Conducting Adhesives for Electrical Connections in a Silicon Calorimeter.

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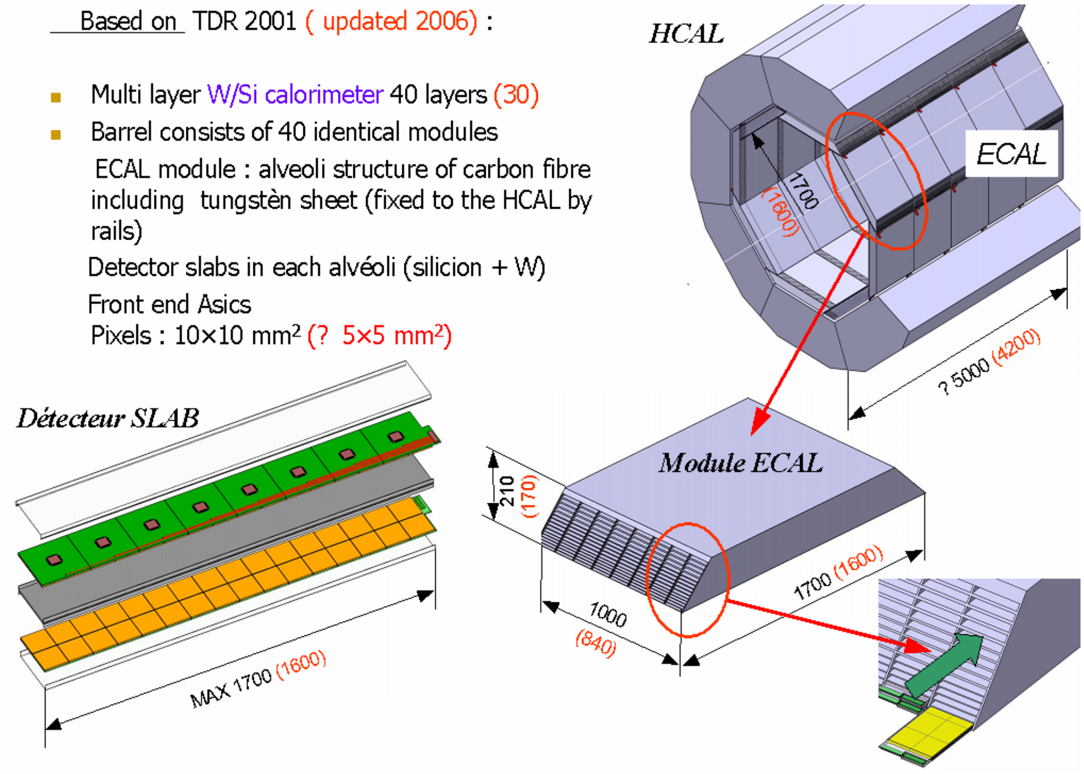
1. Introduction.

The aim of this note is to review the use of electrically conducting adhesives as a means of forming connections to the silicon detector pixels of a proposed silicon /tungsten calorimeter for the International linear collider.

The CALICE collaboration has proposed the construction of a fine grained large silicon/tungsten electron calorimeter using an energy flow approach [1].

The baseline concept [2] consists of layers of silicon detector alternated with layers of tungsten converter, typically 2mm thick. There will be about 30 active layers
The silicon wafers are divided into small pixels, typically 1cm^2.Wafer size will be dictated by economic considerations at the time of assembly. Each pixel provides analogue information giving fine grained resolution of the shower. Such a detector would be a factor of 10 larger in area than current detectors. A calorimeter surface area of ~50m^2 x 30 layers would require 1500m^2 of silicon or 1.5x 10^7 pixels.

Fig 1 shows a possible mechanical design. A 'chest of drawers' structure is formed of alternate layers of tungsten sheet supported in a carbon fibre matrix. The 'drawers' consist of a single tungsten plank supporting on both sides a layer of silicon detectors supported on a thin pcb layer. Electrical connection is made from each pixel using electrically conducting glue via pcb vias to a local readout asic on the reverse side of the pcb. The multiple asics per plank are connected electrically or optically to a readout controller at the end of each plank.
Possible alternative pixel sizes of 5mm or even 3mm are under study.



