM.

3.3 Construction

To minimize the shrink fit friction affects the alumina core and the titanium binder surfaces were spray coated with magnesium disulfide. The arrangement both before and after press fit is shown in Figure 18. The force required to press the alumina core into the titanium binding ring was approximately 230,000 lbs (~1,020 kN). As shown in Figure 18 two aluminum parts (6061-T6 alloy) were fabricated to aid in the press fit process (engineering drawings are shown in Appendix C on page 44). The aluminum ring below the titanium ring (the bottom-most part in Figure 18) was present to capture any alumina fragments from a possible failure. The top aluminum tool comes into contact with the titanium ring to prevent pressing the alumina core beyond flush. Figure 19 shows the completed alumina titanium binding ring arrangement alongside the remaining fabricated components. The electrode piston shown in Figure 19 was later modified to a reduced thickness as shown in Figure 14 to allow for use in an SPS machine outside of ORNL.

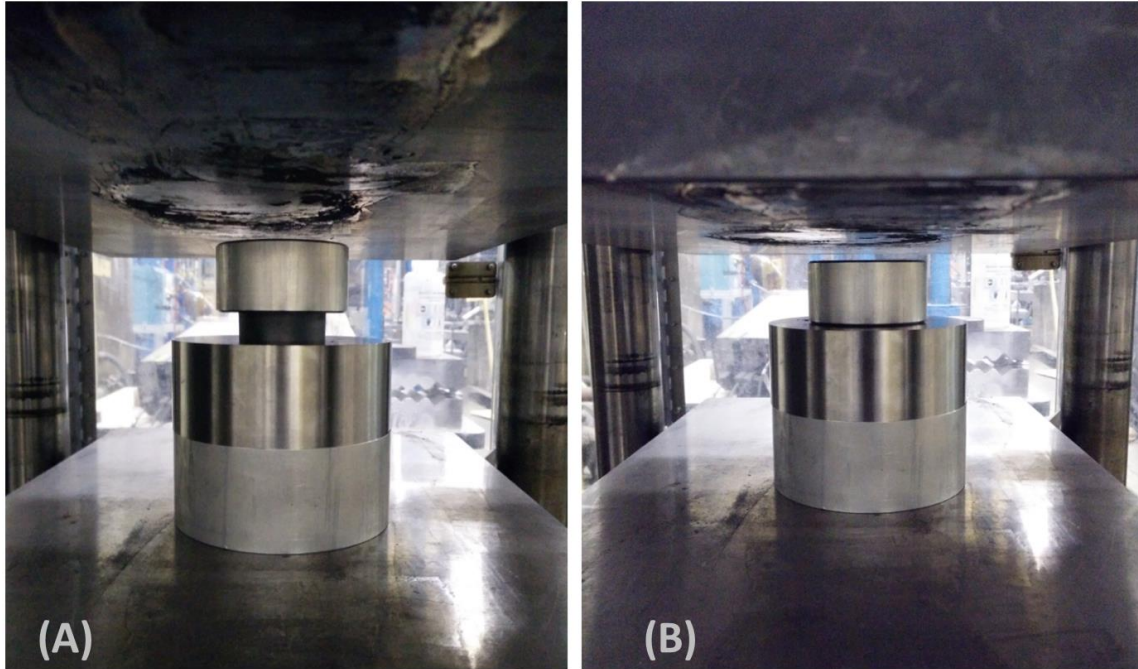


Figure 18 - The alumina titanium press fit process is shown. (A) The alumina ring before the press fit. (B) The alumina ring pressed into the titanium binding ring after application of force.

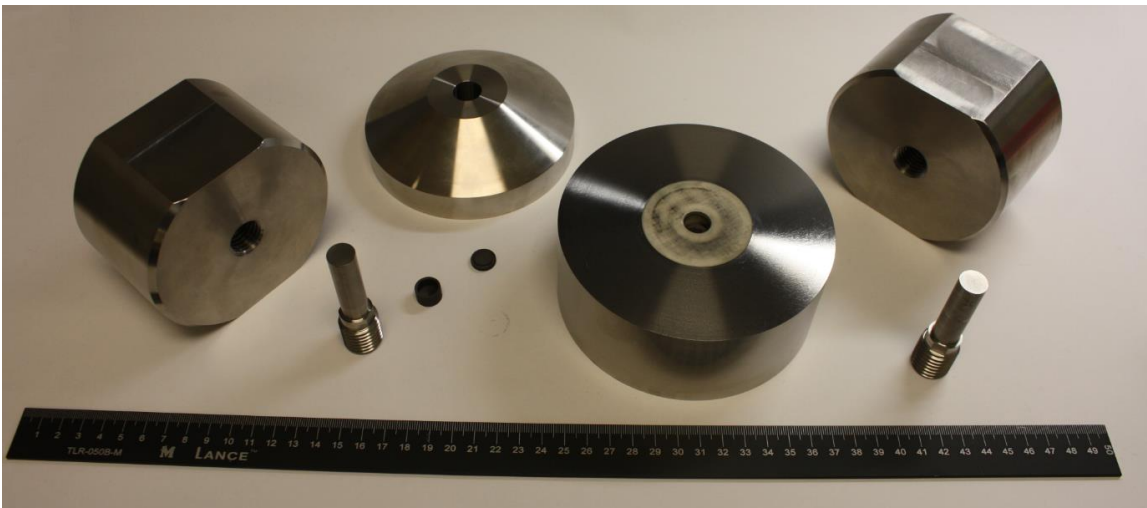


Figure 19 - The components of the prototype SPS cell. The ruler in the foreground is in centimeter units. The electrode pistons shown were later reduced in thickness to match the cross-section shown in Figure 14.

The completed cell assembly is shown in Figure 20. Engineering drawings for the various components of the cell can be found in Appendix D on page 46.



Figure 20 - The assembled prototype high pressure SPS cell. The ruler in the foreground is in mm units.

3.4 Compaction Cell

The graphite capsule (item 6 in Figure 14) was designed to accommodate a small volume sample for testing purposes. If the initial sample is a loose powder with high porosity the resulting compaction would be a thin fragile sample difficult to use and characterize. In this and many other cases it is advantageous to first compact the powder sample at high pressure and room temperature, and use the resulting compaction in SPS. For this reason, a room temperature sintering press was designed.

Without the constraints of high temperature and electrically insulating materials, attaining high pressure is fairly straightforward. A cylinder made from any high strength alloy is all that is required, assuming the safety factor is adequate and

the material fairly ductile for safety. A heat treated maraging steel alloy was chosen for due to its exceptionally high tensile strength and fracture toughness (both being greater than comparable tempered martensite steels, as shown in [27]). Figure 21 shows a labelled cross-sectional view of the compaction cell (drawings are shown in Appendix E on page 50).

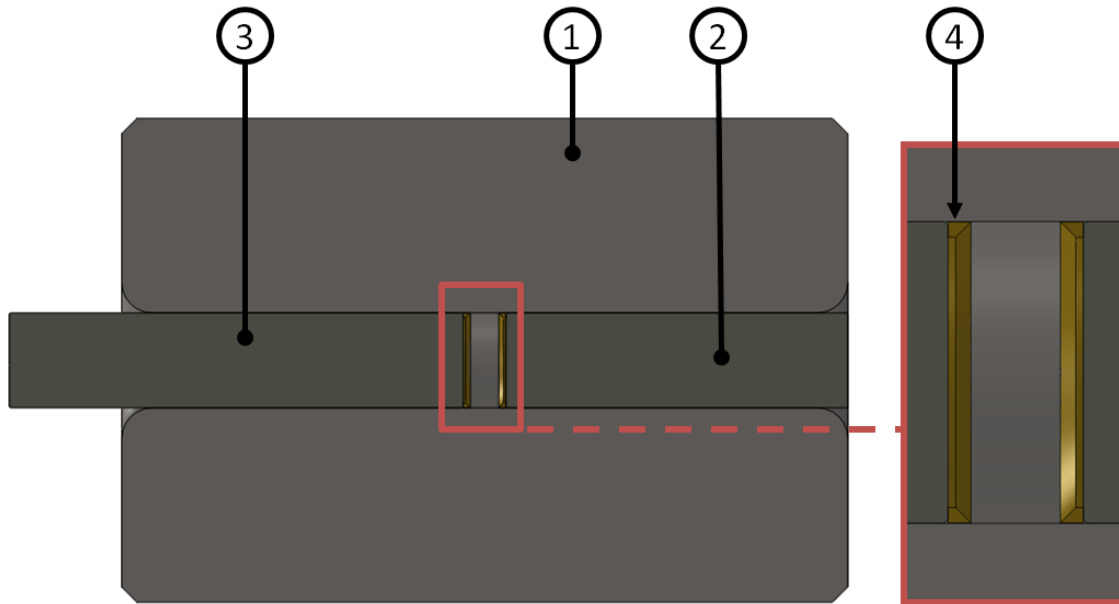


Figure 21 - The compaction cell. Labels: (1) Cell body, (2) lower tungsten carbide piston, (3) upper (moving) tungsten carbide piston, and (4) the brass anti-extrusion rings.

Item 1 in Figure 21 is the heat treated alloy 350 maraging steel body, items 2 and 3 the tungsten carbide pistons, and item 4 optional brass (alloy 360, ASTM B16) anti-extrusion rings for fine powder. Ejection rods for removing the sample are shown in Appendix E on page 50). The completed assembly is shown in Figure 22.

The name “maraging” is a portmanteau of “martensitic” and “aging”, as the high strength and toughness of the alloys comes from carefully aging a steel alloy that had previously undergone a martensitic transformation. The strength-to-weight ratios of the maraging alloys exceeds even those of titanium based alloys. Before aging of the alloys the annealed state is ductile and easily formed, and the aging process introduces only minor distortion. [27] The maraging alloy selected for use was alloy 350 (18 wt. % Ni, 12% Co, 4% Mo, 1.6% Ti, and balance Fe nominal composition). This alloy, purchased as a rod, underwent the following heat treatment: first brought to 1,700 °F (~927 °C) for one hour and air cooled to below 400 °F (~204 °C) then rapidly water quenched, then brought to 1,500 °F (~816 °C) for one hour and air cooled again. Following this heat treatment the rod was machined to the final dimensions with some allowance for post

machining due to distortion. This part was aged at 850 °F (~450 °C) for seven hours to achieve a final yield strength of at least 330,000 psi (~2.28 GPa) as estimated by local hardness testing.



