Development of a 20x20cm^2 'hot' indium-alloy hermetic seal in an inert atmosphere for photo-detector assembly

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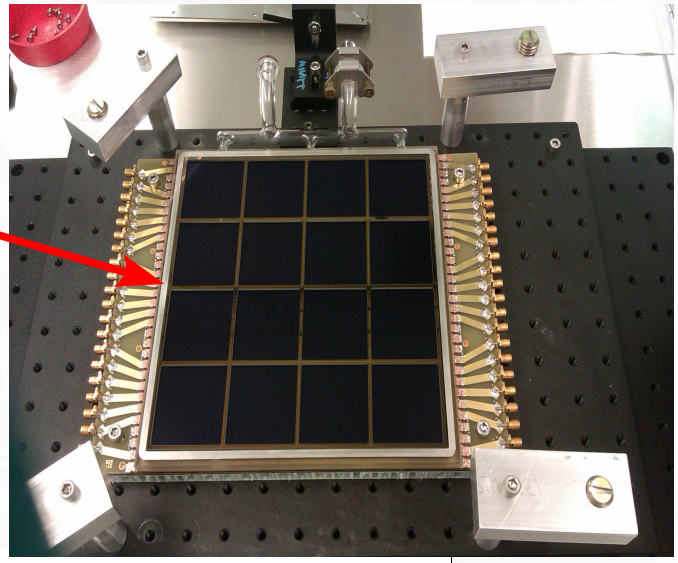
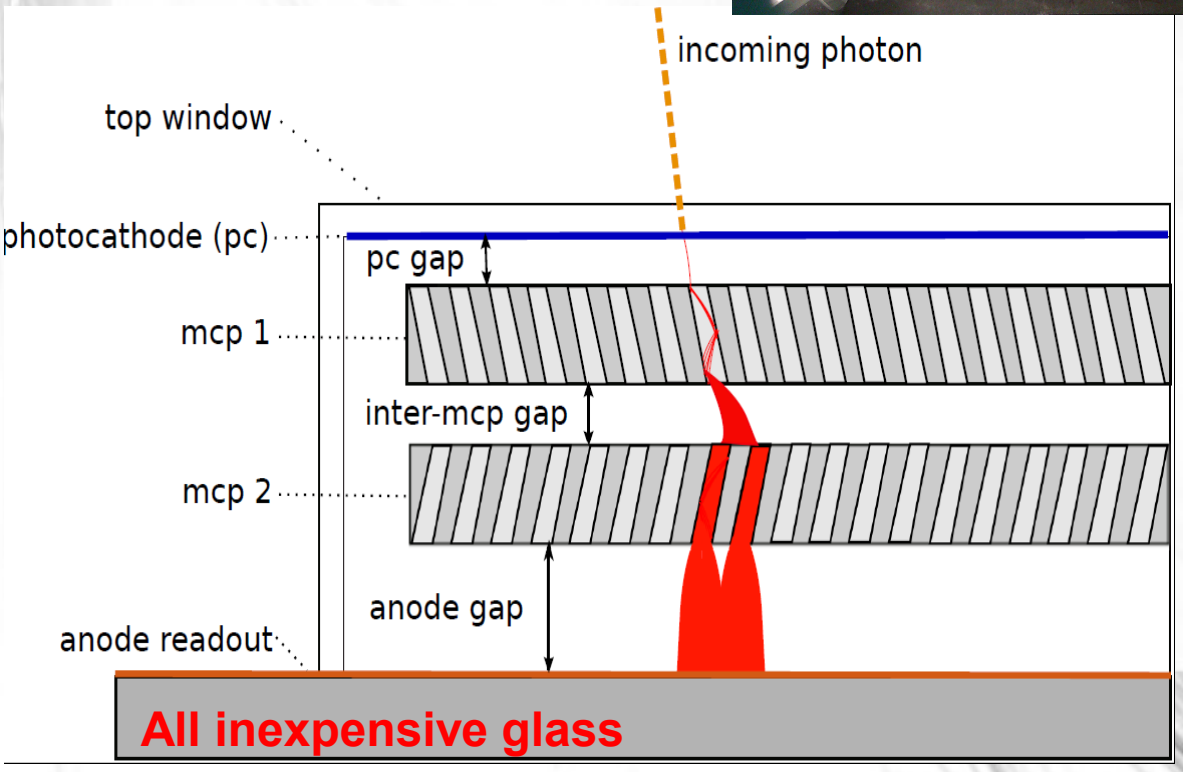
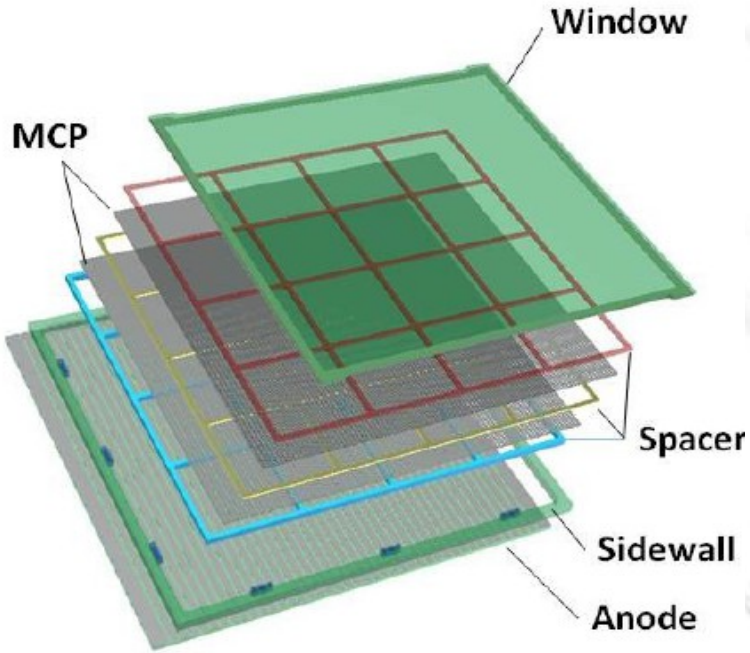
TIPP, June 4, 2014

Large-Area Picosecond Photo-Detectors (LAPPD)