Based on TDR 2001 (updated 2006) :

- Multi layer W/Si calorimeter 40 layers (30)
- Barrel consists of 40 identical modules

ECAL module : alveoli structure of carbon fibre including tungstèn sheet (fixed to the HCAL by rails)

Detector slabs in each alvéoli (silicion + W)

Front end Asics

Pixels : 10×10 mm^2 (? 5×5 mm^2)

Fig 1. Tungsten plank showing detectors bonded to pcb.

A prototype detector segment [3] has been assembled for beam tests. This contains 30 layers each made up of nine 6 cm square detectors cut from 4” wafers, each comprising a 6x6 pixel array giving approximately 10 k 1cm^2 pixels in total
Spots of conducting glue EPO-tek 4110 deposited by a pneumatically driven syringe dispenser mounted on a robot provide the signal connections from the silicon to the PCB. For the prototype, signals are routed to front end electronics at the side of the boards. This prototype has been (partially) assembled since late 2005.

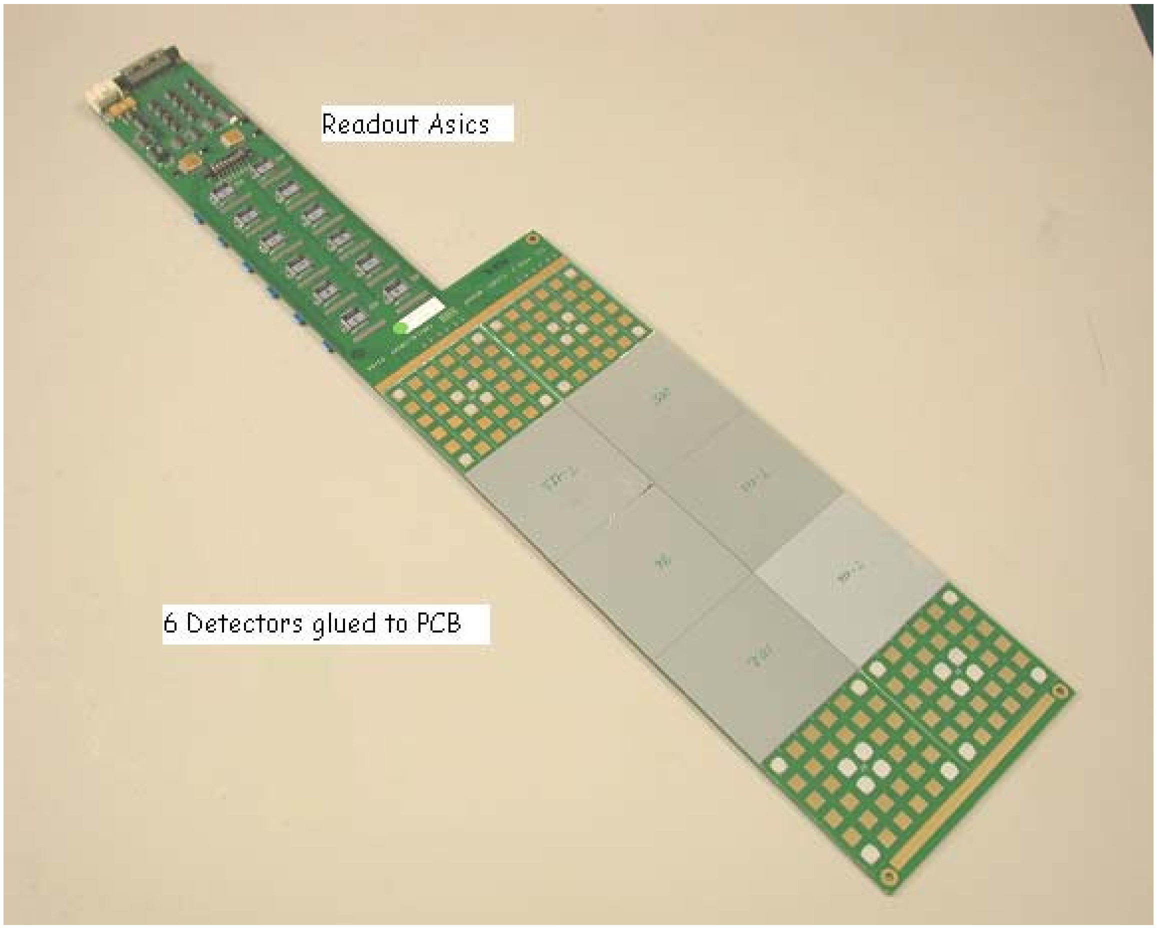


Fig 2 PCB + detectors for CALICE test beam prototype .

