Calice Glue Report Section II
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Section II   Experimental

Section 1 has described in detail the properties of conductive glues and their use with Silicon detectors. Much work has concentrated on the thermal and mechanical properties of these glues. Here we describe some experimental work on long term electrical properties in the context of the Calice silicon tungsten Calorimeter, where the glue provides electrical connection between the 1 cm^2 pixels of a silicon wafer and a pcb.
As previously described we believe the Silver/ aluminium interface to be the most troublesome.

1. Snake test philosophy

The test philosophy is to continuously monitor the electrical resistance of the joint whilst it is thermally cycled to continually stress the joint via thermal expansion. Resistance is the most sensitive monitor of joint behaviour. Unlike most mechanical properties it is capable of continuous monitoring.

In the Snake test (see fig 1) glue dot pairs connect the aluminium layer of each pixel to corresponding gold pads of a double sided pc board in a similar manner to the calorimeter design. The pads are connected in series as shown. A standard wafer with 6 X 6 1 cm square pixels thus has 72 dots. This has the advantage that:

- The larger total resistance removes errors of multiple low resistance measurements
- individual dot pair measurements are still accessible by probing the corresponding pads on the rear of the Pcb
- A continuous current is applied to the joint – this important if ion migration effects are relevant.

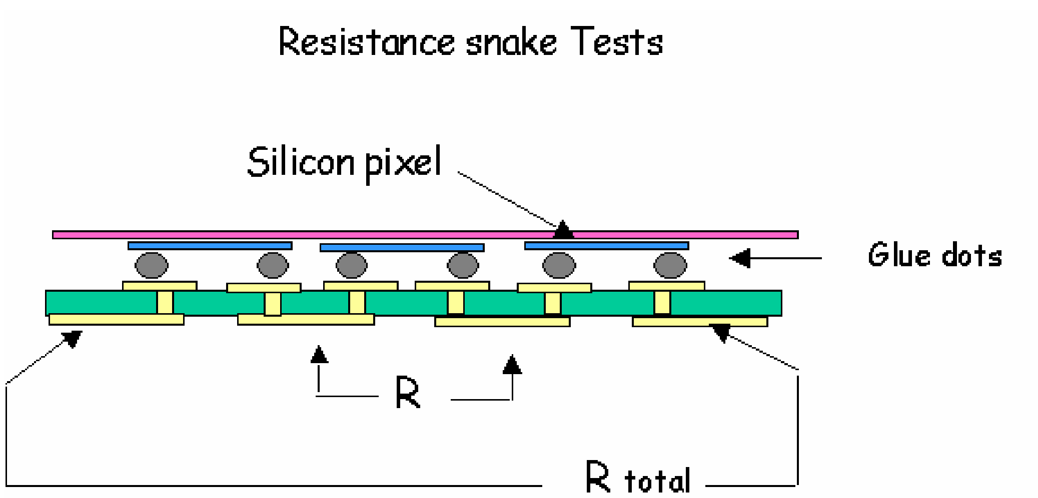

Fig 1   Resistance Snake test concept - By probing the rear pads individual dot pair resistances can be measured or the resistance of the entire string can be measured - 72 dots.

1.1 Test pcb

Fig 2 shows the realisation of the test pcb shown schematically in fig 1. This is a standard 1.6 mm thick double sided board - made by Lyncolec [1] Two pads 5mm x 3.2 mm correspond to each silicon pixel. Plated through holes connect to corresponding pads on the rear. Tracks interconnect pads to form the 'snake' . The resistance of individual dot pairs can be measured by probing the individual pads. The plating is the industry standard gold on nickel process. Previous studies (see section 1) suggest there are few problems at the gold/glue interface. Pcb layout was provided by courtesy of Scott Kolya.

