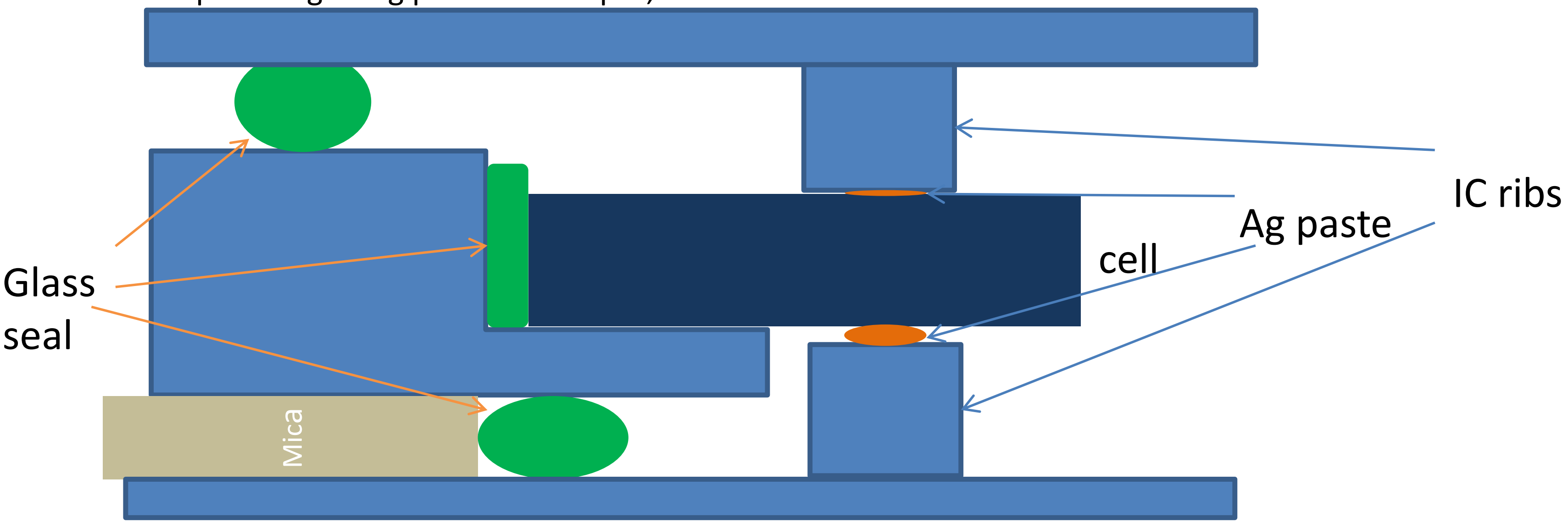
The Stack Variability Project

Steps in Stack Fabrication

Stack Fabrication steps – page 1

1. Record IDs of the ICs being used (scribe IDs on the ICs if new set is being used), Clean ICs in the ultrasonic bath in IPA and dry in air
2. Place the cathode IC on dispensing jig using short SS alignment tubes
3. Stick mica sheet using super glue, stick two ~ 0.9 cm wide mica strips on the sides of the flow pocket,
4. Apply Ag bead on to the cathode IC
   i. Calculate the height of Ag bead to be dispensed using the Excel sheet (depends upon the IC raised rib height and WF thickness etc.)
   ii. Use Program# 32, use 21 gauge metal tip needle (purple color),
   iii. Align the tip using dry runs – lower the tip so that it touches the metal surface
   iv. Adjust the height of the tip by (a) first allowing the metal tip to touch and gently scape the metal rib surfaces, and (b) then raising the tip by a 0.1 mm for a adhesion coat (group edit , press 2 → offset, press 6 → Z offset, (- value) for raising the tip and (+ value)for lowering the tip, use increment of 0.2mm if additional layers are needed)
   v. Dispense Ag using pressure >70 psi , and set the cathode IC aside