Alternative approaches
An obvious alternative approach to a conducting adhesive is to mount the readout asic on to a silicon wafer directly using ball grid bonding.This would of course require additional metalisation patterning on the silicon. Fig 3 shows a proposal by Frey et al [4] in which a 6 inch wafer provides ~ 1000 5mm hexagonal pixels leading to a central 7x7 mm pad array on to which the readout asic is ball bonded. Output signal etc are wire bonded to the support pcb.

Maps

A more advanced idea is to integrate the detector and asic on the same piece of silicon. There would be a significant cost advantage in being able to use using conventional CMOS silicon [5]

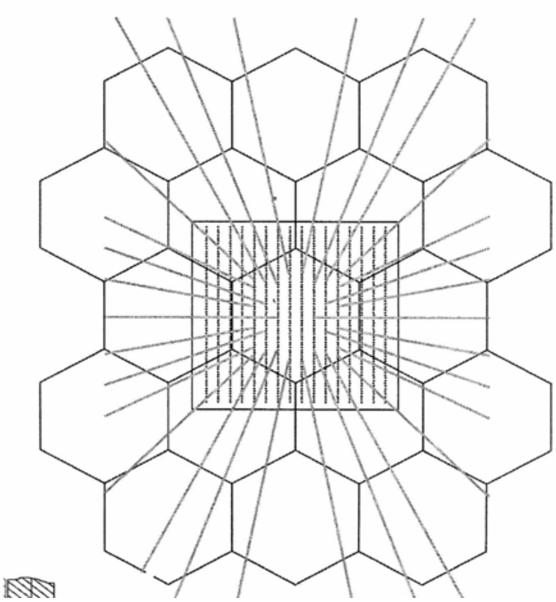


Fig 3. The centre of a 1000 hexagonal pixel wafer showing the bump bond array at the centre for a single readout chip. A few representative traces from the pixels to the bump bond array are shown

2. Information search

Adhesive joints are complex physical and chemical systems, with many interfaces and behaviour scales. There is no overarching theory of adhesion. For a specific joint one or more adhesion theories may be useful.
For the subset of conducting glues the mechanisms of conduction provide yet another dimension of interest.

An information search has concentrated in two main directions
1. A literature search from the electronics industry.
Electrically conducting adhesives have been known and utilized for integrated circuit die mounting for some decades together with some use in hybrid circuit assembly. These were relatively undemanding applications in terms of the material properties required of the adhesives.
Attempts to expand out of these niche markets, to exploit the move to surface mount technology initially via flip chip bonding, by specialist companies provide most of the accessible papers of relevance – for instance early development work lead by Estes and colleagues. [6] ,[7] These are relatively open as they are trying to promote and justify an infant technology.
As conducting adhesives have colonised more mass consumer markets, exploiting the requirement to connect to glass or plastic substrates such as LCD displays,

processes which require lower temperatures than solders have become more proprietary.

Current environmental concerns outlawing the use of lead, driving up both the cost and more importantly the operating temperatures of allowable solders have provided an even wider potential market for solder alternatives.

Industry hence has very large data samples, and the resource and motivation to do long term detailed investigation ,however the commercial imperative rules and to quote a previous researcher [8] “still there is a lot of secretiveness in the adhesive world” and many papers tend to be highly specific.

Dominant concerns for the industry have been on bonding to SnPb materials (the standard component solder surfaces) nothing found on gold bonded to aluminium Similarly the environmental operating concerns pertinent to consumer devices such as operating temperature ranges, exposure to humidity, shock resistance (think of a mobile phone) don’t tend to correspond to particle physics operating conditions which seem to be less onerous in these respects.
Hence industry tests such as 85/85 would seem to have limited applicability

Useful summaries together with many references are to be found in[8],[9],[10],[11]

2. The HEP world

In the absence of comprehensive theory, engineering often proceeds by what is sometimes referred to as the Roman method - in other words to use previously demonstrated techniques.

Wafer scale silicon detectors have been used in HEP since the 1980s, from first generation uses at the 1m^2 scale such as the vertex detectors and very forward calorimeters of the LEP era leading up to the 100m^2 scale of the recent generation of experiments such as the LHC.

