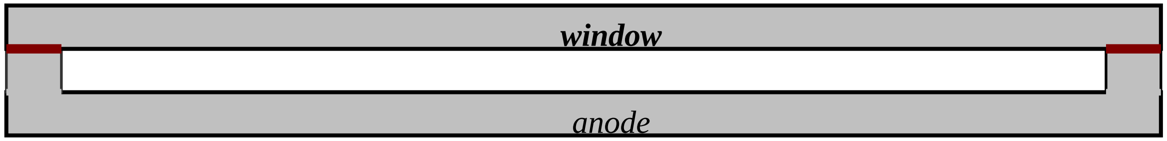
Low Temperature Thermocompression bonding for photodetector sealing



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Argonne National Laboratory
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JSAMRL
Joining Science and Advanced Materials Research Laboratory

The University of Illinois at Chicago

Outline

- Objectives.
- Background
  - Technical issues
  - Sealing technique selection
  - Indium oxide layers
- Experimental Approach
- Preliminary tests
- Remarks

Objectives

- Develop a ***sealing*** for the photodetector that is ***hermetic*** and ***mechanically*** sound.
- Implement a ***low temperature*** (< 150°C) bonding process by thermocompression based on ***In*** and ***In-based alloys***.
- Evaluate initial ***aging*** characteristics of the ***sealing*** material.

Background -- Technical issues

- Bonding of dissimilar materials:
  - Wetting of glass surface by metal (liquid): difference in surface energies.
  - Alteration of the glass surface:
    - Metallizing of borosilicate with Cr coating.
  - Interaction of solder with the Cr coated glass (***adhesion layer***).
- Solder composition: ***In*** and ***In-based***.
- Metallurgical reactions between bonding layer (sealing) and adhesion layer.
- Aging and degradation of sealing:
  - Short term aging.

Background -- Thermocompression

- Bell Laboratories (1950s) bonded Au to Ge:
  - Applied pressures: 5,000-10,000 psi.
  - Applied temperature = 250°C,
  - Note: Au-Ge eutectic = 356°C.
  - This is a solid state diffusion bonding.

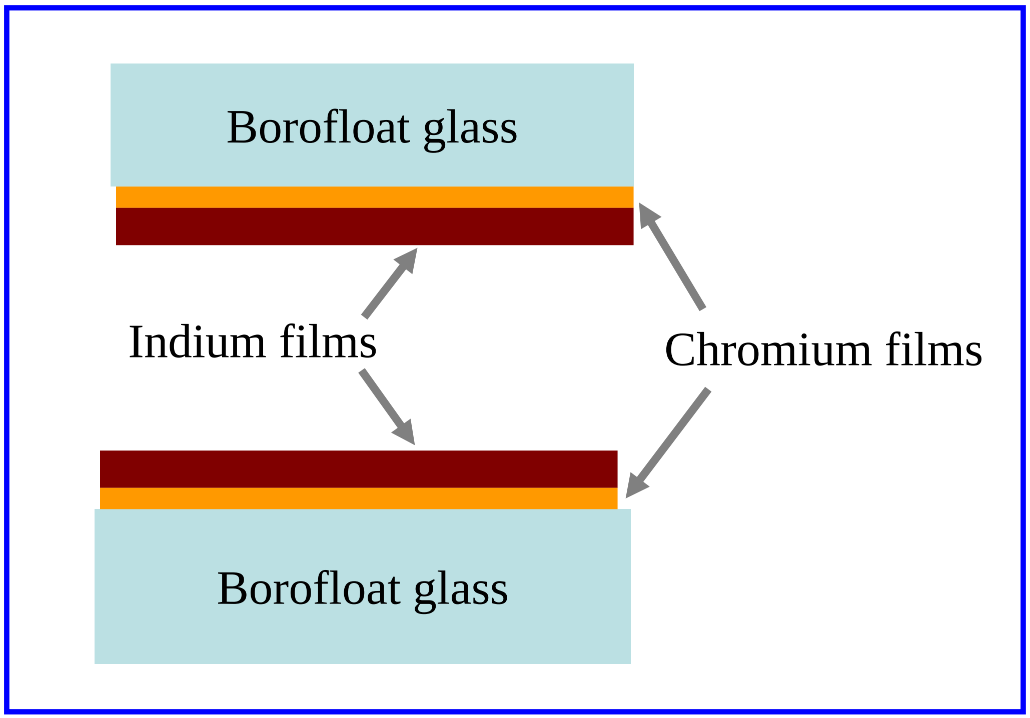
J. Applied Physics, vol. 28, August 1957

- University of Glasgow (1970) reported:
  - Thermocompression bonding of sapphire.
  - **Indium** layer deposited (0.5 µm)
  - Sapphires coated with 0.1 µm thick **Chromium** layers
  - Process:150^{o}C for 24 hours at 400 psi pressure .