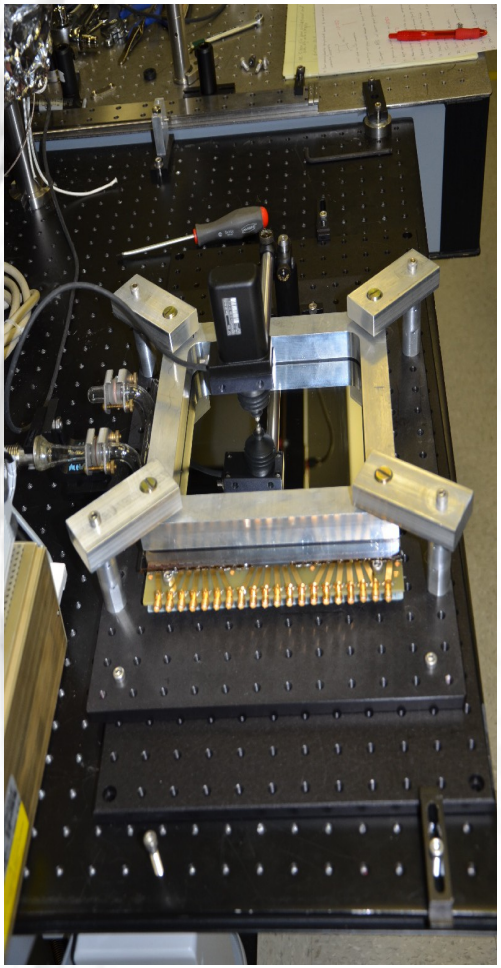


Glass Package Frugal Design

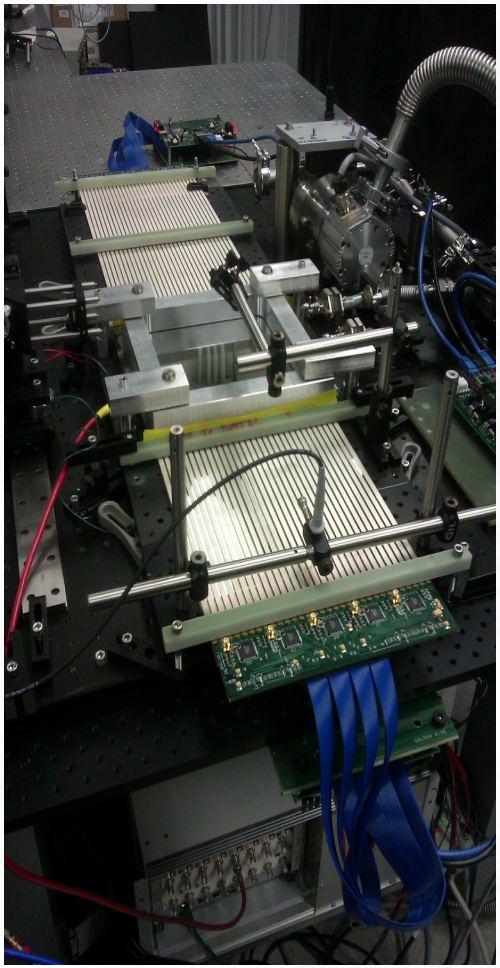


LAPPD Prototype: Demountable

single-tile



1x4 module



See results and performance in poster by Matt Wetstein

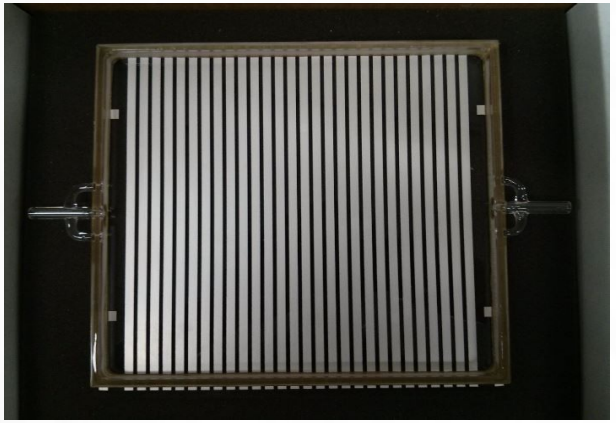
Demountable is a successful demonstration of the LAPPD glass body packaging

- A complete LAPPD glass tile except for
  - an aluminum photo-cathode
  - top seal by compression on a viton o-ring
  - active pumping
- Successful tests of the mechanical. electrical and vacuum properties of a fully sealed tube

This talk is about development of a top seal technique compatible with producing a bi-alkali photo-cathode on the top window and/or with a vacuum transfer assembly process

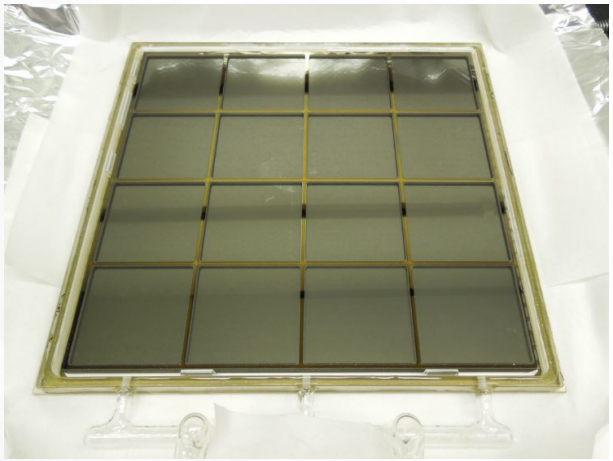
Hermetic Packaging Strategy

1) Make a tile-base (a glass sidewall hermetically sealed over anode plate)
- this is done by a frit-seal
- reliably reproducible by Joe Gregar at the ANL Glass Shop



2) Load internal components (MCPs and grid spacers)
- in vacuum (standard vacuum transfer assembly)

OR
- in inert atmosphere in a glove box



3) Seal at the top with a photo-cathode window
- Top Seal Challenge: hermetic seal between the tile-base and the top window has to be done at moderate temperatures

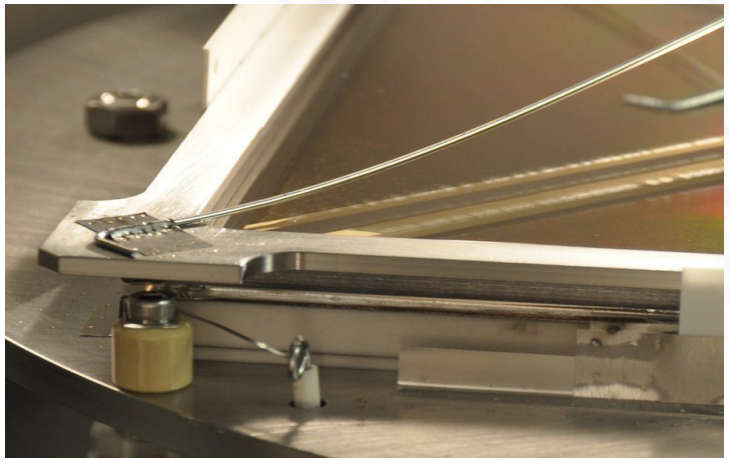
Use indium alloys:
- industry standard approach
- soft metal
- low melting point
- essentially zero vapor pressure

Note squared geometry

SSL Recipe for Ceramic Package

Copper well is brazed into the ceramic body and filled with indium alloy (InBi)

Top window has a NiCr-Cu layers deposited along perimeter



O. Seigmund, et al.

Indium alloy wets copper surface and makes a strong NiCr-Cu-InBi interface between two sealing surfaces