Stack Fabrication steps – page 2

5. Place the anode IC on the dispensing jig and apply Ag bead on to the ribs (follow same steps as above), set it aside
6. Place the cathode IC on the dispensing jig and apply glass bead around the flow pocket
    i. Use program# 60, use 20 gauge plastic tip (green)
    ii. Raise the tip approx. 1 mm above the IC surface
    iii. Dispense one ring of glass paste around the flow pocket using ~70 psi pressure
7. Place the anode IC back on the jig and apply glass around the flow pocket (follow same step as above)
8. Change the alignment rods, use ceramic rods from here on
9. Apply super glue on the anode IC and gently place the WF/cell assembly on to the anode IC (anode cell surface facing down and contacting the anode IC ribs)
10. Seal the assembly by apply gentle uniform load using fingers around the pocket area
11. Apply super glue on mica covered area of the cathode IC
12. Drop the cathode IC gently on through the ceramic rods on top of WF
13. Place 3 SS load blocks on top
14. Measure and record the stack internal resistance using an ohmmeter
15. Let the assembly dry overnight

Hpothesis # 1 - Paste Misalignment and Excessive Smearing

Paste Misalignment and Excessive Smearing - Fishbone Diagram

HELP

Date: 2014-07-01

Highest Score	6.6
2nd highest	5.4
3rd highest	5.0

Cause → **Effect**: *Paste misalignment and excessive smearing*

Composite score by the team

Materials

Avg	Powder/Chemicals
1.4	Powder size/composition varies across batches
1.0	Binder composition varies by batch
1.0	Solvent composition varies by batch

Avg	Cell
2.6	Cell obliqueness causing rotational misalignment
4.2	Cell thickness different from expected/assumed value

Avg	WF
2.6	Pocket obliqueness causing rotational misalignment
3.4	Excessive diameter of alignment hole

Avg	ICs
4.2	Raised ribs height too short or too tall
4.2	* Caused by recycling
1.8	* Caused by machining errors
3.0	Excessive diameter of alignment hole

Machine

Avg	Robotic Arm X-Y Stage
3.4	Excessive play between jig and its holder
3.8	Syringe tip initial location and movement inaccuracy and misalignment

Avg	Pneumatic Air Dispenser
3.8	Start/stop times not accurately controlled (delay in response of air pressure to commands)
6.6	Flow rate affected by paste viscosity variations
3.4	O-ring cleanliness and age affects tightness of seal allowing varying degrees of air leakage

Process

Avg	Making Binder Solution
4.2	Evaporation of solvent over life of batch

Avg	Paste Making
5.4	Hand mixing leads to inconsistent agglomerate size distribution

Avg	Loading of Paste into Syringe
2.2	Air bubbles introduced during loading

Avg	Sealing of Cell and WF
5.4	Misalignment of cell relative to WF (tilted or rotated)

Avg	Dispensing of Paste onto ICs (2)
4.6	Elapsed time (days?) since paste was loaded onto syringe
5.0	No defined procedure for initial positioning
5.4	Lack of alignment / reference marks
2.2	Insufficient accuracy of pressure gauge

Avg	Assembly of Parts
1.8	Elapsed time from dispensing to assembly too long

Post Assembly Events

Avg	Transportation and installation
2.2	Shifting of IC/WF relative to each other

Avg	Vibrations during testing
1.4	Shifting of IC/WF relative to each other

Interface/s

Avg	Alignment Between X-Y Stage and Stack Parts
3.8	Excessive play in alignment pins

Avg	Paste to IC/WF
3.8	Wetting properties not consistent

Most likely cause/s for Paste Misalignment and Excessive Smearing

- The paste flow rate through the pneumatic air dispenser is affected by the paste viscosity variations causing paste misalignment and excessive smearing.