Figure 22 - The completed compaction cell, showing the fabricated cell body, tungsten carbide pistons, and anti-extrusions rings. The ruler in the foreground is in cm units.

The useful pressure of the cell can be estimated by looking at the stresses given by the Lamé equations (Eq. 10 through Eq. 12) and using the von Mises equivalent stress as the failure criterion (Eq. 13). For example assuming the yield stress of the maraging steel body is ~330,000 psi (2.27 GPa) the onset of yield occurs at a pressure of 183,169 psi (1.26 GPa). Figure 23 shows the stress distribution in the cell at the onset of yield prior to plastic deformation. However, the burst pressure is greater than the onset of yield and can be estimated by the Faupel equations:

$$p_{cr_lower} = 2 \frac{\sigma_y}{\sqrt{3}} \ln \bar{D} \quad \text{Eq. 14}$$

$$p_{cr_upper} = 2 \frac{\sigma_y}{\sqrt{3}} \ln \bar{D} \left[2 - \frac{\sigma_y}{\sigma_u} \right] \quad \text{Eq. 15}$$

where ' σ_y ' is the yield strength of the maraging alloy (~330,000 psi), ' σ_u ' the ultimate strength (estimated to be ~340,000 psi [27] [28]), and ' $\bar{D}$ ' the ratio of outer to inner diameter (~5.09). With these numbers we find the lower bound burst pressure to be ~620,076 psi (~4.28 GPa), and the upper bound burst pressure to be ~638,313 psi (4.40 GPa). [29]

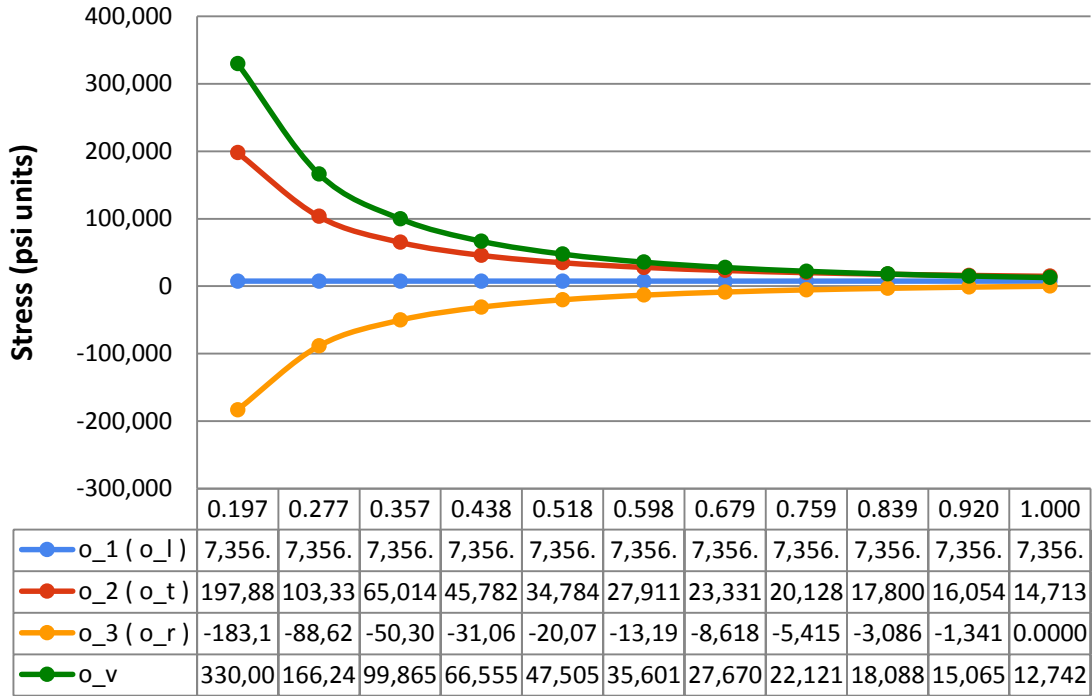


Figure 23 – Stress distribution in the compaction cell prior to the onset of yield. The bottom axis top row is the radial distance from the axis of the cell (inch units), while the stress components are given in psi units.

This indicates that the cell can likely be used to compact powders at 2-3 GPa, given that standard safety precautions are undertaken to shield the user in the event of rupture. While the stored energy is small since only solid samples are used, the strain energy in the cell wall can still lead to fragments posing a hazard if unshielded.

3.4 Cell Testing

Before testing the cell in the high temperature, high pressure environment created by the SPS equipment, the cell was tested to full pressure at room temperature. High purity alumina powder was placed in a graphite capsule and loaded in the SPS cell, and the entire SPS cell was then placed in a hydraulic press located at ORNL. The NiCrAl electrode has a diameter of 0.491 inch

(~12.47 mm), which with an applied pressure of 1 GPa will correspond to a force of 27,462 lb or ~122 kN. The press was programmed to quickly ramp up to 30,000 lbf (~133 kN). The graphite capsule was extracted from the cell and the alumina powder compaction removed, as shown in Figure 24.



Figure 24 - The extracted graphite capsule and alumina powder compaction following the room temperature pressure test.

While the pressure should be fairly isostatic due to the low friction of the graphite and small volume, it was attempted to estimate the pressure by correlating the final compaction density of the alumina with the applied pressure. This was not so straightforward since older references would build semi-empirical models based on the compaction of macroscopic particles. For example Cooper and Eaton suggested the following correlation

$$V^* = \frac{V_0 - V}{V_0 - V_\infty} = a_1 e^{-(k_1/P)} + a_2 e^{-(k_2/P)} \quad \text{Eq. 16}$$

where ' V^* ' is the fractional volume compaction, ' V_0 ' is the initial fractional volume, ' a_1 ' and ' a_2 ' dimensionless constants, ' k_1 ' and ' k_2 ' constants with pressure units, and ' P ' the applied pressure. The values for the coefficients in Eq. 16 can be found in Table 6. [30]

Table 6 - Coefficient values for use in Eq. 16. From ref. [30].

Material	k_1 (psi)	k_2 (psi)	a_1	$a_1 + a_2$
Alumina	3,100	50,000	0.50	0.85
Silica	2,400	54,000	0.60	0.85
Magnesia	2,400	49,000	0.65	0.90
Calcite	1,450	42,000	0.68	1.00

The measured density of the sample was found to be ~47% that of fully densified alumina, and Eq. 16 suggests the density should be ~74% for an applied pressure of 1 GPa – a significant discrepancy. Reference [5] however suggests that if nanopowders are being compacted much of the energy is spent breaking down agglomerates. A Sherrer analysis performed on the original alumina powder sample used in the SPS cell indicated grain sizes on the order of 10 nm, implying that such effects are present. Reference [5] experimentally investigated the compaction of alumina nanopowders of various grain sizes including 66 nm particles and 13 nm particles, finding compaction at 1 GPa to be ~60% and ~45% respectively. While the reference points out that particle size distribution is also important to take into account, the results agree with the densities found in our compaction (although this is by no means a straightforward or exact method of pressure verification).



Figure 25 – The high pressure SPS cell undergoing testing at Clemson. In (A) the cell is visible before raising the vacuum chamber, and (B) the cell is enclosed and the vacuum pumped down.

With the cell shown to successfully handle the full design pressure without failure, the entire setup was brought to Clemson University (Clemson, South

Carolina) for testing in SPS equipment. The equipment available at the university could only reach a maximum force of 10 kN, corresponding to a pressure of only ~81.9 MPa. This pressure was combined with a current of ~600 A, which in similar cells led to a temperature well in excess of 1,000 °C on the sample. One disadvantage of the present cell design is the difficulty in measuring temperature either by probe or pyrometry – the temperature could only be estimated based on previous experiments with current running through a graphite die. LaPO_4 powder was pressed at 8 kN (corresponding to a pressure of ~65.5 MPa) with 700 A of current for ten minutes, yielding a final density of 4.1 g/cm³ (~81% the theoretical density of 5.067 g/cm³ [31]). Figure 26 shows the high pressure cell in use within the vacuum chamber of the SPS equipment at Clemson. The NiCrAl electrode can be seen incandescent about the titanium binding ring. After several runs the cell was disassembled and inspected. No damage was visible on the alumina core or NiCrAl electrodes.

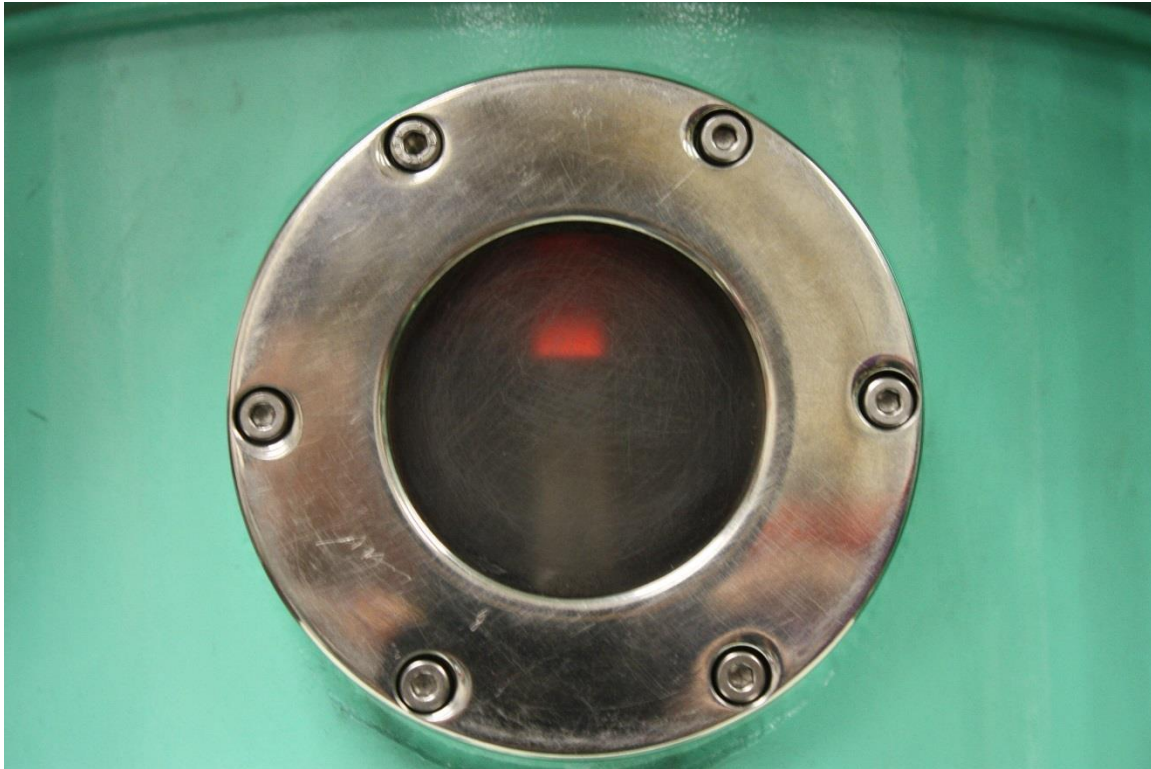


Figure 26 - The cell in use within the SPS vacuum chamber. The NiCrAl electrode can be seen incandescent above the titanium ring.

