

**Analysis Of Low-Temperature Indium Seals
For Hermetic Packaging Of Large-Area Photodetectors**

BY

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THESIS

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SUMMARY

The optimization of process parameters for sealing the sealing of large-area photodetector glass packages using low-temperature indium seals has been investigated in this report. Small scale 2.54×2.54 cm samples were used for experiments in order to gain an understanding of how the processing parameters will effect the seal integrity when scaled up to larger sizes such as 22×22 cm. First, substrates of B33 borosilicate glass were used for seal fabrication to validate that sealing indium to glass was indeed feasible. Second, seals were made using glass substrates which were coated in an 80 wt% Ni/20 wt % Cr alloy (Nichrome A) to investigate its merit as an intermediate bonding layer to glass. The hermeticity of the samples was characterized by using a helium leak detector. The samples were also characterized by means of a lap shear test to evaluate the effect the applied load, load duration, and fabrication temperature played on the robustness of the joint. Nichrome-coated seals were evaluated using Energy-dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS) to identify if any new phases were present as a result of sealing. It was established that both glass and 80Ni-20Cr are acceptable for making hermetic seals at room temperature using pure indium wire. Bonding strength can be enhanced by increasing the applied load and the load duration when sealing to either glass or NiCr. An increase in fabrication temperature was found to increase the strength of the seals made with glass, however, it had no effect on seals made with NiCr.

1. INTRODUCTION

There exists a desire to advance the state-of-the-art of photo-multipliers for applications in the fields of particle physics, astrophysics, nuclear sciences, and medical imaging. The advancement of such technology is being pioneered by the Large-Area Picosecond Photo-Detector (LAPPD) group, a joint effort of teams from the University of Chicago, Argonne National Laboratory, Fermilab, University of California-Berkley, and others. The overall goal of the research project is to develop a 22×22 cm, planar, photo-detector capable of mm-scale spatial resolution and psec-range time resolution. Applications for these photo-detectors which are currently being explored consist of:

- Large water Cherenkov detectors
- Nuclear non-proliferation and transportation security
- Compensating 'dual-light' high-granularity pseudo-digital calorimeters
- Direct Dark Matter searches with noble gases
- Positron Emission Tomography (PET) cameras

In order that these devices work as designed, all functioning components must be hermetically sealed inside of a non-conductive package such as glass. This has been accomplished in other, similar products by using low-temperature indium-based lead-free solders. [1-6] Indium-based solders are an attractive solution

because of their affinity to adhere to most metals and ceramics as well as remaining ductile yet robust, even at cryo-temperatures. [2, 7]

The purpose of this thesis is to optimize the processing parameters for hermetically sealing a 22×22 cm glass package using indium solder. Experimentation will be conducted using 2.54×2.54 cm B33 borosilicate glass parts which will act as scale replicas of the final product. Seals will be fabricated using a gasket of indium wire by means of thermocompression bonding. The processing parameters to be investigated are the load to be applied for bonding, the duration which the load should be sustained, and the temperature during fabrication. The addition of a coating layer on the glass substrate will also be investigated. Seals will be characterized by means of helium leak-checking for hermeticity and a lap shear test for robustness.

Following this introduction, Chapter 2 presents a survey of the literature pertaining to the methods of vacuum sealing using indium solders and the various substrate materials. Chapter 3 outlines the experimental plan and describes the equipment and methods used for fabrication. The results of experimentation and a discussion of the data are presented in Chapter 4. Finally, the conclusions and suggested processing parameters are compiled in Chapter 5.

2. LITERATURE REVIEW

The immediate purpose of this investigation is to further the research on photosensor seals at temperatures lower than 150 °C. This sealing entails the bonding of two pieces of borosilicate glass using a composite of metallic coatings and indium-based filler materials. This sealing must be hermetic and reliable for relatively long periods of time (around 10 years). This investigation explores the application of the main principles of indium sealing and thermocompression bonding techniques where an indium layer between two metallic coated layers on glass surfaces are the materials to be studied. The joining procedure to be utilized is based on current suggested practices considered from information in the open literature, from private communications with parties involved in this project, and our laboratory experience on bonding of dissimilar materials.

It appears there is a lack of consistent and detailed technical information on low temperature bonding of glasses in the published literature. The experimental results offered on the interactions between the different materials used in bonding two pieces of glasses appears to be deficient and do not directly address the situation of the photocathode. The purpose of this chapter is to investigate and interpret the available literature on related topics to develop a sound understand of the methods currently used in the art.

2.1 Methods for Using Indium as a Vacuum Seal

A number of methods for making indium seals have been found in a review of the literature. A report by A. W. Knudsen from 1952 stated that indium could be used as a gasket in high vacuum situations where gaskets made of certain materials such as neoprene could not be used. [1] Indium was suggested because of its ability to easily deform at room temperature and its capacity to stick to clean metal surfaces. Initially, gaskets of indium were cut from 0.178 mm sheet stock to a width of 1.6 mm. The joint would be made by tightening screws around the perimeter of the parts being joined causing indium to “flow” during compression. This was seen as sufficient for many laboratory applications where clamping forces could be used. In cases where more strength was desired, several gaskets 0.178 mm thick would be stacked on top of each other in order that more material would flow during compression. However, if the two parts needed to be separated, the gasket would have to be destroyed. [1]

In his investigation, Knudsen established that indium wire between 0.76 and 1.52 mm in diameter can be fashioned into a gasket for sealing. [1] Two ends of the indium wire were overlapped and then cold welded under an applied force, creating a leak tight seal. A schematic of this is shown in Figure 1. He also indicated that a continuous gasket could be made by cold welding two freshly cut ends of indium wire together in a butt or miter joint. The joint can be made complete with the use of



Figure 1 - Overlapping two ends of indium wire to make a complete gasket. [2]

localized heating and a flux. When compressing the wire between two flanges, the joint is said to be made when the indium is compressed to 15-25 percent of its original diameter. [2]

It is important to understand the difference between a gasket and a true seal. A true seal forms a chemical bond at the mating surfaces while a gasket forms only a vacuum-tight barrier. The chemical bond can be achieved in two ways: 1) by a reaction occurring between the metal gasket/solder and metal flange to produce an alloy layer or intermetallic compound, or 2) by means of an oxide bond. The term “oxide bond” is not clearly defined for these applications; however it is believed that this type of bond occurs due to the very rapid oxidation tendency of the soft metals when fresh, unoxidized surfaces of the metals come into contact with oxides such as glass and ceramics. [3]

2.2 Materials Considered in the Literature

One objective of this research is to select the most suitable materials for making a vacuum seal which is long-lasting, leak-tight, and robust. Two considerations that need to be taken into account are the gasket material and the surface material (coating) when dealing with nonmetallic substrates.

2.2.1 Gasket Materials

Pure indium has been used extensively over the past sixty years as a means for making high and ultra-high vacuum seals. Indium alloys such as In-Sn, In-Bi, In-Ag, and others have also been used where lower melting temperatures or higher strengths are needed.

2.2.1.1 Pure Indium

Indium's low melting temperature of 157°C makes it very useful for low temperature soldering and cold welding. Reed *et al.* have noted that at room temperature (295 K) indium is in close proximity to its melting temperature. ($T_m = 429$ K, $T_r/T_m = 0.69$) It is said that this is the primary reason for its softness at room temperature. The laws of recrystallization outlined by Mehl show that the high amounts of plastic deformation associated with thermocompression induce

tremendous internal energy in the indium which is the driving force for recrystallization. [8] Coupled with indium's relative closeness to its melting temperature at room temperature, recrystallization could happen very rapidly and may be a key mechanism for producing indium seals.

Similarly, it is important to understand the behavior of indium while undergoing plastic deformation. Flow stress is the instantaneous magnitude of stress needed to continue deforming a material at a certain strain rate. Reed et al. has reported the values of flow stress of indium at temperatures from 4 to 295 K. [7]. Temperature has an effect on the flow stress, decreasing with increasing temperature. At room temperature, Reed et al. reports that the flow stress of indium is 1.1 MPa. The thermal expansion of indium ranges between 42×10^{-6} [4] and 33×10^{-6} /°C [9] which is generally higher than desirable when joining dissimilar materials. However, indium has also been seen to have the innate ability to bond materials with vastly different thermal properties without failure during extreme thermal cycling. [4]

The growth rate of indium oxide (In_2O_3) is of particular interest to this research since the thickness of indium oxide on the indium surface has an impact on the strength of the vacuum seals. [9] It is necessary to know how long freshly cleaned indium can be exposed to air before the oxide layer becomes a dominant factor in the integrity of the seal. Figure 2 shows the relationship between temperature and indium oxide thickness after 2 hours of heating in air. Below indium's melting

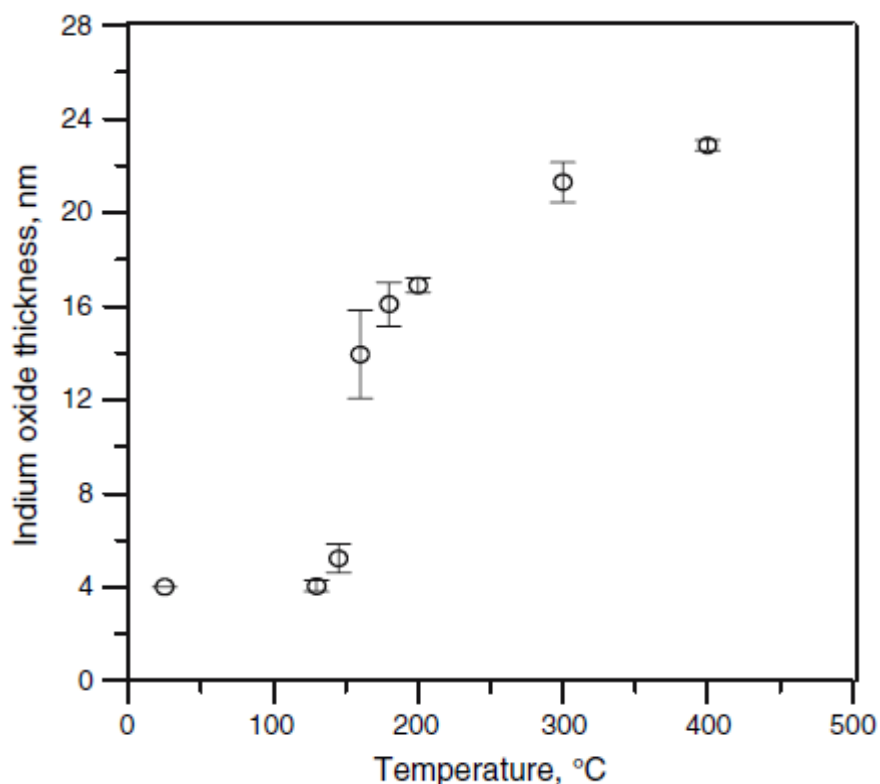


Figure 2 - Growth of indium oxide layer after 2 hours compared to heating temperature. [9]

temperature, an oxide layer will grow to about 5 nm (50 Å) after 2 hours. This is similar to Indium Corporations literature which states indium oxide is self-limiting at 80-100 Å at room temperature. [10] Faster oxide growth occurs at temperatures greater than its melting point up to more than 20 nm (200 Å). Similarly, it is suggested that after ridding the indium surface of its oxide, a new layer of oxide of about 30-40 Å thick will form immediately when exposed to the atmosphere. The rate which indium oxide forms could be important when discussing the possible mechanisms for bonding to ceramic surfaces.

The existence of indium oxide has a detrimental effect on the strength of indium bonds. [9] The correlation between bond strength and the thickness of indium oxide on the surface of the indium is shown in Figure 3. It is clear that with increased oxide thickness, the bonding strength decreases. This notion seems intuitive since a thicker oxide layer means there will likely be larger islands of indium oxide at the interface, restricting the surface area of fresh indium to make contact with the substrate. Greer *et al.* has shown a viable option for oxide removal is with the use of an argon-hydrogen plasma. [11]

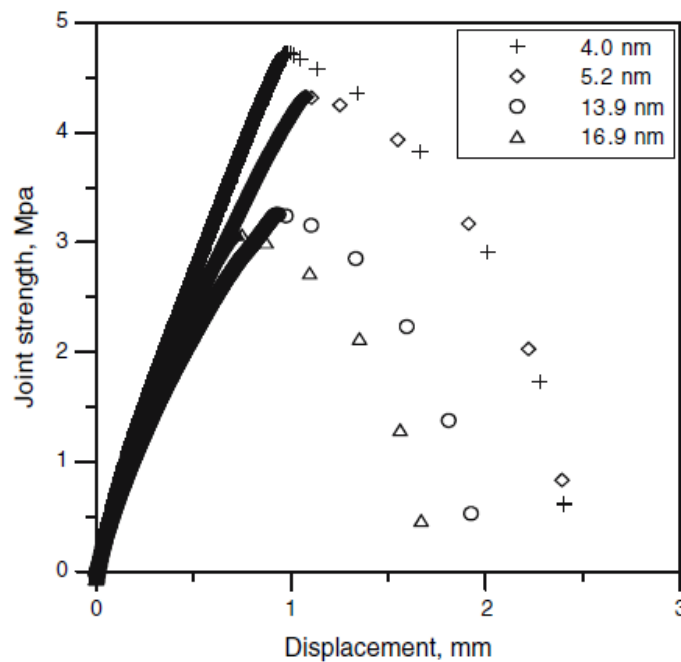


Figure 3 - Joint strength of indium seals with various initial indium oxide thicknesses. Substrates: Au/Cr/glass. [9]

2.2.1.2 Indium Alloys

Indium-tin alloys are viable options for low temperature bonding as explained in the literature. [5] Studying the phase diagram of the indium-tin system in Figure 4 shows that the two equilibrium phases are terminal Sn and In, two intermediate phases of β and γ exist, and that there is an eutectic point between the two intermediate phases. There are no known intermetallic phases. The most common solder used from this system is the eutectic at 52%In-48%Sn which melts at 118°C. The shear strength of this alloy is reported by Indium Corp as 11.24 MPa, almost