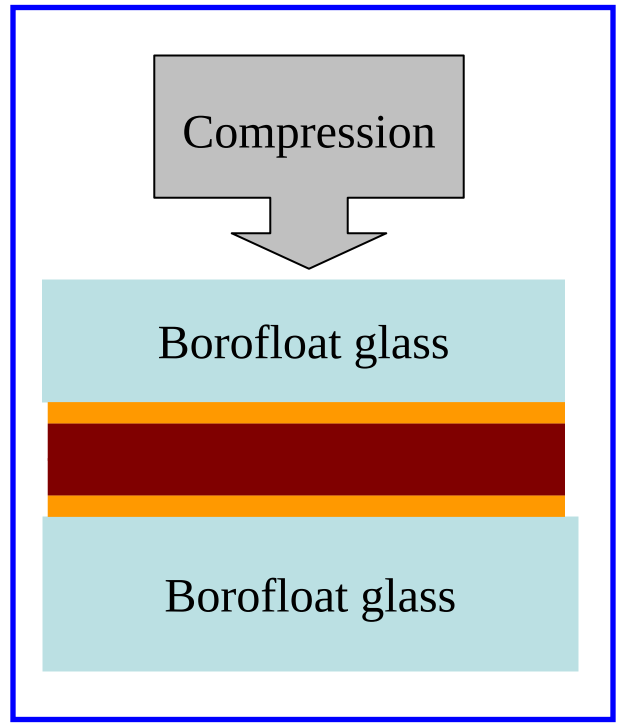
J. Phys. E: T Sci. Instrum. 3, 740, 1970

Background --
Indium Sealing (Thermocompression)

- It is based on the ability of ***In*** to ***cold weld*** itself,
- May require a mild pressure to join the ***In*** surfaces together.



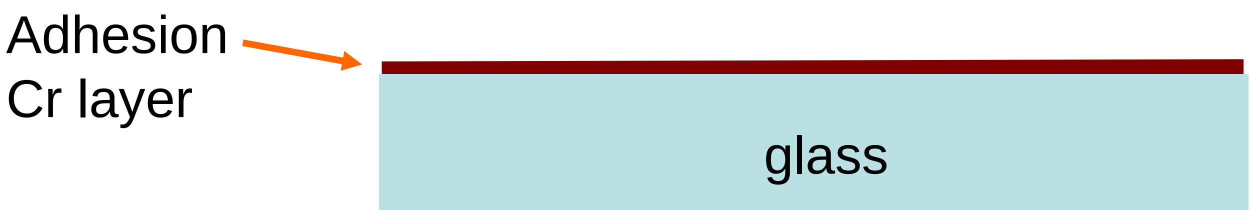
Step 1



Step 2

Background --
Glass Metallization - *Adhesion layer*

- ***In*** does not bond directly to the glass.
- Most common approach to change the surface chemistry of the glass is to ***metallize*** it.



- ***Cr*** is most common for the adhesion layer.
- Ti and Pt are other materials used.
- Adhesion layer acts as oxygen-barrier and forms the bond with the solder.

Background --
Glass composition

- Borosilicate glass (B33 - window):