Clearly information here is in some ways far more pertinent, and the particle physics world is far more open. However it is also rather more fragmented and tends to the anecdotal.

The main recent use of relevance has been the provision of high voltage bias and ground connections to silicon detectors for LHC together with a few more demanding applications in satellites.

Meaningful long term data samples are still relatively small. For example the 4000 modules of the recently built Atlas inner tracker for the LHC use conducting glue for the hv bias connections – possibly 20 k joints, but assembled at a large number of different sites under subtly different protocols.
Once assembled in a detector glue joints are not usually directly accessible, either physically or electrically, hence ageing behaviour is often not detectable until it is very advanced.
Unlike major electronics assembly companies, physicists tend not to be interested in glue as a subject-at best it’s a means to an end. As a result problems tend to be bypassed rather than deeply investigated, for example see section (9.2) If you have a problem sticking two bits of wood together the tendency is to find a nail and a hammer rather than an infrared spectrometer.

Some more rigorous testing has taken place in the context of silicon trackers for satellite applications [Glast,AMS,Pamela]. This has concentrated more on mechanical and thermal stress concerns due to space conditions, The Calice glue E4110 was chosen for Glast [12]

One can hence usefully compile a selection of glues used and pitfalls to be avoided, although lifetime predictions of conductivity remains something of a dark art.

3 Taxonomy of conducting adhesives

As stated above the general idea of conductive adhesives has been known for some decades: an organic resin base loaded with conducting particles is used to surface mount a component to some substrate. The adhesive provides the means of mechanical attachment, and conducting paths are established though contact between the metallic particles.

Electronically conducting adhesives can be divided into two main types:

Isotropic -(ICAs) which conduct in all directions – these are usually resin pastes heavily loaded (~40% by volume) with conducting particles such that multiple point to point contact exists when the adhesive cures.

Anisotropic –(ACAs) adhesives which conduct in a designed direction, sometimes called Z-axis adhesives. Although paste forms exist, these tend to be thin thermoplastic films with a significantly lower conducting particle density (5-10% by volume) such that a single particle provides the conducting path between substrate and component (the z –axis) whilst particles are sufficiently isolated to provide no conduction in the plane of the film.
Anisotropic adhesives tend to use initial pressure to ensure contact between the conducting particle and the substrates – the glue matrix merely maintains this.

Further subdivisions are by
1**) The adhesive matrix,** either thermosets which are permanently changed by heating (curing) or thermoplastics which just melt and reset and can in principle be reworked .

2) **The conducting material**.In principle a variety of conducting powders could be used as the filler .The obvious conductors gold, silver, copper, nickel have been used together with carbon for high resistance uses such as antistatic or shielding applications. For isotropic use the most common filler is silver. For anisotropic use the normal material is gold or gold/nickel coated polymer spheres

For the Calice application we are primarily interested in Silver loaded epoxy based ICAs and hence what follows concentrates in this area.

Isotropic Conducting Adhesives

In practice the dominant usage has become silver because apart from the metals high conductivity, unusually silver oxide itself is relatively highly conducting, unlike most metal oxides. Also silver powders can be produced (usually by precipitation techniques with a high degree of control over particle size, porosity and form (a typical manufacturer's catalogue lists 20 or so silver powder formulations). The preferred form is usually silver flake, with a broad size distribution where overlapping flakes will improve the conductivity. The silver flakes or powders are pre-treated with organic lubricants to control the silver dispersivity and the rheology (flow properties) of the adhesive.

Gold does not form any oxide layers, but remains a niche use, restricted to specialist space and military applications where cost is not an issue.

Metal plated particles can be used. The main use of such particles is for anisotropic adhesives. For isotropic adhesives the main idea has been to reduce cost by reducing the silver content.
In the HEP world there are applications where one would like to lower the silver content - those where one is fighting to reduce radiation length, and those in high radiation environments where the activation of silver can be undesirable.
Eg the Atlas barrel has used Master bond EP79 which consists of silver coated nickel balls in an epoxy matrix to reduce radiation lengths[13]