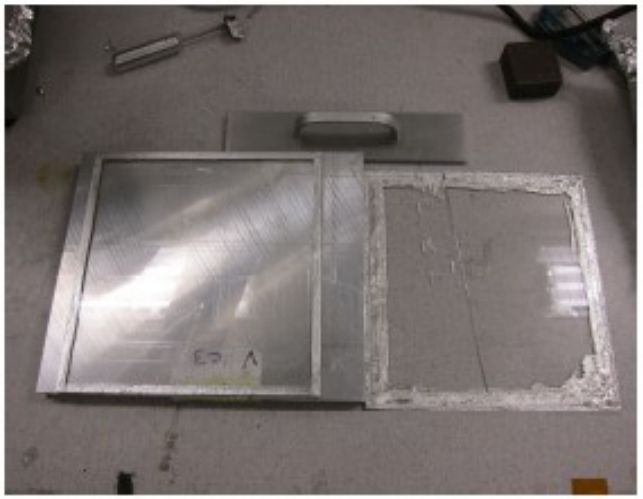
This technique has been proven to work
- small size photo-detectors by SSL
- 1st attempt to seal a ceramic tube last summer (only one well understood leak)

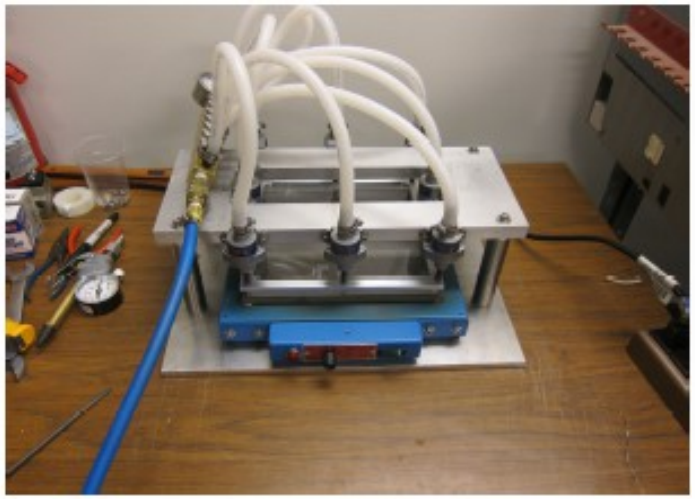
Details of the technique has to be tuned for each application

We adapt this recipe by using glass-NiCr-Cu-InBi interface to seal two flat glass surfaces (edges of the tile-base and top window)

First Attempts to Make a Seal

Started with a simple interface: pure In applied directly onto glass in air





Worked well for 1x1" test samples

Didn't scale to 8x8" easily due to formation of indium oxide (best result was a seal with a leak at 10^-6 cc/s of He)

- Moved assembly into the glove box:
  - + no indium oxide formation
  - – melted indium doesn't wet glass in oxygen free environment (also reported in NIM A 567 (2006) 205-208 by D.Ferenc et. al.)
- Simple indium solder seal becomes incompatible with vacuum transfer assembly.
- Indium wets NiCr-Cu layer very well

'Hot' Seal

- Step 1: Preparation
  - Clean glass parts
  - Sidewall and window surfaces are coated with 200nm of NiCr (80:20%) and 200nm of Cu
  - If long time in air before sealing – clean with Micro-90 and rinse with DI water

'Hot' Seal

- Step 2: Assembly in the N2 glove box

→ Heat glass parts to ~80-90C

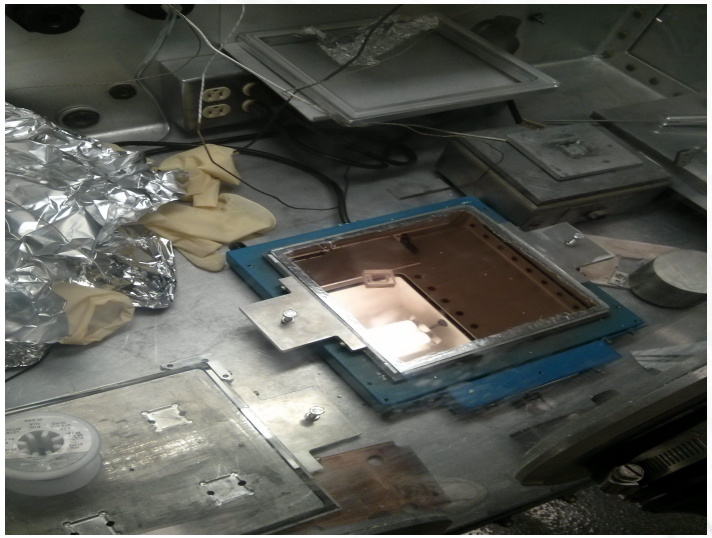
→ Using nickel felt applicator (small metal brush by Indium Corporation) apply InBi alloy onto Cu layer on the sidewall and window

→ Wait when parts cool down and place the window on top of the sidewall

→ Re-heat to 80-90C

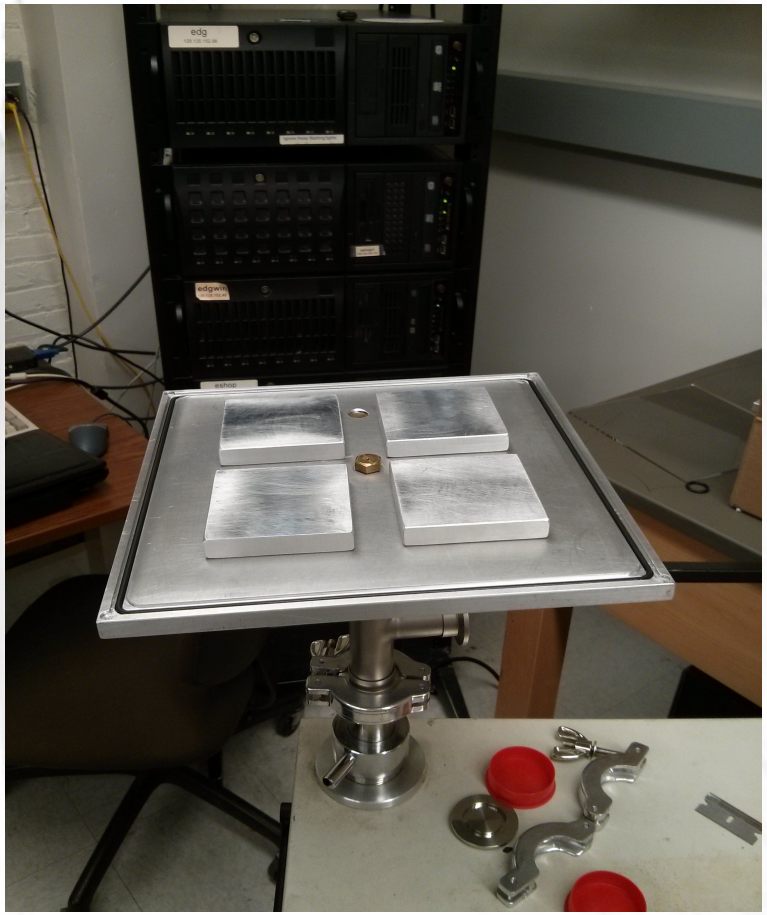
→ Move/slide window within ~1/2 of the sidewall width