  81.0 wt% SiO_2

  13.0 wt% B_2O_3

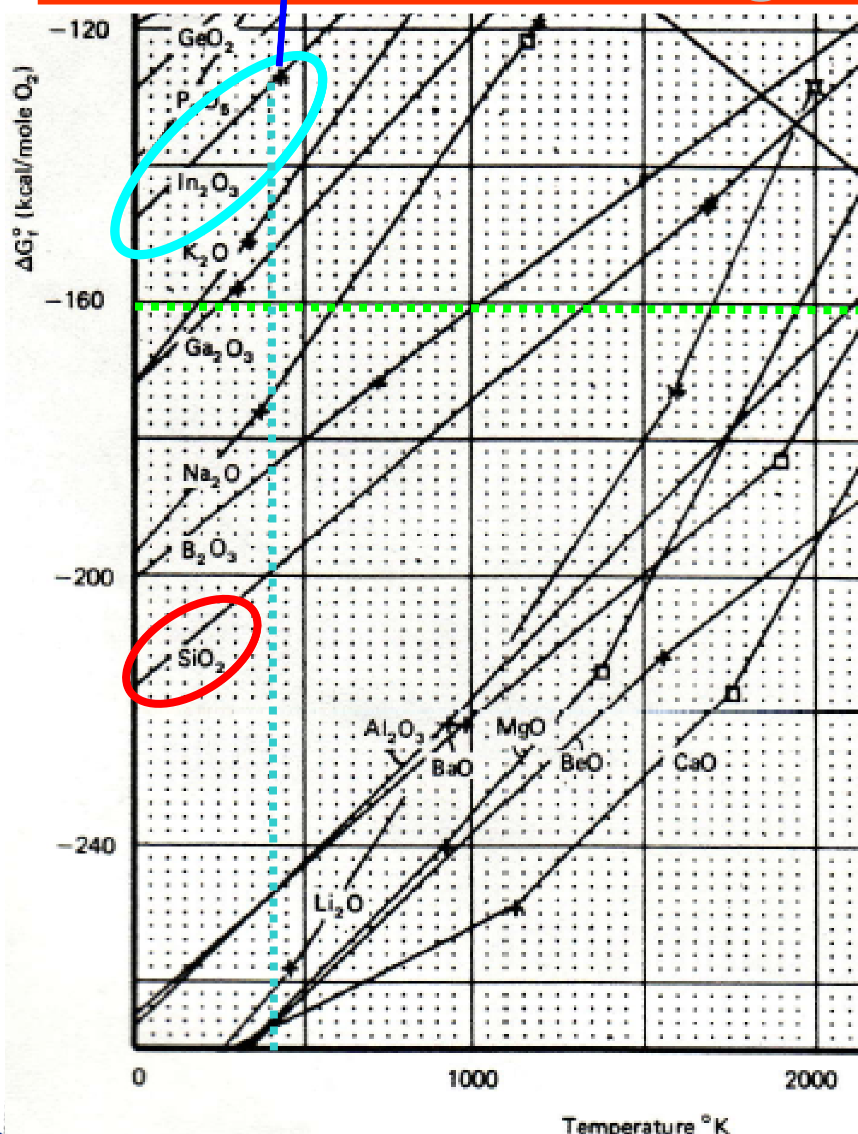
  4.0 wt% $Na_2O + K_2O$

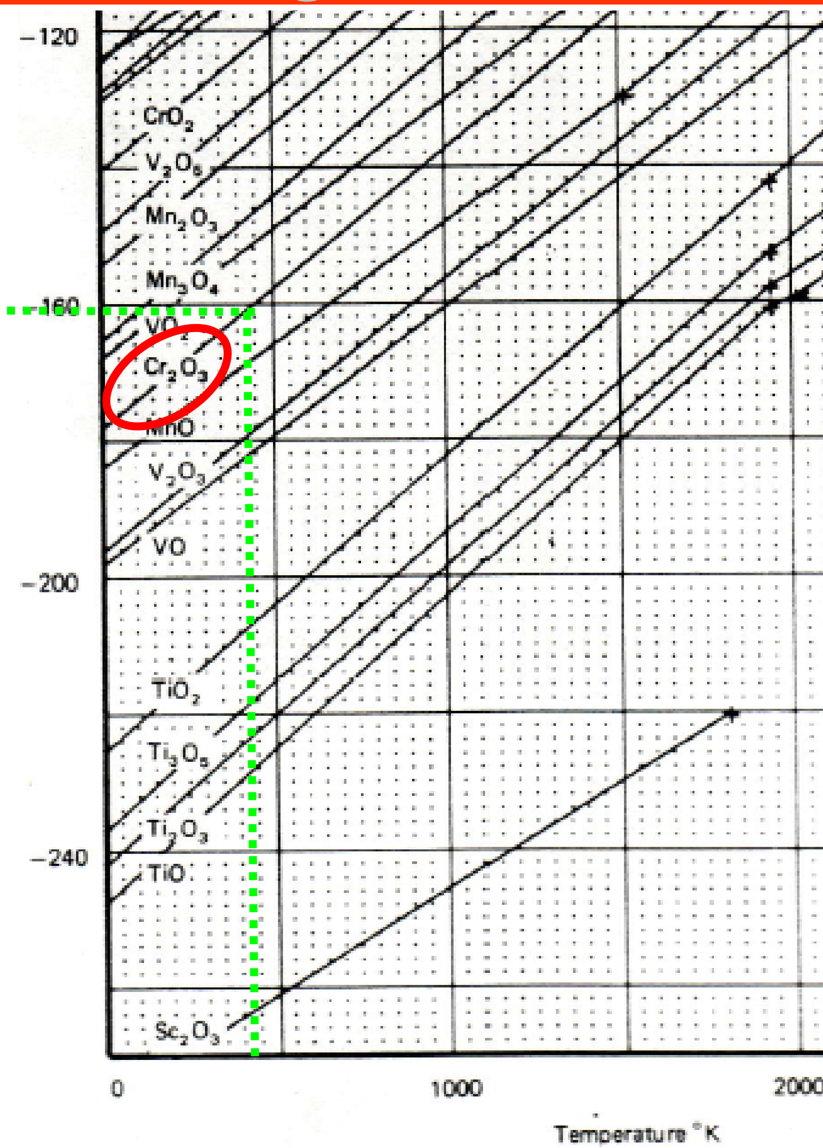
  2.0 wt% Al_2O_3.

- Filler metal: ***In***

Background -- *Ellingham Diagrams*

In m.p. 429.7 K (156.7º C)

