Hot Indium Seal Status and Plans

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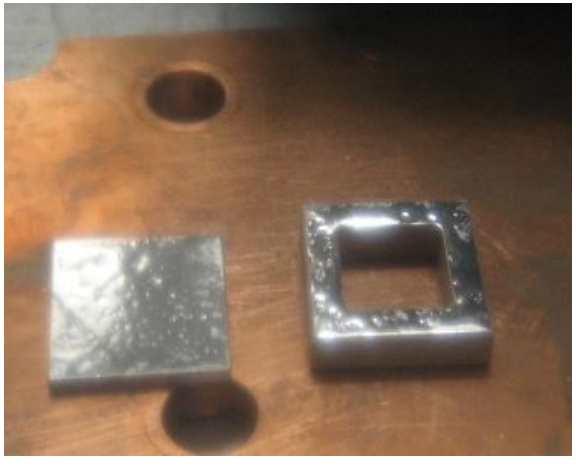
Hot Seal Concept

Step 1:
apply melted indium to the glass parts



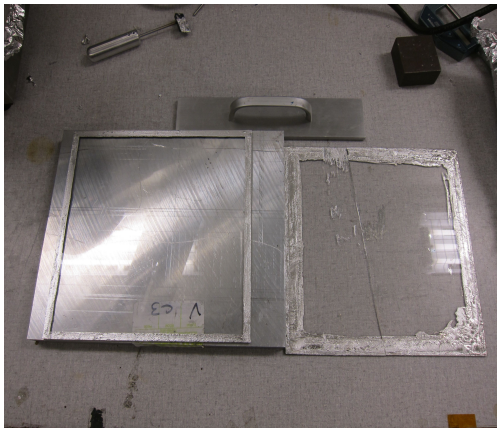
Step 2:
bring parts into contact and press

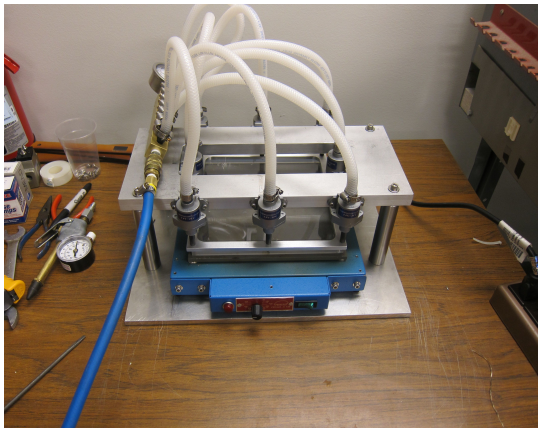
Step 3:
pump from open side and leak check

Prerequisites for leak-tight seal

- **Strong Indium-Glass bond**
- **Strong Indium-Indium bond**

Phase I (in air)





- **Indium seal fundamentals**
  - interface (good adhesion of indium to the glass surface)
  - oxide formation

- **Proof of principle using 1x1" test samples**
  - little oxidation (assembly is fast)
  - many successful reproducible leak tight samples

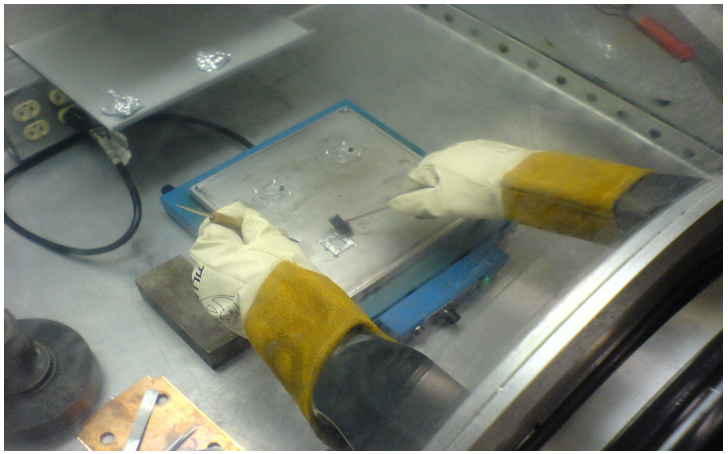
- **Several (>10) attempts to make 8x8" seal**
  - oxide formation becomes limiting factor (slow assembly)
  - best result is a part with 10^{-6}cc/s leak at a single pinpoint

...indium oxidizes quickly...

