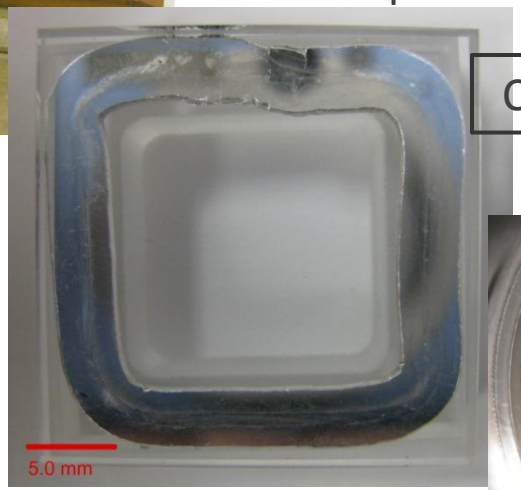
Status Update for Indium Cold Sealing Process

Marc E. Kupfer
University of Illinois at Chicago
Dean R. Walters
Argonne National Laboratory

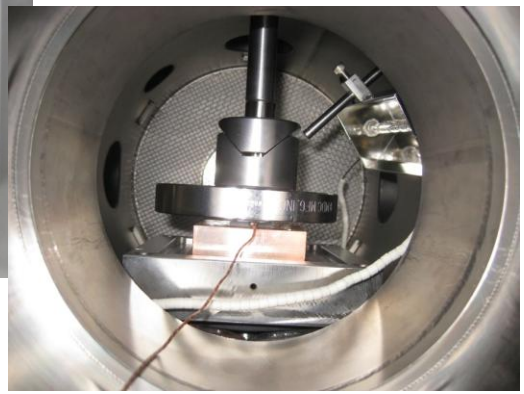
Seal Fabrication - 1 inch

Hydraulic System

- Capable of 3000+ lbs pressing force
- Heated platen and internal quartz halogen heating bulbs
- High vacuum (~10^{-7} torr)
- Laser displacement sensor to measure compression
- Fabricates both 1 inch and 4 inch samples



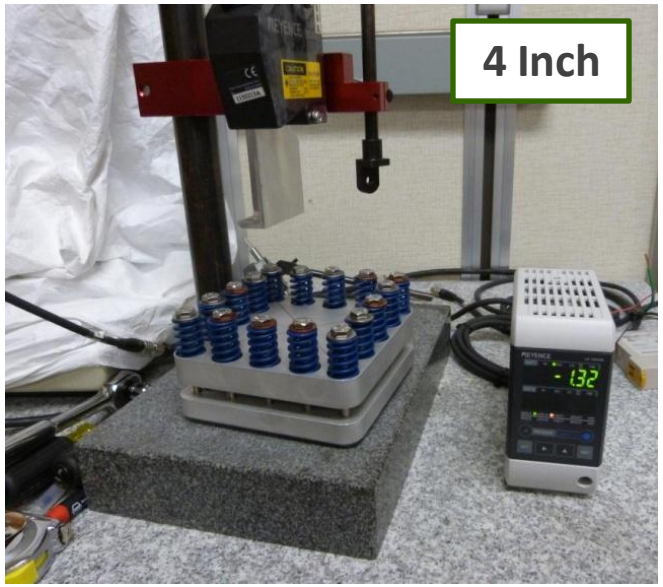
Compressed 2.0mm wire

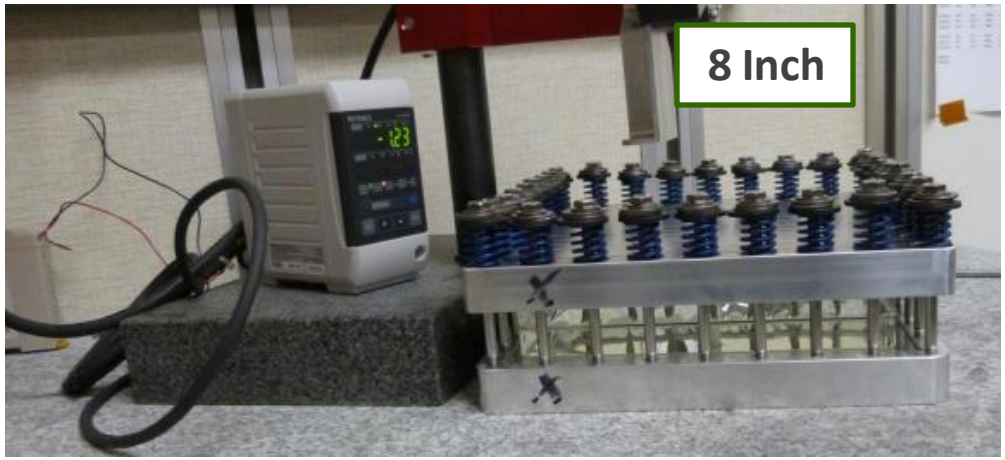


Fabrication process has been optimized over time to consistently make leak-tight 1 inch seals using uncoated glass parts.