Focus

- Hand mixing of paste leads to inconsistent agglomerate size distribution which causes paste misalignment and excessive smearing
- Misalignment of cell relative to WF (tilted or rotated) during hand sealing causes paste misalignment and excessive smearing
- Lack of alignment / reference marks during paste dispensing on to Ics cause paste misalignment and excessive smearing

- Lack of a well defined procedure for initial positioning during paste dispensing on to ICs cause paste misalignment and excessive smearing

Validation of the most likely cause for the paste misalignment and excessive smearing of paste in stack development

The paste flow rate through the pneumatic air dispenser is affected by the paste viscosity variations (causing the paste misalignment and excessive smearing)

Variables

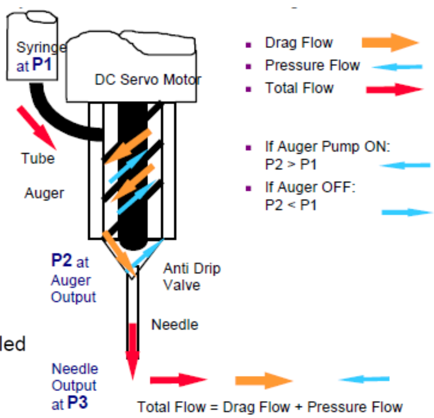
a) Dispensing system (such as pneumatic air dispenser or Auger dispenser or positive displacement pumps etc.)

b) Paste viscosity changes (due to 1. Batch-to-batch variation
2. Aging - sedimentation/solvent evaporation)

A. Dispensing systems

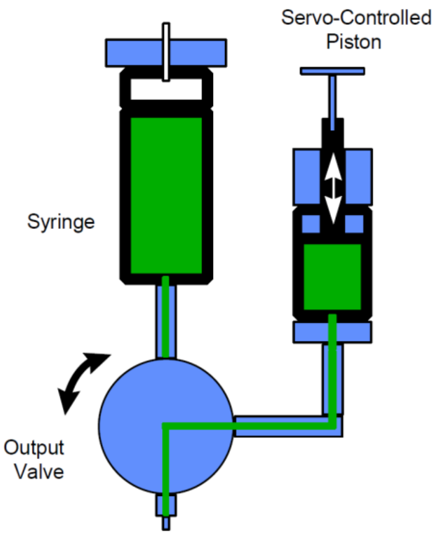
- Time pressure dispense – sensitive to viscosity variation

- Auger Pumps – less sensitive to viscosity variation, works well with high viscosity and small syringe tips



- Positive displacement pumps - less sensitive to viscosity

- Etc ..



B. Factors affecting paste viscosities

A) Materials – particle size and distribution, solids loading, binder and solvent system etc.

B) Process – method of mixing, sequence of addition, amt of dispersants etc.

C) Aging (sedimentation, evaporation solvent and agglomeration)

Viscosities are measured using a viscometer at a fixed temperature

Validation experiments

A. Assess the misalignment and smearing of std Ag paste using different kinds of dispensing pumps
   - Use outside companies and demo services to perform a demo experiment

B. Experiments which are possible with in-house equipment
   - Use Pneumatic air pressure dispenser and change the viscosity (no viscosity measurements possible but, by changing the solid/liquid ratio viscosity can be changed) and measure the misalignment and smearing

Validation experiment – A) Assess the misalignment and smearing of std Ag paste using different kinds of dispensing pumps

Experimental Plan

Objective – Assess and compare the misalignment and smearing of std Ag dots/beads dispensed using pneumatic air pump and Auger dispensing pump

Literature -

Z:\Current data\Stack Development\Fine dot and bead dispensing literature

Materials - std (24 v% Ag) Ag - Pastes – a) freshly prepared Ag-paste, b) 2 days old Ag-paste, and c) 7 days old Ag-paste

Equipment - I) Auger pump (have contacted IDS and Nordson for demo experiments), and II) Pneumatic air pump

Experiment –

1. Prepare 3 batches of pastes (as described above) using std techniques
2. Dispense 100 Dots (10 x10 each 3 mm apart) and 20 3-cm long beads (2 mm apart) using 20 and 22 gauge tips on Silicon substrates using std dispensing technique (use three paste as described above)
3. Take digital images of the dispensed dots and beads

Analysis - Devise a rating scale based on the thickness (diameters) of dots/beads, head and tail uniformity, and consistency. Analyze the pictures and rate the pictures.