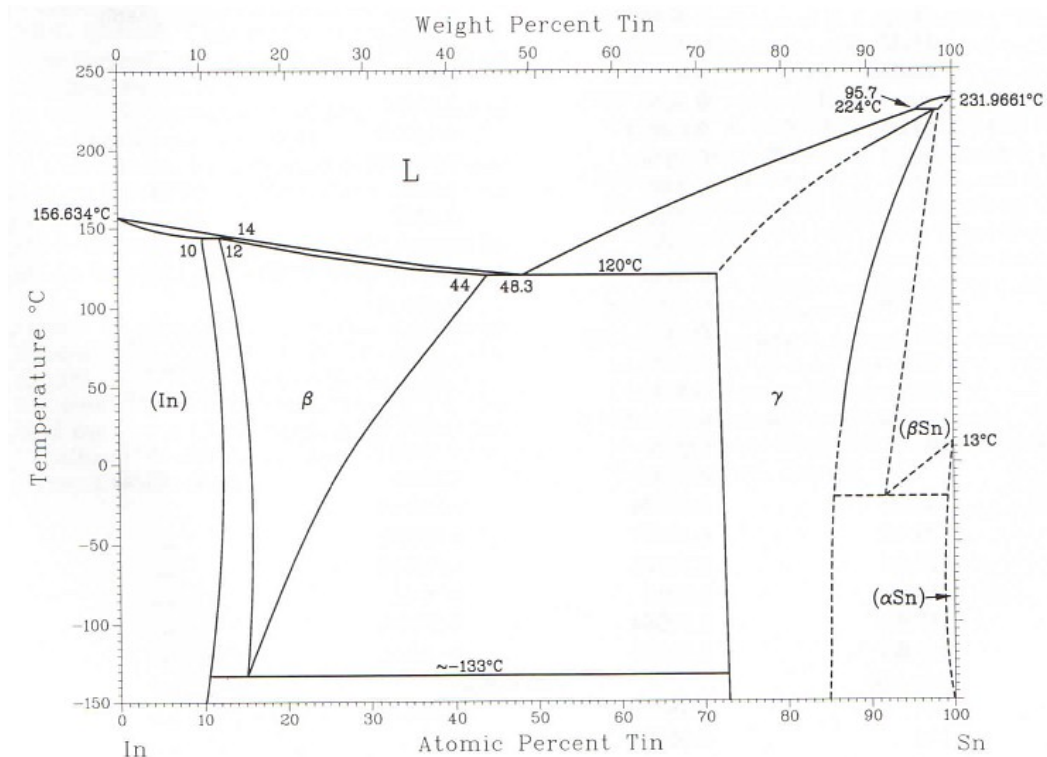


Figure 4 - Diagram for the indium-tin system. [10]

twice the strength of pure indium. [13] Having a higher shear strength could result in a higher joint strength; however the force required to deform the gasket to break the oxide layer and cause the solder to flow will also be much greater. Therefore it is more advantageous to use In-Sn as a molten solder or in a diffusion bonding processes. The problem still remains that if a molten solder is used for sealing to glass, high residual stresses will form during solidification due to the thermal expansion mismatch. Adhesion could be enhanced in this case by using a metalized layer such as chromium on the glass. [5]

An alloy of indium-bismuth is used in the industry as a very low temperature solder for the sealing of vacuum tubes. [14, 15] The phase diagram in Figure 5 shows that there is an eutectic point with a composition of 67 wt% In and 33 wt% Bi and an eutectic temperature of 78.5 °C. This melting temperature is significantly lower than the melting temperature of pure indium (157 °C). A value for the shear strength of this alloy could not be found in the literature. Only 0.01 atomic% of indium is soluble in bismuth while between 4 and 8.2 atomic% of bismuth is soluble in indium. There are three intermetallic compounds which are stable at room temperature: BiIn , Bi_3In_5 , and BiIn_2 . All three phases have melting temperatures equal to or below 110 °C. There also exists a phase ϵ which has a composition range of 88 to 90 atomic% In. The phase has a peritectic formation temperature of 92.5 ± 0.5 °C and an eutectoid decomposition temperature of 49 ± 1 °C. [13]

Studying the phase diagram in Figure 6 for the indium-silver alloy system shows that an eutectic point occurs at a temperature of 144 °C with a composition of 97 wt% In and 3 wt% Ag, making this another usable alloy for low temperature sealing. This temperature is slightly lower than the melting point of pure indium (157 °C). Indium Corp reports the tensile strength of an alloy with this composition has a tensile strength of 5.5 MPa which is more than double that of pure indium (1.88 MPa). [13] No shear strength values could be found. According to Okamoto *et al*, the only phase which will form when the temperature is kept below the eutectic temperature is an intermetallic phase with the stoichiometry of AgIn_2 . [13] More about the formation of this phase is discussed in section 2.2.2.2.

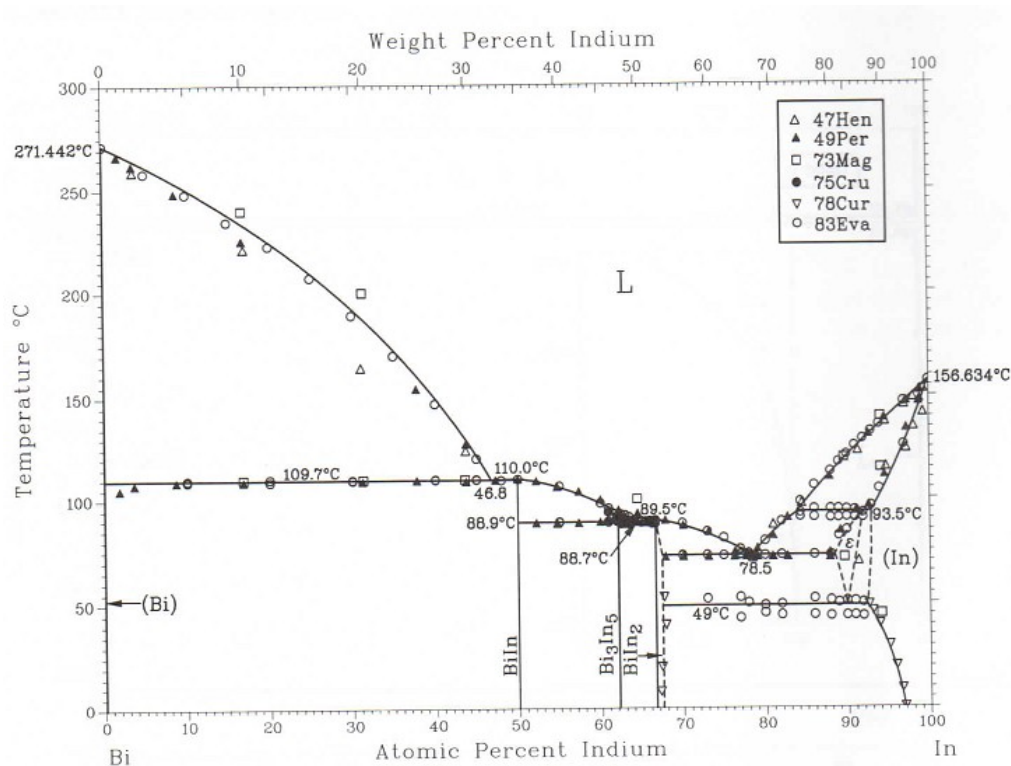


Figure 5 - Phase diagram for the indium-bismuth system. [12]

2.2.2 Substrate Material and Coatings

It is an objective of this investigation is to determine which surface materials will work best for producing a robust, hermetic seals with indium. In the as-received condition, the substrate surfaces are borosilicate float glass which inherently has a smooth finish. Some sources claim indium will bond to borosilicate glass effectively without the need for an intermediate layer or a flux. [6, 16, 17] It has been seen that indium will bond to glass simply by rubbing an ingot of indium onto a clean glass surface. [6] Common thought in the industry is that the bond is a result of indium flowing into the asperities of the glass which forms a mechanic bond. However,

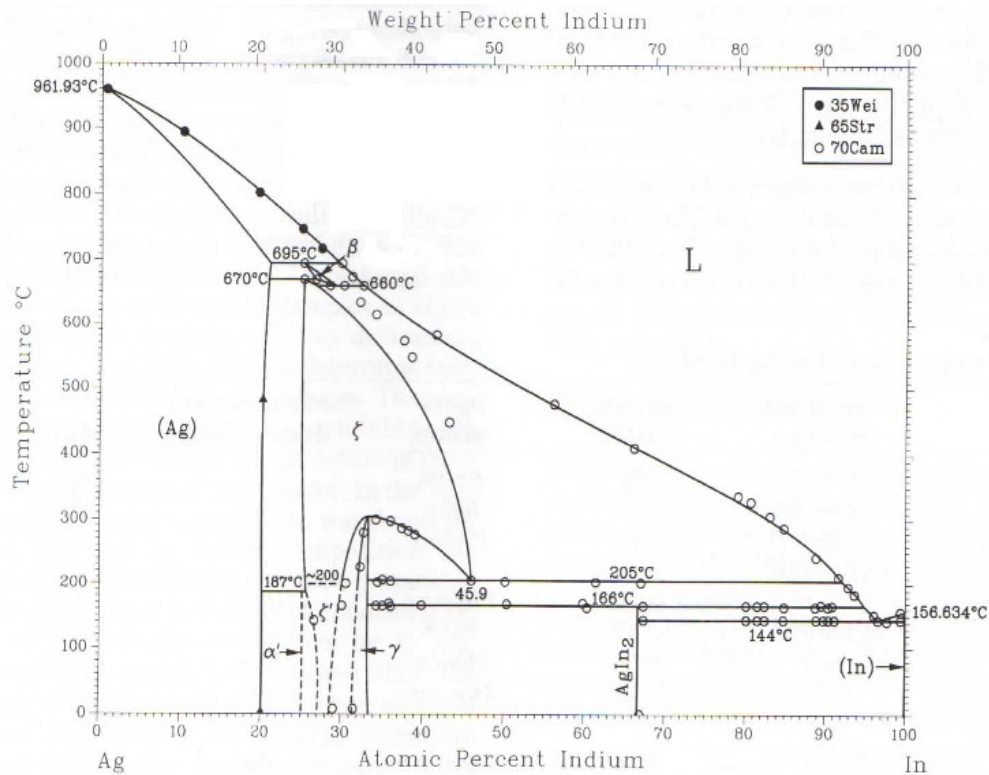


Figure 6 - Phase diagram for the indium-silver system. [12]

Neuhauser suggests the bond forms because of very rapid oxidation of the freshly exposed indium when it comes into intimate contact with a glass or ceramic surface. [6] Based on the literature found for this review, no other work has been found which supports this claim about an “oxide bond.” Literature from AIM Materials does say that in order to make a chemical bond between glass and indium, the indium must be plastically deformed in order to break about the indium oxide layer to expose “fresh” indium to the surface of the glass. [18] The particular mechanisms for bonding indium to glass are not investigated in this thesis.

There are conflicting results from the literature on indium’s solderability to borosilicate glass. Sources claim that in order for indium to wet glass and produce a robust, hermetic seal, a metallic layer is required. [4, 19, 20] There is no consensus in the literature about which materials are best for bonding to indium although gold seems to be the most widely used. It has been suggested by Helber that the material used for the metalized layer should form a primary-intermetallic-free alloy with indium since intermetallic phases will weaken the bond. [19] A primary intermetallic is a compound of two or more single phase metals or metal alloys forming the bond, which is a separate stoichiometric phase of a fixed composition. Materials which form intermetallics with indium include antimony, arsenic, bismuth, copper, gold, magnesium, manganese, nickel, silver, and thallium. [19] The weakening of the joint is most likely due to interface coherency mismatch between the intermetallic compounds and either of the base metals. In some cases, the formation of a primary

intermetallic phase is beneficial. [4, 14, 21, 22] Typically the strength of the intermetallic is greater than that of the two base-metals alone. However, after extended periods of time and/or thermal cycling, solid-state diffusion will occur and the intermetallic layer can become thick and very brittle making the joint integrity unpredictable. In order for this type of bonding to be effective, layers of the material need to be deposited with great precision in order to limit the size of the primary intermetallic region. [19]

Another important consideration for metalized surface is its thermal expansion match to indium. As mentioned in section 2.2.1.1, the thermal expansion of indium is approximately $37.5 \times 10^{-6} / ^\circ\text{C}$ while the thermal expansion of borosilicate glass is $3.3 \times 10^{-6} / ^\circ\text{C}$. [23] Large mismatches in thermal expansion coefficients could result in spontaneous failure in the joint during thermal cycling of fabrication or general operation due to the residual stresses incurred from the thermal mismatch. It is possible to bridge the thermal mismatch by using a metalized layer which has a thermal expansion coefficient somewhere between those of glass and indium.

Cleaning of glass substrates prior to sealing has been mentioned in some literature sources. Organic contaminants must be removed from the surface of the glass in order to make a quality seal using indium. [10, 18] This is most commonly achieved with washing with acetone and alcohol or using a mild detergent.