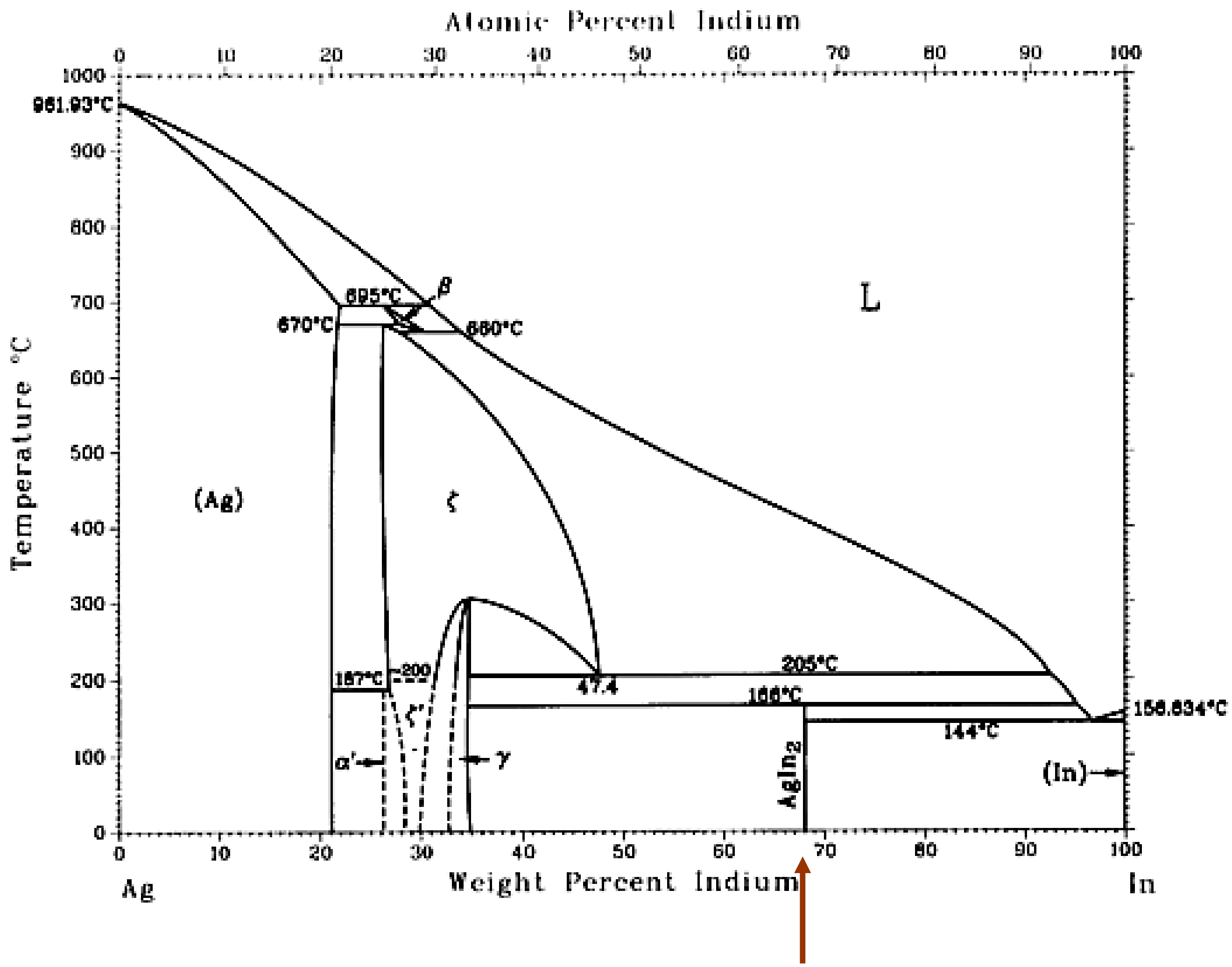




Background -- Other Bonding Techniques: *Fluxless glass-to-glass bonding*

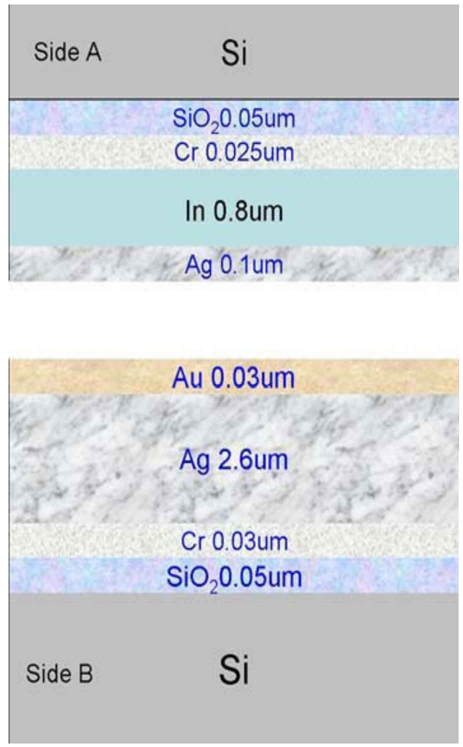
- UC-Irvine's work was similar to UC-Davis (UC-Davis used Au):
  - ***In** was **melted** in both cases.*
  - *UC-Irvine at 157°C and UC-Davis at 250°C*
- UC-Irvine considered the following:
  - In diffuses into Ag (D_o= 2.4 x 10^{-12} m^2/s).
  - The ***$AgIn_2$ intermetallic*** compound show stability even after 15 years at room temperature (prevent oxidation of indium layer).
  - In also diffuses into Au (D_o= 6.05 x 10^{-4} m^2/s)
  - ***$AuIn_2$*** remains stable after 9 months at R. T.

In-Ag Phase Diagram



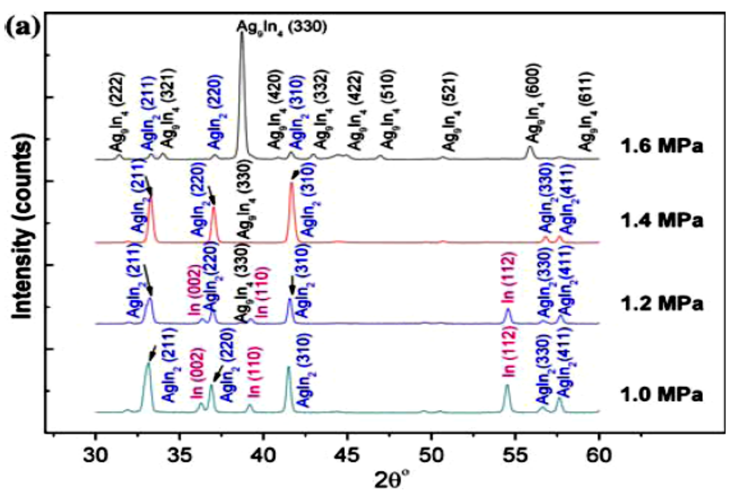
Background
Characterization of Interfaces

"Study of low-T thermocompression bonding in Ag-In solder.."
Journal of Electronic Materials, Vol. 38, No. 2, 2009, 365-371



Aging Effects:
"room temp."

After bonding



RT aging

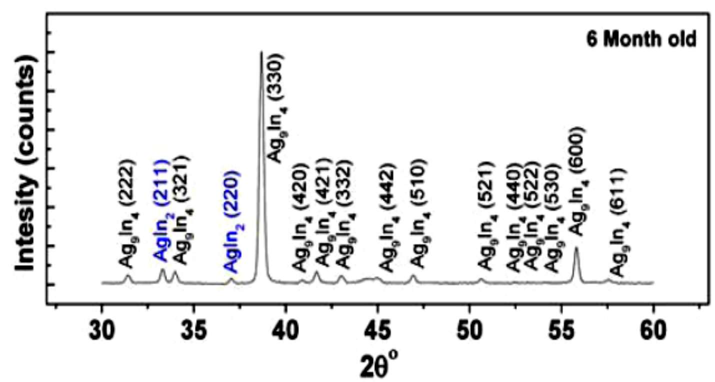


Fig. 8. Results of an XRD scan of samples aged at room temperature for 6 months; samples bonded at 1.4 MPa at 180°C for 10 min.