Chapter 4: Discussion and Future Work

While the preliminary testing of the cell described in this report was successful, considerably more work remains to be undertaken. More testing is required for both the NiCrAl alloy and the high pressure cell to determine if this design is an improvement over existing high pressure cells. For the NiCrAl alloy room temperature and elevated temperature compression data is needed to better understand the strength of the electrodes. Microscopy and phase data with electron microscopy will help to understand why the NiCrAl alloy is as strong as it is, and if Ni_3Al phases present. The high pressure cell needs to be brought to the full design pressure once equipment at ORNL is available for use. Nevertheless the cell has shown to be useful with current SPS setups and looks to be fully reusable with typical pressures and temperatures.

The cell was designed to be a simple and (relatively) cheap prototype to test the general concept of a swaged-core arrangement. With this shown to work, many improvements could be made. For example, the alumina core could be replaced with a stronger ceramic such as hot isostatically pressed (HIPped) Yttria Partially Stabilized Zirconia (YTZP) with a compressive strength of ~ 2.5 GPa and flexural strength of ~ 1.72 GPa (compared to Alumina's 2.1 GPa and 0.35 GPa respectively). The thermal conductivity is also $\sim 6\%$ that of alumina, although this could deleterious for thermal shock resistance.

While titanium was chosen for its low thermal expansion (less effect on the shrink fit stresses due to expansion) combined with high strength, it was noted that the operating temperature might be high enough to reduce the residual stresses in the cell. Water cooling will almost certainly be necessary in a future version. Figure 27 shows how such a cell might look. Water cooling channels can be added to an additional outer metal layer (or directly to the titanium binding ring - or other alloy). Multiple swaged layers could be used consisting of different alloys.

It might be possible to remove the graphite capsule from the cell design by coating the inner surface of the ceramic core with a sacrificial conductive layer such as graphite. This would help to reduce the cost of operation and help to accommodate samples of varying thicknesses.

The cell design described here suffers from the inability to measure the temperature at the sample. With a graphite crucible this is commonly done by drilling a small hole through the graphite close to the sample volume and inserting a standard thermocouple. With this design, even if a hole could be made through the metal binding ring and hard ceramic, it would almost certainly lead to failure during operation. It might be possible to fabricate an electrode with

a small hole through the center to facilitate the use of pyrometry to directly measure the temperature of the sample volume. Both on-going testing of the existing cell and the designing of a more advanced cell employing these concepts is planned.

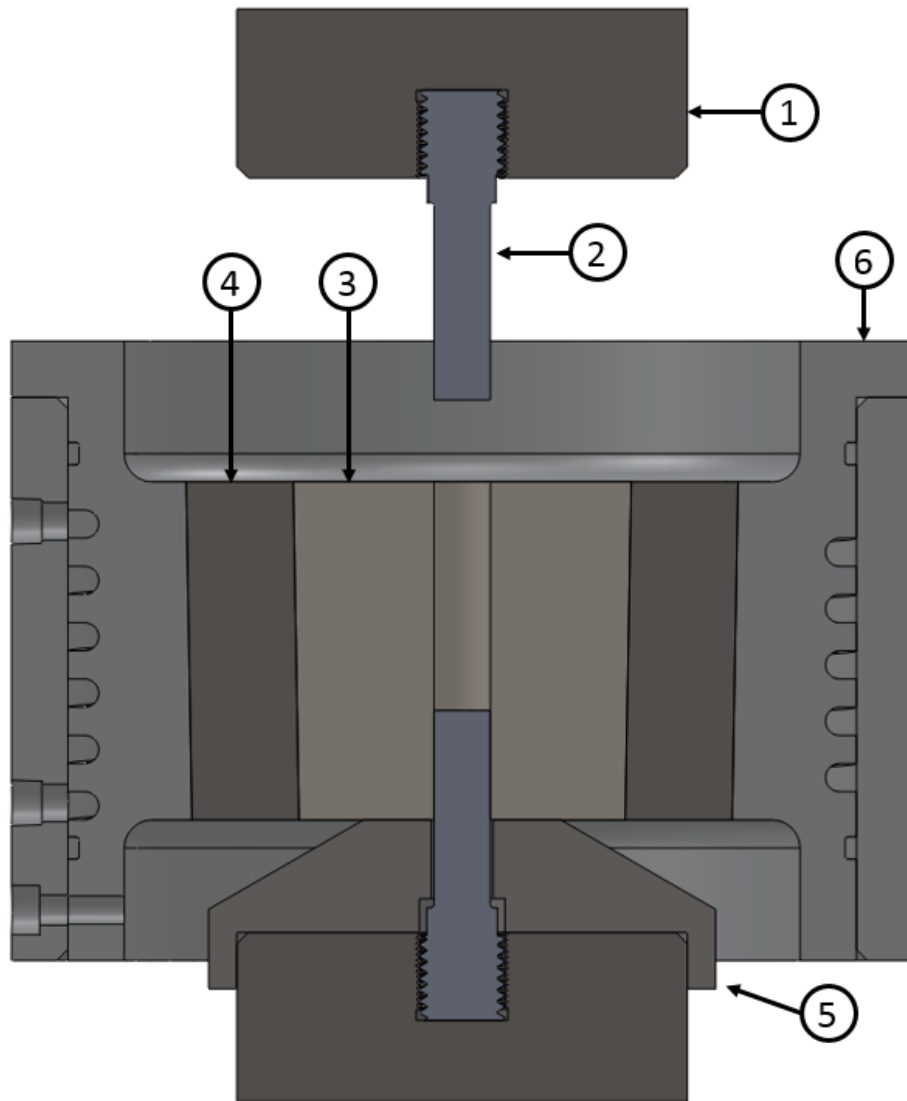


Figure 27 – A concept of a new high pressure SPS cell. Labels: (1) Electrode piston, (2) NiCrAl electrode, (3) YTZP core, (4) titanium binding ring, (5) bottom spacer, (6) press-fit water cooled jacket.

References

- [1] M. B. Waldron and B. L. Daniell, Sintering, Hayden & Son Ltd, 1978.
- [2] R. M. German, "History of Sintering: Empirical Phase," *Powder Metallurgy*, vol. 56, no. 2, pp. 117-123, 2013.
- [3] I.-H. Oh, N. Nomura and S. Hanada, "Microstructures and Mechanical Properties of Porous Titanium Compacts Prepared by Powder Sintering," *Materials Transactions*, vol. 43, no. 3, pp. 443-446, 2002.
- [4] G. M. Fryer, "The Pressure-Sintering of Alumina," *Transactions of the British Ceramic Society*, vol. 68, pp. 185-189, 1969.
- [5] B. P. Saha, V. Kumar, S. V. Joshi, A. Balakrishnan and C. L. Martin, "Investigation of compaction behavior of alumina nano powder," *Powder Technology*, vol. 224, pp. 90-95, 2012.
- [6] G. Dieter, Mechanical Metallurgy, McGraw-Hill, 1986.
- [7] W. Smith and J. Hashemi, Foundations of Materials Science and Engineering, 4th ed., 2006: McGraw-Hill, 2006.
- [8] N. Sata, K. Eberman, K. Eberl and J. Maier, "Mesoscopic fast ion conduction in nanometre-scale planar heterostructures," *Nature*, vol. 408, pp. 946-949, 2000.
- [9] O. Guillon, J. Gonzalez-Julian, B. Dargatz, T. Kessel, G. Schiernin, J. Räthel and M. Herrmann, "Field-Assisted Sintering Technology/Spark Plasma Sintering: Mechanisms, Materials, and Technology Developments," *Advanced Engineering Materials*, vol. 16, no. 7, pp. 830-849, 2014.
- [10] R. M. Hazen, The Diamond Makers, Cambridge University Press, 1999.
- [11] M. Tokita, "Mechanism of Spark Plasma Sintering," Sumitomo Coal Mining Company, Ltd., Kanagawa, Japan, 1993.
- [12] F. Maglia, I. G. Tredici and U. Anselmi-Tamburini, "Densification and Properties of Bulk Nanocrystalline Functional Ceramics with Grain Size below 50 nm," *Journal of the European Ceramic Society*, vol. 33, pp. 1045-1066, 2013.
- [13] Z. A. Munir, U. Anselmi-Tamburini and M. Ohyanagi, "The effect of electric field and pressure on the synthesis and consolidation of materials: A review of the spark plasma sintering method," *Journal of Materials Science*, vol. 41, no. 3, pp. 763-777, 2006.
- [14] U. Anselmi-Tamburini, J. E. Garay and Z. A. Munir, "Fast low-temperature consolidation of bulk nanometric ceramic materials," *Scripta Materialia*, vol. 54, pp. 823-828, 2006.
- [15] S. Klotz, Techniques in High Pressure Neutron Scattering, CRC Press, 2012.
- [16] I. R. Walker, "Considerations on the Selection of Alloys for use in Pressure Cells at Low Temperatures," *Cryogenics*, vol. 45, pp. 87-108, 2005.
- [17] I. R. Walker, "Nonmagnetic Piston–Cylinder Pressure Cell for use at 35 kbar and above," *Review of Scientific Instruments*, vol. 70, no. 8, pp. 3402-3412,

1999.