Phase II (in inert atmosphere)

Phase IIa (in inert atmosphere)

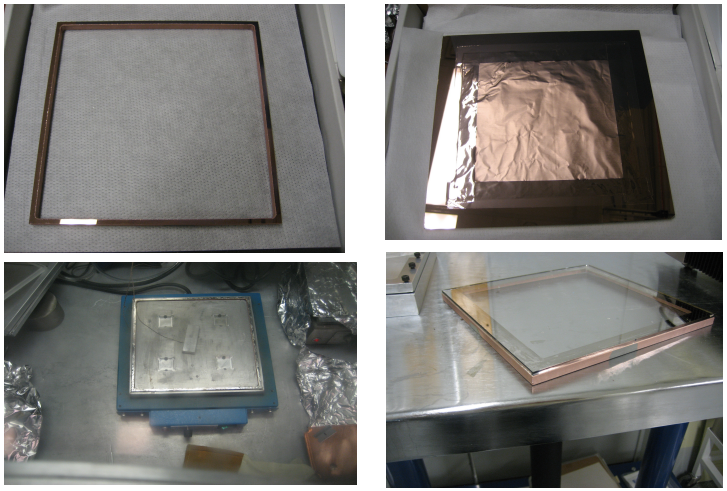
Nitrogen filled glove box:
O_2 and H_2O concentration ~5ppm



...indium doesn't stick to glass if no O_2...

This is not understood:
what's the mechanism of the bond formation between indium and B33 glass?

Phase IIb (add NiCr-Cu layer)



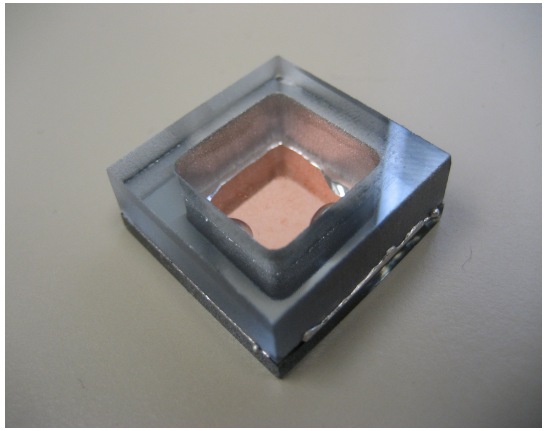
Borrowed from SSL window design (200nm of NiCr+Cu)

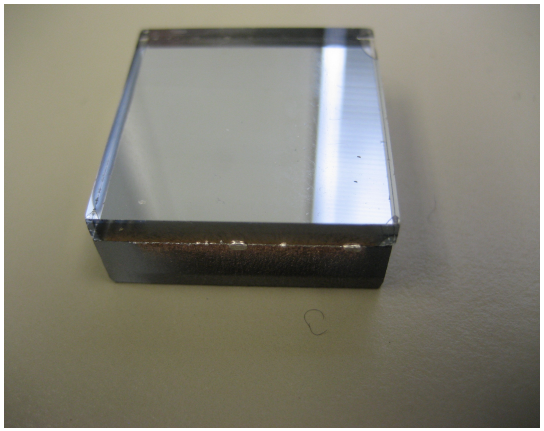
Known facts:

- **Indium wets copper surface**
  - Alloy is formed at the interface over time
- **NiCr interface to glass is essential**
  - NiCr is a good match to glass in terms of thermal coefficient
  - Cu would not stick to bare glass but does so on NiCr

NiCr-In Seal Results
(1" parts)

Total 8 small size seals made: 5 are leak tight
3 have leaks (oxidation of Cu surface or electroding peeling off the glass)





Shear tests results

Leak tight samples:

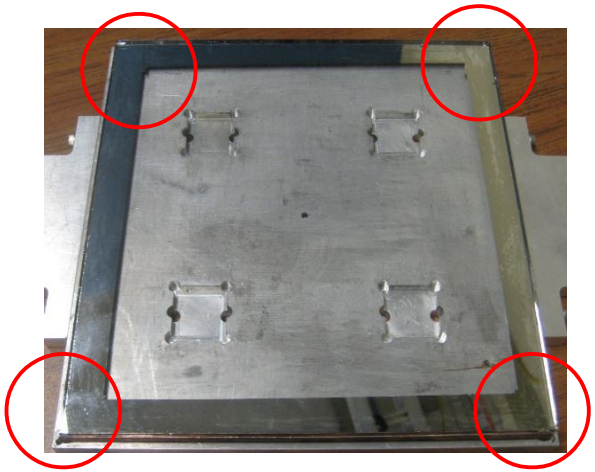
Bare glass #1	190 lbs
Bare glass #2	278 lbs
Bare glass with groove	268 lbs
Cu coated glass #3	390 lbs
Cu coated glass #4	345 lbs

Samples with a leak:

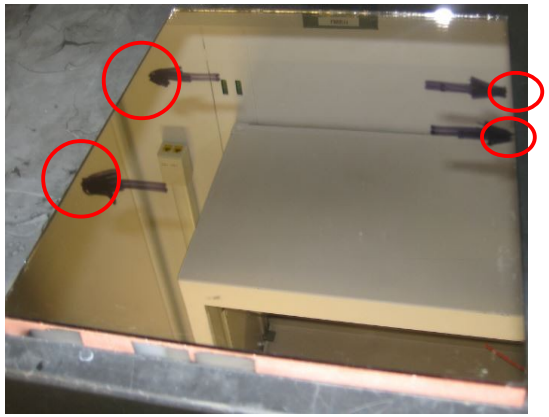
Bare glass #4	47 lbs
Cu coated glass #1	213 lbs
Cu coated glass #2	221 lbs

NiCr-In Seal Results
(8" parts)

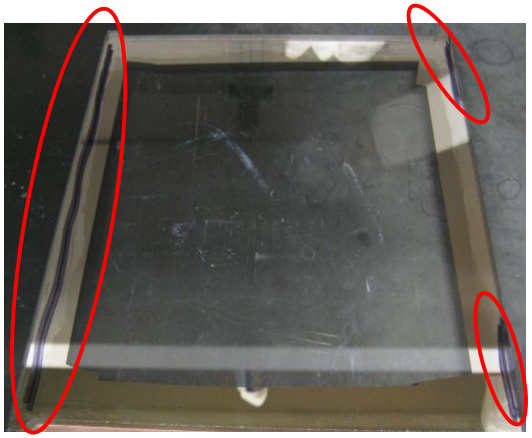
Total 3 large size seals made: all have leaks



Copper surface got oxidized before contact with indium:
- parts held in air after coating
- poor indium wetting observed



Under investigation
- sidewall sat in air between NiCr and Cu depositions
- non uniformity in compression?



Under investigation
- coating non-uniformity
- small size gun used for Cu
- compression problem?

All little details matter. Need more electroded parts.

(first successful 1" seal was attempt number 7)

Status of electroding capabilities

- Argonne sputtering system

  - the only source we have used so far for 8" seals

  - at the moment Cu target for small gun only (1.5x8")

- E-gun evaporation at MRSEC Uchicago

  - at the learning curve (match materials to the right crucible, plasma cleaning, etc.)

  - several successes with 1" but was not able to ensure good bond between glass and NiCr layer on 8" parts

- SSL thermal evaporation

  - 8" parts has been sent, Sharon is working on them

- Commercial way (e.g. H.L. Clausing Inc. Optical Coatings)

  - order is being placed to electrode two 8" sets

- Fermilab ?

Open questions

- What's the exact mechanism of bond between indium and B33 glass?
- What's the mechanism of NiCr-B33 bond?
- Is NiCr-Cu bond stronger than Cu-In?
- Is 200 nm the optimal thickness for NiCr and Cu layers?
- Does Cu-In diffusion affect the seal in the long run?
- Can we completely get rid off the indium oxide (there is always some on the wire even if we assume no oxidation in the glove box)?