→ Position window, apply weight (~50lbs) along the perimeter and cool

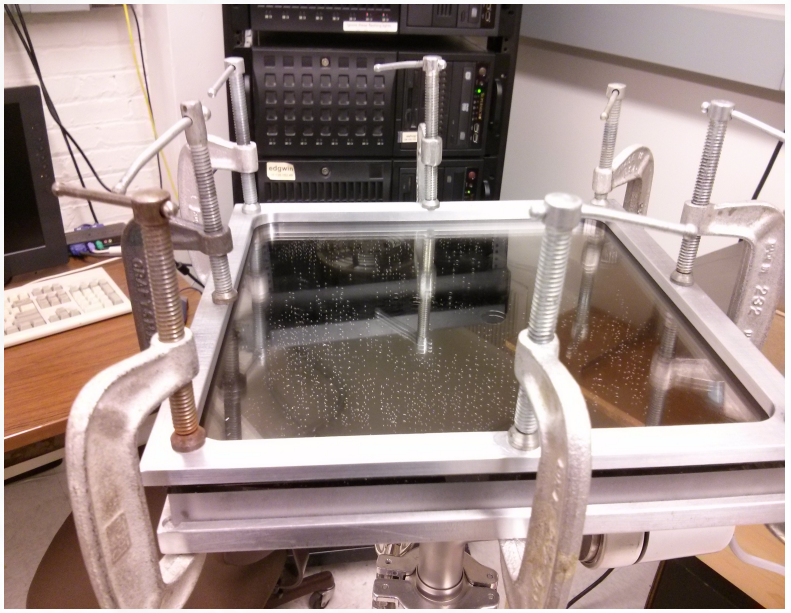


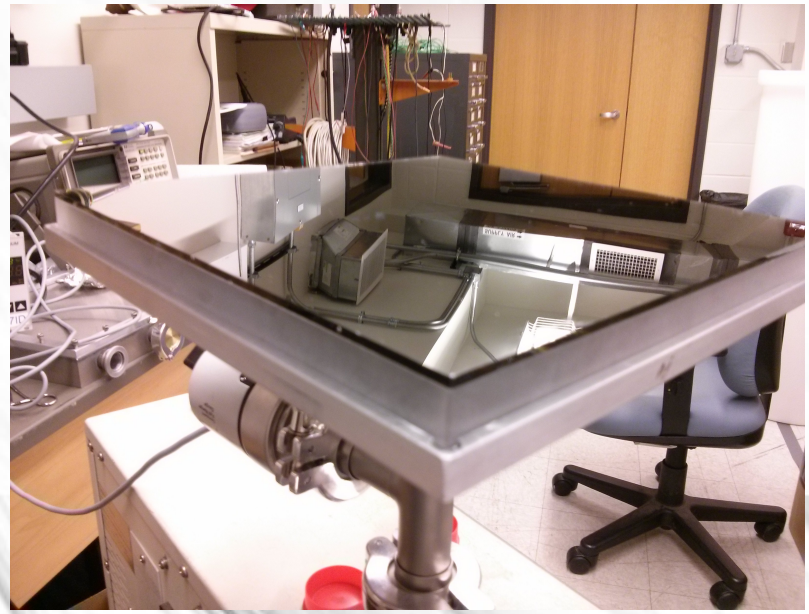
'Hot' Seal

- Step 3: Leak test (sensitivity ~10^{-8} cc/s of He)



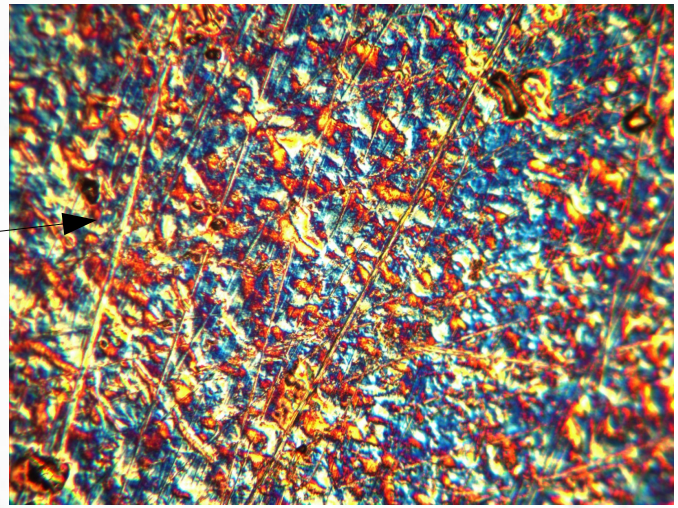
A lot of metrology and tooling
by Bob Metz and Richard Northrop

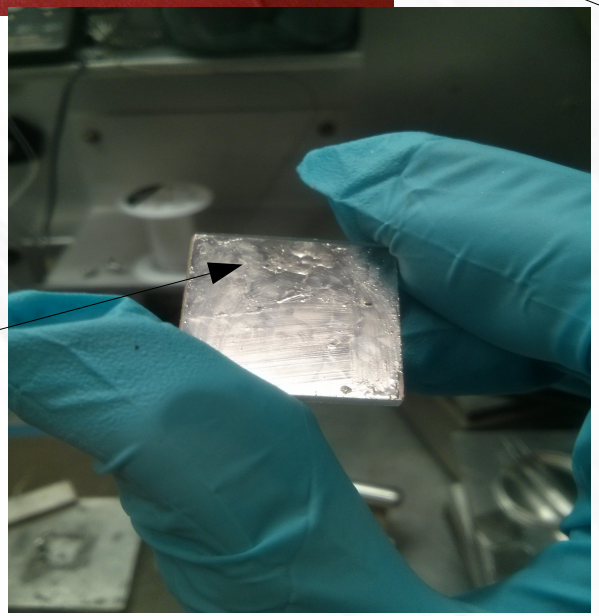




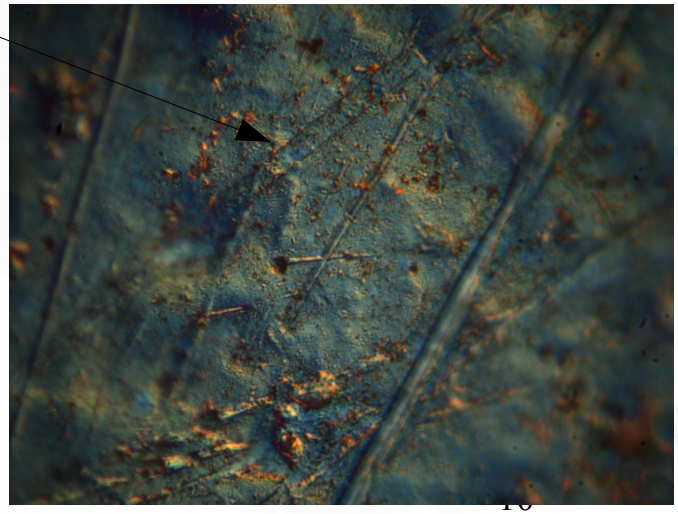
Cu Scavenging Problem

x200





x1000



The layers can be stable

We observe two very different "phases" of the glass-NiCr-Cu-InBi interface

Microscope photos courtesy of H.Clausing

Cu Scavenging Problem

- Success rate for a good glass-NiCr-Cu-InBi interface is ~30%
- None of the following parameters alone explains scavenging