Time line – Contact dispensing companies and arrange for the demo experiments - July 11th 2014

Send them the paste and substrates – July 15th 2014

Perform the experiment – July 25th 2014 (will depend on availability of lab time the the companies)

Analysis – July 29th 2014 (or 2 days after pictures of the dots/beads is received)

Report - July 31st 2014

Auger Dispensing companies

Following two companies have been contacted on 03/07/14 for the experiment using Auger pumps

- Brad Donohue, Integrated Dispensing Solutions (Will get back after discussing with his supervisor)
- Brian Duwsnap, Nordson EFD (Will get back after discussing with his laboratory staff)

Compilation of data - Misalignment in paste dispensing and paste viscosity inconsistencies

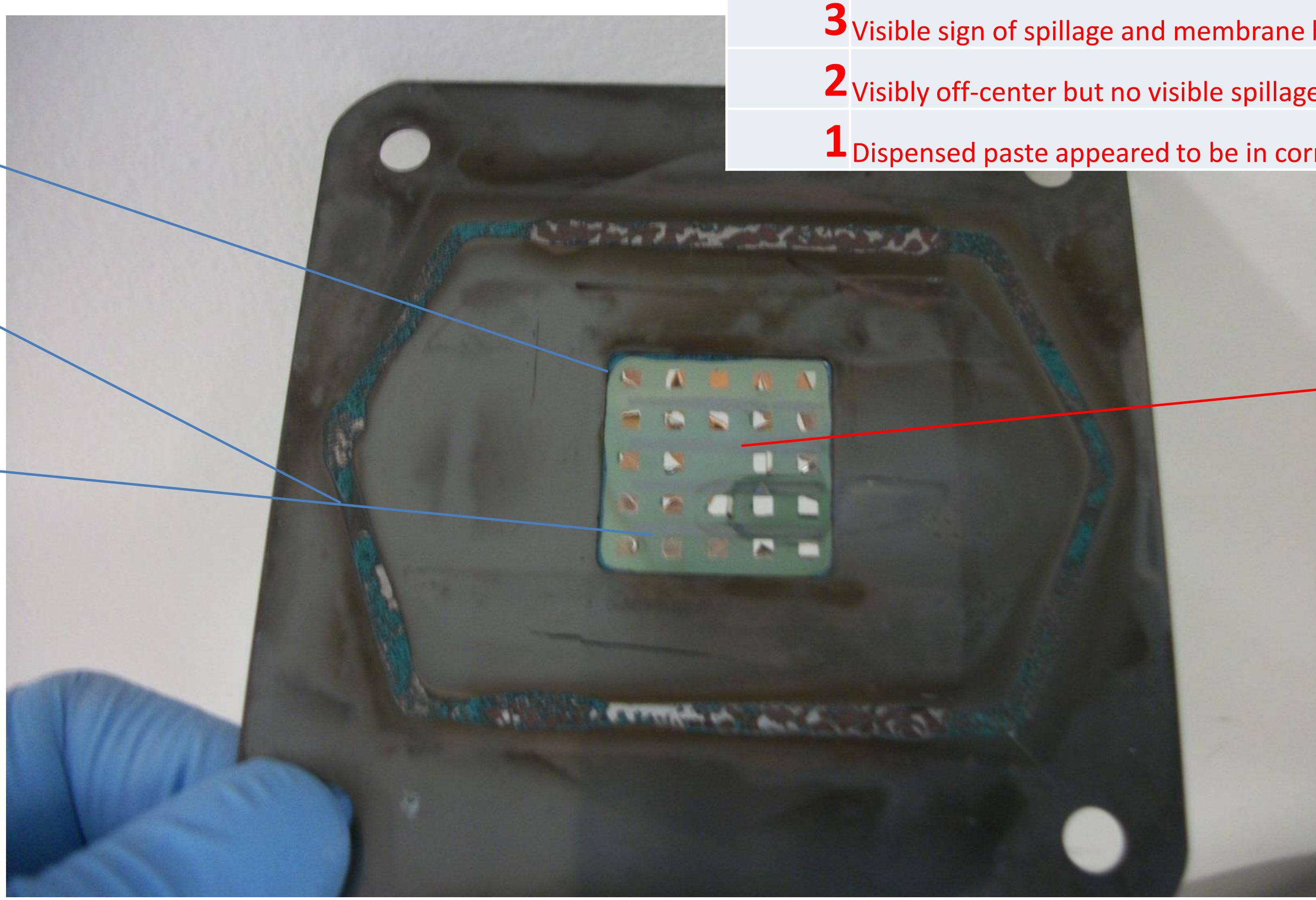
Rating Methodology

The pastes at five different locations were rated by examining the digital images from Post Test Analysis reports

1. Quality of glass paste dispensing by hand on to window frame lips to seal the cell to WF - width and shift
   - Severity – HIGH (leak is in the flow path) for OCV, and hence power
2. Quality of glass paste dispensing using robotic arm to seal WF to cathode IC - width and shift
   - Severity – LOW, may result into pressure imbalance and membrane cracking,
3. Quality of glass paste dispensing using robotic arm to seal WF to anode IC - width and shift
   - Severity - MEDIUM, may result into pressure imbalance and membrane cracking,
4. Quality of silver dot/beads dispensed using using robotic arm on to cathode IC - width and shift
   - Severity – HIGH, width and shift could cause spillage and membrane failure, low height of dispensed beads may not touch the cathode surface, could result into high resistances
5. Quality of silver dot/beads dispensed using using robotic arm on to anode IC - width and shift
   - Severity – HIGH, width and shift could cause spillage and membrane failure. The anode side design which comprises a slanting 500 micron thick silicon, may tolerate slightly wider beads, low height of dispensed beads may not touch the cathode surface, could result into high resistances