2.2.2.1 Silver Coating

Sources agree that high rates of interdiffusion take place between indium and the noble metals; tin and bismuth can also readily react with the noble metals. [24, 25] P. J. Wang *et al.* show that In atoms electroplated onto an Ag substrate will immediately form an intermetallic compound with the stoichiometry AgIn_2 . [26] After one day of storage at room temperature, the intermetallic layer will grow to approximately 4 μm thick (Figure 7a). Continued aging for 12 days at room temperature results in a thick layer of AgIn_2 which has completely consumed the entire layer of indium (Figure 7b). Figure 7 shows that the diffusivity of silver into indium at room temperature is approximately $2 \times 10^{-9} \text{ cm}^2/\text{s}$. It has been stated that silver will diffuse into indium primarily by the interstitial mechanism while indium will diffuse into silver through grain boundaries. [26, 27] With increasing temperature, grain boundary diffusion of indium into silver begins to dominate since the activation energy of grain boundary diffusion (0.34 eV) is less than the activation energy of lattice diffusion (0.55 eV). XRD data shows that indium and AgIn_2 have the same crystal structure, body-center tetragonal. However, other sources indicate that indium has a crystal structure of face-centered tetragonal with a c/a ratio of 1.07. [27] As for the growth mechanisms of AgIn_2 , it is possible that In atoms will diffuse through the AgIn_2 and react with the Ag or the Ag will diffuse through the

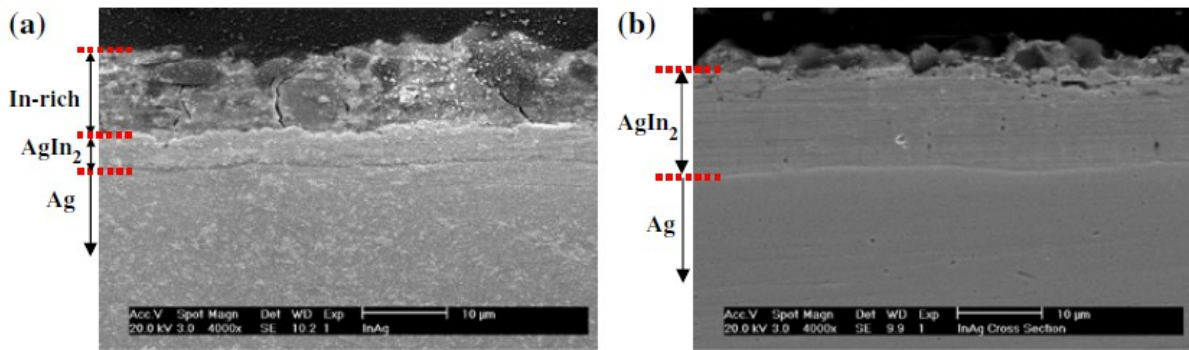


Figure 7 - Cross section of Ag-in (10μm) samples. (a) Sample aged at room temperature for 1 day. AgIn₂ layer has grown to 5μm. (b) Sample aged at room temperature for 12 days. The AgIn₂ layer continued to grow until it effectively absorbed all of the indium (15 μm thick). [24]

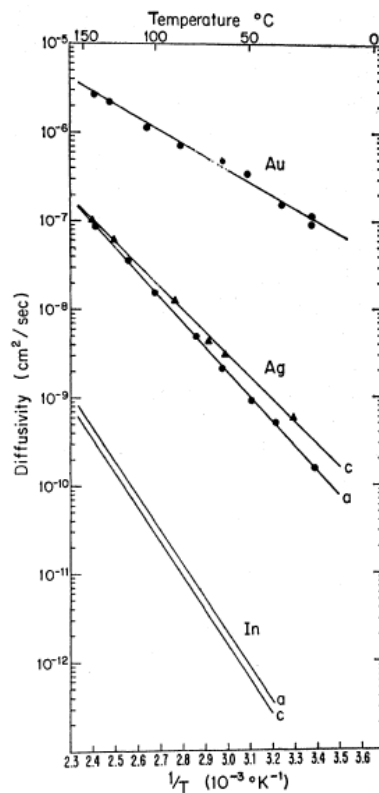


Figure 7 - Diffusivity measurements of indium, gold, and silver in indium. “a” and “c” refer to the direction of motion along the crystal structure of indium. [27]

AgIn_2 to react with the In. Wang *et al.* does not offer data to show which diffusion path dominates.

R. I. Made *et al.* have studied how the structure of Ag-In compounds will change with increase in time, temperature, and pressure in wafer level packaging under thermal compression conditions commonly seen in flip-chip fabrication. They determined that while under a load of 1.4 MPa and at a temperature of 180 °C, short duration (10 minutes) bonding produces the compound AgIn_2 that dominates even with ample amounts of Ag available. When the duration was increase to 40 minutes, it was seen by XRD (Figure 8) that the phase Ag_9In_4 dominates. They suggest that Ag_9In_4 may not initially be stable at room temperature. Longer aging times may increase the stability of Ag_9In_4 as it allows for extra Ag to remain in the un-reacted pure metal layer. [28]

2.2.2.2 Gold Coating

Using gold as a metalizing layer has many advantages and disadvantages when it comes to indium solder joints. Several sources indicate that indium wets very well to gold. [4, 14, 19, 20-22, 29] This makes it an excellent material to use as a bonding layer for indium alloys since it is said that these alloys do not exhibit good wettability to ceramic and glass substrates. Gold can also be used as an oxidation

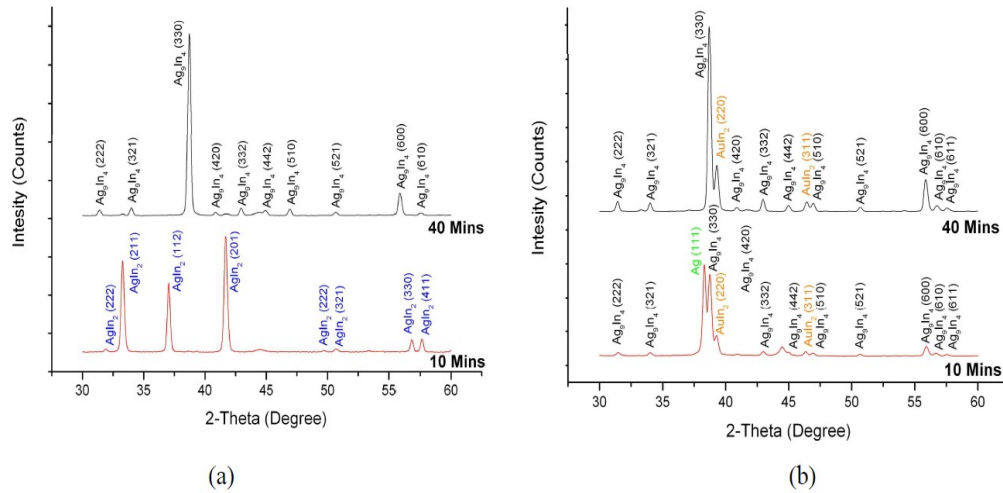


Figure 8 - XRD spectrum of aged samples at 70°C with different aging times. Samples Loaded with 1.4 MPa at 180°C for 10 minutes. (a) is of an In-rich sample and shows the presence of co-existing AgIn_2 and Ag_9In_4 . (b) is of a Ag-rich sample which shows the preference of Ag_9In_4 . [28]

barrier for metals such as silver, copper, and indium when thin films of these materials are deposited under vacuum. [5, 14]

The caveat of using gold is that it readily alloys with indium even at room temperature resulting in strong but brittle intermetallics which could jeopardize the integrity of a joint. It has been found that indium will begin to react with gold immediately after vacuum deposition. [12] According to the phase diagram in Figure 9, two intermetallic compounds are stable at room temperature: AuIn and AuIn_2 . C. Helber claims that indium joints using gold as a wetting layer are initially very strong, but after extended aging or thermal cycling, the bonds become weaker and may fail entirely. [19] The growth rate of these compounds has been investigated by J. E.

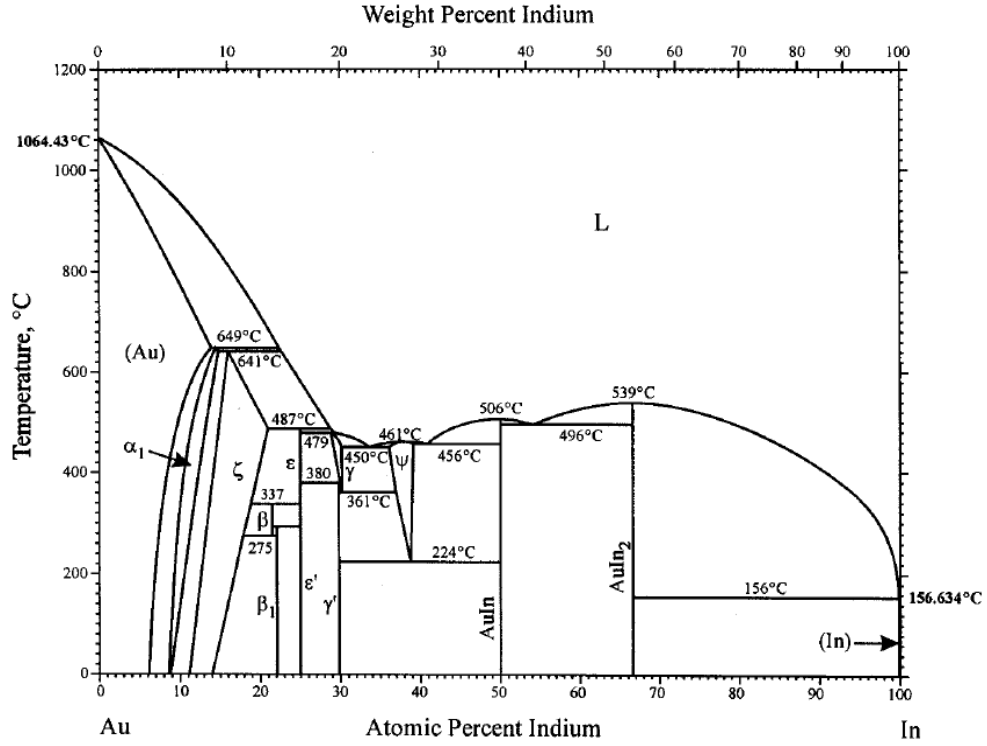


Figure 9 - Phase diagram for the gold-indium system. [30]

Jellison. [29] She concludes that “the growth rate of AuIn₂ is not negligible at room temperature.” [29] Jellison defined the equation for the growth rate of AuIn₂ as

$$\beta = \left(6.88 \times 10^8 \mu\text{m}/\text{hr} \right) \cdot \exp \left(- \frac{0.72\text{eV}}{k_B T} \right)$$

where β is the growth rate, k_B is the Boltzmann constant and T is the absolute temperature in kelvin. This equation is plotted on the graph in Figure 10 as the line labeled “This work.” Solving this equation for 20 °C (293 K), the growth rate is found

to be $0.00023 \mu\text{m/hr}$ or $2.0 \mu\text{m/year}$. Jellison nor any other author has shown the correlation between the thickness of the AuIn_2 layer and the strength of the joint. Though it has been reported that seals with gold layers $0.2\text{-}0.4 \mu\text{m}$ thick applied to Pyrex then bonded with indium have withstood major thermal cycling tests. [4]

2.2.2.3 Nickel-Chromium Alloy Coating

A nickel-chrome alloy (nichrome) is a suitable material to study for its use as a glass coating in an indium seal. Nichrome is typically used in the design of photomultipliers as a means for connecting the vacuum-sealed internals to the external high voltage. [14, 31] Sources do not explicitly identify which alloy is used

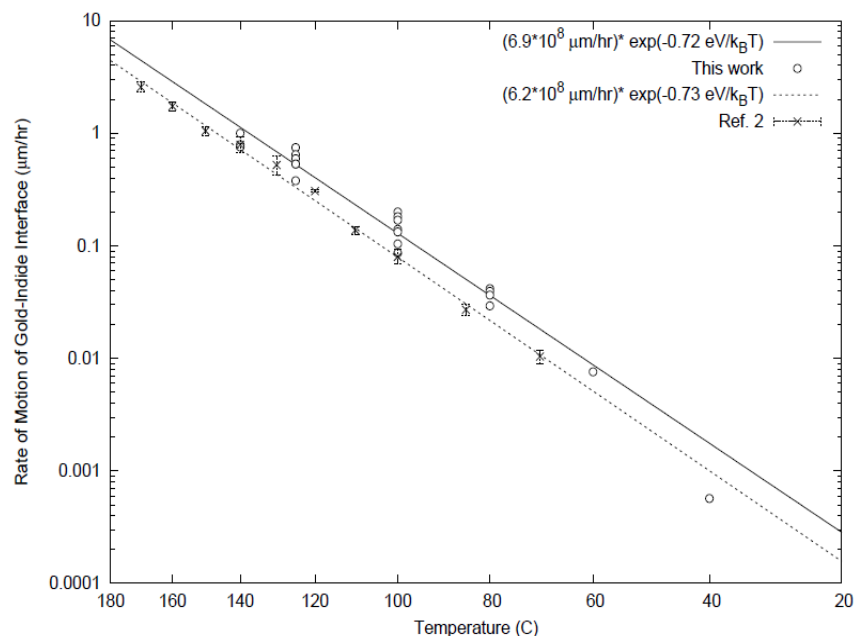


Figure 10 - Arrhenius plot of the motion rate of the gold-indium interface. [29]

on their photomultipliers; however an alloy of 80 wt% Ni and 20 wt% Cr (NiCr A) is commonly used in the electronics industry. An alloy of this composition has a coefficient of thermal expansion of $13.4 \times 10^{-6} / ^\circ\text{C}$ making it fairly compatible with borosilicate glass. [32]

Chromium plays an important role in the functionality of nichrome as a coating for indium sealing. Chromium alone has been used in numerous indium seal designs found in the literature since it exhibits good adhesion to glass. [5, 20, 28] However, no literature was found where indium was bonded directly to chromium without the addition of a gold layer. It is well known that a thick layer of Cr_2O_3 will form on the surface of the chromium when exposed to air. In this case, the mechanism for bonding indium to chromium may be more like that of an oxide bond rather than the formation of an alloy bond like other metals.

It is also necessary to study the nickel-indium system as nickel makes up 80% of the nichrome. Studying the phase diagram in Figure 11, several intermetallics exist, however in a study by Kim *et al*, only $\text{In}_{27}\text{Ni}_{10}$ was found to form at room temperature. [33] The thickness of this intermetallic compound with respect to aging temperature is shown in Figure 12. Diffusion rates of indium in nickel are very slow making nickel a good diffusion barrier if it is deposited over a metal sensitive to alloying with indium.