- [18] Y. Uwatoko, S. Todo, K. Ueda, A. Uchida, M. Kosaka, N. Mori and T. Matsumoto, "Material Properties of Ni-Cr-Al Alloy and Design of a 4 GPa Class Non-Magnetic High-Pressure Cell," *Journal of Physics: Condensed Matter*, vol. 14, pp. 11291-11296, 2002.
- [19] V. K. Sikka, "Intermetallic-Based High-Temperature Materials," Oak Ridge National Laboratory (ORNL), Oak Ridge, TN, USA, 1999.
- [20] C. T. Liu, C. L. White and J. A. Horton, "Effect of Boron on Grain-Boundaries in Ni₃Al," *Acta Metallurgica*, vol. 33, no. 2, pp. 213-229, 1985.
- [21] ASTM International, "E831-12 - Standard Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis," ASTM, 2012.
- [22] R. Vepa, Dynamics of Smart Structures, West Sussex, UK: John Wiley & Sons Ltd, 2010.
- [23] T. Lay and T. C. Wallace, Modern Global Seismology, London, UK: Academic Press, 1995.
- [24] H. T. Hall, "Ultra-High-Pressure, High-Temperature Apparatus: the "Belt"," *Review of Scientific Instruments*, vol. 31, no. 2, pp. 125-131, 1960.
- [25] J. E. Shigley, Mechanical Engineering Design, McGraw-Hill, Inc., 1977.
- [26] D. M. Fryer and J. F. Harvey, High Pressure Vessels, Springer, 1998.
- [27] F. R. Decker, Source Book on Maraging Steels, American Society for Metals (ASM), 1979.
- [28] A. A. Iannelli, "Processing of 18% Ni Maraging Steel (350 Grade)," Army Materials and Mechanics Research Center, Watertown, Massachusetts, 1972.
- [29] C.-x. Zheng and S.-h. Lei, "Research on Bursting Pressure Formula of Mild Steel Pressure Vessel," *Journal of Zhejiang University SCIENCE A*, vol. 7, pp. 277-281, 2006.
- [30] A. R. Cooper and L. E. Eaton, "Compaction Behavior of Several Ceramic Powders," *Journal of the American Ceramic Society*, vol. 45, no. 3, pp. 97-101, 1962.
- [31] D. Bregiroux, S. Lucas, E. Champion, F. Audubert and D. Bernache-Assollant, "Sintering and Microstructure of Rare Earth Phosphate Ceramics REPO₄ with RE = La, Ce or Y," *Journal of the European Ceramic Society*, vol. 26, pp. 279-287, 2006.
- [32] D. M. Hulbert, A. Anders, D. V. Dudina, J. Andersson, D. Jiang, C. Unuvar, U. Anselmi-Tamburini, E. J. Lavernia and A. K. Mukherjee, "The Absence of Plasma in "Spark Plasma Sintering"," *Journal of Applied Physics*, vol. 104, p. 033305, 2008.

Appendix

NOTES:

1. SPECIMEN DIMENSIONS PER ASTM E831-12 : STANDARD TEST METHOD FOR LINEAR THERMAL EXPANSION OF SOLID MATERIALS BY THERMOMECHANICAL ANALYSIS.
2. MATERIAL HARDNESS APPROXIMATELY 60 HRC. MATERIAL MUST BE GROUND OR MACHINED WITH ELECTRICAL DISCHARGE MACHINING.

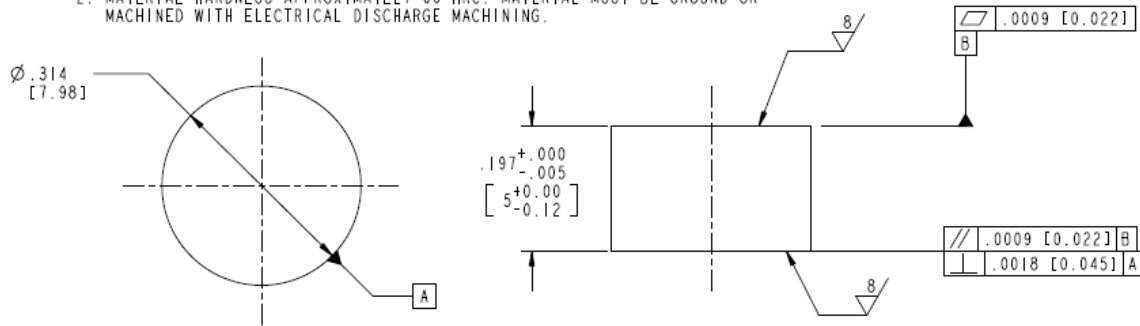


Figure 29 - The specimen used for thermal expansion measurements. Units are dual dimensioned (dimensions in brackets are mm units).

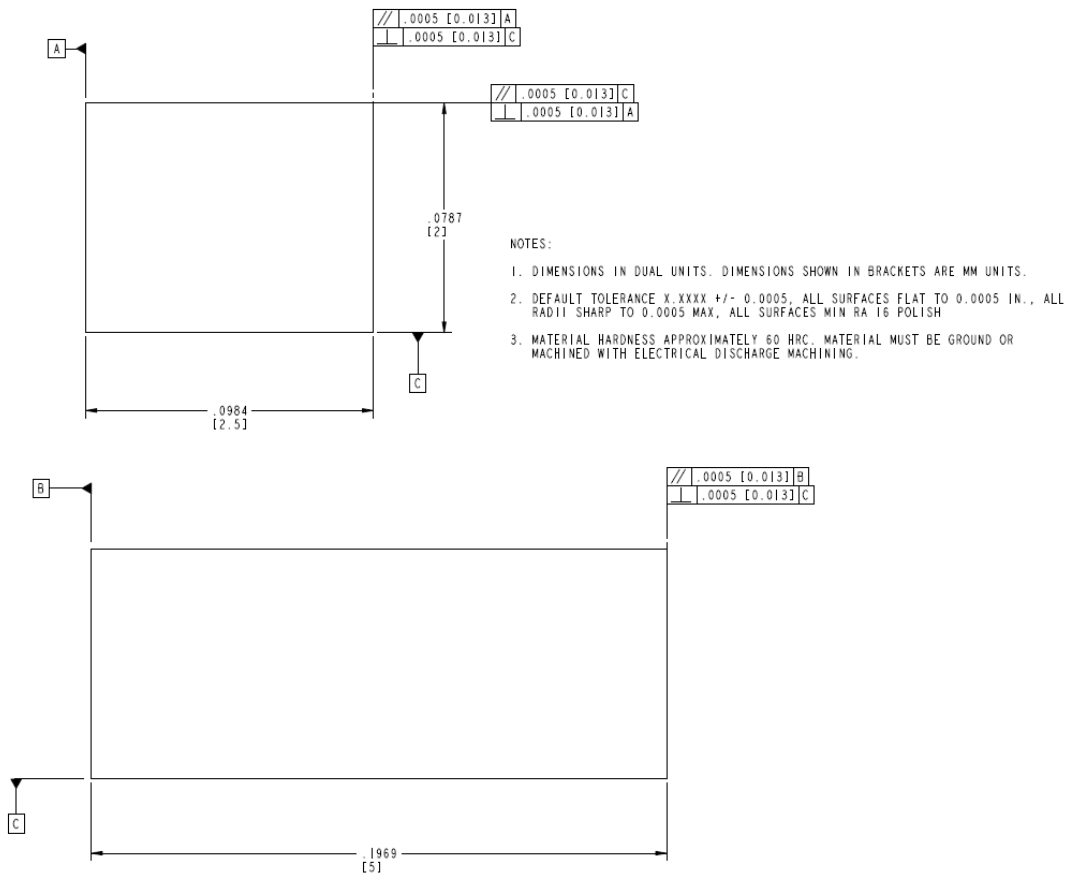


Figure 30 - The specimen used for Resonant Ultrasound Spectroscopy (RUS) measurements. Units are dual dimensioned (dimensions in brackets are mm units).

Appendix B: Python Code for Calculating Press Fit Stresses

The code below calculates the residual stresses in a press fit. By setting the “pressure_inner” to a nonzero value the code can also calculate the operating stresses. Values are compared to FEM results imported from a text file and graphed.

```
# -*- coding: utf-8 -*-
"""
Created on Fri May 9 20:00:45 2014
@author: Justin Carmichael
"""

from __future__ import division #force real results
from pylab import *
import csv

"""
INPUT PARAMETERS
"""

# global a, b, c, delta, E
a = 0.5/2
b = 2/2 # + delta...
c = 5/2
delta = 0.008

pressure_inner = 0 #psi
pressure_outer = 0 #psi

E_i = 44000000
v_i = 0.21
E_o = 16500000
v_o = 0.33

sig_o = c/b
C_o = ((sig_o**2)+1)/((sig_o**2)-1)
K_o = (1/E_o)*(C_o + v_o)

sig_i = b/a
C_i = ((sig_i**2)+1)/((sig_i**2)-1)
K_i = (1/E_i)*(C_i - v_i)

p_c = (1/(K_o + K_i))*(delta/b)

x_points = 500 # Number of points between r_i to r_o to calculate.

def txt_data_open(file_name, columns):
```

```

line_count = 0
data = []
for line in open(file_name):
    items = line.rstrip("\r\n").split("\t")
    items = [item.strip() for item in items]
    line_count += 1
    data.append(items)
M = zeros((line_count,columns+4) )
for h in range(M.shape[0]):
    for j in range(columns):
        num = h-1
        M[(h),j] = data[h][j]
return M

U = txt_data_open("NewResults/no_applied_pressure_proc.txt",5)
for h in range(U.shape[0]):
    # shift r over by r_i, calculate FEM von Mises stresses, then convert to ksi
    U[h,0] = U[h,0] + a
    U[h,4] = (((U[h,1]-U[h,2])**2 + (U[h,2]-U[h,3])**2 + (U[h,1]-U[h,3])**2)/2)**(1/2)
    # U[h,1] = U[h,1]*(10**2)
    # U[h,2] = U[h,2]*(10**2)
    # U[h,3] = U[h,3]*(10**2)
    # U[h,4] = U[h,4]*(10**2)

```

```

.....
PROCESSING : STRESS DUE TO PRESS FIT ONLY
.....