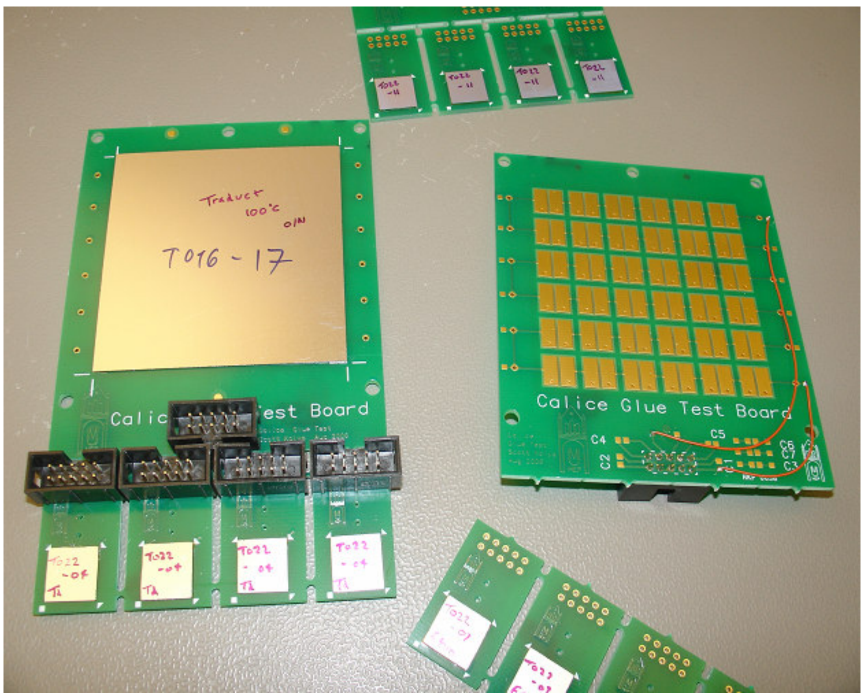

Fig 2 Glue Test boards

2 Silicon wafers

10 silicon wafers were provided by the Calice Prague group courtesy of P. Sicho. They were rejects from those used to assemble the calorimeter test having low breakdown voltage or similar faults.
All assembly and handling took place within a class 10000 clean room.
Wafers were used out of their sealed packets. No surface cleaning was attempted.

3.1 Glue Mixing

Glues of interest are two part epoxies. For Epotek 4110 the recommended manufacturer's ratios are 10:1 by weight. Typically 5 g / 0.5 g amounts were weighed out using a good electronic balance into a plastic mixing pot. Care was taken to maintain the specified ratio. The two components were then mixed by handed using a small spatula for 5 minutes before being transferred to a 10 ml syringe. With such small samples there appeared no evidence of bubble formation and no vacuum degassing was used. Glue remaining in the pot was kept to check pot life.

3.2 Glue Dispensing

The glue is dispensed by a Sony Cast Pro Assembly robot to ensure repeatability. See Fig 3. The robot provides programmable xyz motion with 20 micron step size. The Z axis supports a volumetric dispensing head (LCC Dispensit) [2].

Freshly mixed glue is placed in a 10ml syringe under 6 bar nitrogen. At the end of the syringe a fine deformable plastic tube connects to the output needle. Two pins driven by pneumatic cylinders pinch the tube in sequence to produce a defined volume of glue. Micrometer control of the end stops provides adjustable volume control.

A small CCD camera mounted on the Z-axis provides a magnified image of the end of the syringe/deposited dot pattern. This helps tuning of the height of the dispenser tip above the Silicon wafer - typically 500 micron. Height adjustment is a sensitive parameter. Too low risks wafer damage, too high gives incomplete glue dot transfer.

The useful pot life is a couple of hours. Most of our samples were dispensed within 30 min or less of mixing.

Sequences of runs were performed to establish dot parameters, optimising size repeatability and eliminating glue tails.

Fig 4. shows a typical Pcb after glue deposition, showing uniformity of production. The dots are typical about 1.6 mm diameter.

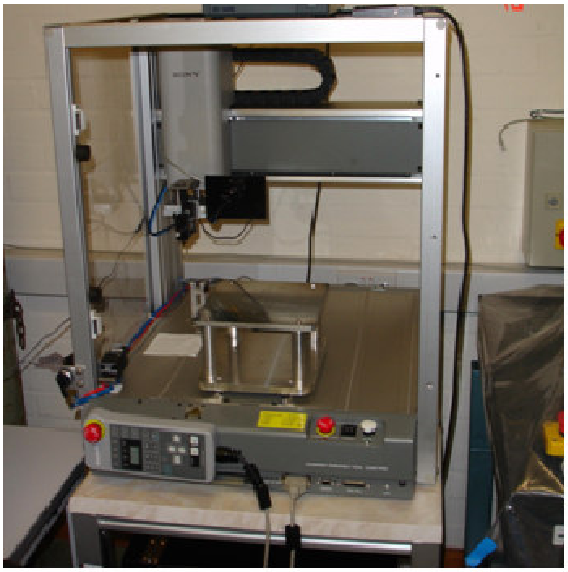

Fig 3 Sony robot with Glue dispenser head

3.3 Wafer assembly.

Immediately after glue deposition a wafer was manually placed on the pcb using a vacuum pickup.
The wafer position on the pcb is defined by the blue corner pads, and the gap thickness is defined by Kapton tape spacers to be about 66 micron. A small weight ~100 g was used to hold the wafer down during curing.