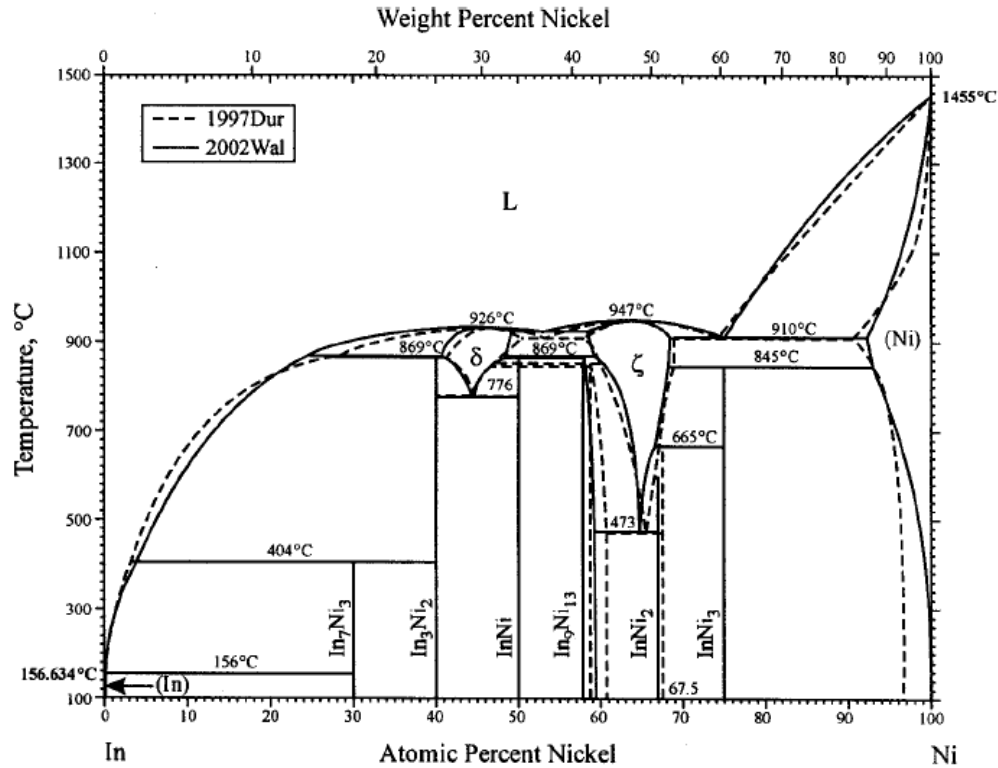


Figure 11 - Phase diagram for the indium-nickel system. [10]

2.2.2.4 Additional Coating Materials

Other materials are available that could be useful for sealing with indium since they form a primary-intermetallic-free alloy with indium as discussed in section 2.2.2. These materials include aluminum, cadmium, gallium, germanium, silicon, and zinc. Helber reports that “primary-intermetallic-free alloys are strong and remain strong and tough with the passage of time and upon thermal exposure.” [19] In the case of aluminum-indium system, the solubility of either element into another is below 0.1%

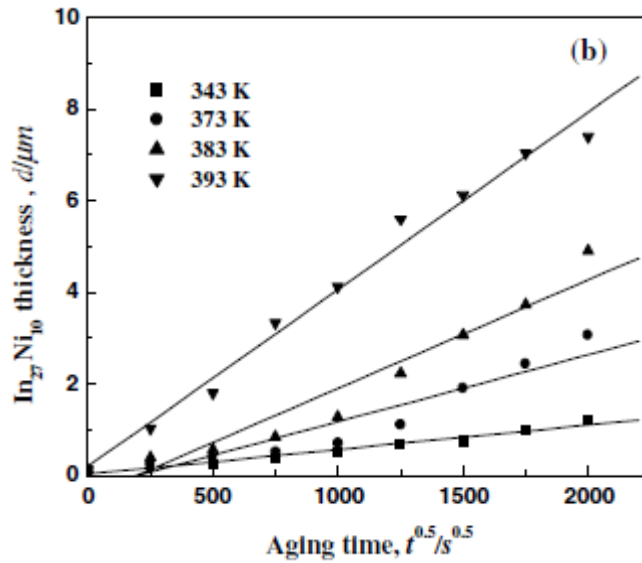


Figure 12 - The thickness of the intermetallic compound $\text{In}_{27}\text{Ni}_{10}$ with respect to aging time at various temperatures. [33]

at room temperature, and there are no intermediate phases which will form at room temperature. The aluminum phase and indium phase have nearly the same crystal structure: face-center cubic ($a = 0.405$) and tetragonal ($a = 0.460$, $c = 0.495$) respectively. [13] Therefore, the alloying region of the bond will contain coherent and semi-coherent interfaces between grains which should be relatively more reliable the grains in a system where intermetallic precipitation occurs.

Another material which could potentially be used for indium sealing but is seldom researched for that purpose is titanium. Several intermetallic phases are known in the indium-titanium system; however, those compounds will only form at elevated temperatures. [13] Few studies have been done on the diffusion of indium

into titanium, but only at elevated temperatures (823-1073K). Even at those temperatures, diffusion rates were lower than 10^{-18} m²/s implying diffusion rates at room temperature would be many orders of magnitude slower. [34] Titanium is also a good thermal match to borosilicate glass with a coefficient of thermal expansion of 8.41×10^{-6} /°C. [23]

A transparent conducting oxide material such as indium-tin oxide (ITO) also has the potential to form a leak-tight seal with indium through the formation of oxide bonds similar to that of borosilicate glass. And investigation of the cold-weldability of ITO is warranted since it is widely used in the electronics industry as a thin film for applications such as photovoltaic solar cells and flat-screen TVs. Current applications use high temperature brazes or organic adhesives to make these joints. Such methods are not acceptable for low temperature, vacuum applications, however. Few studies have been done on bonding to ITO thin films for optical applications, but none have been found involving indium solders. [35]

3. EXPERIMENTAL PROCEDURE

3.1 Experimental Plan

The purpose of this investigation is to determine the optimal procedure for producing a long-lasting, robust vacuum seal between two borosilicate glass substrates using indium as a solder. It is important to this investigation to understand the effects of bonding temperature, applied load, and process duration on the formation of a low-temperature indium seal. There appears to be a lack of concise information on these topics in the literature. In order to study these effects, bonding tests will be carried out using B33 borosilicate glass substrates obtained from Cat-i Glass, both coated and uncoated. Material coating to be focused on is nichrome alloy (80 wt% Ni, 20 wt% Cr) Indium wire with purity 99.995% was obtained from Indium Wire Extrusion. A helium leak detector will be use to characterize the samples' hermeticity and measure any existing leaks. Joint strength will also be investigated by means of a lap-shear test to characterize the seal robustness. Both characterization methods will include a small number of samples which have undergone an aging procedure. Scanning electron microscope with an electron diffraction spectroscopy attachment as well as X-ray diffraction will be attempted to characterize surface coatings as well as any compounds which may form during bonding.

3.2 Experimental Setup

All samples were fabricated in a custom built vacuum chamber at Argonne National Laboratory shown in Figure 14. The chamber is capable of reaching a pressure of 10^{-8} Torr. This is a suitable vacuum pressure for fabricating the seals. Attached to the chamber is hydraulic cylinder and accompanying manifold capable of applying loads up to 3000 pounds. Hydraulic pressure to the cylinder is controlled by a regulator with a digital pressure monitoring gauge attachment. Speed of the loading can be roughly controlled using flow control valves. Measurements of the travel speed of the cylinder and the indium deformation during loading is obtained from a laser displacement monitor. Processing temperature can be increased with

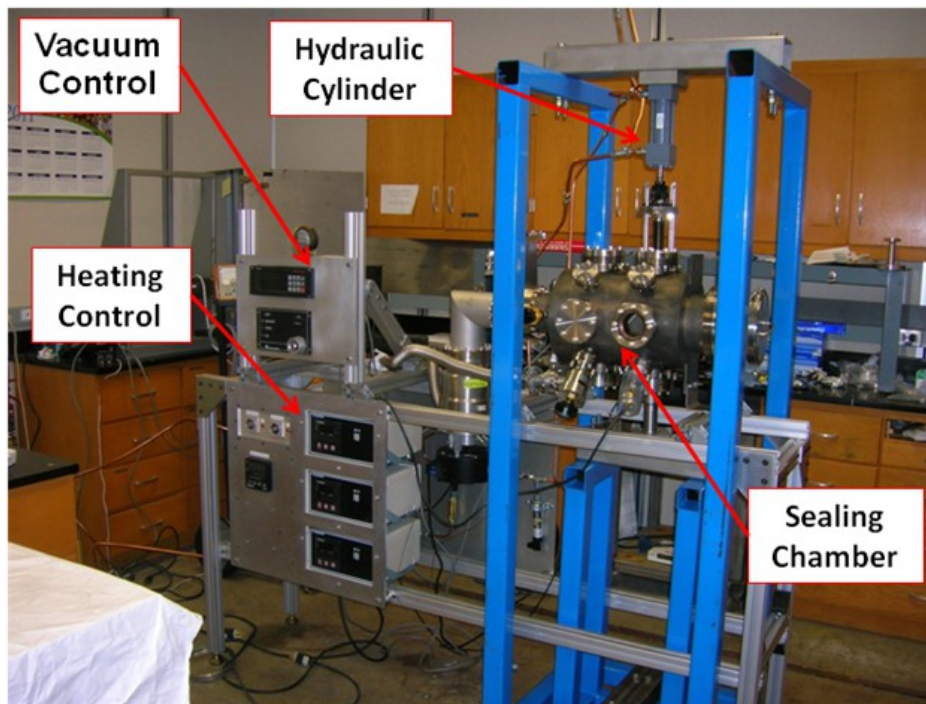


Figure 14 - Diagram of vacuum sealing chamber.

the use of two halogen bulbs above the sample stage and a resistive heating element below. One thermal couple is mounted into the sample stage adjacent to the sample and another is rested on the indium which is removed once the desired temperature is reached.

3.3 Fabrication of Samples

Samples fabricated for testing consist of two glass substrates and an indium gasket. One substrate is fashioned into a glass frame $2.54 \times 2.54 \times 0.66$ cm with a wall thickness of 0.5 cm. This part will be referred to as the “sidewall” in this report. The other substrate to be used in testing will be a $2.54 \times 2.54 \times 0.275$ cm part. This part will be referred to as the “window” in this report. A cross-sectional diagram of these parts is shown in Figure 15. An image of the actual parts can be seen in Figure 16. Hollow samples are necessary for this investigation since the seals will be

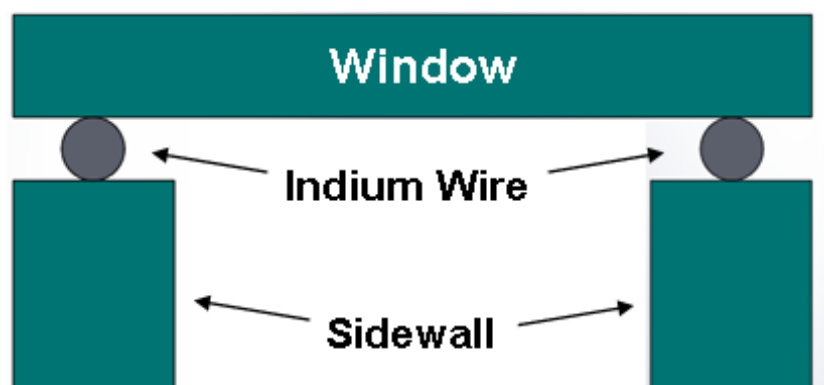


Figure 15 - Cross-section of borosilicate glass parts and indium wire gasket.

characterized by their hermeticity. All samples will be machined in the shape of a square in order that the experimental procedure is easily scalable to larger sized packages.

Uncoated glass pieces were cleaned using ultrasonic baths of acetone and alcohol for 15 minutes each prior to sealing. Samples which had been coated were left in the as-coated state as this was considered to be the cleanest state.

3.3.1 Indium Gaskets

Several methods for making an indium gasket were considered based on the literature. Initially, indium foils 50 μm thick were cut to the same shape as the

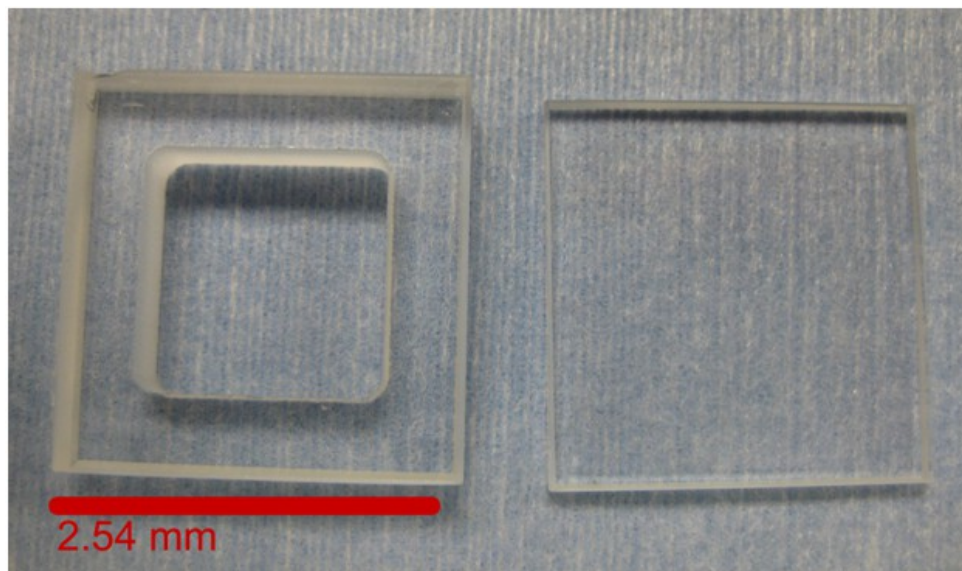


Figure 16 - Image of uncoated, borosilicate glass parts.

sidewall. It was found that the plastic deformation of the indium during the compression of the foil was not adequate enough to break the oxide layer to form a robust bond to the glass. Increasing the thickness of the foil to 100 μm did not improve the sealing. Neither chemically nor physically removing the oxide improved bonding using the foils. Increasing the applied load improved the plastic deformation; however, the glass substrate was nearly always damaged as a result.