Seal Fabrication - 4 and 8 inch

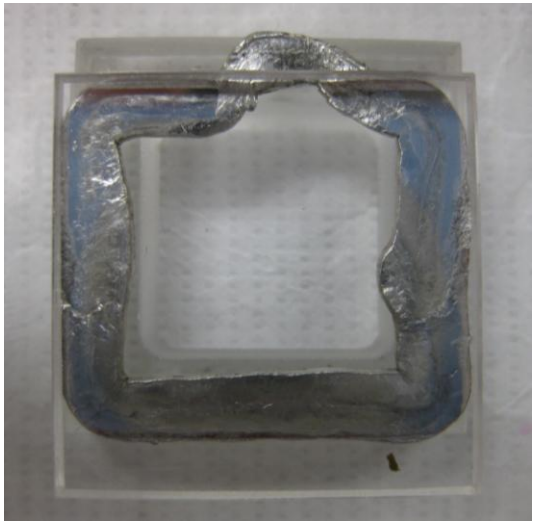
- 4 inch and 8 inch seals are made by the use of a “spring press.”
- Fabrication process uses two heavy aluminum platens compressed together using bolts and springs.
- Compression is controlled by laser measurement of the change in thickness of the indium.
- This method has the advantage of holding samples under compression for extended periods of time. Spring press can also be placed inside an over while compressing.

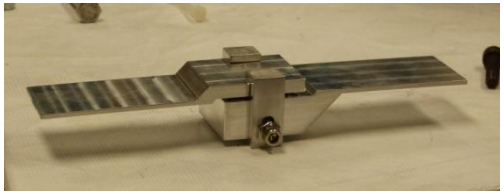




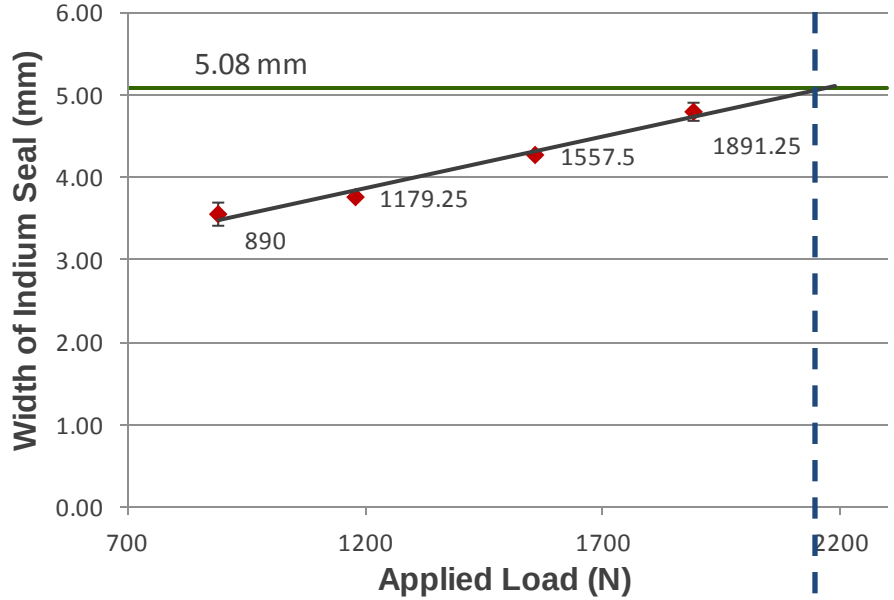
Seal Characterization

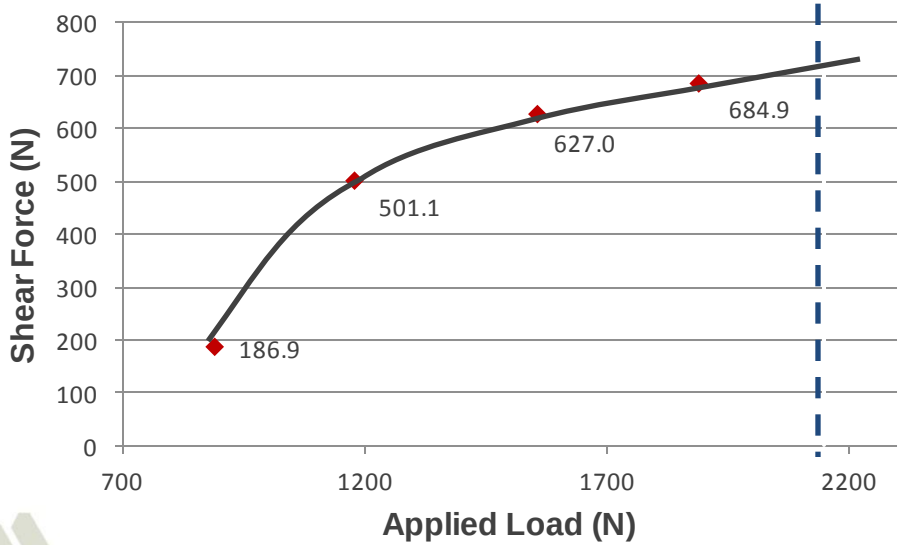
- Hermeticity is measured using an Alcatel helium detector. Seals are considered leak tight if the base reading is $<10^{-10}$ ccm of He per second and there is no response from the gauge when the sample is hit with helium.
- The strength of the seal is measured using a lap shear test.