Neither of these problems are particularly relevant to the CALICE design

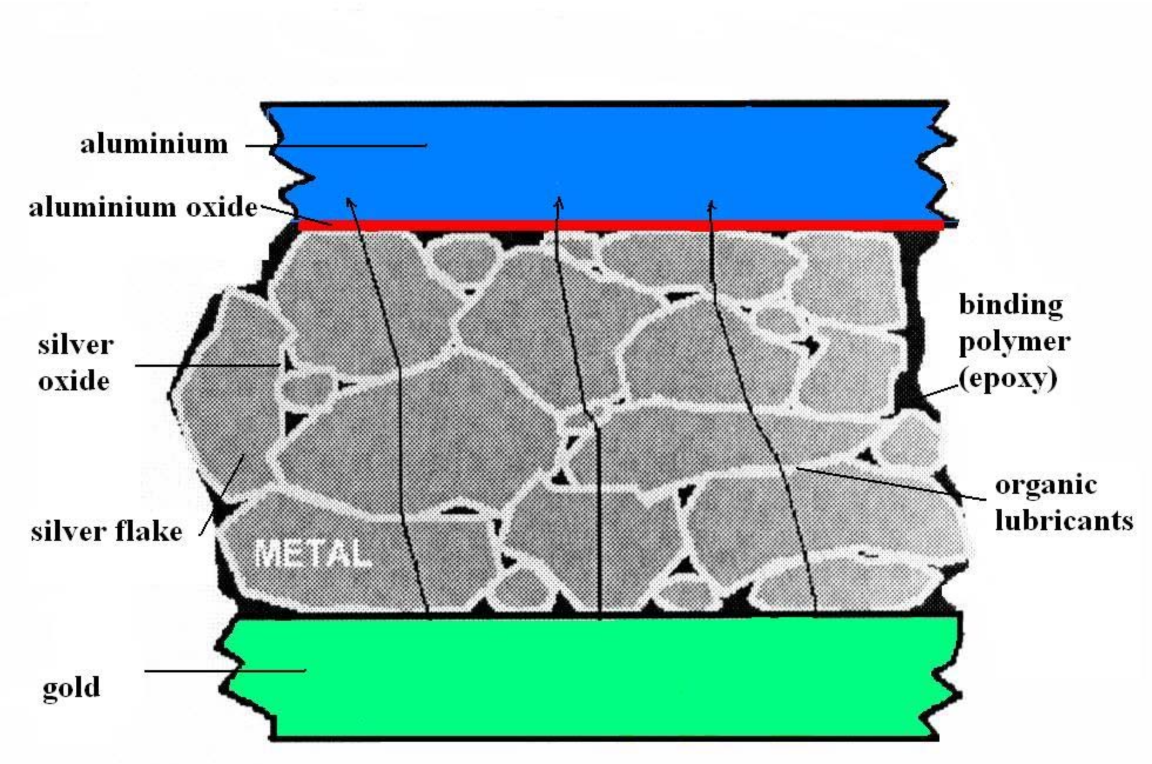


Fig 4. A symbolic representation of the structure of an ICA joint , between the aluminium metallization of a silicon wafer and the gold coating of a pcb
Note the variety of possible interfaces

4. The adhesive matrix

4.1 Thermosets

Thermosetting polymers as their name suggests undergo permanent change on heating. Single component systems are solvent based. They are of mainly historical interest, as the solvent evaporation makes them porous and hence mechanically weak.[14] Thermosets used for practical ICAs are nowadays two component systems epoxies or silicones .The two component epoxies typically consist of a relatively low molecular weight liquid with reactive epoxy groups and a co- reacting hardener. Heat causes the components to react forming high molecular weight cross-linked polymers. The cross links or bonds between adjacent chains form a 3D network which irreversibly fixes the materials shape.

4.2 Thermoplastics

Conversely, in thermoplastic polymers there is no cross linking of chains. Heating allows the individual chains to move relative to each other and hence the material can be reshaped – in effect it melts and sets. As thermoplastic binders tend to be solids they are little used in ICAs which require selective application. Instead they are used in thin film form for anisotropic conducting adhesives

A potential advantage of thermoplastics is their rework capability.

5.1 Application

A major practical production consideration is how to use two part adhesives. Pot life considerations require the components to be combined within hours of use. Batch mixing at the point of use can have several drawbacks

1. Measurement errors - particularly when ratios are asymmetric
2. Inadequate mixing
3. Entraining of air and water vapour
4. Small batch use tends to be wasteful

Clearly with care and appropriate protocols such as vacuum degassing these can be guarded against, but they remain potential trouble spots and can cause considerable difficulties in interpreting fault and lifetime data

The alternative is mixing by the manufacturer to give a one-part product which is stored at very low temperature until use – as the rate of chemical reaction halves for every 10^{o}C , storage at -40^{o}C for example can give a few hundred fold slowing making a 6 month storage practicable. Heraeus PC3001 for example has a usable processing life of 20 hr. at room temperature and can be stored for up to 6 months at -40^{o}C.