The best option was to use a gasket made of indium wire, either 1.5 or 2.0 mm in diameter. Majority of tests were carried out using 2.0 mm wire since more material is available to flow under compression, creating a larger bonding area. Gaskets were made by hand from indium wire by cold welding two freshly cut ends of the wire together, forming a region referred to as the “splice.” Many problems were associated with how the splice was formed. Simply overlapping two ends of the wire resulted in non-uniform loading and caused the glass parts to break. The perfected method of making the splice was to cut both ends of the wire at an angle then immediately connecting the ends before oxidation can occur. Tweezers were used to massage the splice back to a rounded shape. Any bumps or valleys which may have formed during this fabrication were smoothed out by gently scraping the region with a metal object.

As mentioned in the literature review, surface preparation and cleaning are very important when making vacuum seals. While various cleaning procedures of

the indium were tested, the majority of tests were carried out using indium in the as-received condition to test the worst case scenario. In some cases, a metal tool was used to scrape the indium surface in an attempt to physically remove the indium oxide.

3.3.2 Deposition of Coating Material

In order to test the effect of surface coating on the seal, a method of magnetron sputtering was used to deposit the material onto the surface of the glass. Glass samples were cleaned thoroughly before coating in an ultrasonic bath of 2% concentrated solution of Citrox and dried with nitrogen. System pressure of the sputtering system before coating was 10^{-8} Torr. An argon plasma treatment was used at a pressure of about 200 mTorr. The various parameters for coating are listed in Table I. Coated parts are examined using X-ray diffraction to confirm structure and composition.

TABLE I - COATING PARAMETERS USED FOR SPUTTERING DEPOSITION OF MATERIALS

Material	Current in Target Material (Amps)	Voltage in Target Material (Volts)	Argon Gas Pressure (Torr)	Deposition Time (min)
Nichrome	1.0	490	0.012	12

3.4 Hermeticity Testing

The primary objective of this investigation was to determine the hermeticity characteristic of indium seals made with coated and uncoated borosilicate glass substrates. Hermeticity of the seals was characterized using a Alcatel helium leak detector with a mounting fixture containing a Viton o-ring (Figure 17). For leak testing, samples are pumped down to a base pressure of 10^{-4} Torr. Ample time was given for the onboard analogue filament gauge to stabilize. Helium was allowed to flow around the joint and a measurement of the gauge was taken. Samples were considered “leak-tight” if the gauge reached the lower limit of 2×10^{-11} cc/s of helium and there is no visible response from the gauge when the sample is completely saturated with helium. A sample is considered to “leak” if there is any measurable response from the gauge regardless of the size of the leak, even if the gauge initially read 10^{-10} cc/s of helium. The location of a leak is found by probing the perimeter of the sealing using a syringe filled with helium. The intensity of a leak is not significant for this investigation since any leak is considered a failure in hermeticity. This makes the hermeticity results very polarizing and further characterization methods are needed to truly understand the seals.



Figure 17 - Alcatel leak detector used for hermeticity testing.

3.5 Shear Testing

An assessment of the robustness of the indium seals was carried out using a method of lap shear testing. The shear strength of the indium seals gives a quantitative measurement of how the processing parameters and coatings affect the hermeticity. It is possible that an indium seal which is hermetically sound could exhibit poor mechanical properties and be prone to failure. This is the case if the indium gasket has formed a gasket-type seal in which the seal is leak-tight, however there may actually be little to no bonding between the two materials.

Preliminary testing of seals made using glass substrates with the dimensions 22 cm by 22 cm have indicated that issues arise in the corners of these seals when there is a pressure difference between in the inside and outside of the vacuum tube. When the air is evacuated from the inside of the vacuum tube, the atmospheric pressure pushes down on the window causing the glass to bow inward. Since the geometry of the window is square, the deflection of the window is not uniform causing it to deflect more at the corners than at the straight edge. Finite element analysis of the parts under atmospheric load confirms this (Figure 18). [36] The analysis shows a deflection of 0.025 mm in the corners and 0.010 along the straight edge under this load. This deflection causes a stress which must be overcome by both the strength of the indium and the strength of the bond in order to prevent the corners from detaching, causing to leaks.

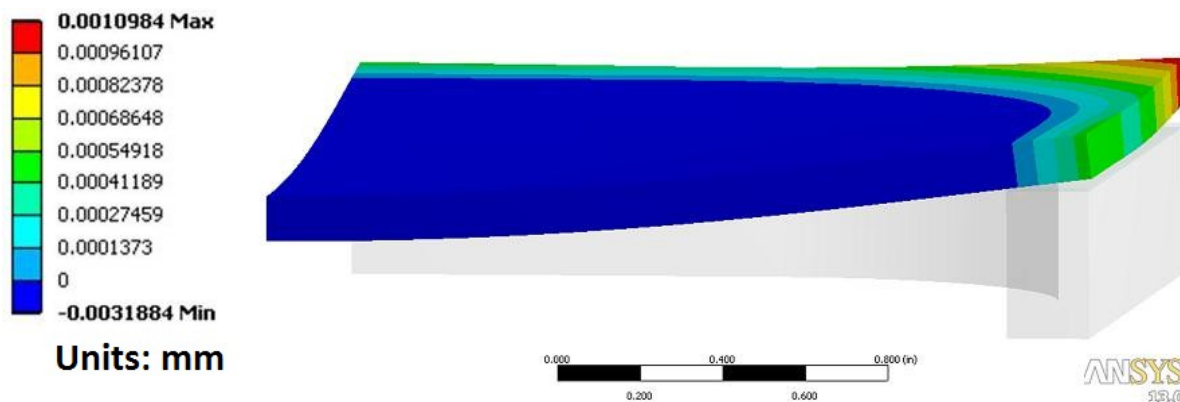


Figure 18 - Finite element analysis of a 22 cm by 22 cm glass window with a load of 1 atm. Blue indicates a deflection <0. The maximum deflection in the corner is 0.025 mm. [36]

Shear testing was conducted on a Tinius Olsen H50kT tensile testing machine (Figure 19). A custom fixture was made such that the load path would be through the indium as to prevent any rotation stresses during testing. The strain rate of the tests was 0.5 mm/sec. The machine delivers the data in units of force so the actual sealing area must be calculated using computer imaging software. It becomes difficult to determine exactly what areas of the indium have bonded to the substrate and if all the bonded areas have the same strength. Because of this, it is more useful to compare the raw shear-force-to-failure measurements than shear strength. For



Figure 19 - Image of Tinius Olsen tensile tester used for lap shear testing.

nominal calculations, an average bond area of 0.55 cm^2 is used for computing shear strength, unless otherwise noted.

The underlying goal of the shear testing was to make a joint in which the bonding between the indium and the substrate was so strong that failure would occur only in the indium. In order to get a baseline of indium shear strength, parts made of pure copper were used since it was found in the literature that the bond between indium and copper is much stronger than indium itself when copper is abundant. In this way, shear failure is guaranteed to take place in the indium. The strength obtained by the tests was 6.0 MPa which is very close the 6.14 MPa published by Indium Corp. [13]

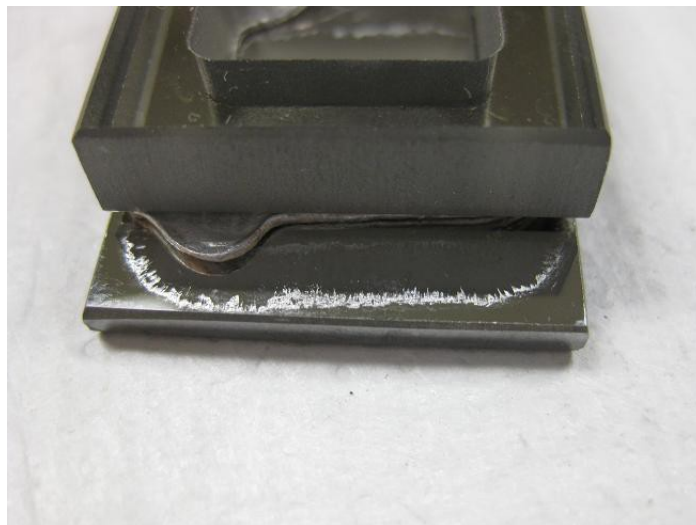


Figure 20 - Fresh indium flows from the sides of the gasket during compression to form the bulk of the bonding in two “tracks” along the inside and outside edges of the wire.

Shear testing revealed that the sealing does not take place over the entire width of the seal. Instead, the bulk of the bonding appeared to take place along two “tracks” of indium located on the inside and outside edges of the compressed gasket. These formations can be seen in Figure 20 of a shear tested sample. When the indium wire is compressed, fresh indium flows out the sides of the wire leaving the center portion of the indium oxide unbroken and therefore un-bonded. A schematic of this result is shown in Figure 21. It is difficult to measure the width of the tracks because shearing the sample causes the indium to smear. In chapter 4, it will be discussed how increasing the width of the gasket by adjusting the processing parameters, and therefore the width of the tracks, will increase the force to shear failure of the seal.

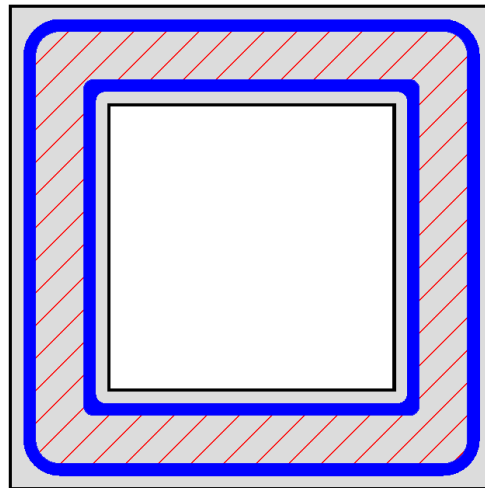


Figure 21 - Schematic of indium “tracks” on the surface of the glass substrate. Blue lines indicate tracks where fresh indium has bonded to the substrate. The red-hatched region indicates the area where indium oxide has prevented bonding.

3.6 Aging tests

Samples were aged at temperatures below indium's melting temperature to simulate the behavior of the seals over extended periods of time. These samples were tested for changes in their hermeticity at various intervals. At the end of the thermal cycling, the samples were shear tested to study the effects of aging on the shear strength. The broken seals were examined using a scanning electron microscope (SEM) with an energy-dispersive X-ray spectroscopy (EDS) attachment. XRD was attempted in an attempt to identify any new compounds which may have formed. However, the regions where failure occurred were too small and too thin to be detected.

4. RESULTS AND DISCUSSION

The results of hermeticity tests and shear tests along with scanning electron microscope analysis are presented in this section. First, the results of the uncoated glass samples are discussed to gain a basic understanding of how the processing parameters affect the seal. Next, results of samples which include glass coated with a layer of nichrome will be discussed in order to understand if coatings can improve sealing integrity. Further analysis of the sustainability of the seals by means of aging tests are discussed. XPS spectra are also presented in order to investigate the formation of alloying phases caused by aging.

4.1 Process Parameters for Indium Seals Using Uncoated Glass

Two indium wires of different diameters, 1.5 and 2.0 mm, were chosen for making the seals. Figure 22 compares the final width of the indium gaskets of the two wires after compression. It was seen that with a compression force of 1180 N over 11 minutes at 20°C, the 2.0 mm gasket produced a bonding area that was 1.03 mm wider than the 1.5 mm wire as was expected because of the greater mass in the larger diameter wire. Figure 23 compares the force to shear failure of the two seals. There is a clear correlation between the increase in wire diameter and the increase in bond strength. Increasing the wire diameter from 1.5 mm to 2.0 mm increased the force to shear failure from 376.9 N to 496.2N. Visual inspection of the samples in Figure 24 shows the width of the seal made

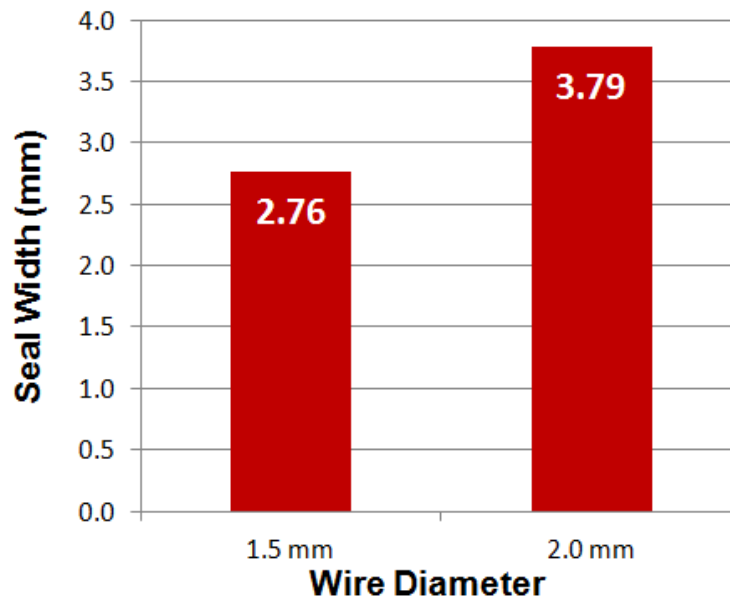


Figure 22 - Comparison of diameter of indium wire to the width of the seal with an applied load of 1160 N for 11 minutes at 20°C.

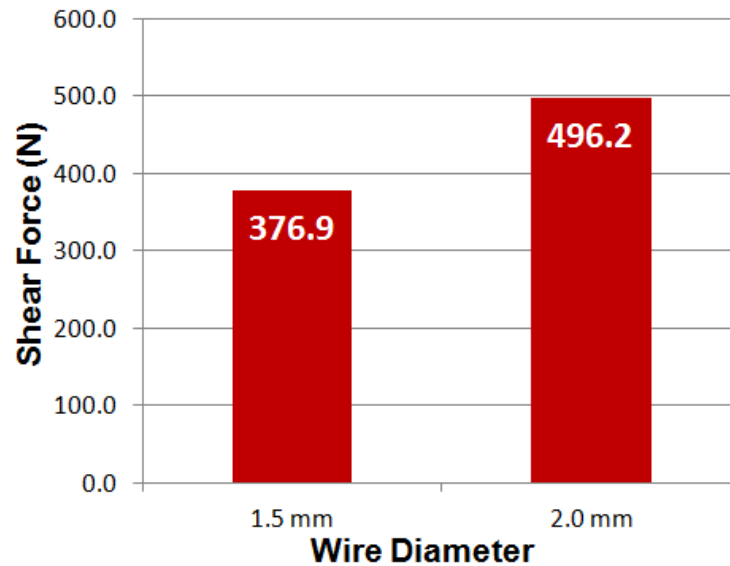


Figure 23 - Increasing the diameter of the wire has a direction relationship on the rigidity of the seal.