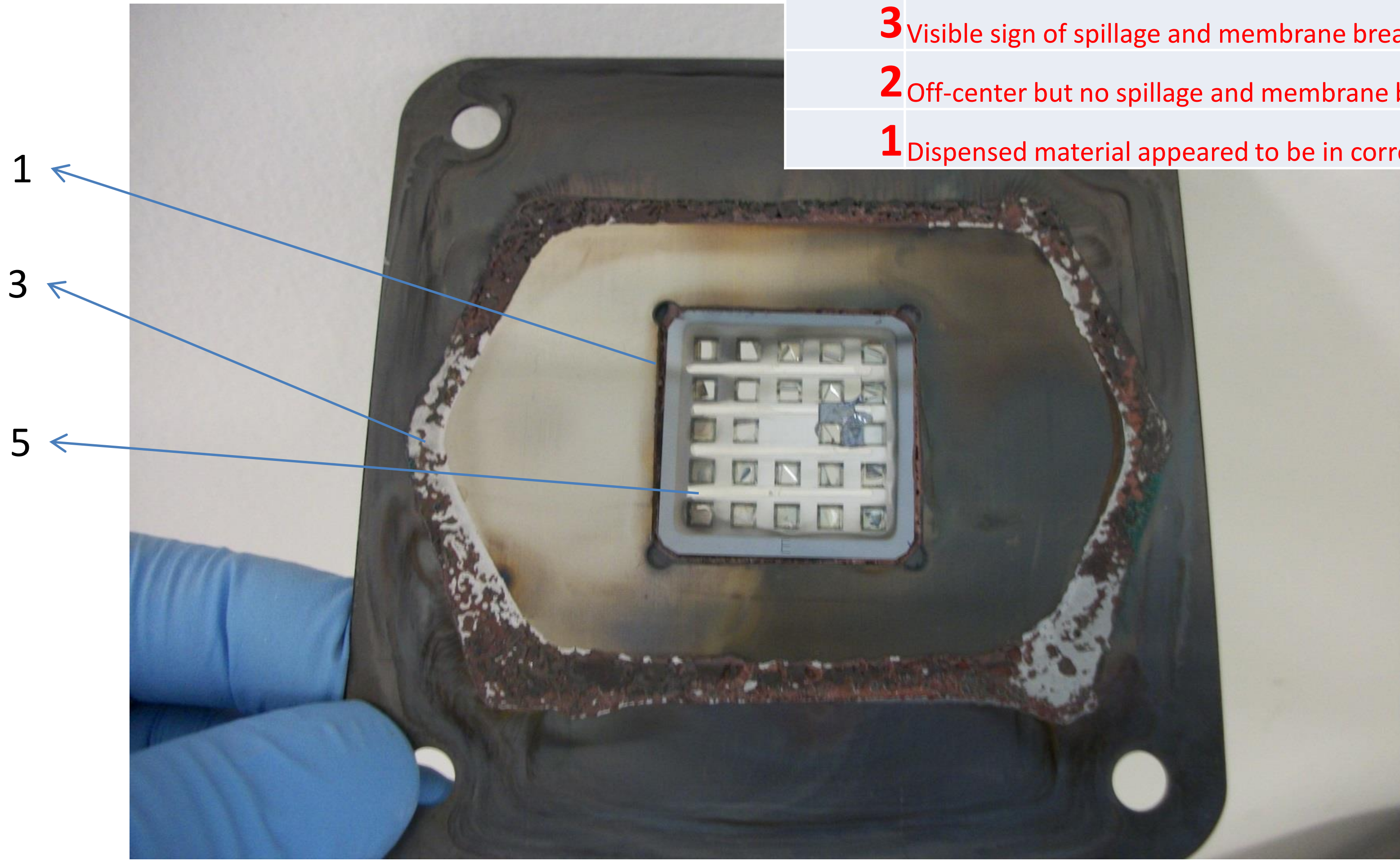
Example - Stack 116 – Cathode surface

Rating	Explanation
3	Visible sign of spillage and membrane breakage
2	Visibly off-center but no visible spillage
1	Dispensed paste appeared to be in correct location



Example - Stack 116 – Anode surface

Rating	Explanation
3	Visible sign of spillage and membrane breakage
2	Off-center but no spillage and membrane breakage
1	Dispensed material appeared to be in correct location



Cell to WF sealing steps

Taken from most recent procedure – emailed 09/11/13
Detailed txt draft is also available

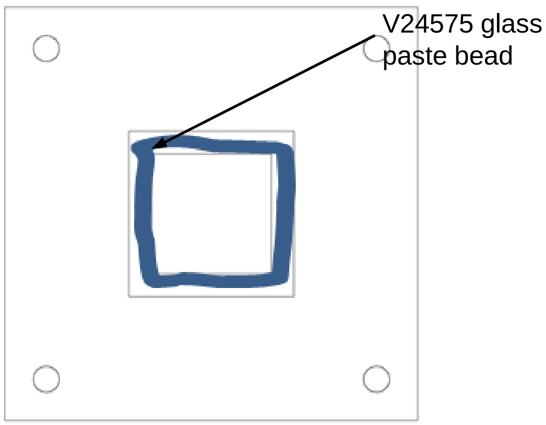
1. Use a new Kovar WF after cleaning in the ultrasonic bath using IPA
2. Apply glass paste all along the edges of the cell pocket using a hand syringe
   1. Use 20 gauge metal tip (pink)

Anode surface up

3. Place the cell in the pocket, cathode side facing down, and apply load using blunt tip on the four corners
4. Use fine tipped hand brush to apply silver paint on the ribs on the anode side (OPTIONAL)
5. Let the assembly dry for > 30 min
6. Examine the cell and membrane quality under light table and 10X magnifier
7. (optional) Record digital pictures of the assembly using digital USB camera

Rating - Window frame to cell seal

- Window frame to cell seal (width and shift),
  - Width (difficult to measure in picture),
    - less quantity could result into leakage,
    - excess quantity could result into spillage on to membrane → develop leakage,
  - Shift could cause spillage on to membrane → leakage
  - Possible causes for variations –
    1. Variation in the paste rheology (sedimentation, aging),
    2. Flatness and dimensional inaccuracies/variations in the machined parts,
    3. Misalignment a) machine (tip alignment and jig play) related, b) operator error