5.2 Dispensing systems

A variety of commercial systems exist for accurate glue dispensing. The most common are syringe dispensers driven by timed pneumatic pulses. Alternatives are Archimedian screw systems (CAM/LOT) for more viscous glues, Peristaltic pumps (Hofer+ Bechtel) and Jetting systems (Asymtec) have also been developed. In most of the relevant HEP uses the conducting adhesive is applied as a small few mm^3 blob. The relatively small batch nature has tended to favour syringe systems. The

pressure pulse profile is often tailored to ensure proper blob detachment to avoid glue tails.
The dispenser head is usually mounted on a small precision robot for automated positioning Fig shows the set up for the calice prototype board.

5.3 Stencil printing

In the mass production environment of the electronics industry, screen or stencil printing of solder pastes is commonplace, a technique that be used for ICA pastes. This has been used primarily for Flip chip bonding, where a chip is directly bonded to a circuit board substrate. Stencil printing of ICAs is capable of producing bumps down to 50 micron on 125 micron centres, comparable with ball grid arrays, at much lower operating temperatures - in principle down to room temperature.

Ref [15] describes the use of a flip chip bonding technique using EPO-tek E4110-PFC for a prototype Cadmium zinc telluride (CZT) pixel detector for space born x-ray and gamma ray telescope use. CZT detectors are temperature sensitive, ruling out solder techniques, and launch vibration is a problem for wire bonding methods.
The CZT detector has 64 gold pixel pads 200 micron in diameter on a 1mm square grid. They are to be bonded to thick film gold pads on a ceramic substrate
1. Silver loaded epoxy ink is printed using a stainless steel stencil with 100 micron apertures on to the CZT electrodes.
2. This is cured at 70C for 5 hours to give conducting bumps typical 20 micron in height
3. A corresponding mirror image stencil is used to deposit slightly larger diameter epoxy bumps on to the pads of the ceramic substrate
4. The 2 components are precision aligned and pressed together – the wet epoxy bumps on the substrate make contact with the CZT bumps - the already cured bumps provide a defined height standoff preventing excessive spreading of the wet epoxy on contact
5. Resulting assembly is cured as step 2. The electrical connections are now complete
6 The low shear strength of the small bumps requires an additional step called underfilling to guarantee mechanical stability. A low viscosity conventional non conducting epoxy is wicked between the two surfaces by capillary action and cured

6.1 Curing.

Cure schedules are strongly temperature /time dependant, typical examples-Traduct 2902 is 24 hours at 25C or 2 hr at 65C or 1hr at 110C or15min at 150C
EPO-TEK E4110 is 3 days at 25C or 4hours at 80C or 1 hour at 150C
For the faster cures ramp rates should not be faster than 20c/min.
Only a small amount of material is lost in the cure 0.1 -0.5 % below 200C
(However see section on vapour etc)

6.2 Mechanical properties of ICAs.

Theoretically if there is good adhesion between the filler particles and the polymer matrix then the strength of the filled system will increase with the volume fraction of filler, and conversely will fall in the absence of adhesion between the two phases. Experimentally it has been found that at filler ratios >35% by volume there starts to be insufficient resin to wet the entire filler, so maximum strengths are found at filler volume fractions of 0.30-0.40. However large conducting filler fractions are required to achieve conduction. This is a design trade off – Practical ICAs tend to maximise the conductivity and tend to be relatively mechanically weak[14]

6.3 Glass transition temperature (Tg)

Extending (compressing) a polymer moves the end chains together or apart). To achieve this rotation individual C-C bonds have to change position from *trans* to *gauche* or vice versa. At low temperatures not enough energy is available for these transitions to happen so the conformation is frozen in - the behaviour is glass like. Above Tg sufficient energy is available and the chains can respond – the material becomes more elastic. This is not usually a sharp transition and is usually determined by differential scanning calorimetry. The change in energy absorbed by a sample as its temperature is increased shows a broad peak at Tg

This is a second order phase transition and gives rise to discontinuous volume expansion, heat capacity and mechanical property changes.