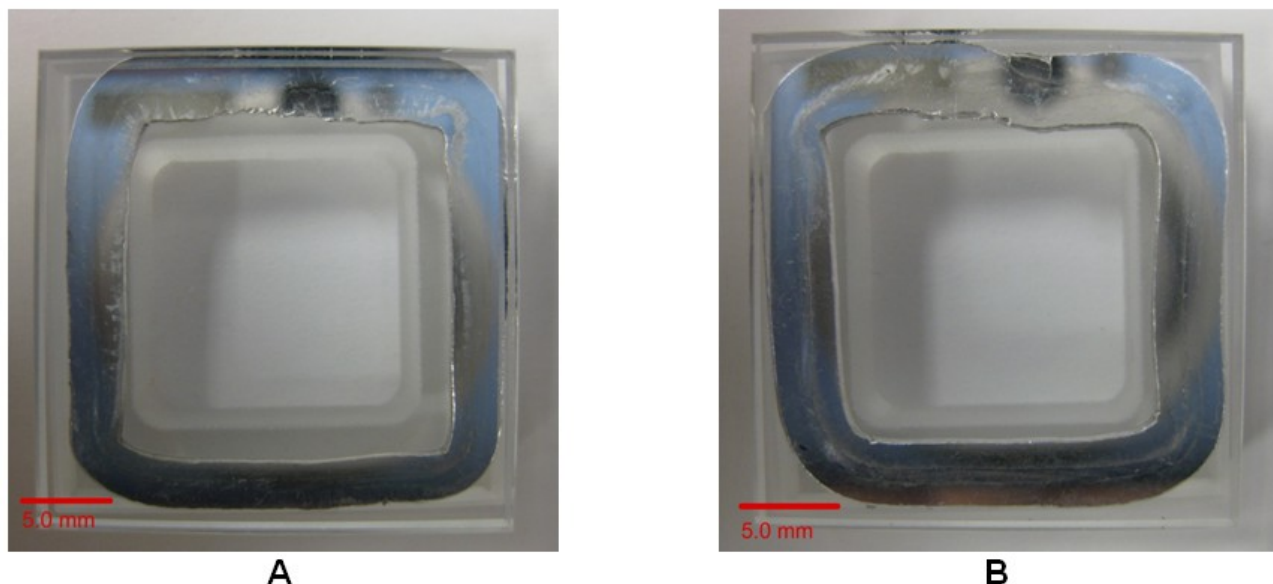


Figure 24 - Top-down view of two seals made with A) 1.5 mm wire and B) 2.0 mm wire in test samples of 2.54 by 2.54 cm.

with the 1.5 mm wire is noticeably more narrow than the seal made with the 2.0 mm wire. Samples made with both wire diameter sizes were entirely hermetic. It is clear that by increasing the area of contact between the indium and glass would improve the hermeticity, but also will enhance the seal strength, as demonstrated in Figure 23.

Tests were then conducted to determine the highest compression load that could be applied without breaking the glass while also increasing the spreading of the indium. The seal width with respect to applied load using 2.0 mm wire is plotted in Figure 25. Fabrication time and temperature were held constant at 11 minutes and 20 °C respectively. As expected, increasing applied load causes the width of the indium seal to increase

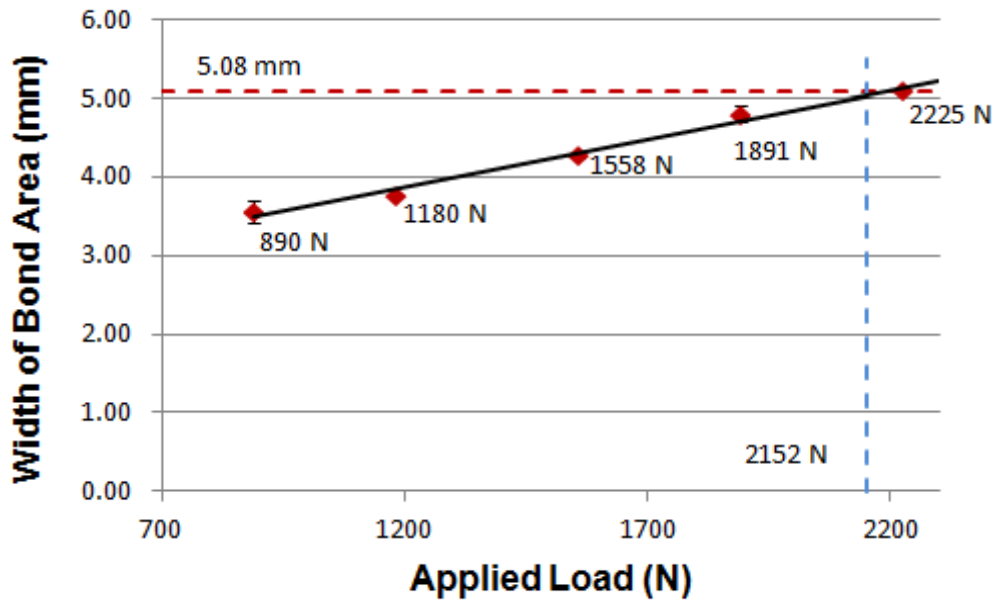


Figure 25 - Indium seal width measurements compared to the applied load. Trend line indicates linear relationship over the range of 890 N to 1891 N. Horizontal line indicates a width of 5.08 mm, the width of the sidewall.

linearly. A line is drawn on the plot to indicate the width of the sidewall, 5.08 mm. The intersection of this line and a trend line of the data is at 2152 N which is the applied load which causes the indium to begin over flowing from the sidewall using a 2.0 mm wire. Over flowing needs to be prevented in order that the indium does not protrude out so far that it make contact with adjacent phototubes in an array and cause a short circuit. Similarly, it should not flow inside the packaging as it may contaminate any other coatings deposited onto the window such as a photocathode. Therefore, the load for this particular application should be kept below 2152 N for 11 minutes at 20 °C. The plot in Figure 26 shows that the

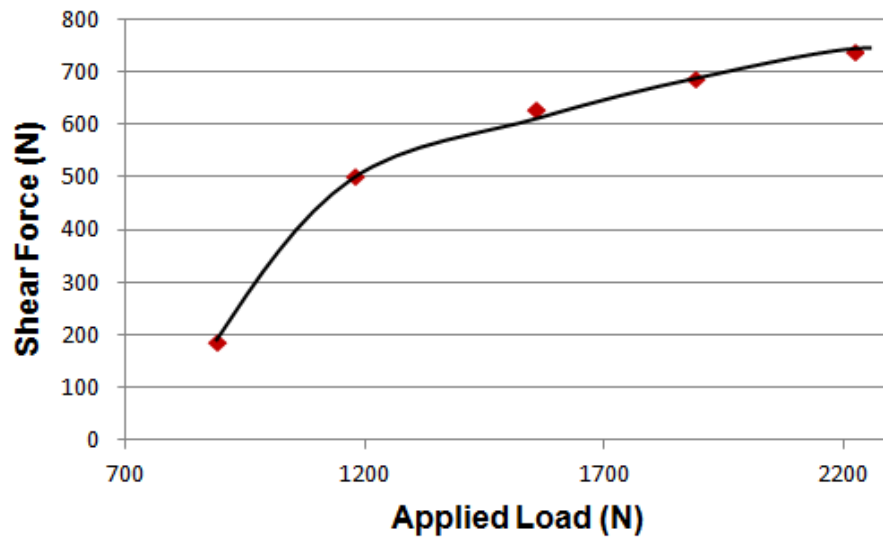


Figure 26 - There is a general trend that with increasing the applied load will increase the strength of the seal. Fabrication time and temperature are held constant at 11 minutes and 20 °C respectively.

force to shear failure indeed increases with increasing indium width. This is a clear result of the increased flow of indium with increasing sealing load.

During fabrication of a seal, the time of manufacturing is a key factor in process efficiency. Tests were conducted where the applied load and temperature were held constant at 1180 N and 20 °C respectively and the time was varied from 10 seconds to 60 minutes. The variance of time did not adversely affect the hermeticity of the samples as all seals were leak-tight. The relationship between indium width and load duration is plotted in Figure 27. Table II shows that with an applied load of 1180 N, 94.4% of the flowing occurs within the first 6 minutes. From 6 minutes to 60 minutes, the indium width

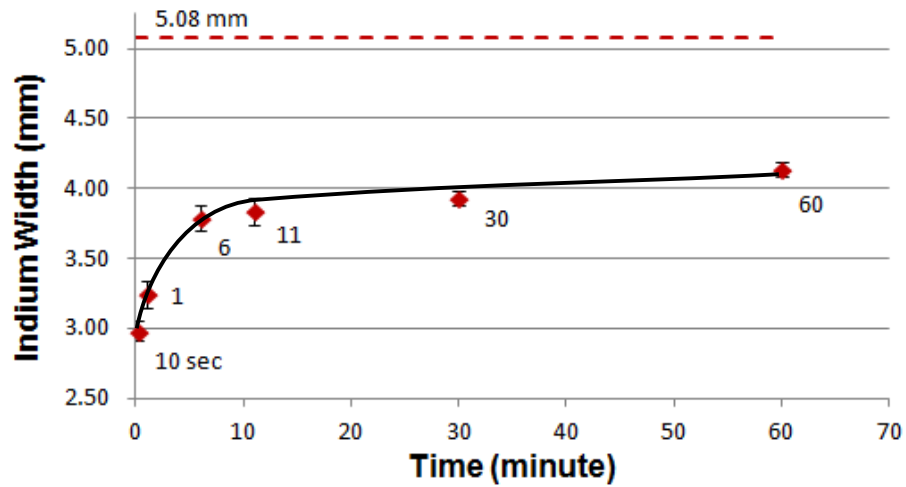


Figure 27 - Relationship between indium width and the duration of applied load. Horizontal line indicates a width of 5.08 mm, the width of the sidewall. An applied load of 1180 N and a temperature of 20 °C were used.

TABLE II - COMPARISON OF THE DEFORMATION OF INDIUM OVER VARIOUS LOAD DURATIONS. DEFORMATION IS RELATIVE TO THE INITIAL 2.0 MM DIAMETER WIRE. APPLIED LOAD OF 1180 N AND TEMPERATURE OF 20 °C WERE USED.

Duration (min)	Deformation (mm)	Percent Deformation (%)
0.17	0.945	76.2%
1	1.03	83.1%
6	1.17	94.4%
11	1.18	95.2%
30	1.2	96.8%
60	1.24	100.0%

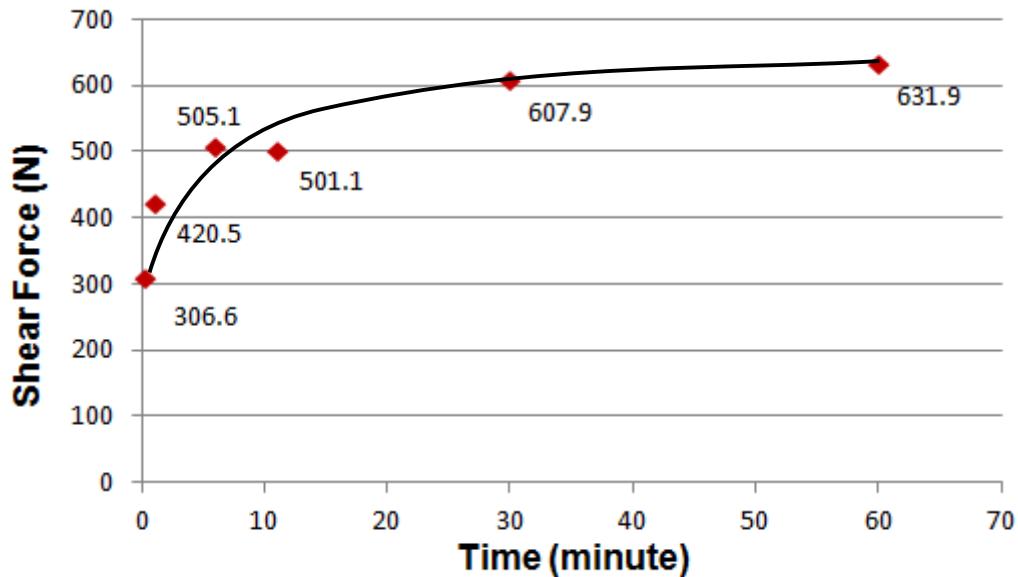


Figure 28 - Relationship between the force to shear failure of the seal and the duration of an applied load of 1180 N.

increased by 0.07 mm, an increase of 6%. Figure 28 shows that over the range of 6 minutes to 60 minutes, the force to shear failure increases from 505.1 N to 631.9 N, an increase of 25%. Again, a line is drawn on Figure 27 to show the width of the sidewall, 5.08 mm. In these trials, the indium was not compressed so much as to cause overflow issues. This also means, however, that indium gasket is being under-utilized, leaving an average of 0.95 mm of sidewall which is not being used for bonding in the case of a gasket compressed for 60 minutes. For a 2.0 mm diameter wire, the cross-sectional circumference of the wire (which is covered in a layer of indium oxide) is 3.14 mm. Subtracting half of this length from the width of the seal would result in the theoretical width of the “tracks” between the indium and one of the substrates. The widths of the tracks at

one bonding interface for samples made with 1180 applied load for 11 and 60 minutes are 2.36 and 2.56 mm respectively, a theoretical increase of 8.5% in the bonding area. This does not coincide with the 25% increase in bond strength, suggested that fabrication time actually has a positive impact on in the kinetics of the so-called oxide bond and is not just beneficial for increasing the sealing area.