(Parameters marked in **red** increase the likelihood for good NiCr-Cu layer)

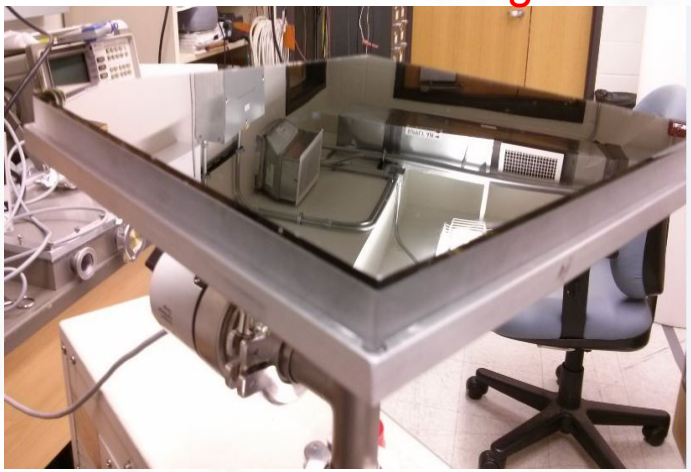
  - Vendor of NiCr-Cu coating
  - Sputtering vs evaporation
  - **No vacuum break** vs vacuum break in between NiCr and Cu deposition
  - **Crystaline** vs amorphous NiCr layer
  - **InBi alloy** vs pure In
  - Soldering temperature
  - Glass surface preparation: e.g. **polished** vs raw surface

Acknowledgments: Qiti Guo (UChicago), Chian Liu (ANL), Ian Steele (UChicago), Ossy Seigmund (SSL), Jason McPhate (SSL), Sharon Jelinsky(SSL), Dean Walters (ANL)

- Current approach to 20x20cm^2 seal: **use good parts with no scavenging**

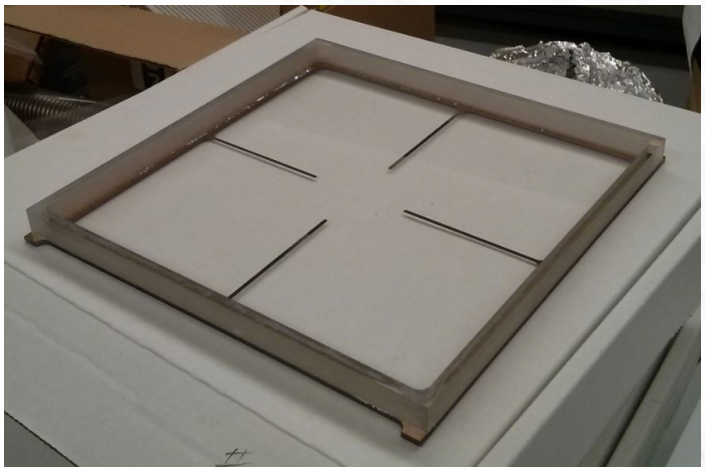
20x20cm^2 Sealing Tests

Seal #1 – August 2013
Good NiCr-Cu-InBi interface
on both surfaces: leak tight.

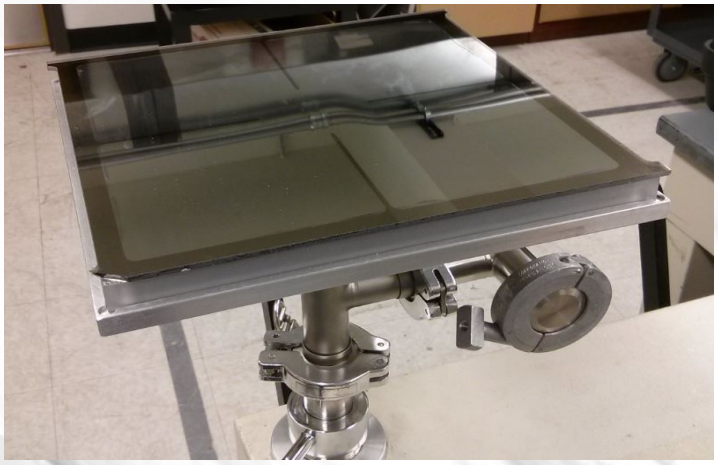


Seal #2 – February 2014
Good NiCr-Cu-InBi interface
on both surfaces: leak tight.

Seal #3 – February 2014
Good NiCr-Cu-InBi interface on the sidewall,
"bad" window (InBi applied only on the sidewall):
leak tight for 5 mins, then broke at the corners.



Seal #4 – March 2014
Good NiCr-Cu-InBi interface
on both surfaces: leak tight.



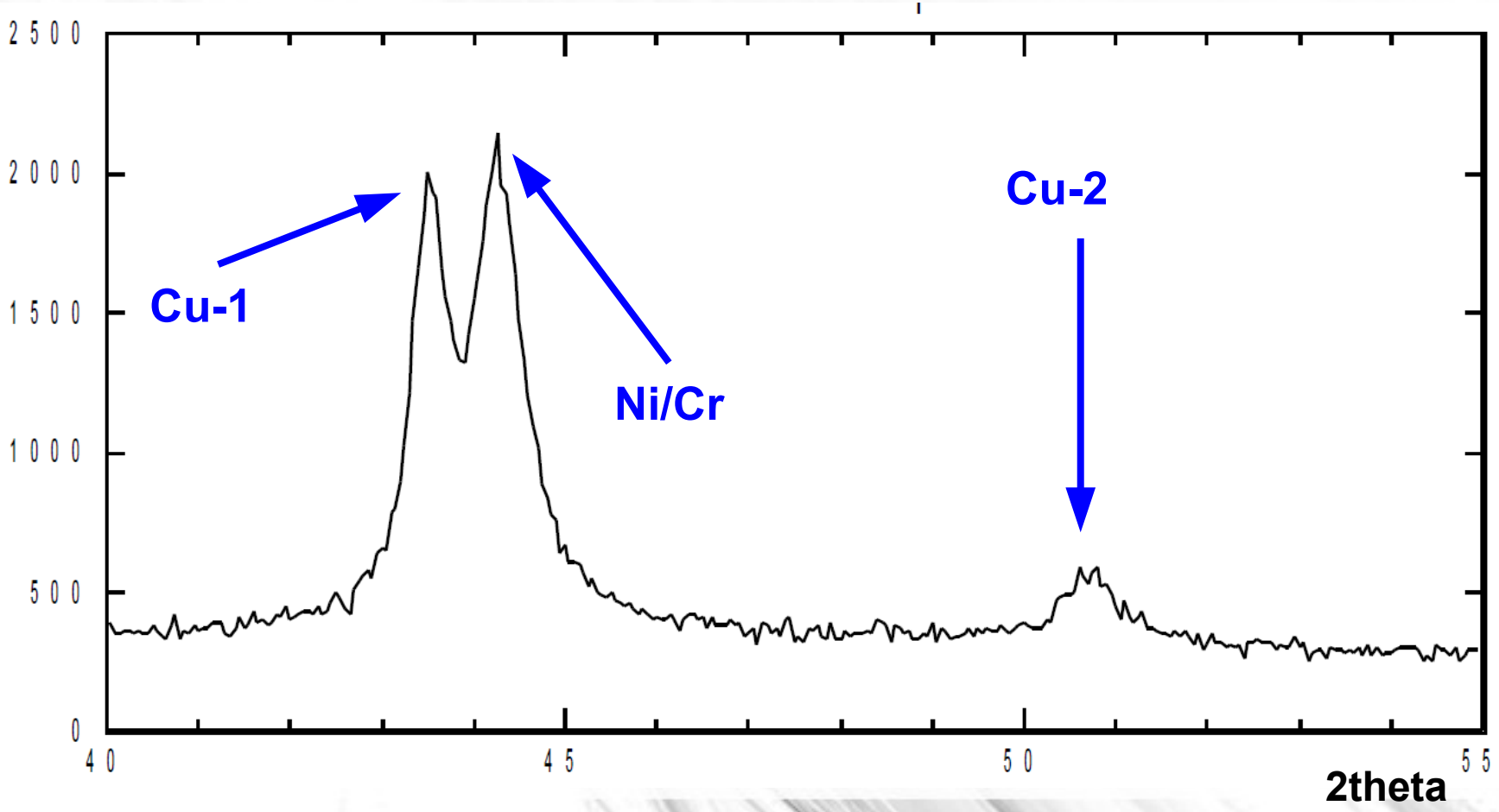
The seal is reproducible if start from good NiCr-Cu layer on the glass