3.4 Curing

The Pcb/ silicon assembly was transferred to an oven and cured, typically at 100 deg C for 2 hours. A high temp cure was chosen as a worst case scenario – more likely hood of stressing glue joints.
Examination of dots produced using glass slides and destructive separation of one wafer/pcb pair show formation of clean round glue ' buttons ' about 1.6 mm diameter , corresponding to a volume about 0.13 mm2. See Fig 5

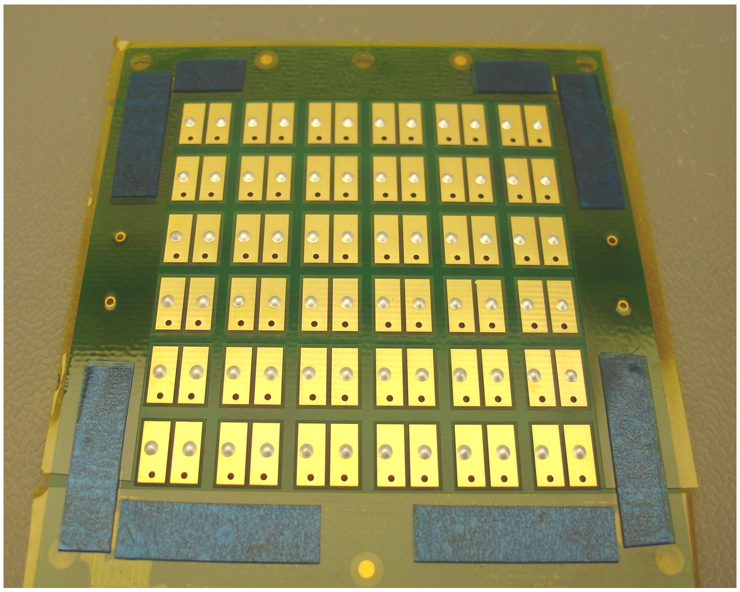

Fig 4 Showing deposited glue dots + yellow kapton spacing film.

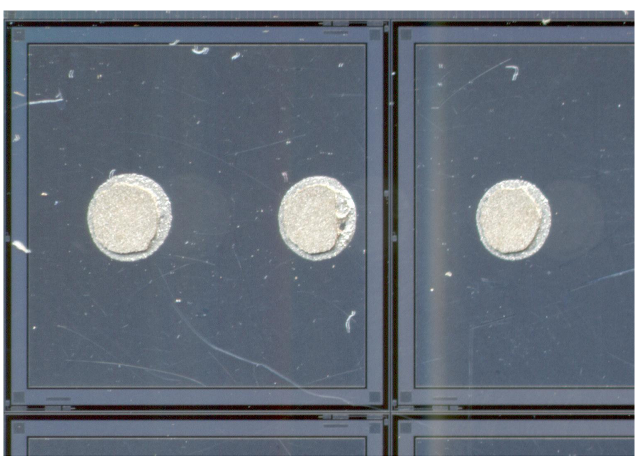

Fig 5 Disassembled wafer showing cured glue dots on a 1 cm2 pixel

3.5 Small 3 mm pixels structures

We also had access to some small 3 x3 test structures with 3 mm pixel size processed with each main detector .We have tried to utilize these .
However Fig 6 shows that the electrode area is only about 1.7 mm square. We found we could not consistently form small enough dots that would function reliably. A move to a lower viscosity glue formulation Such as E4110- LF should improve the ability to make smaller dots, but conversely the glue is more mobile.