Precipitation of Ag_9In_4 with high pressure and aging.

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Background -- *Summary of Issues: Fluxless Techniques*

- Glass-to-glass bonding with low-temperature solders and uncoated glass:
  - no data reported.
- Coated gold and indium films:
  - intermetallic compounds
- Thermocompression bonding with coated silver and indium films:
  - intermetallic compounds, and
  - pressure effect

Background --
Indium Oxide Studies

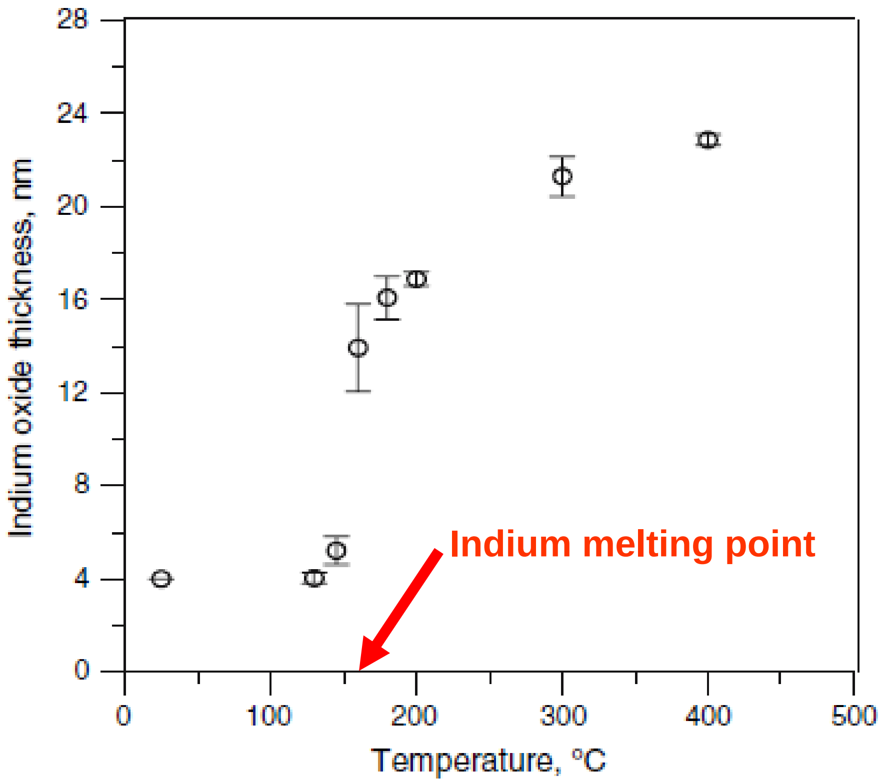


Fig. 1. The growth of indium oxide versus heating temperature.

Each indium sample was on a hot plate exposed to air for 2 hours.