Analysis of 1 inch Data

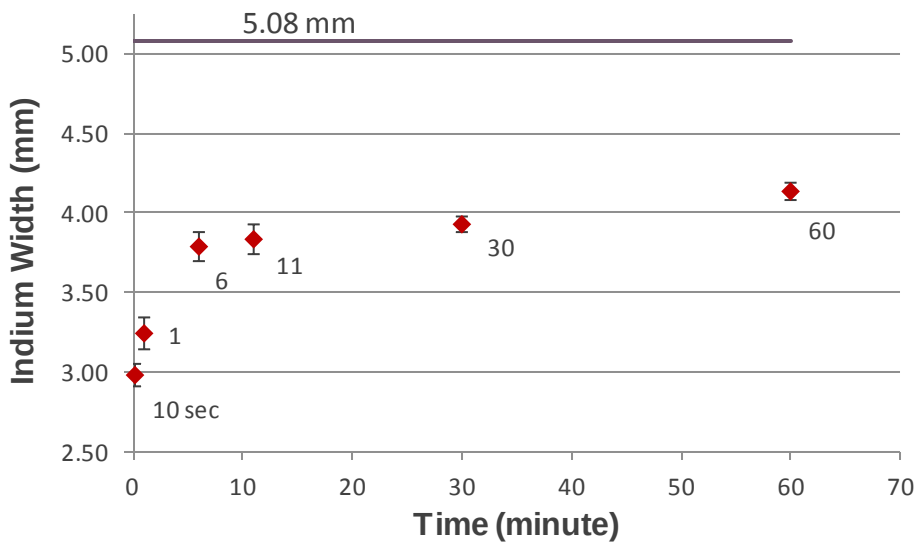


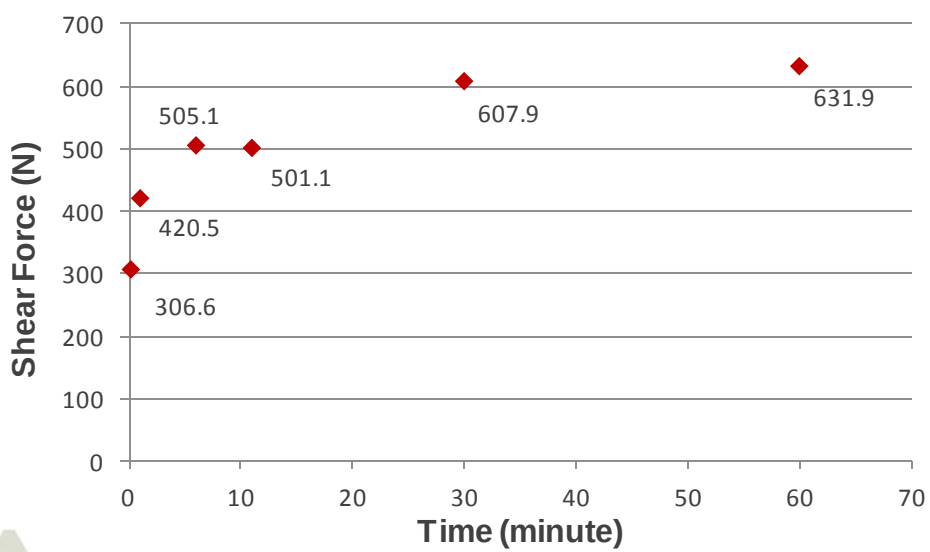


- There is a linear correlation between the width of the indium seal and the applied load. (Temperature = 20ºC, time = 11 minutes)
- This correlation shows that in order to cause the indium gasket to flow to the entire width of the sidewall, a compression force of 2150 N (483 lbs) is needed.

- There is a general increase in force to shear failure with the increase in applied load.
- This trend suggests that the maximum strength achievable with the given fabrication procedure with uncoated glass is approximately 720 N.