Tests were carried out by varying the process temperature while holding the applied load and processing time constant at 1180 N and 11 minutes respectively. Seals were made in air so oxidation of the indium could not controlled. Varying the temperature did not adversely affect the hermeticity of the seals. The width of the indium seals with respect to process temperature is plotted in Figure 29. As was expected, there is an increase in the indium width with an increase in temperature. A trend line shows there is a near linear relationship between temperature and width over the range of 20 °C to 60 °C and plateau between 60 and 80 °C. A line is drawn on the plot to indicate the width of the sidewall, 5.08 mm, which intersects the trend line at approximately 55 °C. Above 55 °C, indium began to over flow from the sidewalls. As stated above, it is necessary to prevent over flow of the indium as to protect the phototubes from creating short circuits with their neighbors in the phototube arrays or from contaminating internal components. The plot in Figure 30 shows there is a plateau of values in the relationship between the manufacturing temperature and the force to shear failure. The maximum shear force obtained without over flow was 644 N at a temperature of 50 °C with an applied load of 1180 N and duration of 11 minutes. This is 40 N less than a seal made at room temperature with an applied load of 1891 N and

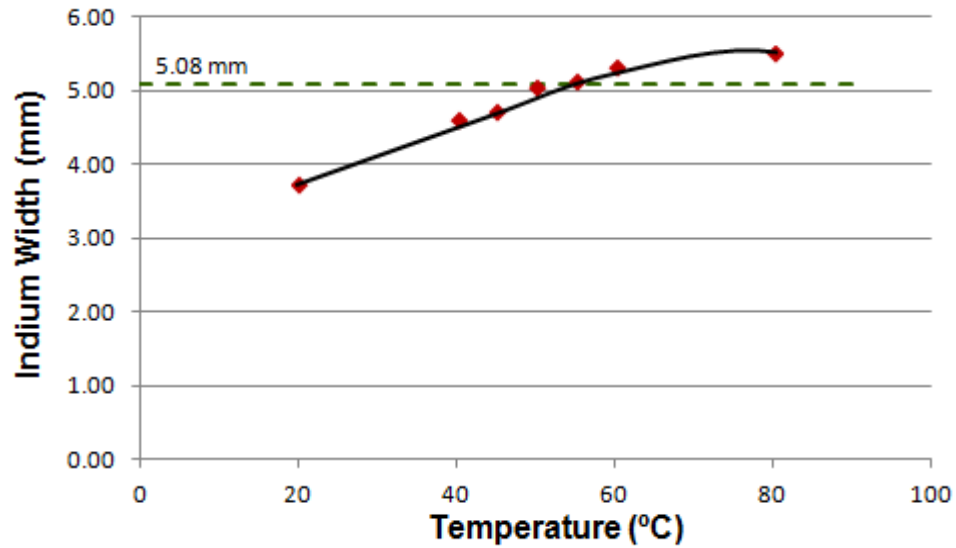


Figure 29 - The width of the indium seals in relation to the processing temperature. Horizontal line indicates a width of 5.08 mm, the width of the sidewall. Applied load of 1180 N and a load duration of 11 minutes were used.

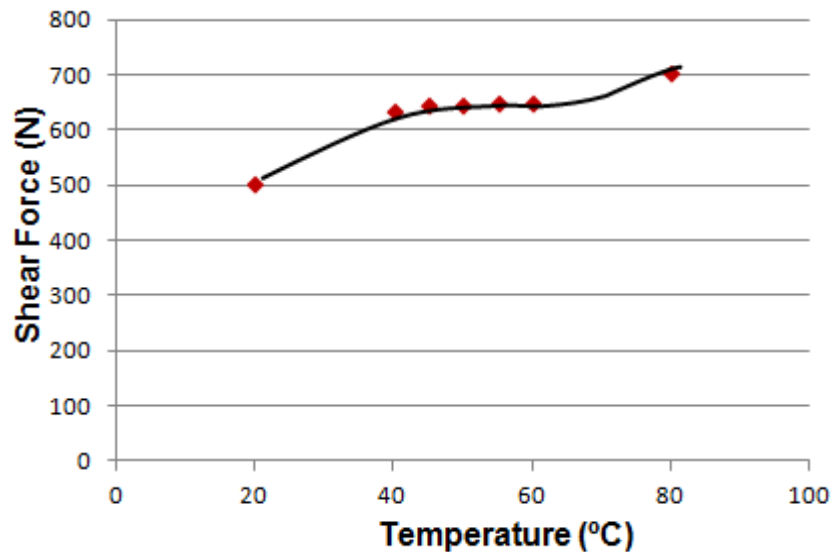


Figure 30 - Relationship between the force to shear failure of the seal and the processing temperature. Applied load of 1180 N and load duration of 11 minutes were used.

duration of 11 minutes. Interestingly, the width of the seal made at room temperature had a seal width of 4.69 mm compared to 5.06 mm of the sample made at 50 °C. This would indicate that temperature is detrimental to sealing to glass in air. The cause is likely because of the increased kinetics of oxide formation over the sealing duration. Therefore, it would be more efficient to increase the applied load instead of increasing the fabrication temperature.

4.2 Sealing Nichrome-Coated Glass Substrates

Several samples were fabricated using glass substrates coated with a thin layer of nickel/chrome alloy of composition 80 wt% nickel and 20 wt% chromium. Figure 31 shows the comparison of force to shear failure between seals made with 1180 N and 1558 N applied loads while at a constant manufacturing time and temperature of 11 minutes and 20 °C respectively. An island of indium that remained on the surface of a nichrome coated sample after shear testing is seen in Figure 32. These results were compared to seals made using uncoated glass from the previous section. Trend lines indicate that the average increase in force to shear failure of parts coated in nichrome is 54% with increasing the applied load from 1180 to 1558 N. The increase in shear force is due to the increase in bonding area as it was shown in the previous section that increasing the applied load increase the width of the tracks. Figure 31 also indicates that there is nearly no difference in the shear force required for breaking the seals between the uncoated and

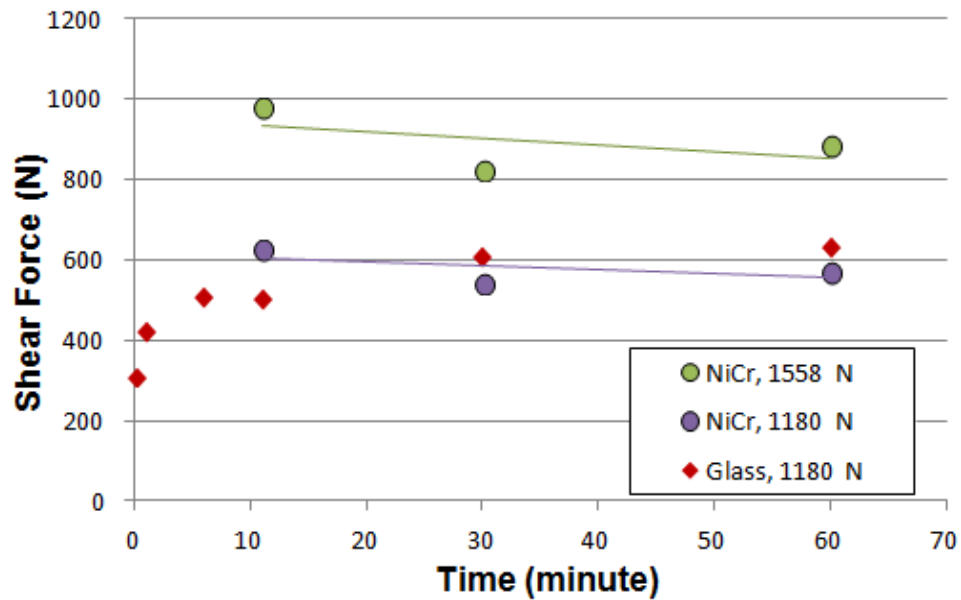


Figure 31 - Correlation between manufacturing times and force to shear failure of samples which are uncoated and coated in 80/20 nichrome. Processing temperature was held constant at 20 °C.

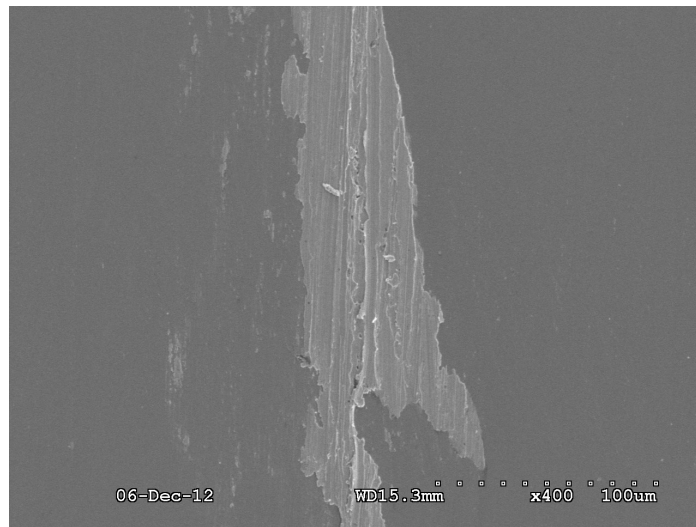


Figure 32 - Scanning electron image of an island of indium left on a nichrome surface after shearing. EDS analysis of this region was inconclusive on discovering new phases forming between the indium and the nichrome.

coated parts made with a load of 1180 N. Since all process parameters other than the coating are the same between these two sets of data, it is reasonable to assume that the area of the seal is approximately the same between the glass and NiCr samples. And the bonding strength is nearly the same meaning that it is reasonable to infer that the mechanism for bonding indium to nichrome could be the same as the mechanism for bonding to glass, i.e. an oxide bond. This is highly possible since it is well understood that a layer of chromium oxide will form on the surface of the nichrome when exposed to atmosphere.

The influence of temperature on the sealing of nichrome coated parts was

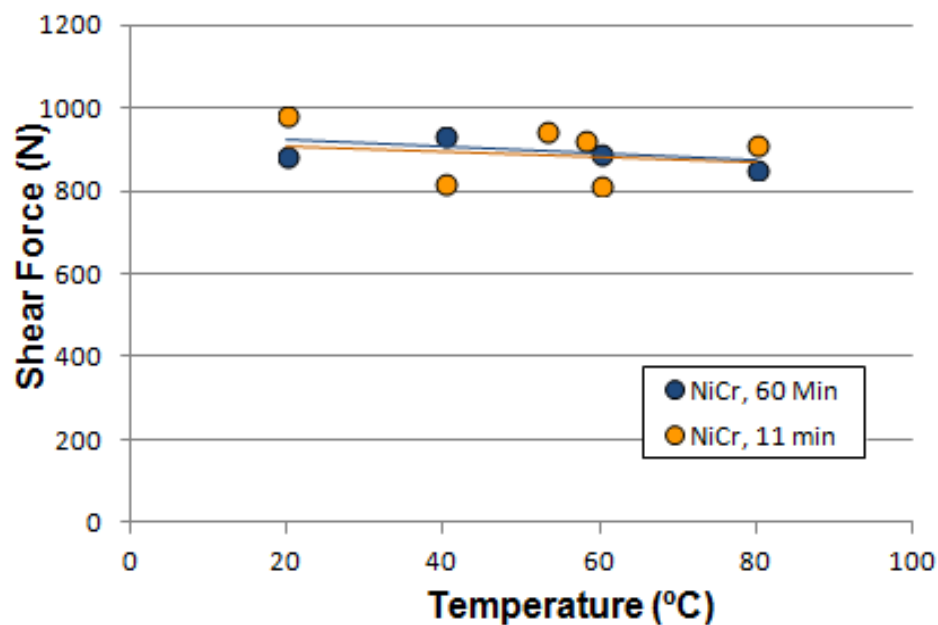


Figure 33 - Correlation between process temperatures and force to shear failure of samples which are coated in 80/20 nichrome. Applied load was held constant at 1558 N.

investigated next. The plot in Figure 33 compares the force to shear failure values of samples made with a manufacturing time of 11 and 60 minutes over the range of 20 to 80 °C. The applied load was constant at 1558 N for all samples. Both trend lines indicate that increasing temperature during manufacturing does not significantly affect the shear force at both 11 and 60 minute manufacturing times. These results are not as expected. It was initially thought that increasing either the manufacturing time or temperature would increase the kinetics of the chemical reaction occurring at the surface leading to higher bond strengths. It appears that any reaction that would take place over a span of one hour actually occurs in less than 11 minutes.

Next, it is necessary to simulate the extended life of the seals and induce the formation of intermetallic compounds by aging at elevated temperatures. Four nichrome coated samples were aged in air at 140 °C (the melting temperature of indium is 157 °C) for 25 days. The surface of the nichrome sample that has been aged and shear tested is shown in Figure 34. It appears that the aging increased the amount of indium which remains on the nichrome surface after shearing, particularly in the center of the seal where the indium oxide has not been broken. Figure 35 compares the aged samples to samples which were shear tested immediately after fabrication. The manufacturing load, duration and temperature for all samples were 1558 N, 11 minutes, and 20 °C respectively. Aging improved the average shear force by only 7.8 N. Although the increase in shear force is small, it is important to note that there was no critical decrease in strength.