As a consequence of becoming move elastic above Tg, the coefficient of expansion changes markedly - by up to a factor of 3. Typical values for the CTE change are 45 ppm/^{o}C rising to 150 ppm/^{o}C

Typical values for the ICAs we are considering are Tgs in the range 50-90^{o}C.

For a given epoxy formulation Tg is affected by the cure temperature regime - lower temperature cures give somewhat lower Tg. E129-4 has a Tg of 99^{o}C after a 1 hour cure at 150^{o}C, but only 43^{o}C after a 4 hour cure at 80^{o}C.

Polymers where Tg is well below zero show marked rubbery behaviour and are usually elastomers. Silicone based elastomers have been used for large area mechanical glueing of detectors to support structures where their rubbery nature minimises stress due to CTE conflicts.

6.4 Electrical behaviour on curing

The cure process is clearly fundamental to the performance of the connection.

Figs x show typical dependences of the joint resistance of a silver loaded ICA, in this case used to make connections to a solar cell array – a relatively high current application. [16] Increased curing times give a lower resistance joint. Similarly increased cure temperature improves the conductivity. In both cases the high standard deviations seen at lower values indicate the joints at these points are incompletely cured.

It has been shown [17] that the contacts between metal flakes are primarily established during cure. The conduction development is associated with decomposition and displacement of the organic lubricants on the flake surface, and increased contact area due to thermal stress and shrinkage. The dominant mechanism turns out to be cure shrinkage. Glues with higher cure shrinkage have higher conductivity. Typical shrinkages are of the order of 1-2%.

Hence Room temperature curing tends to give higher values of resistance.

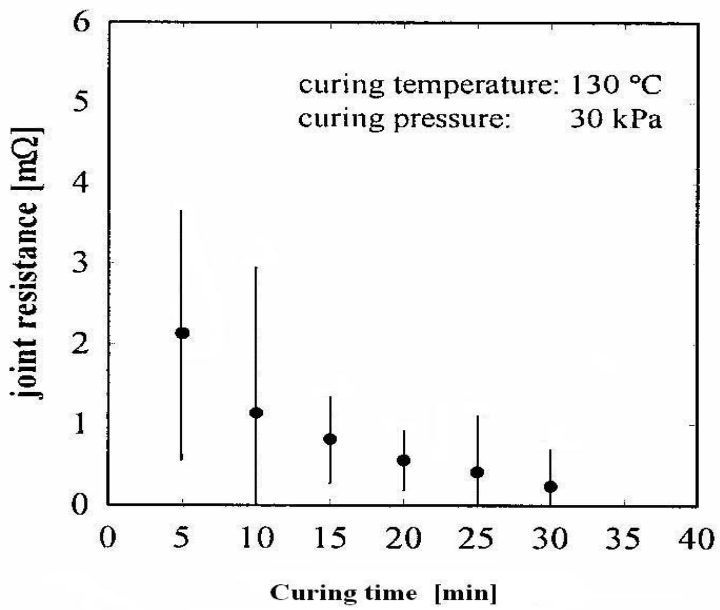


Fig 5 Joint resistance of adhesive junction v curing time

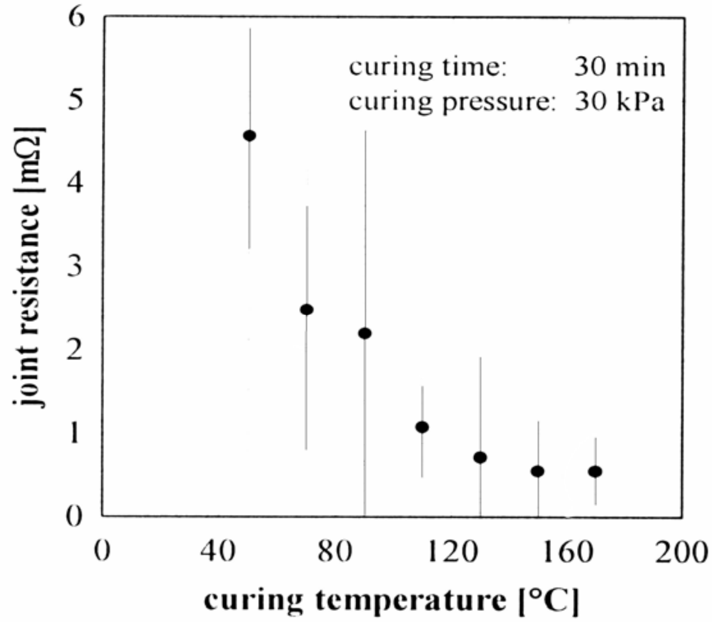


Fig 6 Joint resistance of adhesive junction v curing temperature

6.5 Adhesion

As it is a complex process, various theories have been invoked to explain adhesion phenomena. Lacking a more general approach, one or more of these may be useful for a particular joint. These are adsorption, chemical bonding, mechanical interlocking, diffusion and electrostatic effects.