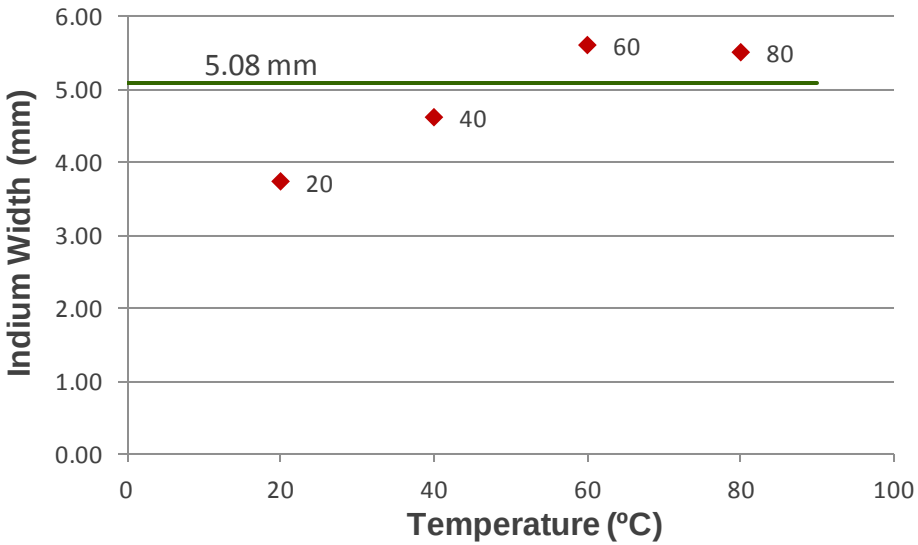
Analysis of 1 inch Data

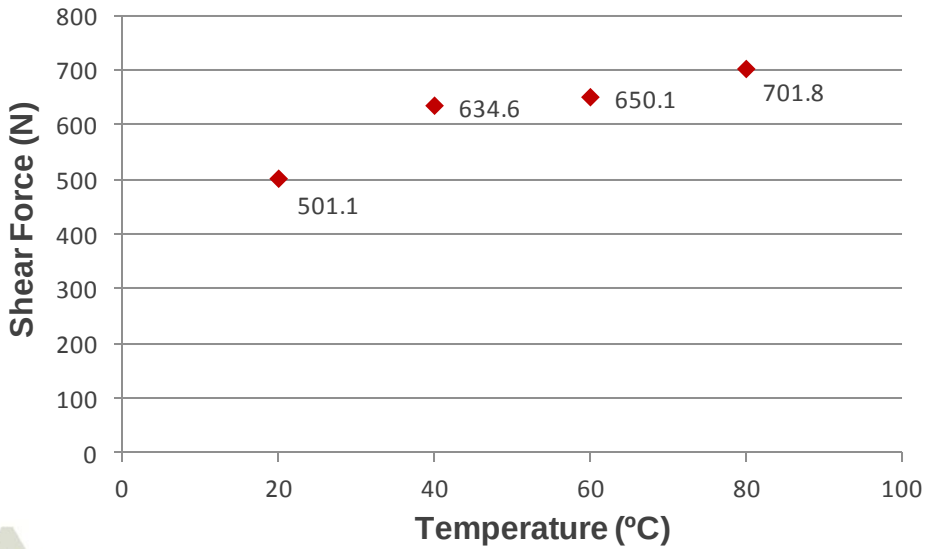




- Comparing the width of indium to the sealing duration shows:
  - 90% of compression will occur after 6 minutes.
  - Indium will continue to flow while under compression.
- Increasing the time from 11 minutes to 60 minutes did not cause the indium to flow enough to fill the entire sidewall.
- Increasing time also did not sufficiently increase the sealing strength. A greater increase was seen when increasing applied load.

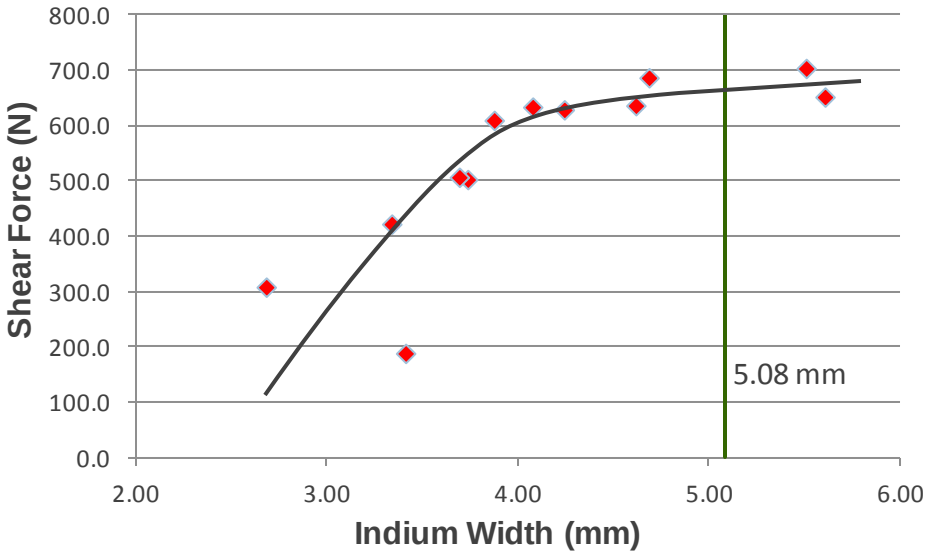
Analysis of 1 inch Data





- Increasing temperature of sealing had a great impact on the width of the indium. Interpolation predicts that only 50 ºC is needed to cause the indium to flow to the thickness of the sidewall in 11 minutes.
- The greatest increase in strength was between 20 ºC and 40 ºC showing that increasing the temperature by any amount will increase the strength of the seal.
- The width at 80 ºC was less than that at 60 º, however the strength is greater. This is evidence that temperature improves the bond as well as the making the indium more malleable.