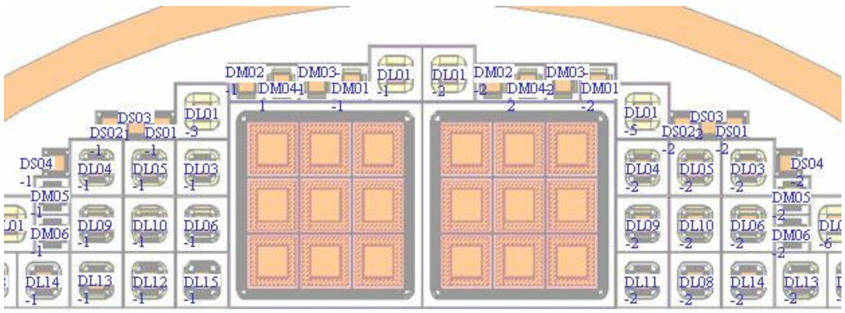

Fig 6. 3mm pixel test structures at sides of wafer.

4. Thermal cycling

The detector/ pcb assembly is thermally cycled to generate mechanical stresses. The detector board is placed in a 40 litre programmable environmental chamber [3]. Temperature and humidity are data logged along with i/v measurements at 1 minute intervals. Fig 11 shows the standard temperature cycle - a 50 degree swing from +20 to +70 deg C at a ramp rate of 1 deg C/min with a 60 minute dwell time at top and bottom of the cycle too allow for thermal stabilisation. Normal room atmosphere was maintained in the chamber.
Humidity in the clean room is maintained at 40% +/- 5 % at 20 deg c.

5. Resistance measurement

The resistance of the detector assembly is derived from i/v measurements using a Keithley 247 source measure unit (SMU).
This enables us to source either current or voltage to high accuracy. For the snake tests measurements were usually made made sourcing I in the range 10 – 50 ma and measuring V.
A Labview program provides control and monitoring, histograming etc.
Figs 9,10 show typical displays. Data is typically logged at 1-minute intervals Measurements of individual dot pairs can be made by probing the pads on the rear of the pc board

6. I/V effects

We have observed interesting I/v measurements on so called " Virgin" glue, Ie cured glue that has not previously had any volts applied. Figs 4 and 5 show typical behaviour as a function of voltage. Individual glue dot pairs were probed via the rear pads on the pcb. A Keithley 237 source measure unit applies an

increasing voltage staircase in 50 mv steps of 10 seconds duration until a current compliance (usually 50 ma) is reached. At low voltage (few hundred mv) there is a relatively high resistance state typically 10 k ohms. As the voltage is increased there appear a chaotic series of transitions of generally decreasing resistance leading to a sudden jump to a low resistance state (few ohms) at voltages above about 2 volts. Once established this low resistance state appears permanent even after removing the voltage.
The effect doesn't appear to be cure related - It still happens the first time the glue is powered even if this is some days after initial cure.

We believe this is understandable in terms of the complex conduction path ways present in the glue as described in part 1. At various interfaces both at the surface and possibly between silver flakes there will be nanometre scale phenomena such as oxide films which limit conduction.
We believe as the voltage is increased these films are disrupted by a variety of mechanisms such as tunnelling to direct breakdown and mechanical expansion due to local heating- this is the chaotic phase about 1 volt. As more current flows feedback occurs - more current gives greater heating, as individual oxide paths breakdown, more voltage appears across remaining high resistance points.
At some point this internal disruption creates a robust conducting path which remains permanent.

This effect can be a source of great experimental confusion, compounded by the general tendency to use handheld DVMs to check resistances. These tend to use low voltages (to minimise taking high currents on low resistance ranges).

It is entirely believable that joints left unpowered for long times in lab atmospheres could redevelop these increased resistance effects.

In the case of the Atlas detectors there is clear evidence that ~20 % of modules gave high resistance values on acceptance tests at CERN when measured with low voltage sources. These modules had previously been unpowered for significant times since construction.
It has been demonstrated (Jones [31] section 1) that hv bias ing the module and sourcing current cures the problem, re-establishing the conducting path. Due to 50kohm Hv protection resistors on the module, this is only applying a small voltage drop across the glue joints, consistent with what we see with new glue.

It would clearly be instructive to look at glue joint resistance after a long times unpowered (> year).
For the Atlas detectors we have a database of 1200 glue resistance values taken in the construction phase of the forward SCT. The measurement protocol explicitly applied 2 volts to the joint before measurement to ensure a low resistance. We have seen no obvious correlations in the cases of modules that have developed high resistance in the Cern acceptance tests.

We have looked at ways of revisiting them, however once assembled we have no direct electrical access to individual glue joints. The 4 detectors are in parallel, with a connection via series bias resistors).Any measurement would require the effective disassembly /destruction of the module, and a reasonable number would be required to be statically meaningful.