```

```

def stress_pressure(a, b, c, delta, points):
    M = zeros(((points+1),5) )
    for h in range(M.shape[0]):
        # points across the wall thickness
        M[h,0] = a+((c-a)/points)*(h)
        if M[h,0] <= b:
            r_i = a
            r_o = b
            p_o = p_c
            p_i = 0
            # longitudinal stress (assume open end, so zero...)
            M[h,1] = 0
            # tangential stress
            M[h,2] = ((p_i*(r_i**2))-(p_o*(r_o**2))-(((r_i**2)*(r_o**2))*(p_o-p_i)/(M[h,0]**2)))/((r_o**2)-(r_i**2))
            # radial stress
            M[h,3] = ((p_i*(r_i**2))-(p_o*(r_o**2))+(((r_i**2)*(r_o**2))*(p_o-p_i)/(M[h,0]**2)))/((r_o**2)-(r_i**2))
            # von Mises stress
            M[h,4] = (((M[h,1]-M[h,2])**2 + (M[h,2]-M[h,3])**2 + (M[h,1]-M[h,3])**2)/2)**(1/2)
        if M[h,0] > b:
            r_i = b

```

```

r_o = c
p_o = 0
p_i = p_c
# longitudinal stress (assume open end, so zero...)
M[h,1] = 0
# tangential stress
M[h,2] = ((p_i*(r_i**2))-(p_o*(r_o**2))-(((r_i**2)*(r_o**2))*(p_o-p_i)/(M[h,0]**2)))/((r_o**2)-(r_i**2))
# radial stress
M[h,3] = ((p_i*(r_i**2))-(p_o*(r_o**2))+(((r_i**2)*(r_o**2))*(p_o-p_i)/(M[h,0]**2)))/((r_o**2)-(r_i**2))
# von Mises stress
M[h,4] = (((M[h,1]-M[h,2])**2 + (M[h,2]-M[h,3])**2 + (M[h,1]-M[h,3])**2)/2)**(1/2)
return M

```

PROCESSING : STRESS DUE TO OPERATING PRESSURE ONLY

```
def lame_stresses(r_i, r_o, pressure_inner, pressure_outer, x_points):
```

```

    p_i = pressure_inner
    p_o = pressure_outer

    N = zeros(((x_points+1),5) )

    for h in range(N.shape[0]):

        # points across the wall thickness
        N[h,0] = r_i+((r_o-r_i)/x_points)*(h)
        # longitudinal stress:
        N[h,1] = 0
        # tangential stress
        N[h,2] = ((p_i*(r_i**2))-(p_o*(r_o**2))-(((r_i**2)*(r_o**2))*(p_o-p_i)/(N[h,0]**2)))/((r_o**2)-(r_i**2))
        # radial stress
        N[h,3] = ((p_i*(r_i**2))-(p_o*(r_o**2))+(((r_i**2)*(r_o**2))*(p_o-p_i)/(N[h,0]**2)))/((r_o**2)-(r_i**2))
        # von Mises stress
        N[h,4] = (((N[h,1]-N[h,2])**2 + (N[h,2]-N[h,3])**2 + (N[h,1]-N[h,3])**2)/2)**(1/2)
    return N

```

PLOT, REPORT

```

M = stress_pressure(a, b, c, delta, x_points)
N = lame_stresses(a, c, pressure_inner, pressure_outer, x_points)

```

```
O = zeros(((x_points+1),14) )
```

```
for h in range(O.shape[0]):
```

```
    O[h,0] = N[h,0]
```

```
    if O[h,0] <= b: # graphite section only
```

```
        # calculate longitudinal strain e_z from press fit only
```

```
        O[h,1] = (1/E_i)*(M[h,1] - (v_i*(M[h,2] + M[h,3])))
```

```
        # calculate longitudinal strain e_z from lame stresses only
```

```
        O[h,2] = (1/E_i)*(N[h,1] - (v_i*(N[h,2] + N[h,3])))
```

```
        # sum the two for total longitudinal strain
```

```
        O[h,3] = O[h,1] + O[h,2]
```

```
        # calculate radial strain e_y from press fit only
```

```
        O[h,4] = (1/E_i)*(M[h,3] - (v_i*(M[h,1] + M[h,2])))
```

```
        # calculate radial strain e_y from lame stresses only
```

```
        O[h,5] = (1/E_i)*(N[h,3] - (v_i*(N[h,1] + N[h,2])))
```

```
        # again, sum the two for total radial strain
```

```
        O[h,6] = O[h,4] + O[h,5]
```

```
        # calculate tangential strain e_x from press fit only
```

```
        O[h,7] = (1/E_i)*(M[h,2] - (v_i*(M[h,3] + M[h,1])))
```

```
        # calculate tangential strain e_x from lame stresses only
```

```
        O[h,8] = (1/E_i)*(N[h,2] - (v_i*(N[h,3] + N[h,1])))
```

```
        # again, sum the two for total tangential strain
```

```
        O[h,9] = O[h,7] + O[h,8]
```

```
        # from strains, calculate the longitudinal stress
```

```
        O[h,10] = O[h,3]*(E_i/(1+v_i)) + ((v_i*E_i)/((1+v_i)*(1-2*v_i)))*(O[h,3] + O[h,6] + O[h,9])
```

```
        # from strains, calculate the radial stress
```

```
        O[h,11] = O[h,6]*(E_i/(1+v_i)) + ((v_i*E_i)/((1+v_i)*(1-2*v_i)))*(O[h,3] + O[h,6] + O[h,9])
```

```
        # from strains, calculate the tangential stress
```

```
        O[h,12] = O[h,9]*(E_i/(1+v_i)) + ((v_i*E_i)/((1+v_i)*(1-2*v_i)))*(O[h,3] + O[h,6] + O[h,9])
```

```
    if M[h,0] > b: # inconel section only
```

```
        # calculate longitudinal strain e_z from press fit only
```

```
        O[h,1] = (1/E_o)*(M[h,1] - (v_o*(M[h,2] + M[h,3])))
```

```
        # calculate longitudinal strain e_z from lame stresses only
```

```
        O[h,2] = (1/E_o)*(N[h,1] - (v_o*(N[h,2] + N[h,3])))
```

```
        # sum the two for total longitudinal strain
```

```
        O[h,3] = O[h,1] + O[h,2]
```

```
        # calculate radial strain e_y from press fit only
```

```
        O[h,4] = (1/E_o)*(M[h,3] - (v_o*(M[h,1] + M[h,2])))
```

```
        # calculate radial strain e_y from lame stresses only
```

```
        O[h,5] = (1/E_o)*(N[h,3] - (v_o*(N[h,1] + N[h,2])))
```

```
        # again, sum the two for total radial strain
```

```
        O[h,6] = O[h,4] + O[h,5]
```

```
        # calculate tangential strain e_x from press fit only
```

```
        O[h,7] = (1/E_o)*(M[h,2] - (v_o*(M[h,3] + M[h,1])))
```

```
        # calculate tangential strain e_x from lame stresses only
```

```
        O[h,8] = (1/E_o)*(N[h,2] - (v_o*(N[h,3] + N[h,1])))
```

```
        # again, sum the two for total tangential strain
```

```
        O[h,9] = O[h,7] + O[h,8]
```

```
        # from strains, calculate the longitudinal stress
```

```
        O[h,10] = O[h,3]*(E_o/(1+v_o)) + ((v_o*E_o)/((1+v_o)*(1-2*v_o)))*(O[h,3] + O[h,6] + O[h,9])
```

```

# from strains, calculate the radial stress
O[h,11] = O[h,6]*(E_o/(1+v_o)) + ((v_i*E_i)/((1+v_i)*(1-2*v_i)))*(O[h,3] + O[h,6] + O[h,9])
# from strains, calculate the tangential stress
O[h,12] = O[h,9]*(E_o/(1+v_o)) + ((v_i*E_i)/((1+v_i)*(1-2*v_i)))*(O[h,3] + O[h,6] + O[h,9])
O[h,13] = (((O[h,10]-O[h,11])**2 + (O[h,11]-O[h,12])**2 + (O[h,10]-O[h,12])**2)/2)**(1/2)

graph = 1

if graph == 1:
    ax1 = subplot(111)
    p1, = ax1.plot(O[:,0], O[:,11], 'b--', label=r'$\sigma_r^R$ (Analytical)$')
    p2, = ax1.plot(O[:,0], O[:,12], 'r--', label=r'$\sigma_{\theta}^R$ (Analytical)$')
    p3, = ax1.plot(O[:,0], O[:,13], 'g--', label=r'$\sigma_{vm}^R$ (Analytical)$')

    p4, = ax1.plot(U[:,0], U[:,1], 'bs', label=r'$\sigma_r^R$ (FEM)$')
    p5, = ax1.plot(U[:,0], U[:,3], 'rs', label=r'$\sigma_{\theta}^R$ (FEM)$')
    p6, = ax1.plot(U[:,0], U[:,4], 'gs', label=r'$\sigma_{vm}^R$ (FEM)$')

    handles, labels = ax1.get_legend_handles_labels()
    ylabel("Stress (psi)")
    xlabel("Distance from axis (in.)")
    hl = zip(handles, labels)
    ax1.legend( loc='best', shadow=True, ncol=2)
    grid(True)

    show()

```

Appendix C: Press Fit Tooling

Below are the relevant engineering drawings used in the construction of press fitting of the cell. Unless otherwise specified dimensions are in inch units, details conform to ASME Y14.100, and tolerances are as follows:

- $X.XX \pm .01$
- $X.XXX \pm .005$
- $\pm .5^\circ$ (angular)