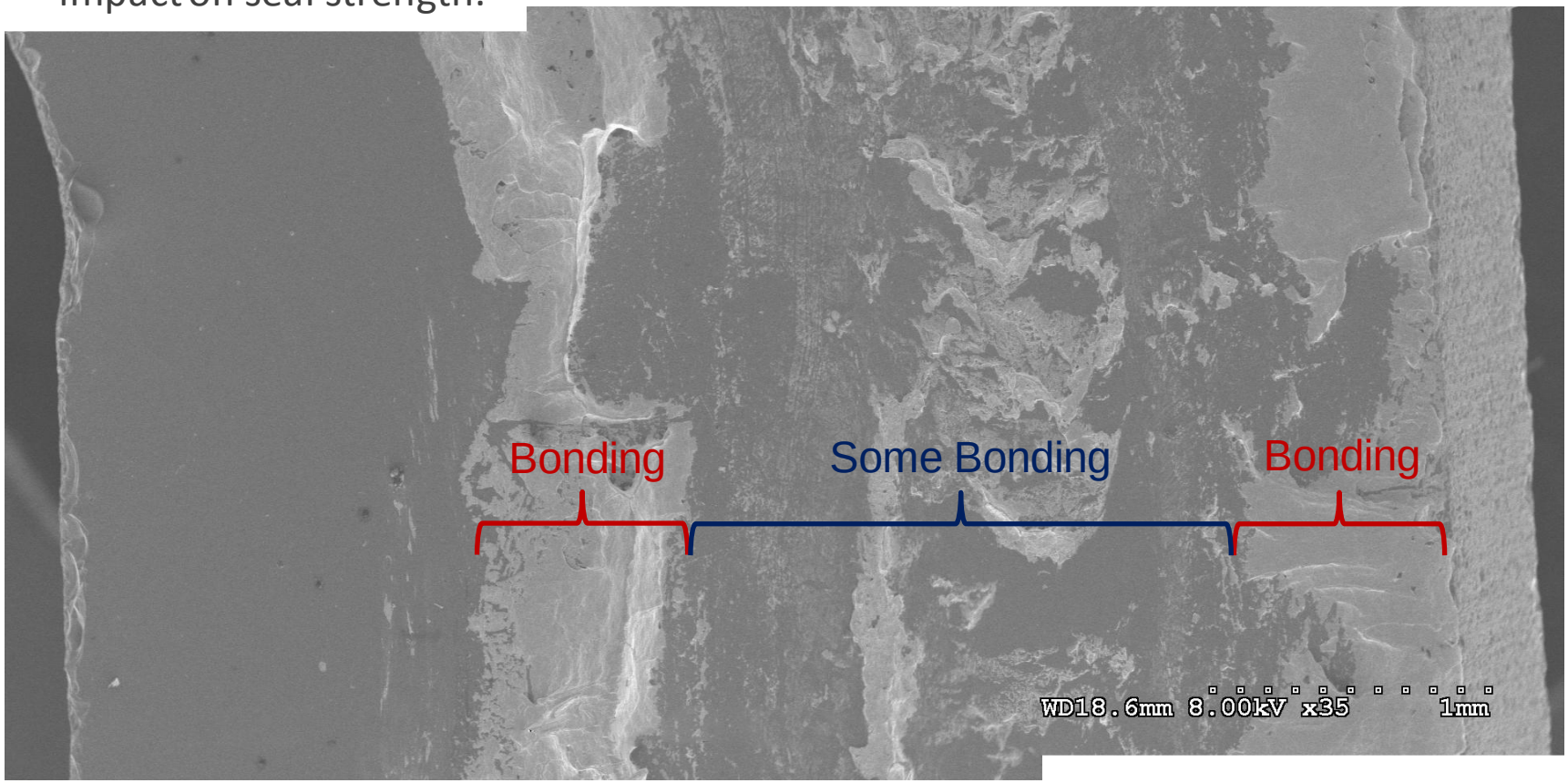
Analysis of 1 inch Data



- This plot combines the data points of all tests to show the correlation between indium width and bond strength.
- Shear force appears to be asymptotic to approximately 700 N.
- It is possible this limiting nature is due to surface oxides on the indium. As the indium flows under compression, the oxide will continue to break and spread out along the bond area.