Investigation of NiCr-Cu Layers

X-ray Diffraction

by Ian Steele at UChicago

Good sample

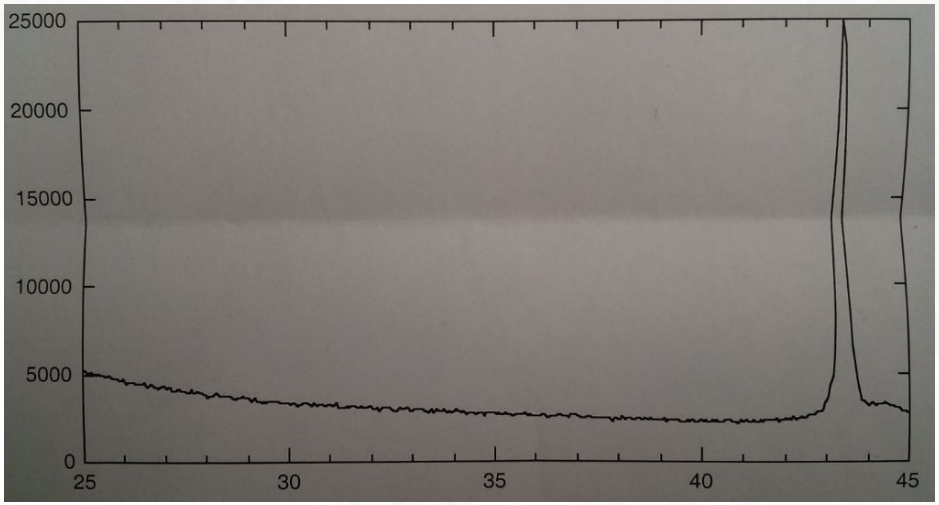


Investigation of NiCr-Cu Layers

X-ray Diffraction

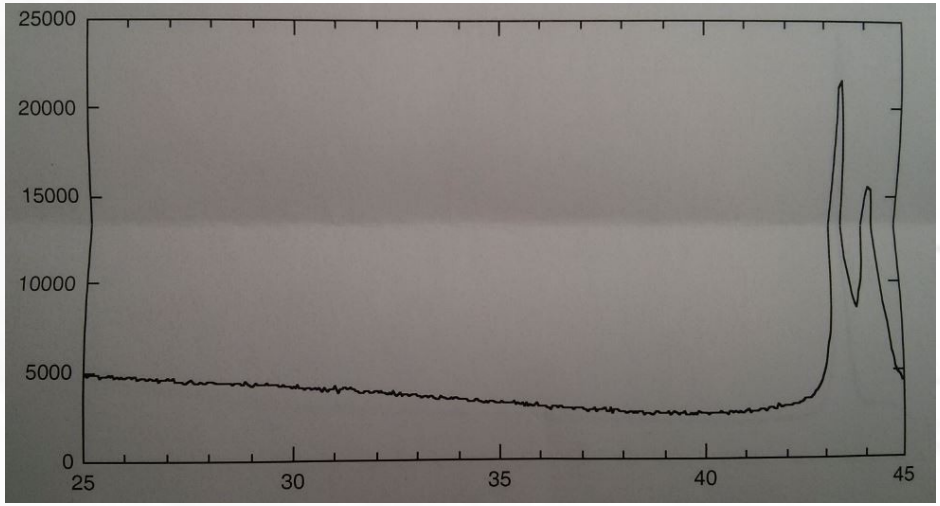
by Ian Steele at UChicago

Typical bad sample



Amorphous NiCr layer

Typical good sample



Crystalline NiCr layer

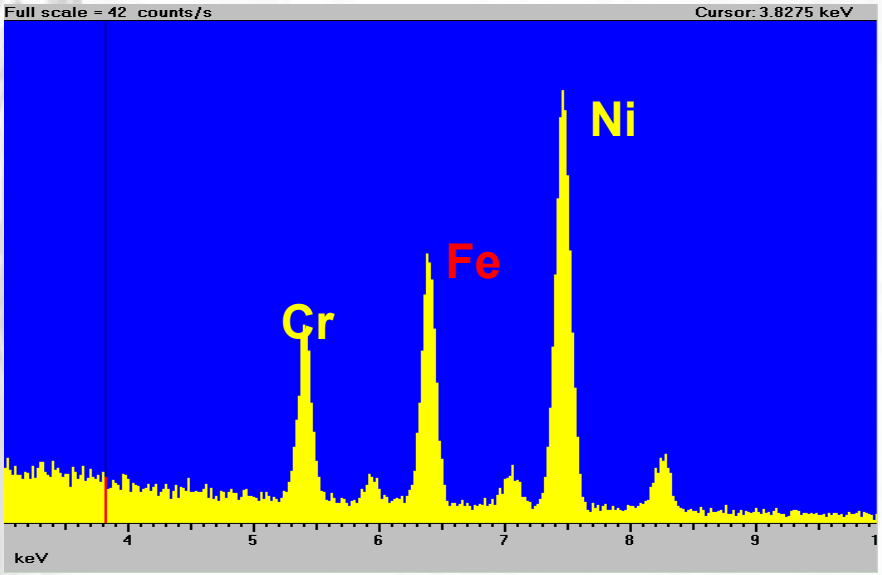
However, not a 100% correlation

Investigation of NiCr-Cu Layers

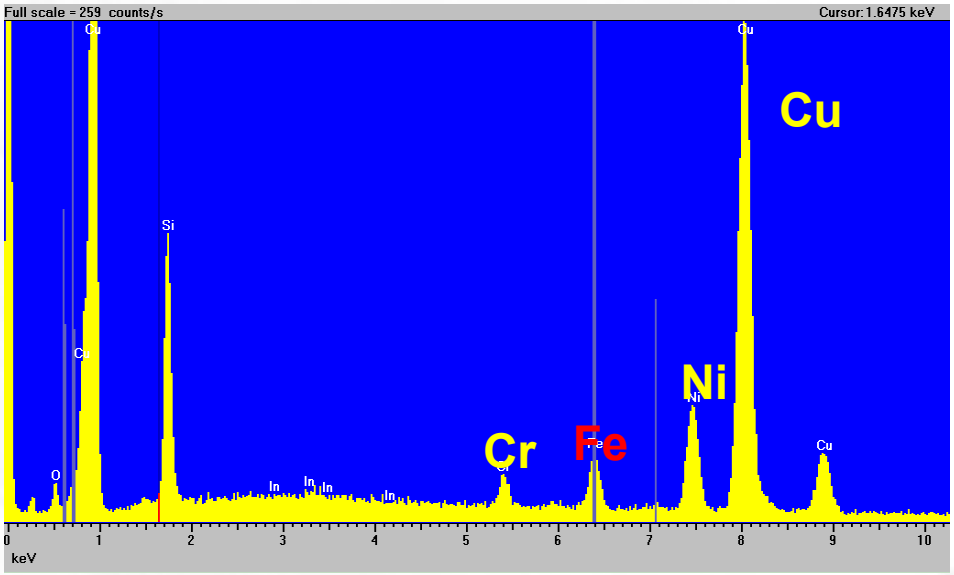
EDS spectra

by Ian Steele at UChicago

Bad samples
looking directly on NiCr layer



Good samples
looking on NiCr layer through 200nm of Cu



We haven't find any difference (yet) between good and bad samples, however

- it is an extra tool to attack Cu scavenging problem at the material level
- happened to be very useful for quality control