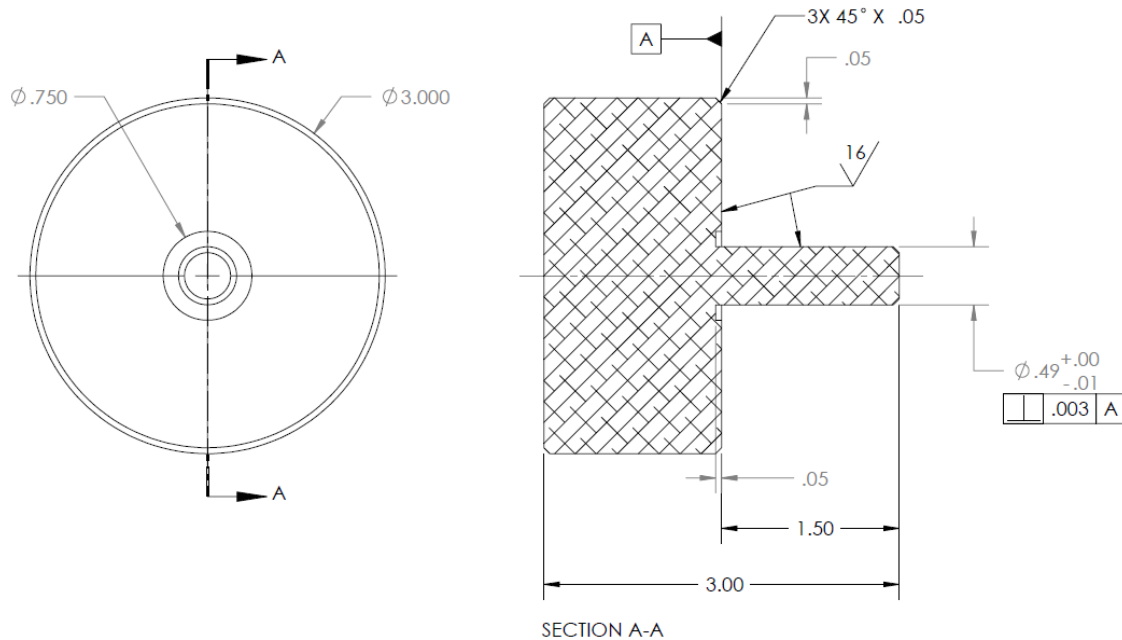


Figure 31 - The top tool used in the assembly of the press fit as shown in Figure 18 (inch units). The material is 6061-T6 aluminum alloy.

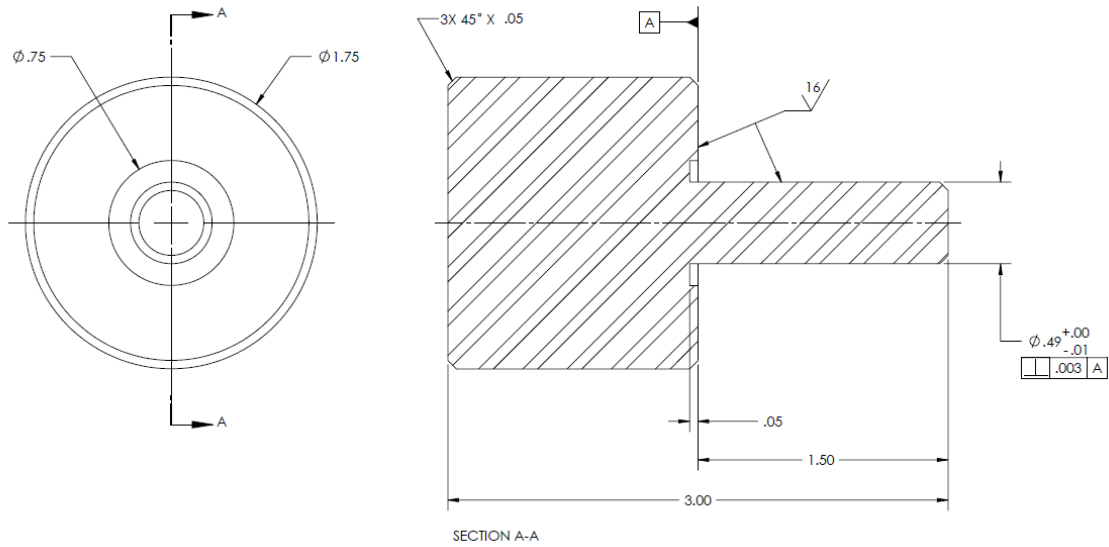


Figure 32 – A tool used for ejecting the alumina core in the event of a fracture (inch units). The material is 304 alloy stainless steel.

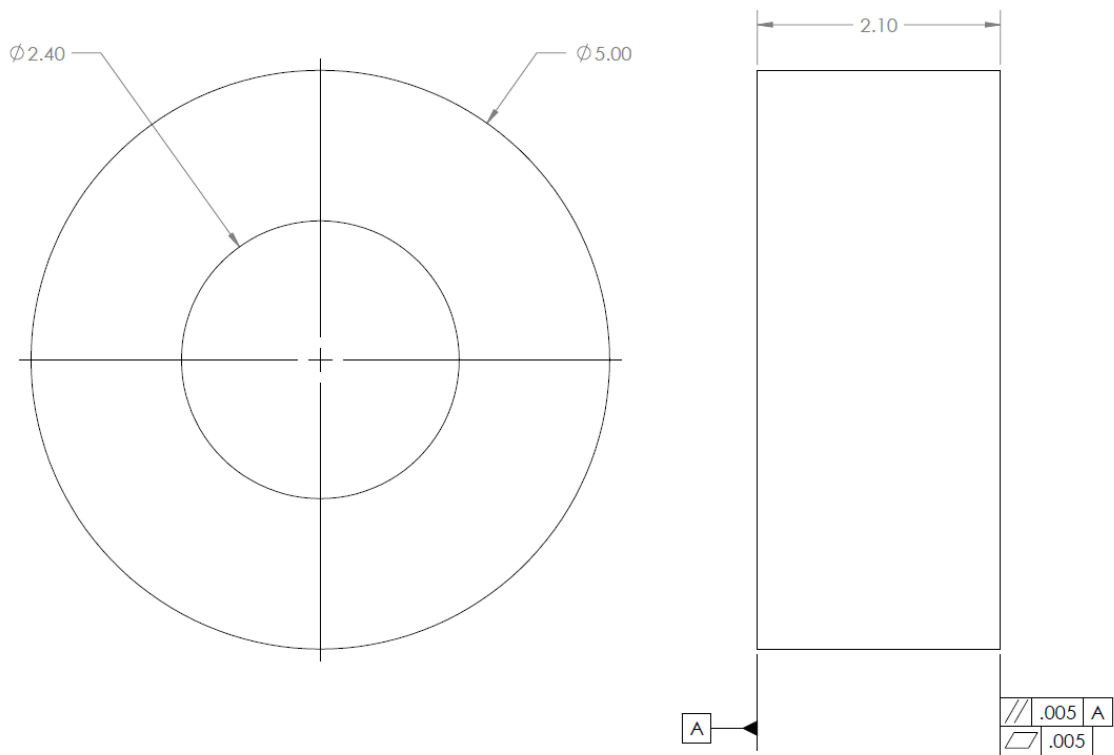
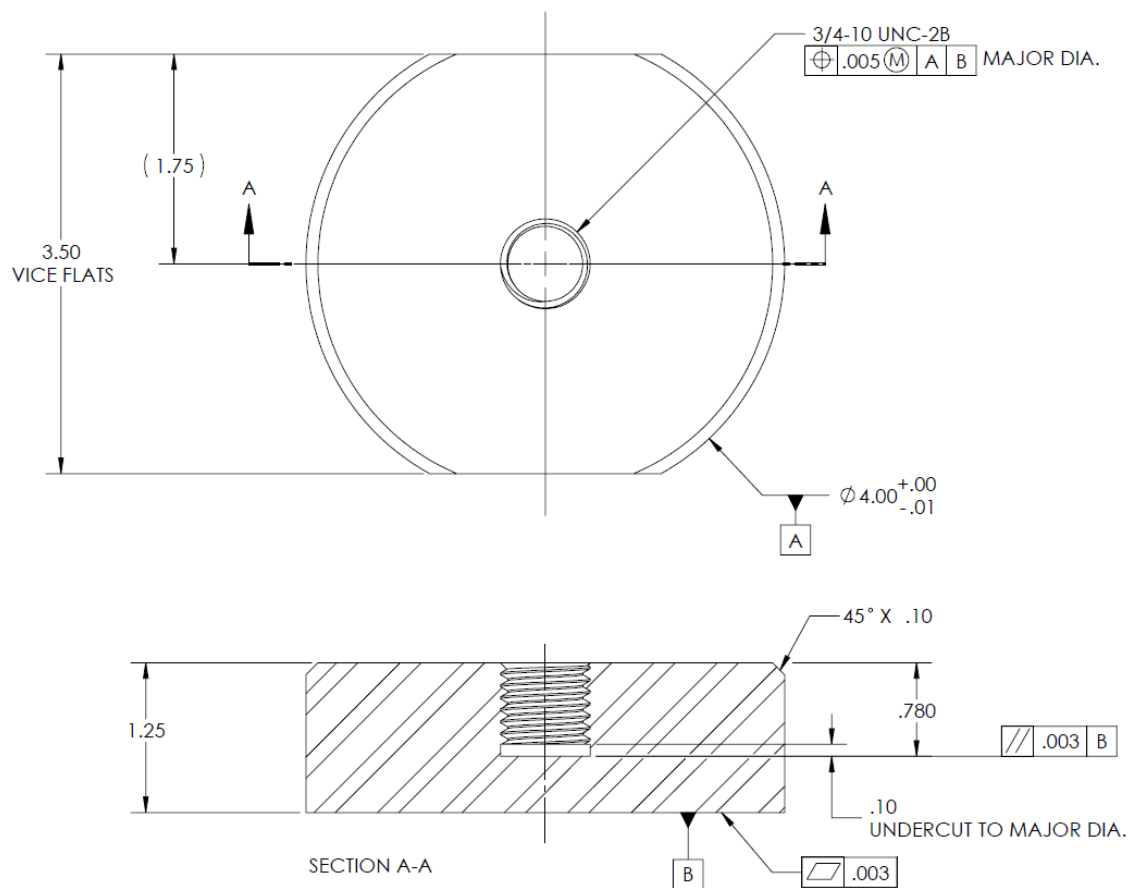
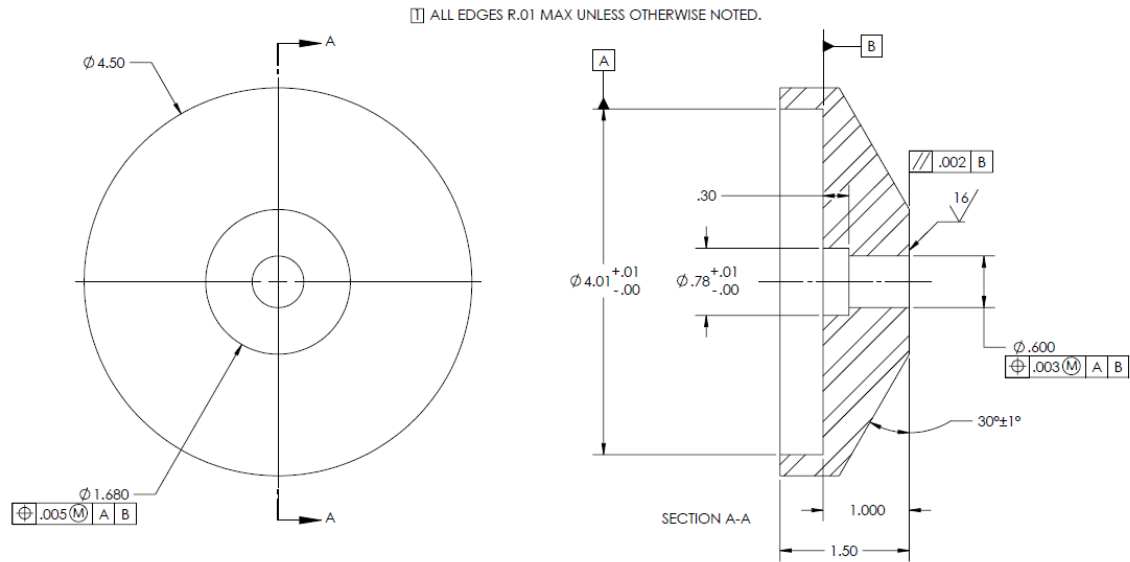