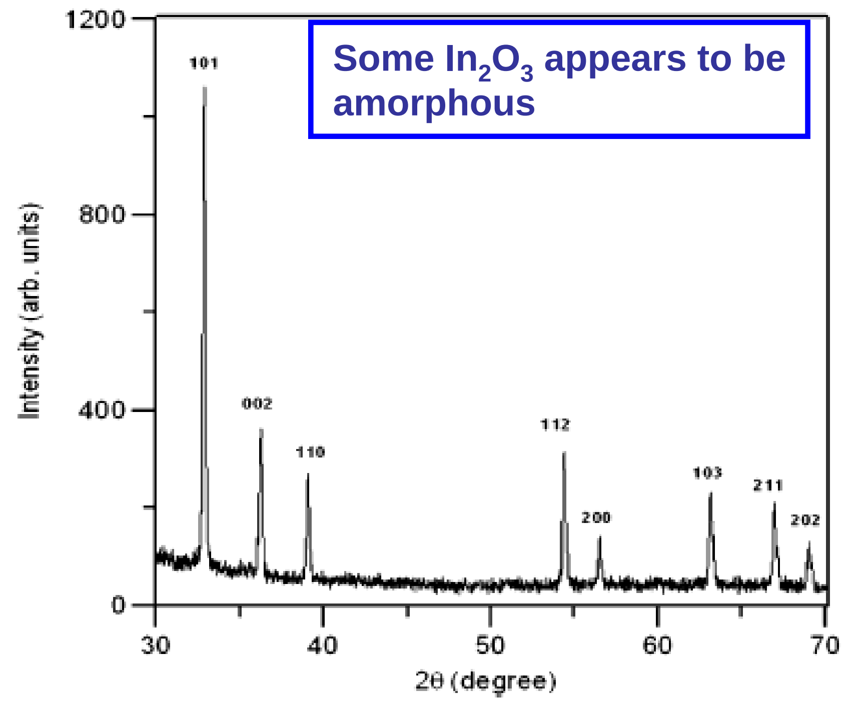
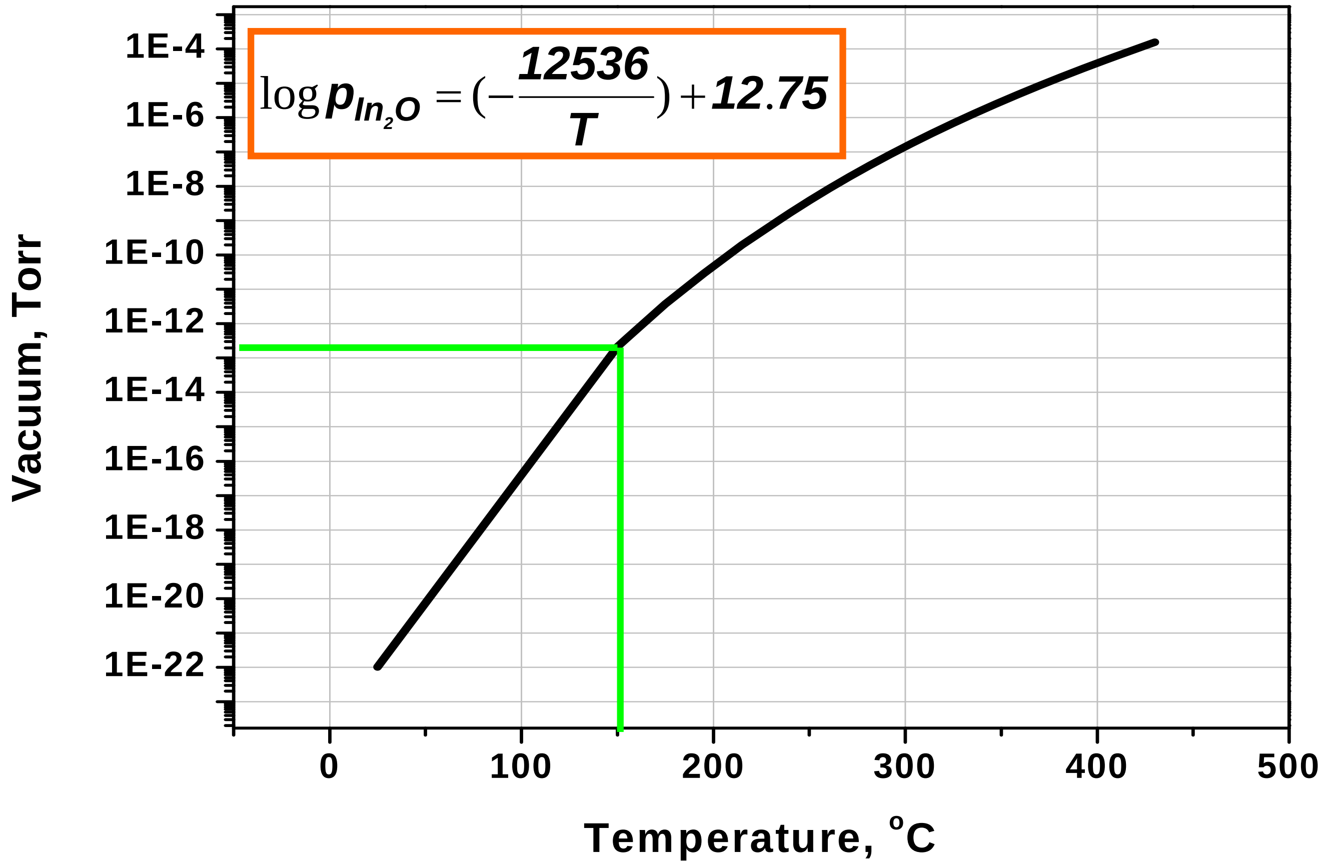


Fig. 4. XRD plot of intensity versus 2θ (scattering angle) for the sample heated at 153°C for 4 h. The peaks labeled are from pure indium.

Effect of Oxidation on Indium Solderability
J. Kim, et al.
Journal of Electronics Materials, Vol.37, No.4, (2008), 483-489.

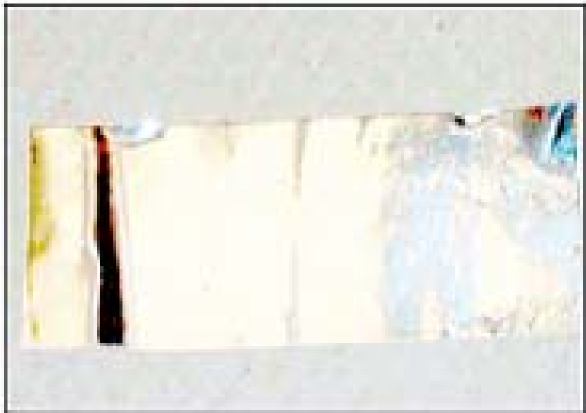
Background
In_2O_3 volatilization



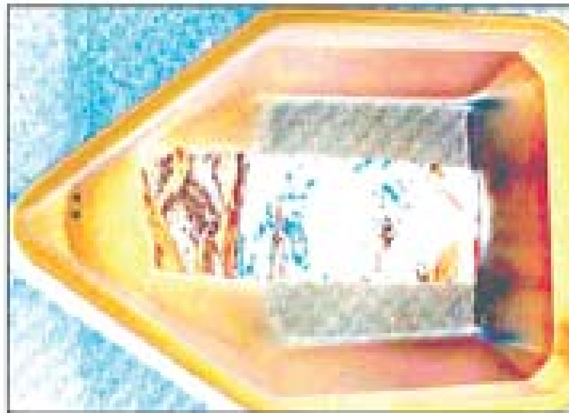
In_2O_3 (solid) + 4 In (liquid) = 3 In_2O (gaseous)

Jenko M., Erjavec B., Praček B.; Vacuum, 40 (1990), 77.

Background --
In oxide dissolution



Pure indium ribbon with normal oxide layer of 80-100 Å (formed at room temperature in ambient conditions).