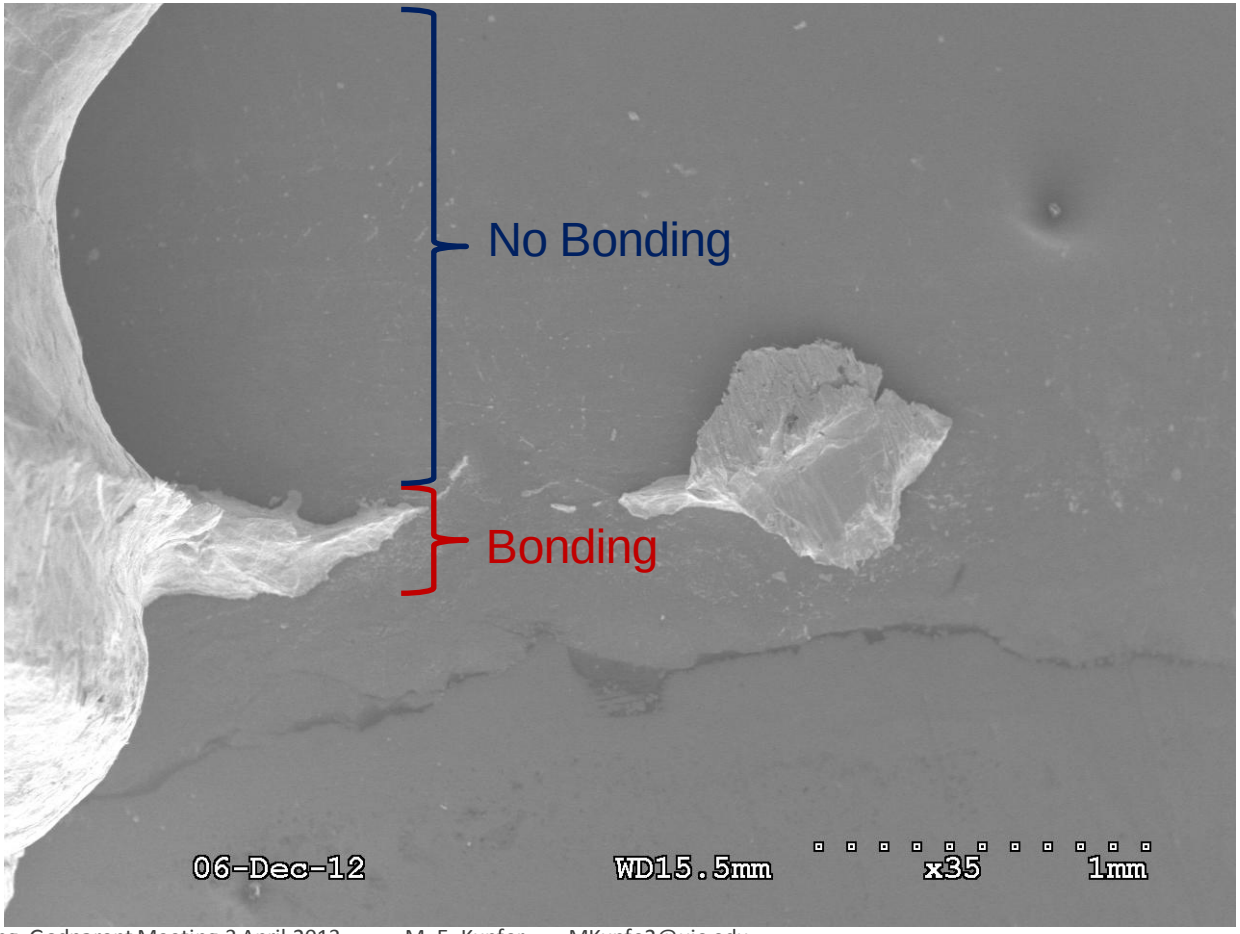
Inspection of Seals - SEM

- Glass coated in NiCr. Sample aged 7 days at 80 ºC and then 9 days at 130 ºC.
- Shear testing revealed bonding at the two outside edges of the indium, as well as some in the middle.
- Few samples were tested, however it was seen that aging NiCr samples had a great impact on seal strength.



Inspection of Seals - SEM

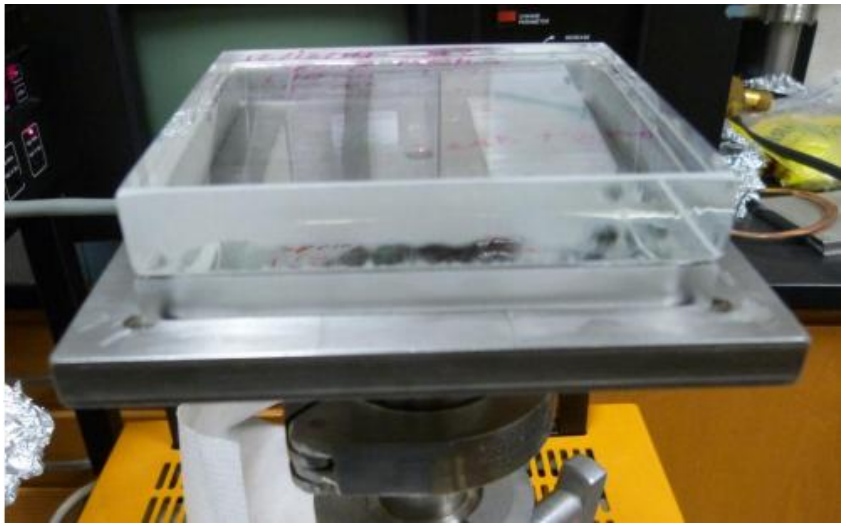
- Glass coated in Cr. Sample aged 7 days at 80 ºC and then 9 days at 130 ºC.
- Clear evidence that bonding occurs only at the outside edges of the indium.
- The strength of this seal was not significantly greater than un-aged Cr samples.