Rating	Explanation
3	Visible sign of spillage and membrane breakage
2	Off-center but no spillage and membrane breakage
1	Dispensed material appeared to be in correct location

Equal likelihood of all four causes

Glass Paste Formulation

Glass Paste	V24575
Date	
Operator	AV
Paste Batch #	140429
Purpose	Standard paste for sealing

The viscosity will depend on the particle size (surface area) of the powder

Adjust the amount of liquids accordingly

"solidv%=" 52.0%

Batch Weight (cc)	5
V % solids	47.0%
w% QPAC in binder solution	20.0%

Solids	v	Wt	Actual	v	v%
V24575	2.6	12.56	12.80	2.65	0.54
Pt					
Ag					
Ni					
LSCo					
Pore Formers					
xx	0	0.00			
Liquids					
QPAC40					
Propylene glycol diacetate				0.00	0.00
Binder (w 20% QPAC) solution	2.4	2.62	2.50	2.29	0.46
	Total Wt	15.18	14.27	4.94	

For Research Purposes Only

Prepared by	AV	V24575 - PbO based glass
Sealing Paste	V24575	V24575 VIOX glass
Paste Batch #		QPAC40
		Propylene glycol diacetate

Ag - 140211 | V24575-140207 | Ag-140311 | V24575-140311 | v24575-140403 | V24575-140410 | **V24575-140429** | Ag-140429

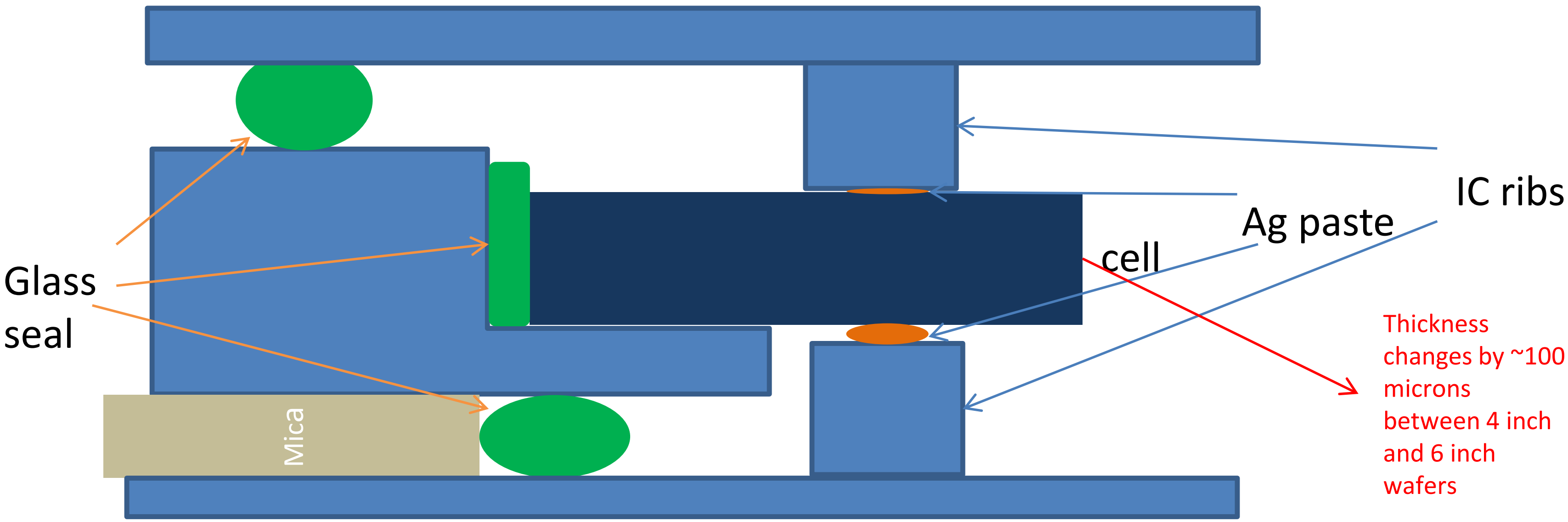
Steps

1. Weigh and mix – QPAC40 (binder) and PGD (solvent)
2. Mix and heat up at 70 C for 30 min
3. Weigh glass powder and add QPAC solution
4. Mix for 5 min and transfer in to syringe