Indium ribbon being etched in the hydrochloric acid solution for 1 minute to remove oxide layer.

Etched indium ribbon wound back on itself and cold-welded to form a loop.

3. Thoroughly rinse twice in DI water.
4. Rinse off the water with acetone (preferred) or isopropyl alcohol.
5. Blow-dry with dry nitrogen.
6. A mild pressure is all that is usually required to join the indium surfaces together.
7. Use caution to properly align the indium before joining, as they will not easily separate without damaging the joint. They will stick like contact cement.

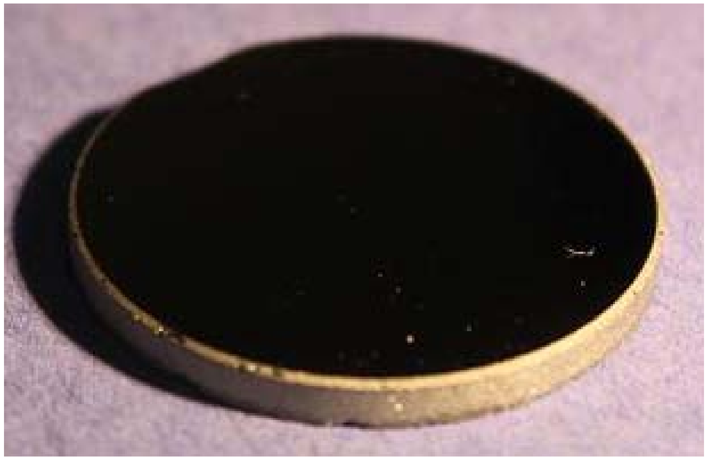
Indium cold welding
Indium Corp. of America
Form No. 97749 R1

Experimental Setup -- *Preliminary Work*

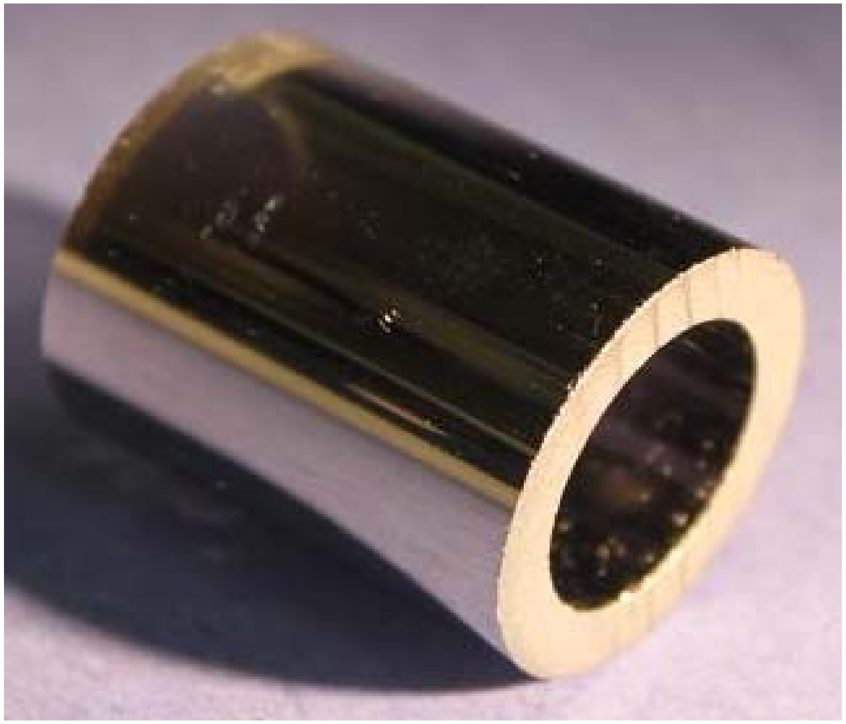
- Tube furnace (controlled atmosphere or high vacuum)
- Vacuum: 10^{-7} millitorr
- Data acquisition using Measurement Computing DaqBoard2000 and DBK81 thermocouple card.
- Used for preliminary bonding tests under high vacuum.

Fabrication of Bond Samples

- ❑ Attempts were made to bond coated samples at 150ºC in vacuum under compression with and without ***In foil***.



Top plate coated with chromium (60 nm) and indium (100 nm)



Glass tube coated with chromium (60 nm) and indium (100 nm)