(e.g. although Fe is expected in some of the NiCr-alloys, in this case it was a surprise to us and to the vendor as the specs were Ni:Cr-80:20%. The vendor identified and corrected the problem)

Summary

We developed a technique to make a 20x20cm^2 hermetic seal between flat glass surfaces by using indium alloy solder

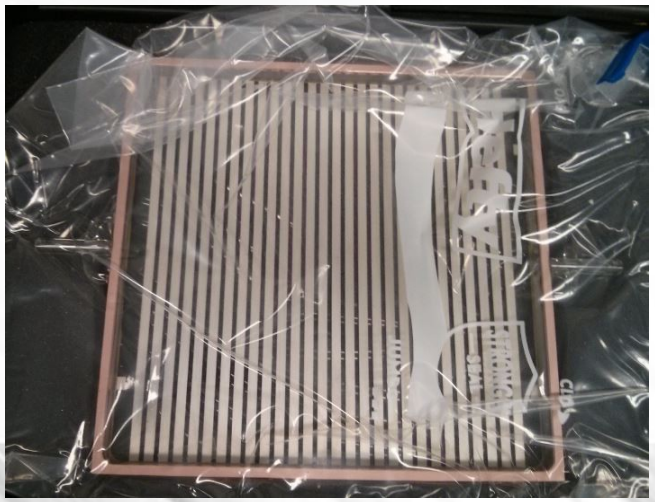
- observed two distinct "phases" of glass-NiCr-Cu-InBi interface
- the seal is well reproducible if good quality NiCr-Cu coatings are used

This technique has been adapted from LAPPD ceramic package

- the technique has been known to work in other applications
- the details of the technique had to be tuned

Work in progress to identify key parameters affecting the stability of the NiCr-Cu-InBi interface (Cu-scavenging problem)

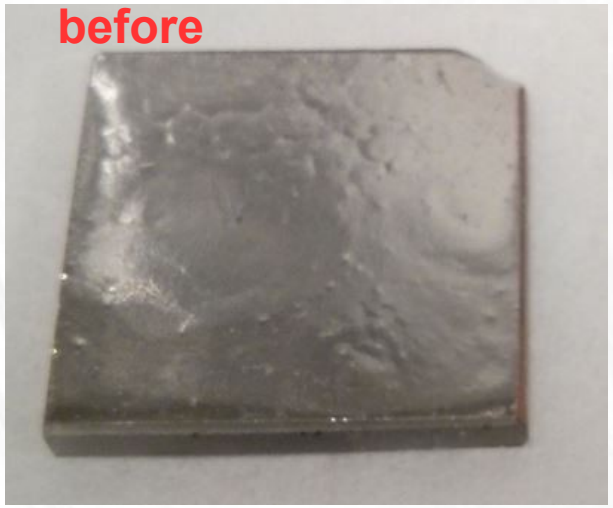
We are moving from proof of principles to sealing an LAPPD tile