Z:\General\paste formulations – master sheet.xlsx

Dispensing process (example)

1. Place the cathode IC on dispensing jig using “short stainless steel” alignment tubes
2. Stick mica sheet using super glue, stick two ~ 0.9 cm wide mica strips on the sides of the flow pocket,
3. Apply Ag bead on to the cathode IC
   i. Calculate the height of Ag bead to be dispensed using the Excel sheet (depends upon the IC raised rib height and WF thickness etc.)



   ii. Use Program# 32, use 21 gauge metal tip needle (purple color),
   iii. Align the tip using dry runs – lower the tip so that it touches the metal surface
   iv. Adjust the height of the tip by (a) first allowing the metal tip to touch and gently scape the metal rib surfaces, and (b) then raising the tip by a 0.1 mm for a adhesion coat (group edit , press 2 → offset, press 6 → Z offset, (- value) for raising the tip and (+ value)for lowering the tip, use increment of 0.2mm if additional layers are needed)
   v. Dispense Ag using pressure >70 psi , and set the cathode IC aside

Changes depending upon paste viscosity -> aging?

#	Test Date	Cell ID	Glass paste WF to cell width	glass WF to cell shift	Glass paste sealing WF to anode IC width	Glass paste sealing WF to anode IC shift	Glass paste sealing WF to cathode IC width	Glass paste sealing WF to cathode IC shift	Ag on cathode width	Ag on cathode shift	Ag on Anode width	Ag on anode shift
112		131127 (L)	1	1	1	1	1	1	2	1	1	1
113	2014	131127(H)	1	1	1	1	1	1	1	1	1	1
114		140129	1	1	1	1	1	1	2	2	2	2
115		131211	1	1	1	1	1	1	2	2	2	2
116		131211	1	1	1	1	1	1	2	2	2	2
117		140131	1	1	1	1	1	1	2	1	2	1
118		140131	1	1	1	1	1	1	2	1	2	1
119		131211	1	1	1	1	1	1	2	1	2	1
120		140206	1	1	1	1	1	1	2	1	2	1
121		140206	1	1	1	1	1	1	2	2	2	2
122		140206	1	1	1	1	1	1	3	2	2	2
123		140206	1	1	1	1	1	1	2	1	1	1
124		140414	1	1	1	1	1	1	2	2	2	2
125		20140225	No images - Shorting in the test stand									
126		20140226	1	1	1	1	1	1	1	1	1	2
127		20140226	1	1	1	1	1	1	2	2	2	2
128		20140421-2	3	1	1	1	1	1	1	2	1	1

ID	Glass paste WF to cell width	glass WF to cell shift	Glass paste sealing WF to anode IC width	Glass paste sealing WF to anode IC shift	Glass paste sealing WF to cathode IC width	Glass paste sealing WF to cathode IC shift	Ag on cathode width	Ag on cathode shift	Ag on Anode width	Ag on anode shift
5	1	1	1	1	1	1	2	1	1	2
6	1	1	1	1	1	1	3	2	3	2
7										
8	1	1	1	1	1	1	2	1	2	1
9	1	1	1	1	1	1	3	2	3	2
10	1	1	1	1	1	1	1	1	1	1
11	1	1	1	1	1	1	1	1	1	1
12	1	1	1	1	1	1	1	1	1	1
13	1	1	1	1	1	1	2	2	2	2
14	1	1	1	1	1	1	2	2	2	2
15	1	1	1	1	1	1	1	2	1	2
16	1	1	1	1	3	1	1	2	1	2
17	1	1	1	1	1	1	1	2	2	2
18	1	1	1	1	1	1	1	2	2	2