  - indium oxide float to the surface if baked in vacuum at ~400C

    (see back-ups)

  - this is standard step in SSL sealing procedure

Conclusions and Plans

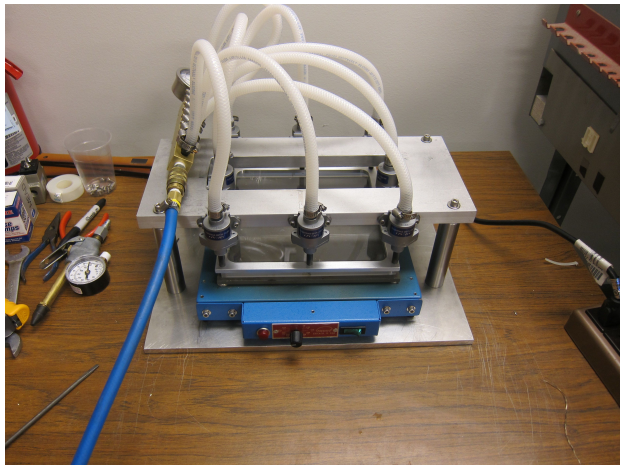
- Sealing in nitrogen atmosphere prevents serious oxidation of indium
- NiCr-Cu-In interface seems to be very strong
- More electroded 8" parts is needed
- We will heat and open last two 8" seals to investigate where the leaks come from

  - we might be able to re-seal them if the probelm is not in glass-NiCr-Cu interface
- Follow up on indium oxide removal by vacuum bake at 400C and scraping
- Seal new electroded parts

Back-up

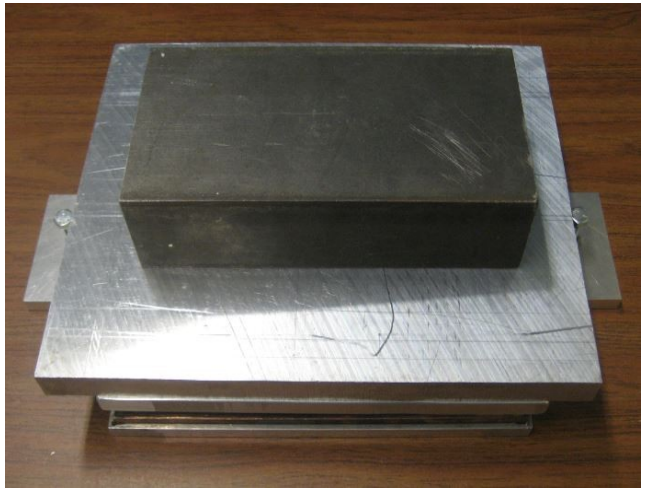
Compression

In the air

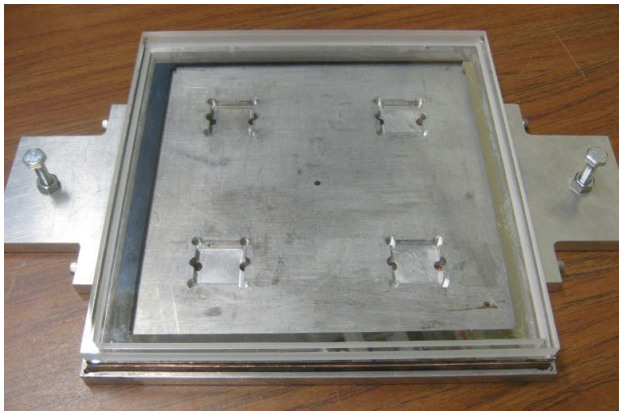


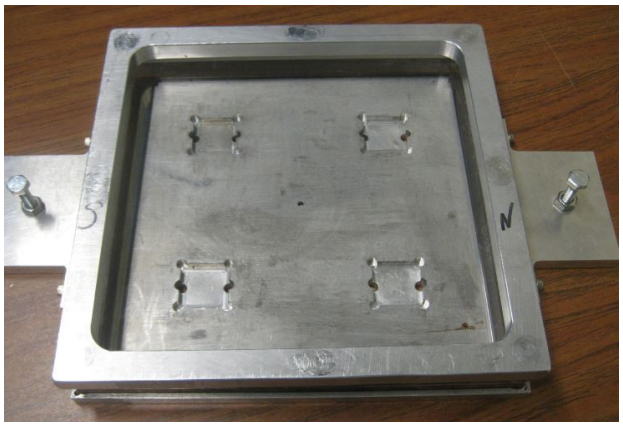
Capable of 80lbs at 8 points
Normally was used at 30lbs