Figure 33 - The bottom tool used in the assembly of the press fit as shown in Figure 18 (inch units). The material is 6061-T6 aluminum alloy.



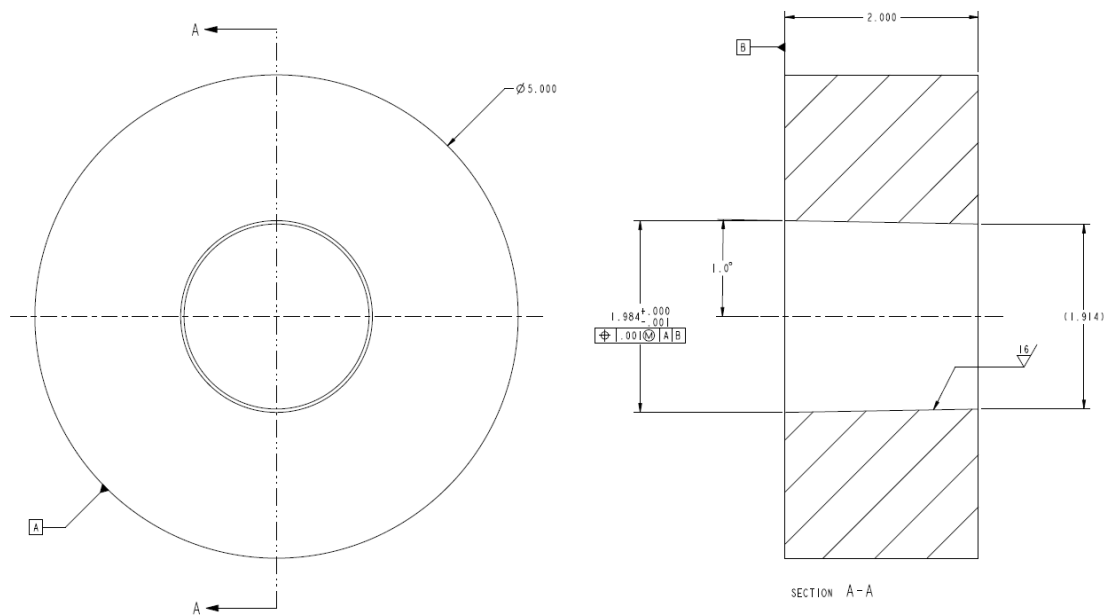


Figure 38 – The titanium binding ring (item 4 in Figure 14).

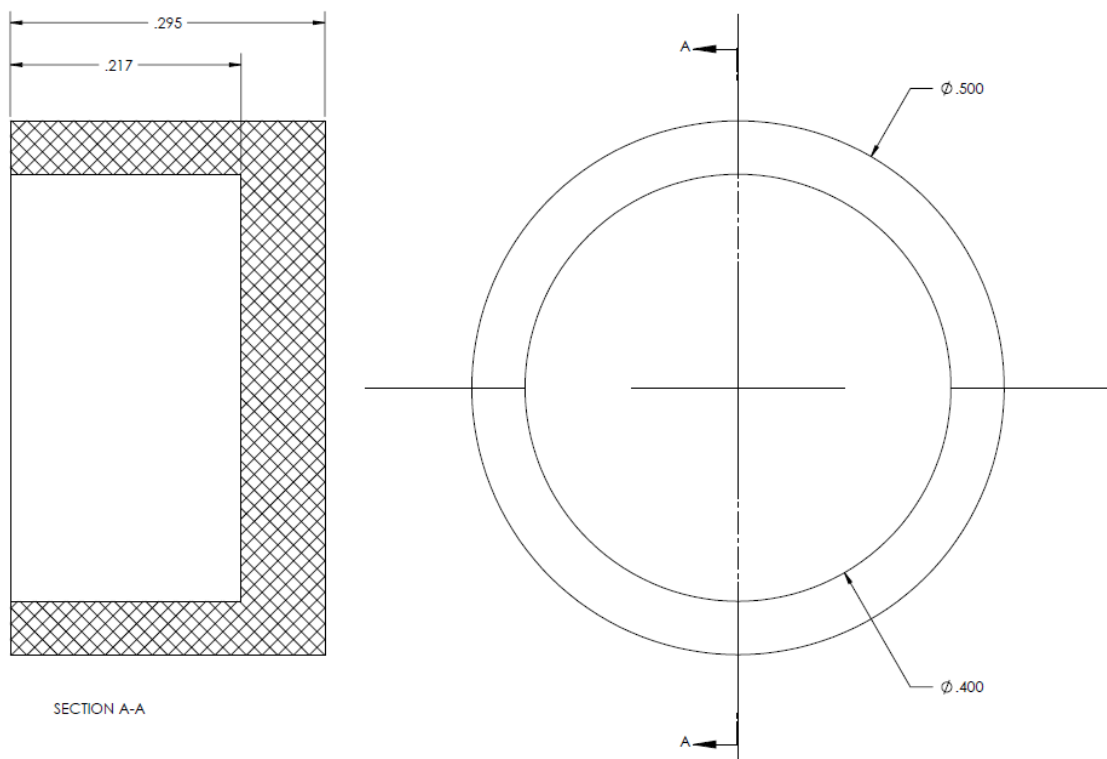


Figure 39 – The graphite capsule bottom (assembly item 6 in Figure 14).

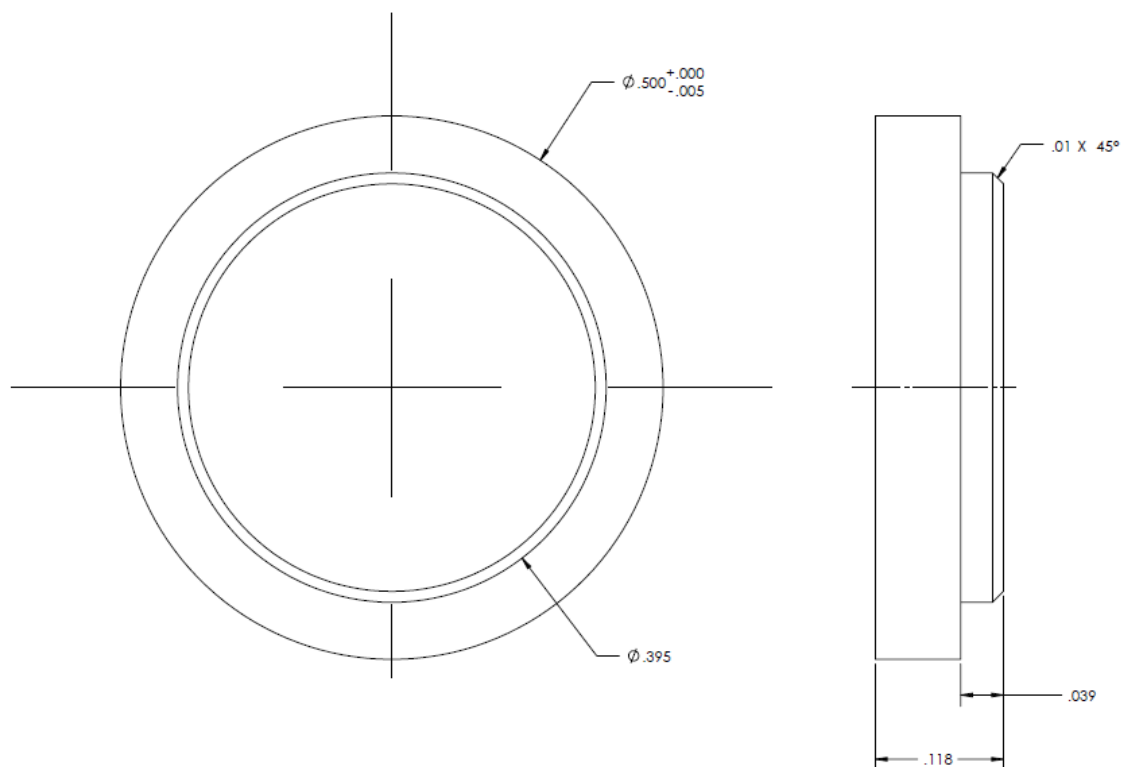


Figure 40 – The graphite capsule lid (assembly item 6 in Figure 14).