Similarly we cannot directly access the glue joints in the Calice beam test protype.

Our test boards have been designed so we can directly remeasure individual dot resistances at various times in the future.

It would also be interesting to see if there was a difference between detectors stored exposed to normal air and those stored under nitrogen.

We believe the resistance effects are generic. We have observed them directly with Traduct 2902 (Atlas) and Epoteck E4110 (Calice). CMS initially used Traduct 2902 then switched to Epoteck 129-4 (a variant of E4110).

It is important to realise that the once off application of a small bias voltage to the joint, sourcing current seems to reset these problems by reforming the conducting paths.

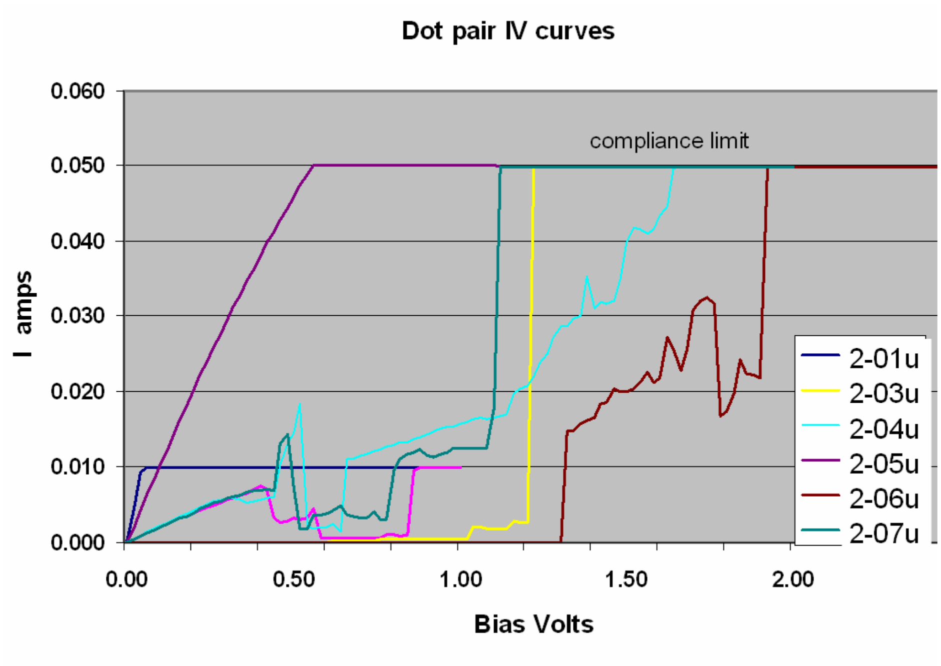

Fig 7 Individual dot pair IV curves for " Virgin" Glue showing chaotic transitions from high to low resistance states as voltage is increased.

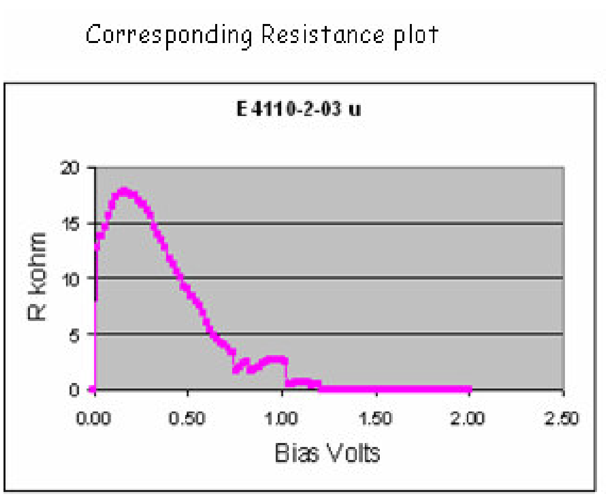

Fig 8 Corresponding resistance plot showing transition.

7 Thermal cycling measurements

Fig 9 shows typical result from thermally cycling the glued wafer/pcb as described above. The red plot shows the temperature cycle, the blue the measured series resistance of the 72 dots. We see an initial resistance of some 400 ohms which seems relatively stable for the first 20 hours. It is possible the glue is still finishing curing.