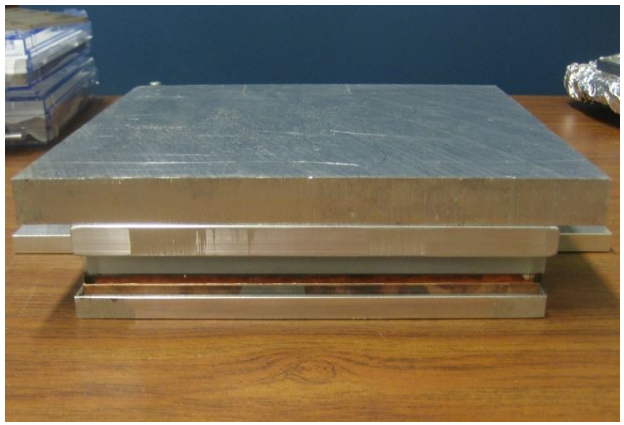
In the glove box



Pile of bricks up to 50lbs



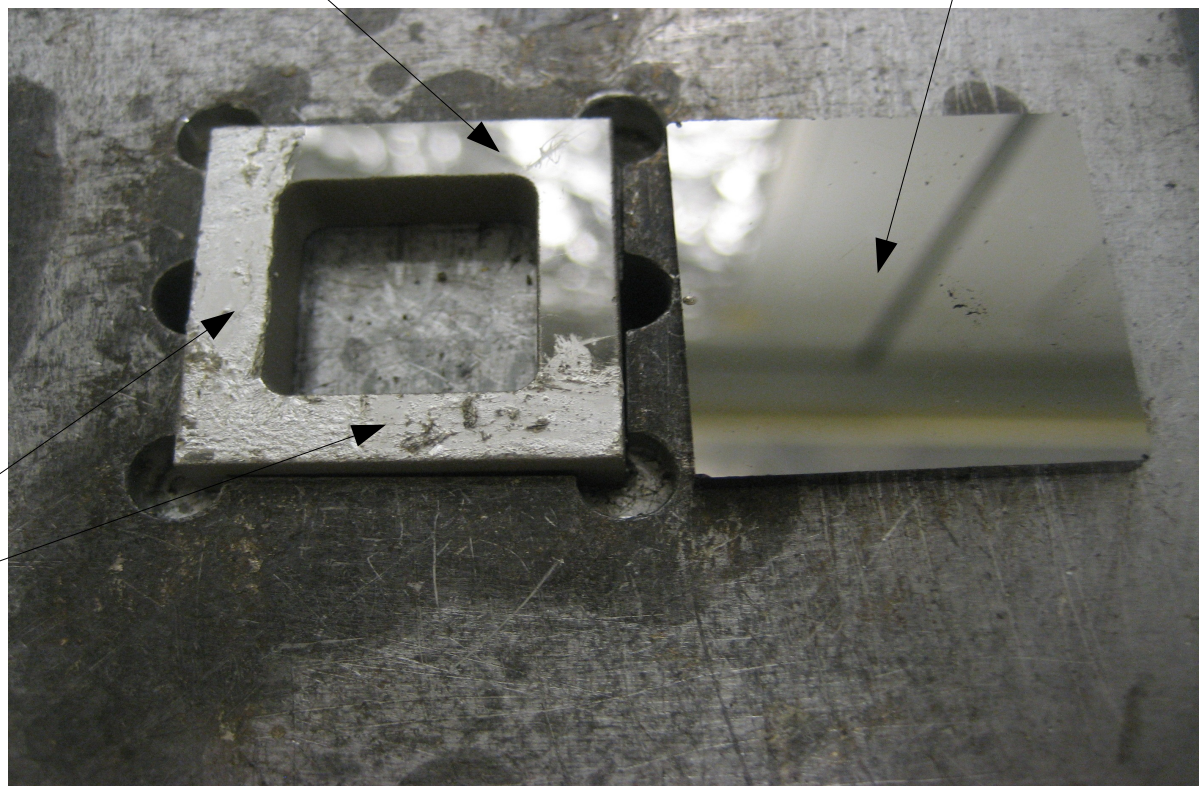




Before vacuum bake but after heating at 300C in air for an hour

NiCr with ~20nm indium sputtered on top

NiCr only

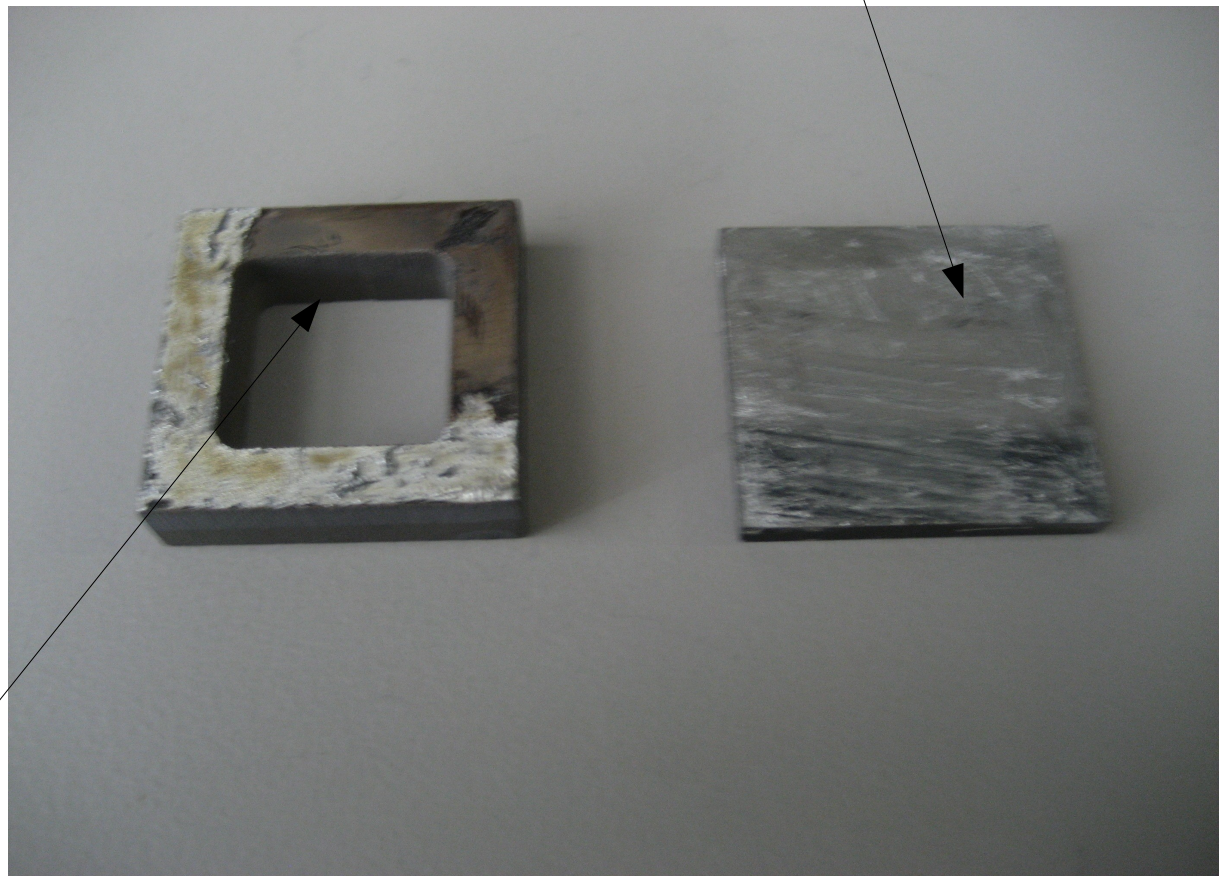


Extra indium applied

This step was done to make sure that In2O3 is definitely there before vacuum bake

After 16hrs bake @ 400C & ~10^{-6} mbar

This is different sample:
bare glass with quick tinning of indium without extensive heating in air prior to vacuum bake.
Stayed unchanged after vacuum bake



Same sample as on previous slide
Many brown spots after vacuum bake