Fabrication of Bond Samples

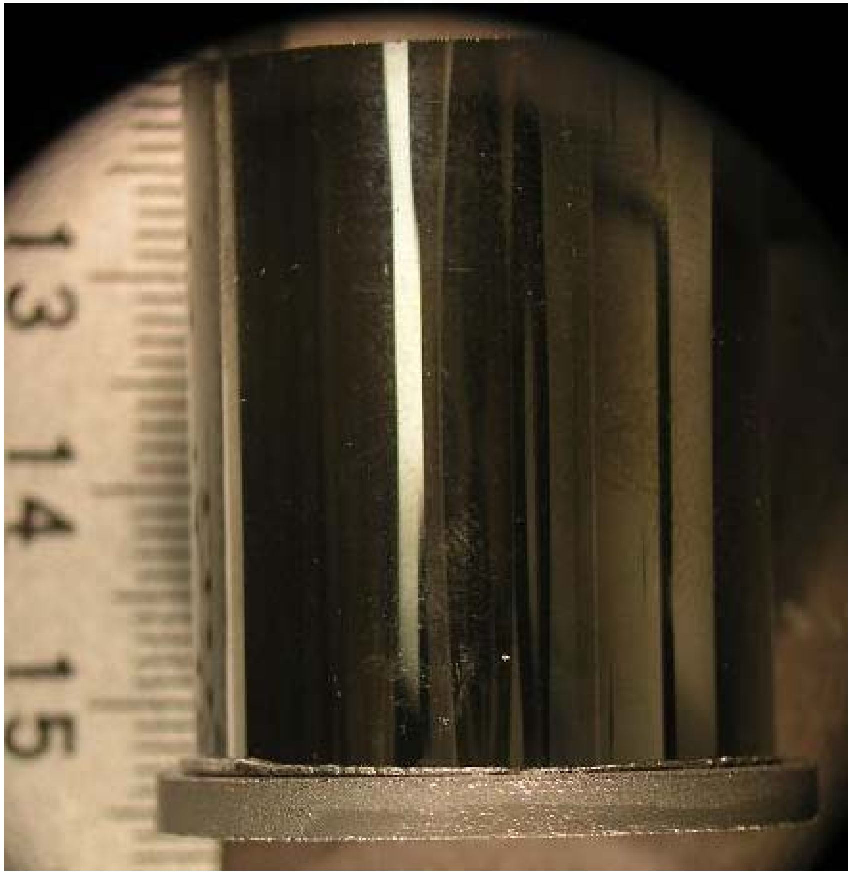
- Samples were kept in a sealed container filled with nitrogen up until immediate use.
- Mechanical rubbing was used to remove the indium oxide layer from the coated samples.
- Tube and top plate were clamped together while subjected to heating cycles.
  - Clamping was only used during preliminary tests.
  - It produced a multitude of problems which will be addressed later.

Fabrication of Bond Samples

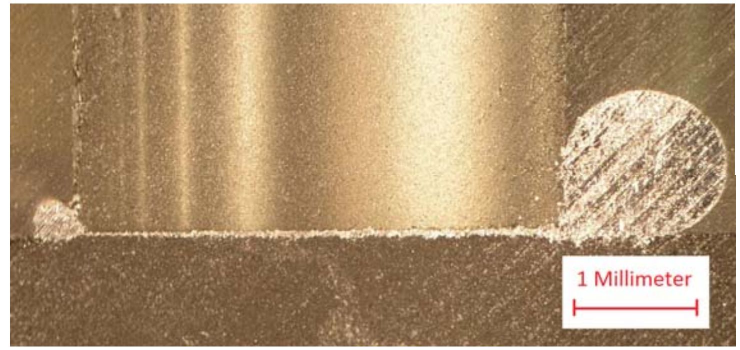
Indium ring prepared from indium foil.

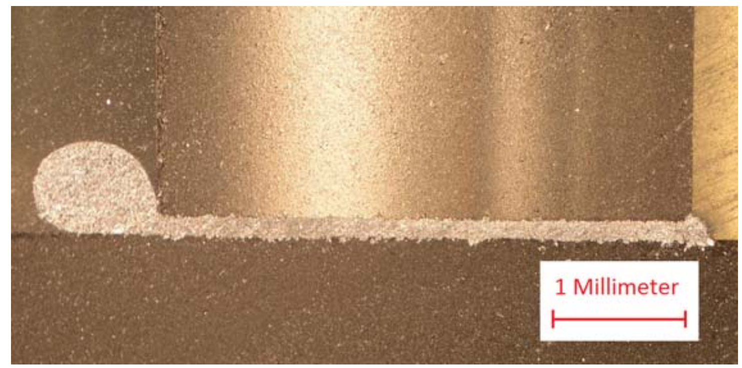
- ***In wire*** was rolled to a flat strip.
- The ring was shaped manually from a straight strip to a an Indium ring.
- The ends were over lapped and pressed together to make the ring continuous.
- Indium oxide was manually removed by scraping the surface with a metal tool.

Preliminary Results -- *Macrographs of bonding*



After bonding at 150^{o}C and at ~300 kPa (0.02 ton/in^2).





These pictures represent opposite sides of the cross-sectioned joint. Non-uniform pressure and varying foil thickness resulted in an uneven joint.

Preliminary Results -- *Procedural Issues and Solutions*

- Samples without ***In foil*** showed no bonding. It appears that In coating is too thin.
- The current set up imposes a limitation on applying required loads.
- A new setup is in the works to apply a uniform load.

Preliminary Results -- *Procedural Issues and Solutions*

- Thermocompression bonding at RT in controlled atmosphere is being considered.
- Mechanical rubbing of the coated samples prior to testing may have removed some of the ‘fresh’ indium layer. For future tests, samples will be washed in a HCl solution before testing.

Preliminary Results -- *Metallography of Indium*

- Standard metallographic procedures do not apply to ***In*** because of its softness.
- Because of In is very soft these procedures cause sawing and polishing debris to imbed into the indium, or the indium will simply ware away.
- A possible solution to this will be to electropolish the In joint using a carbonate, HCl, HNO_3 solution.
- Standard procedure involves cross-sectioning the joint with a diamond saw, grinding with increasingly finer grit silicon carbide paper, and polishing with alumina or diamond.

Remarks

- A thorough review of literature of possible glass-to-glass bonding techniques at 150^{o}C (max), implies that the use of ***In*** is likely the most viable alternative for sealing.
- Several samples have been coated already at Argonne with Cr and In. Preliminary work is underway
- ***In foils*** have been used in preliminary work.
- Cleaning of ***In surfaces*** before sealing is a major factor to produce bonding.

Remarks

- The short-term goal of a mechanical bond with In foil on Cr coated samples was made.
- Early results using ***In*** as a sealant are encouraging.
- Bond samples using flat glass specimens are in progress.
- Further bonds using thicker ***In coatings*** and ***foils*** are programmed.