We would expect a conventional change in conductor resistance on each temperature cycle for silver of ~ 40 x 10^{-4} (Au, Al are similar). For a 50 deg C swing this would be 50 x 10^{-4} = 20 %. In the first few cycles we see changes of lower than this, implying as one expects a significant component of the resistivity variation is not metallic but due to the interfaces. Over the next 100 or so cycles we see the variation increases significantly, typically 100 ohm changes suggesting that on each cycle as the glue expands and contracts the interface contacts are changing. Note that the values on each cycle are different. Remember we are integrating over 72 separate dots, 144 junctions. However the overall resistance is dropping. Presumably oxide layers etc are being mechanically disrupted here. With no cycling the resistance is stable at about 90 ohms. Resumed cycling continues with 100 ohm variations on each cycle, but the underlying resistance is now stable between 200 to 400 hours. One can think of this in terms of the silver flakes etc settling down to some sort of optimum minimum energy arrangement.

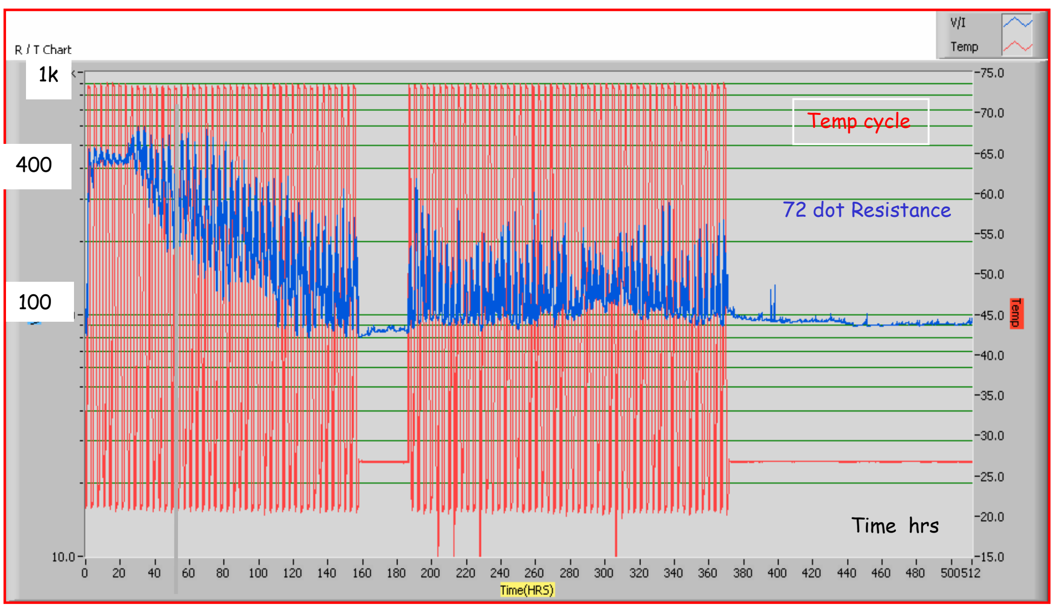

Fig 9. 500 hr thermal cycle

Fig 10 shows a different example, this time running for 1000 hours. Here we see a similar resistance of 400 ohm being reached after the first 20 hours. For the next 500 hrs the large variations of typically 100 volts on each cycle appear, but with the mean value varying fluctuating in the range 400-300 v. After 500 hours a more stable regime appears, settling down to about 300 ohm. The amplitude of

Fig 10 1000 hr   Thermal Cycle

the variations on each cycle slowly reduces to about 60 volts, consistent with the 20 % change expected of just the metallic contribution, suggesting again a stable state has been reached.

In all our examples we see no evidence for obviously increasing resistance or joint failure. If anything, against expectations thermally stressing the joint when powered seems to give some slight improvement. This is possibly understandable in terms of the flakes reaching some optimum packing density, akin to shaking a packet of cornflakes

8 Deliberately induced Failure

We have attempted to simulate bad surface oxide effects by constructing a snake test using old silicon wafers that had been in the lab for a long time. Fig11 shows the results. The Temperature cycle is as before :+ 20 to +70 deg C at 1deg/min. On each temperature cycle we see a rise in resistance. This due to a mixture of effects a) resistance rise with temperature of the conductors, and b) a component due to contact changes on expansion. Normally these would both be reversible on cooling. In this case we see that on the cooling part of the cycle there is a drop due to the metallic conduction, but the contact resistance component stays high. Clearly on each cycle the joint resistance is getting worse, fairly rapidly in this case.