Leak Checking of 4 inch Seals

- Initial leak checking of 4 inch samples was difficult. Several types of spacers were used in an attempt to mitigate the deflection of the window while under vacuum.
- Most samples appeared to be leak tight at first, only to have leaks forms at the corners during testing.

- To remove the problem, the 4 mm thick window was replaced with a 16 mm thick window to reduce the deflection.
- Since making the switch, no leaks have formed in the corners. (Leaks have been found in other places.)

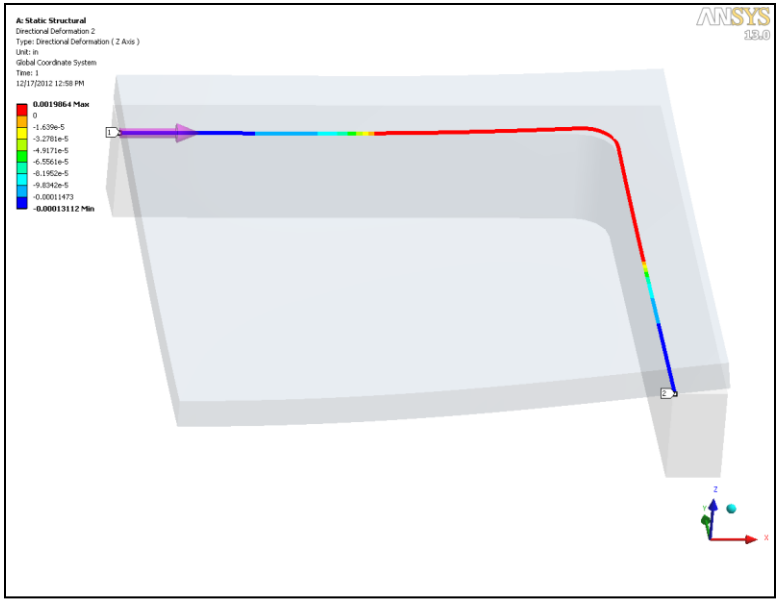


Leak Checking of 8 inch Seals

- The same issue was occurring with the 8 inch seals. Finite element simulation of the seal shows that when the window bows downward under vacuum, the corners lift up, breaking the seal.
- 16 mm thick plates were used to prevent this deflection in order to move forward with experimentation.

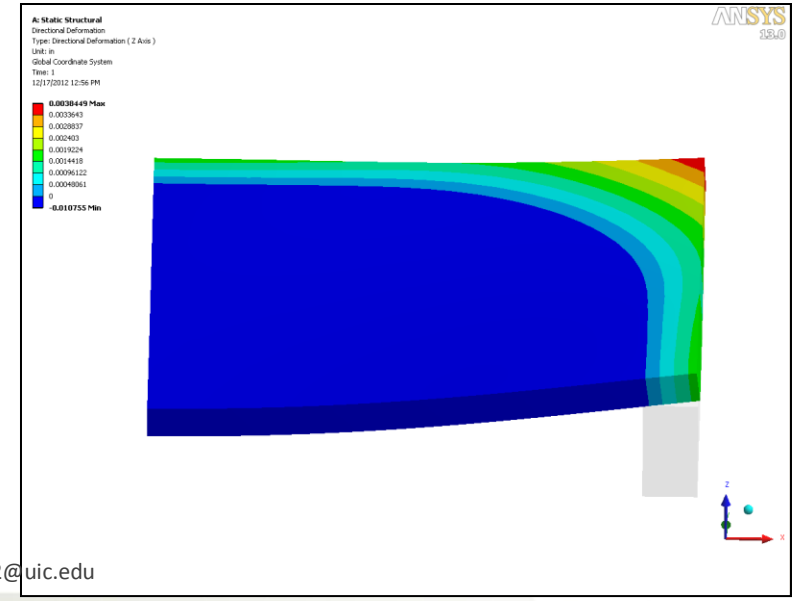
FEA of 8 inch Seals

Glass on glass, 1/8 inch corner radius



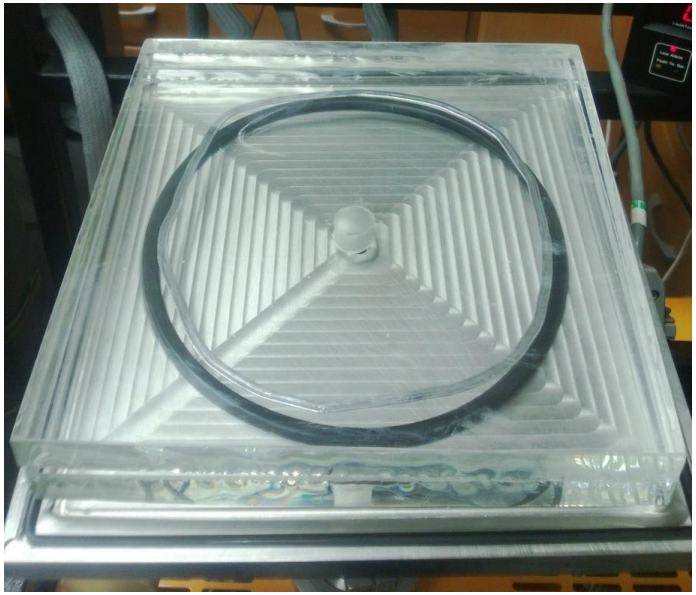
- Z Deflection on plate at inside edge of wall.
- Red is >0.