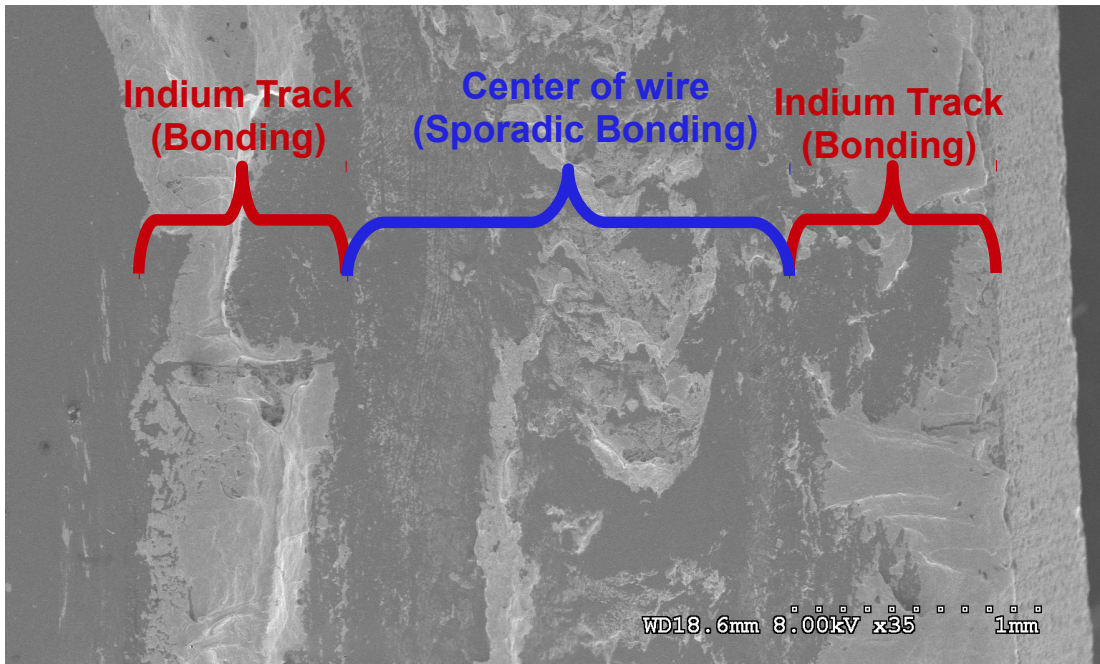


Figure 34 - SEM image of an aged nichrome coated sample after shear testing. Indium tracks remain on the surface as well as some periodic bonding in the center of the wire.

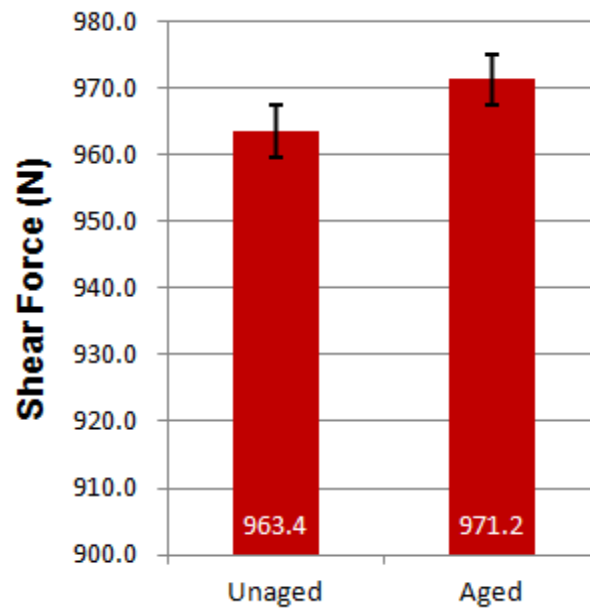


Figure 35 - The relationship between the average force to shear failure and aging condition of nichrome coated samples. Aging duration was 25 days at 140°C. 1558 N, 11 minutes, and 20 °C were the processing parameters for all samples.

EDS, XRD, and X-ray photoelectron spectroscopy (XPS) were used in an attempt to identify any new phases which may have formed. Typical metallographic procedures of sectioning and polishing the samples were attempted but to no avail. The dramatic difference in hardness between the glass and the indium lead to chipped glass and smeared indium at the interface after polishing. Analysis had to be done on the tracks of indium from the top-down perspective after the samples had been sheared apart. An SEM image of the area studied is shown in Figure 34. EDS was unable to clearly identify any changes in the composition that would indicate the formation of new phases. Similarly, XRD was not able to identify any new phases. It is possible that the phases which formed are too thin to be detected or that the signal was drowned out by the bulk material. A survey scan (Figure 35) of XPS did reveal anomalous peaks which could pertain to an interaction between the indium and the nickel. Figure 36 shows the In3d peaks in the XPS spectrum taken of the sample in Figure 34. It was expected that there would be peaks for pure In and for In_2O_3 , the most stable form of indium oxide. However, there is also a peak associated with an unknown state of In. The XPS database is unable to confirm which compound this peak is linked to. It is logical to speculate that this peak could be related to either an In-Ni or In-Cr compound. Looking at the peaks for Ni2p in Figure 37, we can see that there is also an anomalous peak which was not identified by the XPS database. It can be speculated that these two peaks may be related. This assumption can be enforced by studying the Cr2p peaks in Figure 38 which does not show the presence of any unknown peaks. However, it is possible that the Ni2p and Cr2p orbitals are so convoluted that

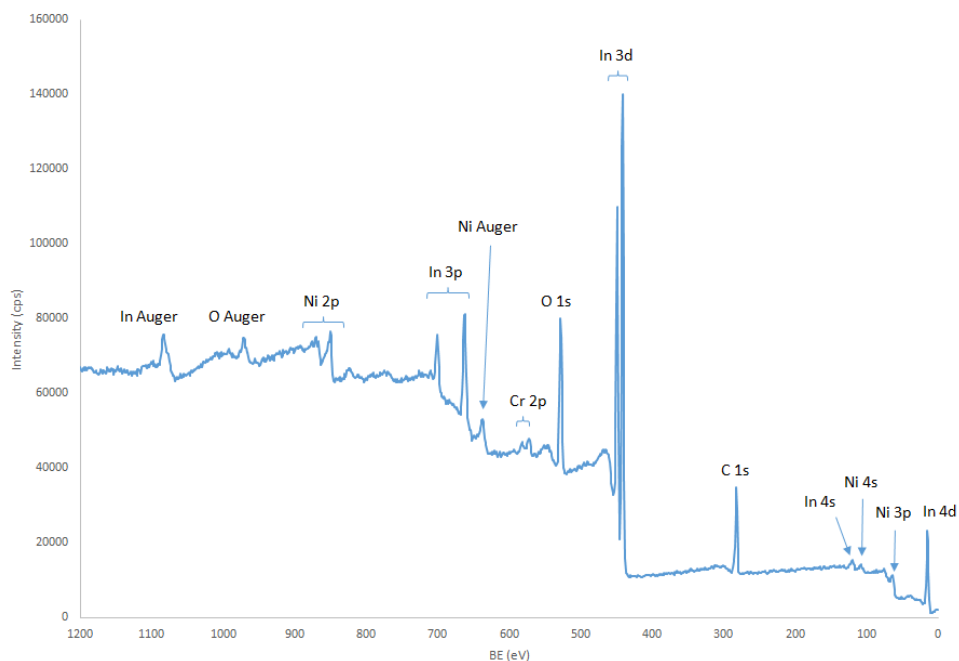


Figure 35 - XPS survey scan of an indium the indium track shown in Figure 34.

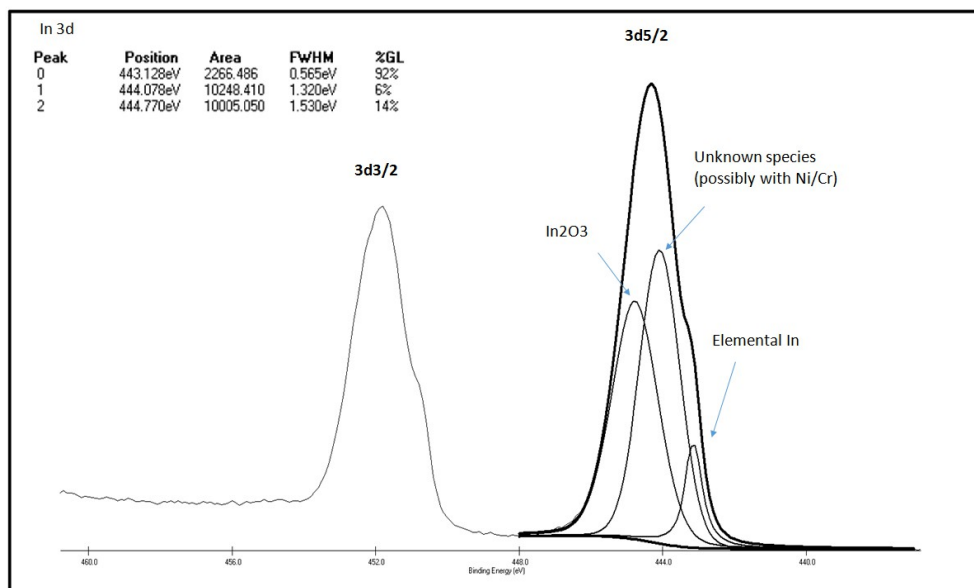


Figure 36 - Indium peaks of the XPS spectrum taken from the specimen in Figure 34. Two stable phases of pure In and In_2O_3 are seen as well as an anomalous third peak which could be related to a compound formed with Ni or Cr.

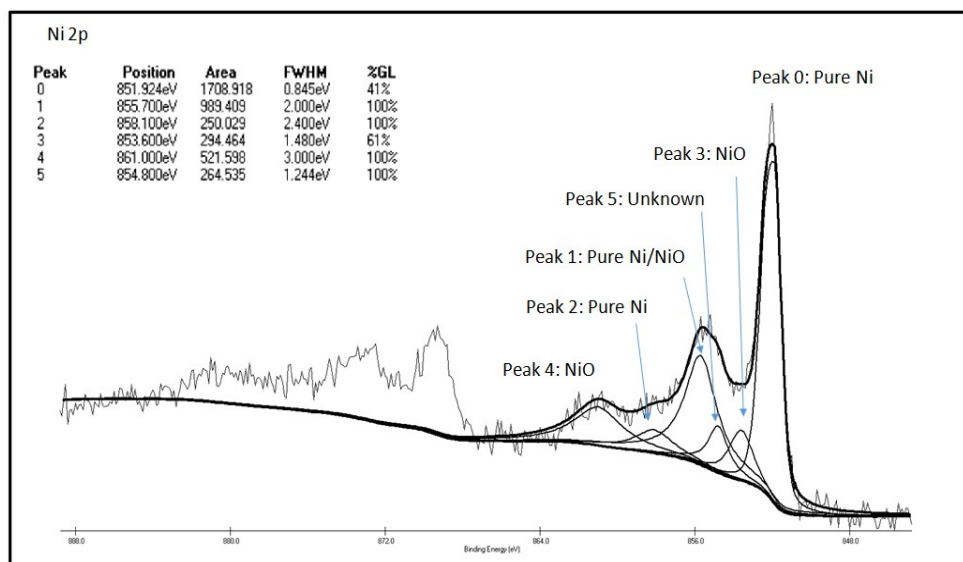


Figure 37 - Nickel peaks of the XPS spectrum taken from the specimen in Figure 34. One anomalous peak was found which could be related to the unknown In peak in Figure 36.

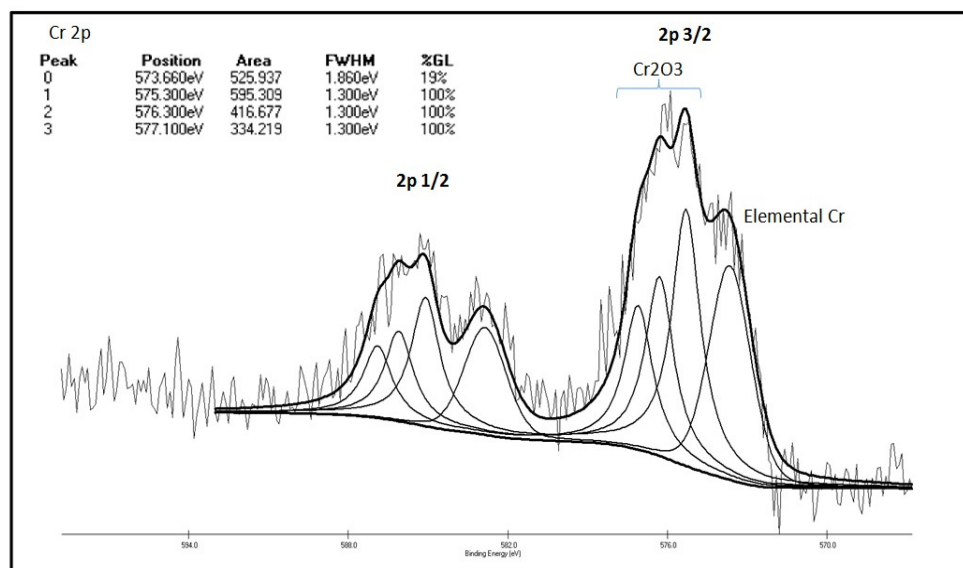


Figure 38 - Chromium peaks of the XPS spectrum taken from the specimen in Figure 34. All peaks have been identified as pure Cr or Cr_2O_3 .

smaller peaks are drowned out by the larger peaks meaning that many compounds may exist but are not being detected. In order to confirm the formation of a In-Ni compound, more thorough and detailed XPS scans are required.

5. CONCLUSIONS

1. Testing has shown that indium can seal borosilicate glass packages at room temperature. Increasing the applied load increased strength of the bond. Time also appears to improve strength while only marginally increasing the width of the seal when sealing for longer than 6 minutes. Increasing temperature appears to cause a weaker bond than increasing applied load when seal widths are similar.
2. Pure indium can effectively seal glass substrates which have been coated in a 80 wt % Ni/20 wt% Cr alloy. Increasing applied load increased bonding strength. Increasing manufacturing time did not appear to have an affect on sealing to NiCr. Increasing temperature was not detrimental to seal strength.
3. EDS and XRD were not able to detect the formation of intermediate phases or intermetallic compounds between the indium and NiCr. XPS data was used to speculate that a compound formed between In and Ni, and not between In and Cr. Further investigation along these lines is necessary to fully understand the mechanisms for bonding.
4. Based on this investigation, the optimal parameters to use for sealing a 2.54×2.54 cm nichrome-coated glass package are: an applied load of 1558 N with a processing time of 11 minutes at 20 °C. Using 1558 N on a 2.54×2.54 cm sample is equivalent

to 195 N/cm of indium wire (approximately 8 cm of indium gasket). To scale up to a 22×22 cm package with a gasket length of 86 cm, an applied load of 16770 N should be used.

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