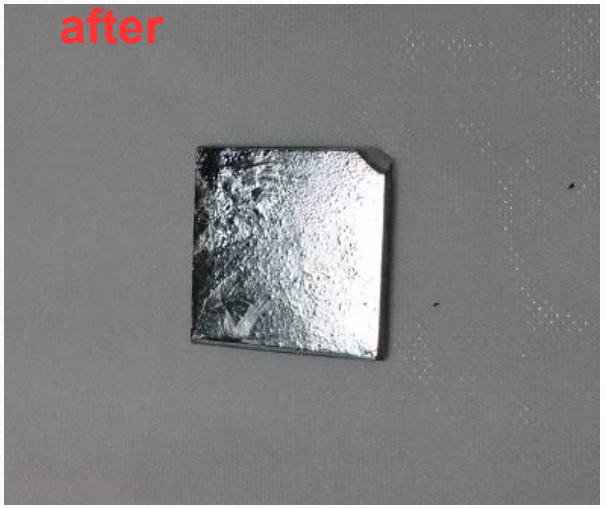


Back-Up

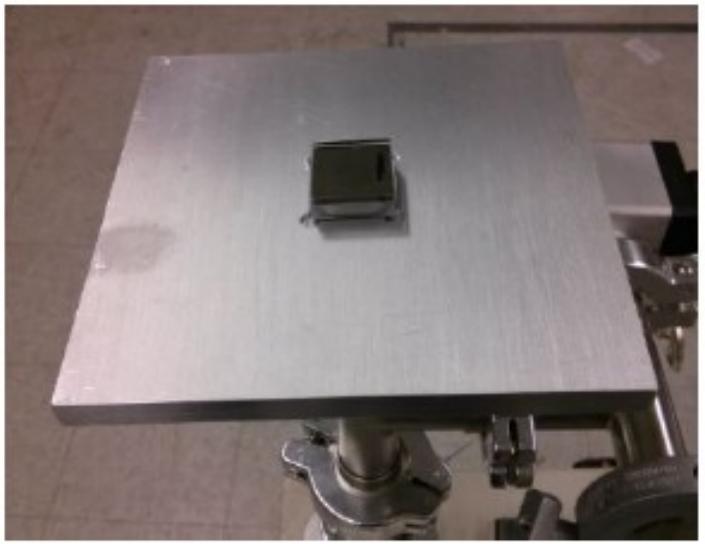
Tools

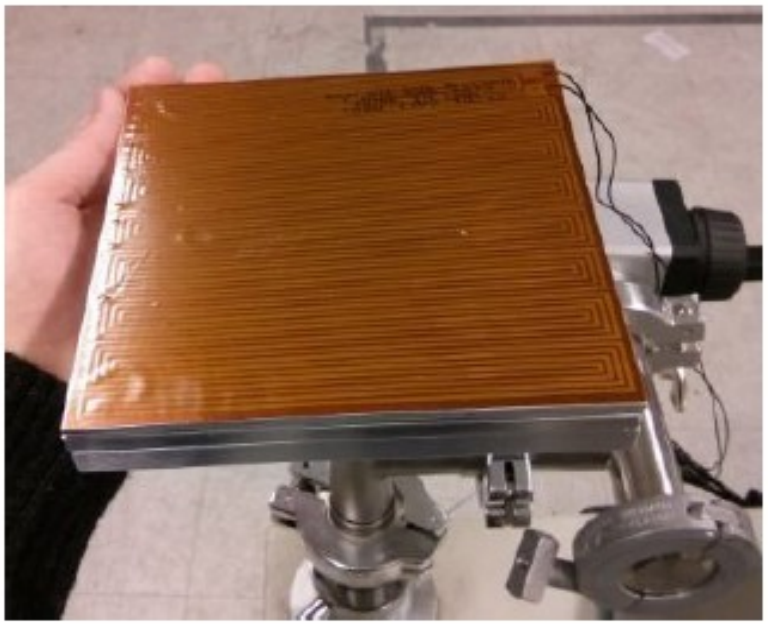
Vacuum bake at 400 C

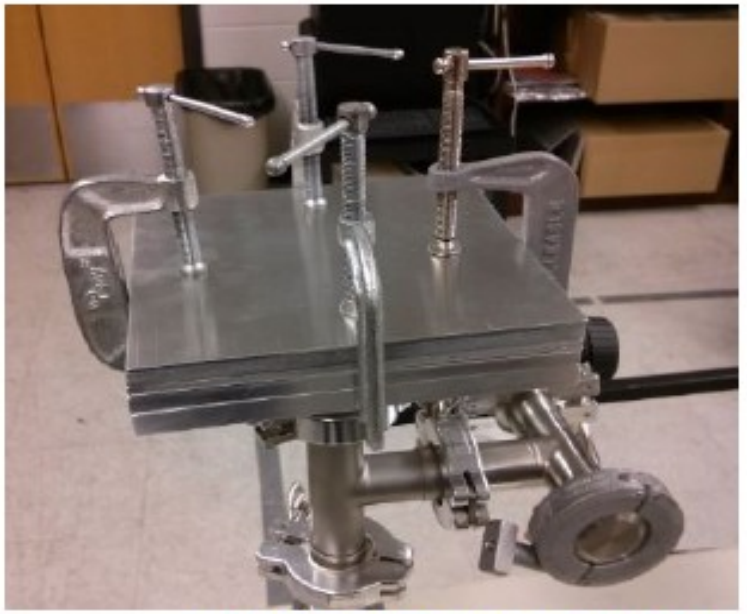




Heating fixture

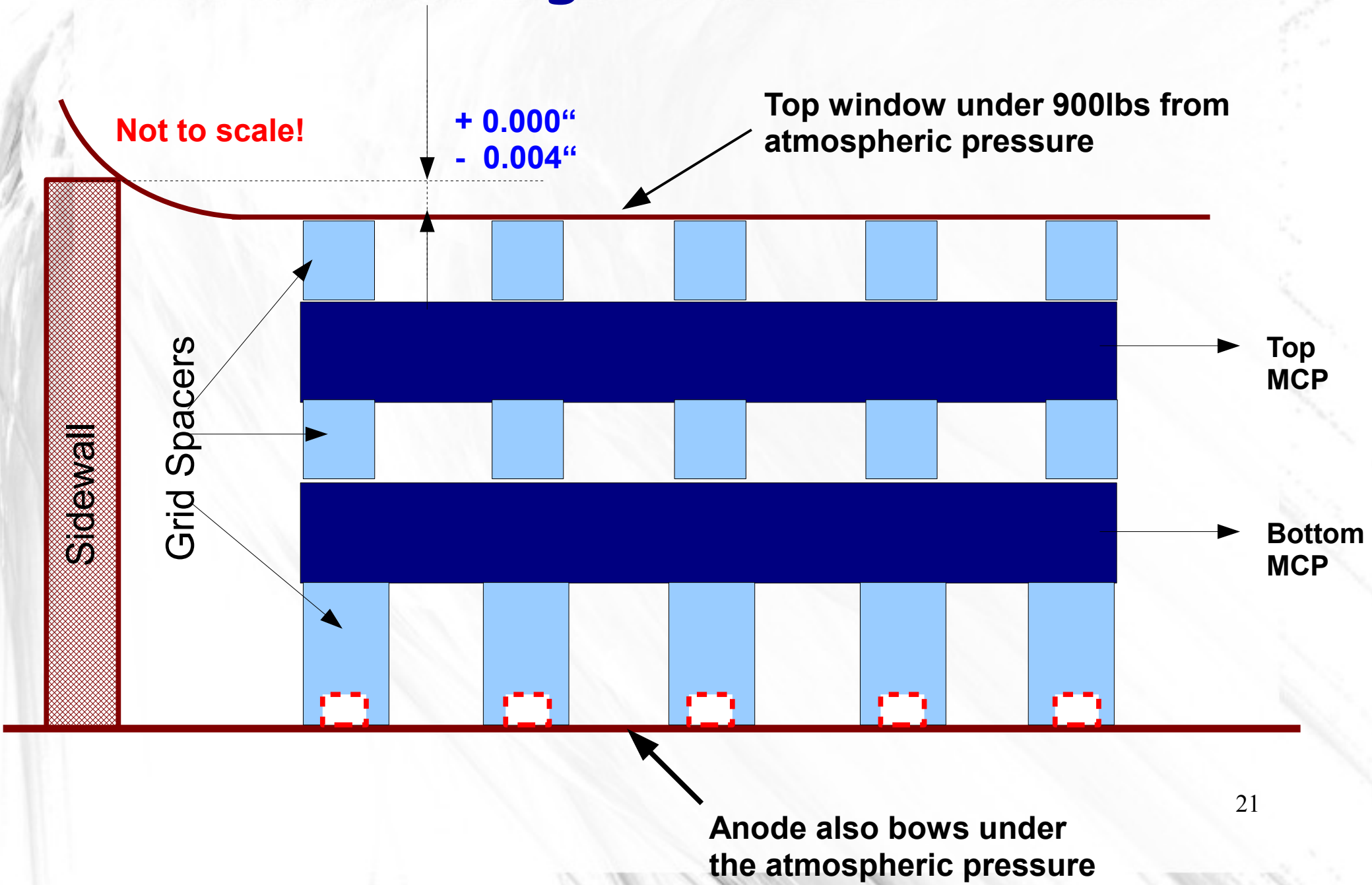






Thanks to Rich and Bob!

Sealing an 8x8" Tile



A lot of metrology and tooling by Bob Metz and Rich Northrop

View through the top window (not to scale)