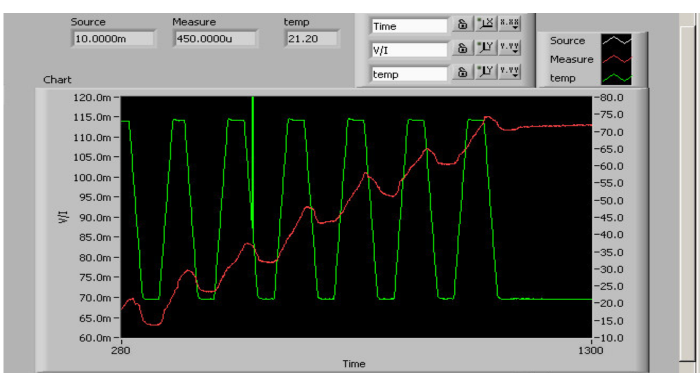

Fig 11 Temperature Cycle old Silicon Snake

9 Radiation effects

Ionising radiation generates free radicals in organic materials due to the rupture of covalent bonds. These can then react chemically, altering the material structure and hence its properties.
A large body of previous work has been done on radiation induced changes in glues (and other materials) by the CERN machine group and other detector groups in preparation for the LHC

See for example Cern reports 79-04 79-08 82-10 89-12 96- 05 2001-008
In general epoxies show no significant degradation up to at least 100 kgray
A variety of silver loaded conducting glues have been validated for the LHC trackers which will have accumulative 10 year doses in the range 10 to 1000 K gray.
Dose rates at the ILC are estimated to be a factor 1000 lower.[Dauncey 2004] .So we do not anticipate any radiation induced effects.

10. Conclusions

1. Virgin unpowered glue shows an initial high resistance state that is changed into a low state by the once off application of a few volts, sourcing say few 10s of microamps

2. When powered, glue connections maintain a low resistance state when aggressively temperature cycled for > 1000 cycles in air. If anything, contrary to expectations stressing glue joints whilst powered seems to lead to slight improvement.

3. Atlas experience suggests that a significant fraction (20%) of joints left unpowered for long times - few months to a year in air develop resistive states

4. The normal low resistance state can be reformed as in (1) by the application of some volts to the joint or sourcing current - Due to 50 kohm series resistors in this corresponds to a high voltage bias.

5. Because normal application of the detector high voltage bias 'cures' the problem, this effect may well be going unnoticed in many other detectors.
It only came to light due to low voltage checks.

These effects have been explicitly observed for large samples of Epotec 4110 and Tracon 2902 connected to aluminium. We believe they are probably generic properties of silver loaded glues.

6. 1.6 mm dots were used. We could not reliably push dot sizes bellow 1mm

7. In short, use of conducting glue to make connections to silicon detectors is clearly a mature technology, with large scale working detector arrays to demonstrate it.
There seems to be little evidence to suggest that long term aging effects when powered are a problem.
If anything problems seem to be associated with unpowered modules.
The resistance effects outlined above, whilst confusing seem curable by simple means. In a normal Hep environment detectors once installed are usually

continually powered anyhow. One should also bear in mind that in the LHC trackers a open circuit glue joint removes an entire module, whereas in the Calice calorimeter it merely removes one pixel. Recall that the use of silicon with up to 1% dead pixels on purely economic grounds has been seriously considered.

8. Whilst a perfectly viable technology, the use of conducting glue is a fiddling one. A purely personal preference is that a cleaner, more elegant solution is that proposed for SID, in which a fan in tracks are incorporated into a further metalisation layer on the silicon, fanning into a central connecting pad which is bump bonded to the readout Asic.

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References

[1] Lyncolec Ltd, 2 Abingdon Road, Nuffield Industrial Estate, Poole, Dorset, BH17 0UG, UK.

[2] Despensit . Integrated dispensing equipment - 8400 Port Jackson Ave N.W North Canton,Ohio 44720- 5464

[3] Environmental Chamber – Design Environmental Ltd 232 Rassau Ind Estate Ebbw Wale Gwent

[4] Cern Yellow reports 2001 -008

[5] Dauncey Oct 2004 Discusion paper on Maps in a LC electromagnetic Calorimeter www.hep.ic.ac.uk/Calice/maps/mapsdd/mapsdd.ps