- Z Deflections
- Blue is <0



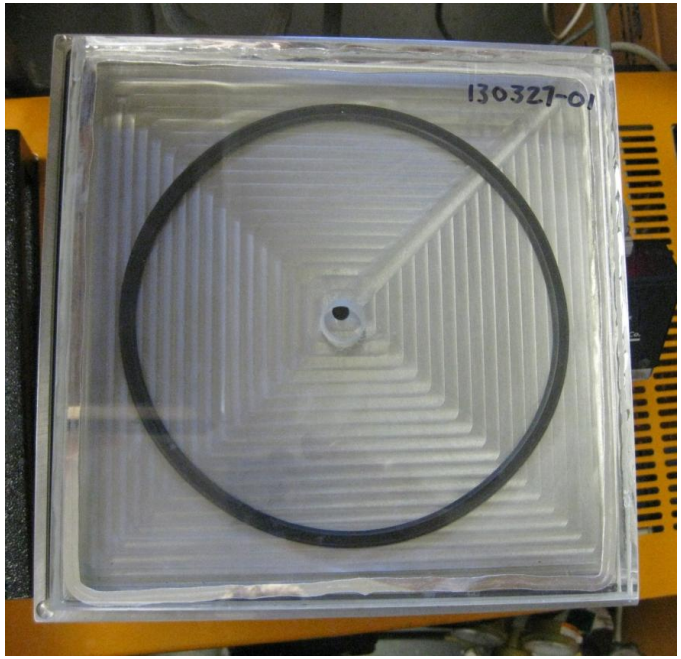
Progression of 8 inch Seals

- Using the 16 mm thick windows, seals were made with large radii corners, working progressively to tight corners to match the width of the sidewalls.
- First seal made was a 14 cm diameter circle gasket. Sample was held under vacuum for 15 days, periodically being exposed to air and pumped down again.

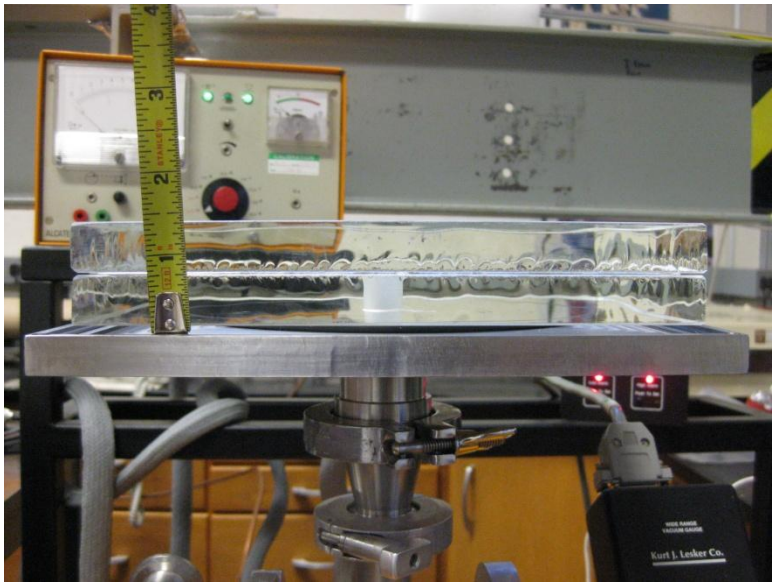


- For the following seal, the radius was increased to 19 cm.
- This seal was held for 12 days at vacuum with periodic vacuum cycling. It remained leak tight over the entire duration.

Progression of 8 inch Seals



- Most recently, a seal was made with corners that match the radius of a typical sidewall, ~6.4 mm.
- After 8 days under vacuum, the part remains entirely leak tight.



Conclusions

- Gained an understand how process parameters affect the thickness, width, and strength of the seal. Two good places to go further:
  - 20 ºC, 1890 N applied load, 11 minutes
  - 50 ºC, 1180 N applied load, 11 minutes
- Consistent success making 8 inch seals using 16 mm thick glass plates.

Future Work

- Complete work with NiCr coated 1 inch parts.
- More work with NiCr coated 4 inch plates, moving to parts with actual sidewalls.
- Making 8 inch seals with fritted anode/sidewall and rigid internal supports (no MCPs)
- Considerations for other indium alloys.