Appendix E: Compaction Cell Engineering Drawings

These drawings were used to fabricate the compaction cell. Unless otherwise specified dimensions are in inch units, details conform to ASME Y14.100 (tolerances: X.XX ± .01, X.XXX ± .005, ± .5° angular)

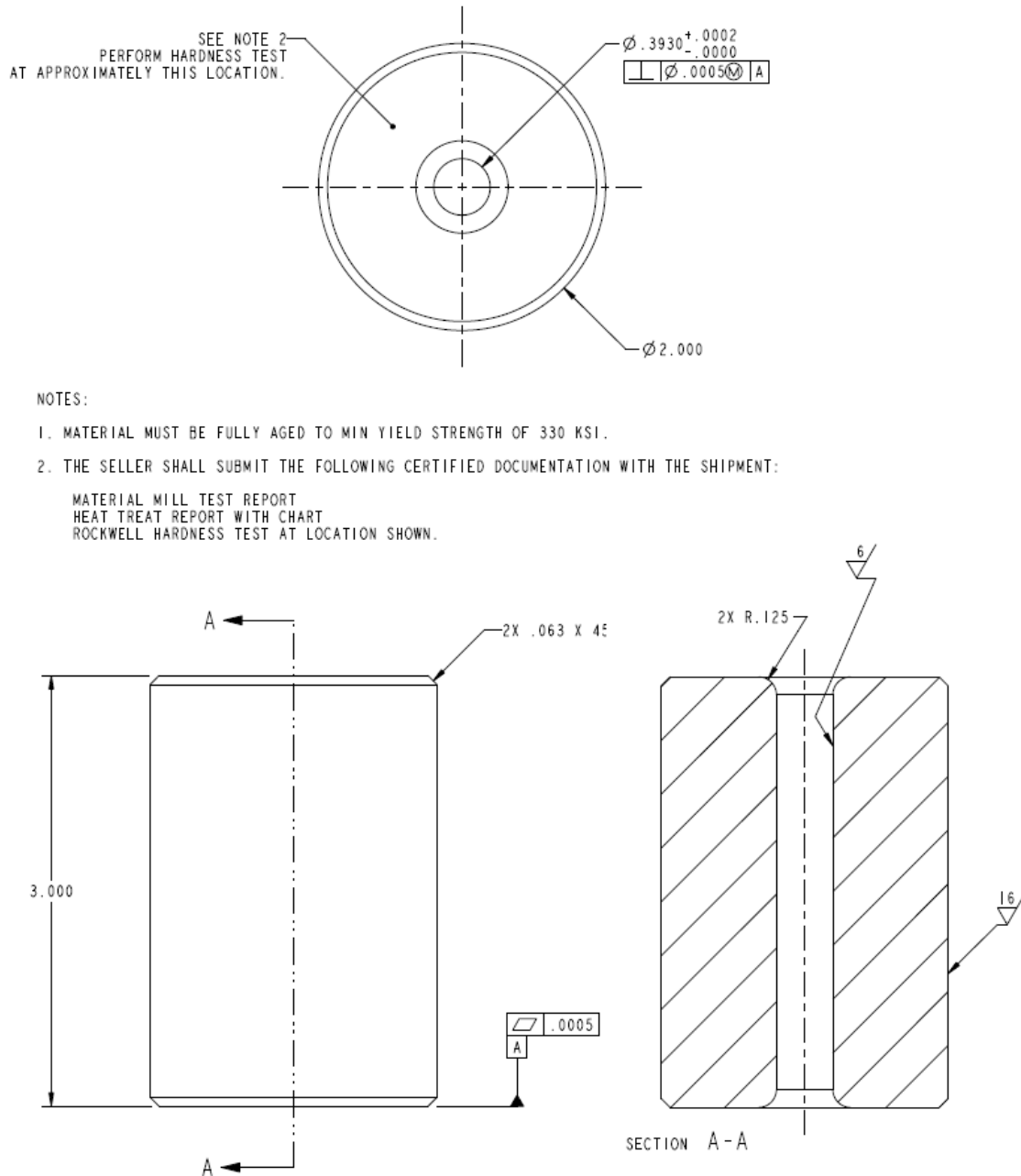


Figure 41 – The maraging steel body of the compaction cell (item 1 in Figure 21).

Figure 42 shows one of the tungsten carbide pistons. The other pistons are identical to the drawing, but with lengths of 1.875 inch (~47.6 mm) and 3.500 inch (88.9 mm, ejection rod).

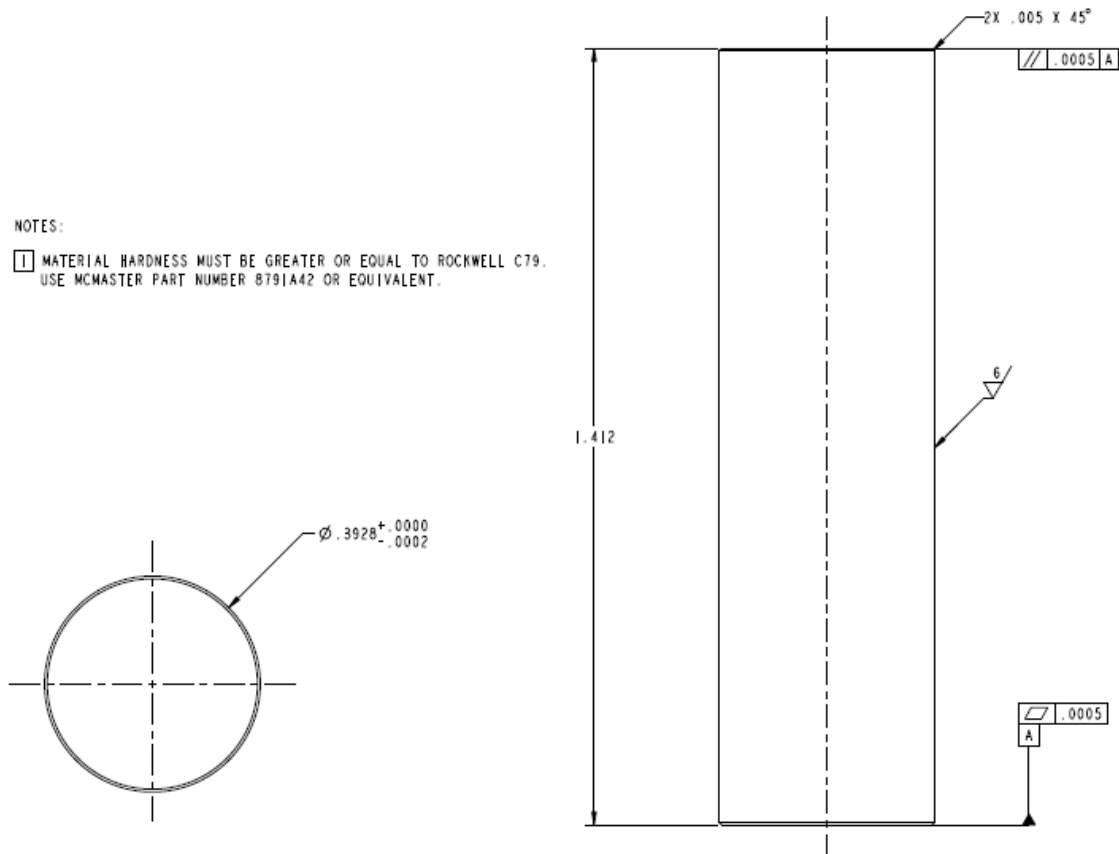


Figure 42 – The lower (fixed) tungsten carbide piston (item 2 in Figure 21).

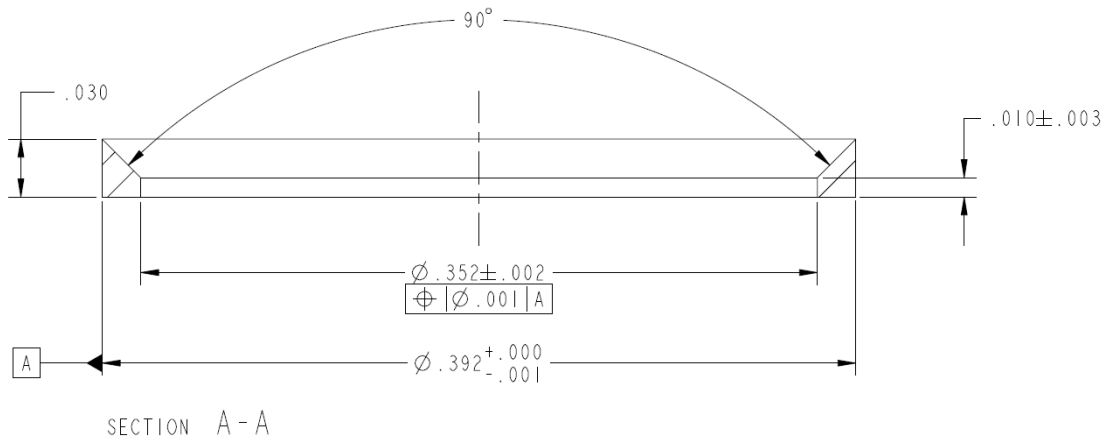


Figure 43 – The brass anti-extrusion ring (item 4 in Figure 21).

Vita

Justin Robert Carmichael grew up in Rindge, New Hampshire. He attended Worcester Polytechnic Institute (WPI) and received his Bachelor of Science Degree in Physics in 2008, and his Master of Science degree in Mechanical Engineering in 2009. As a student he was an intern at the Spallation Neutron Source (SNS) at Oak Ridge National Laboratory (ORNL), and assisted with the H⁻ ion source development. After completing his Master's degree he joined the SNS full time as a mechanical engineer in the Sample Environment group. After five years with this group he then moved to the ORNL Fusion Energy Division to perform work for the International Thermonuclear Experimental Reactor (ITER). After six years of working for ORNL Justin moved to Chicago with his wife Zijing, where Justin currently works for Fermi National Accelerator Laboratory (Fermilab).