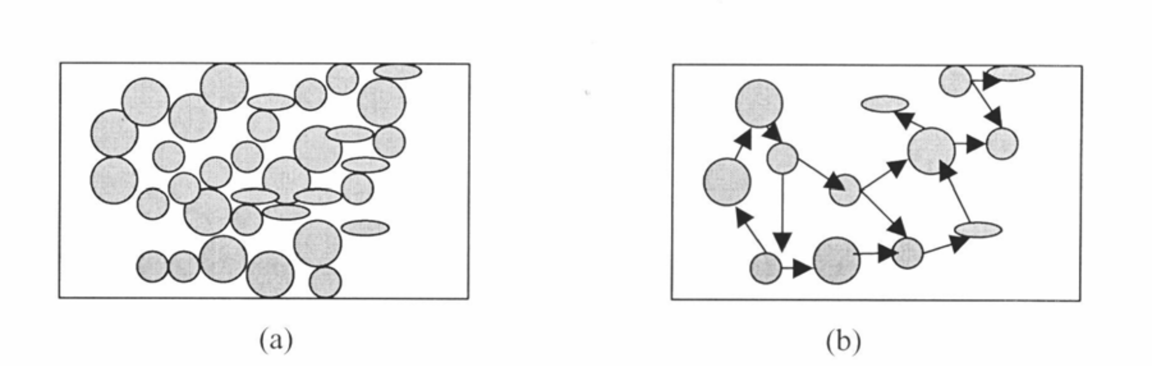
For our application the most important is probably adsorption. Here adhesion involves intermolecular contact between the 2 surfaces, giving rise to van de Waals forces and weak hydrogen bonding. It has been shown that in many cases only secondary interfacial forces are involved.[18] An apparent exception is gold which does not allow hydrogen bonding due to the lack of oxygen at the surface. As in many solid state problems experimentally measured strengths are much weaker than theoretical derivations, possibly reflecting the existence of flaws, voids and other defects on the macroscale.
To achieve the necessary molecular contact, good surface wetting is required I.e. the surface tension of the adhesive must be similar or lower than that of the substrate.
Epoxies typically have relatively high surface tensions in the range 80 dyne /cm [Si units??].Most clean electronics use metals and ceramics also have equally high tensions and there should not be any compatibility problems
A quick test – if water wets the surface its usually ok, if not the surface needs cleaning.
Plasma etching ashes organic surface contamination
SURFACE cleanliness is probably the most important factor in good bonding.

7. Conduction Mechanisms in ICAs

A typical conducting epoxy containing ~ 40% by volume (> 80% by weight) of silver has a volume resistivity of 10^{-4} /10^{-5} ohm–m whereas solid silver is 1.6 x 10^{-8} ohm-m demonstrating the point contact nature of the conducting paths.
Thermal conductivities show a similar pattern - typically ~1-3 W/m.K against a value for pure silver of 427 W/m.K
Note this can imply quite high local current densities, a 1ma current (typical of an irradiated detector bias current) over $1mm^2$ becomes a current density of 50A/cm2 allowing for this factor.

As fig shows there can be a considerable number of different interfaces involved in the conduction paths

Two possible conduction modes are known to exist. The first is true conduction, in which particles are in direct point to point contact within the matrix. The second mechanism is called percolation, in which quantum electron tunnelling occurs between particles close enough to allow dielectric breakdown of the surrounding epoxy/ oxide. It is suggested [19] that percolation is the dominant mode to start with, as the applied voltage polarises the adhesive causing the resistance to drop through charge effects.
As currents continue to be applied, polarised particles move and further coalesce and direct particle to particle contact conduction comes to dominate the process.



(a) Particle to Particle

(b) Quantum electron tunnelling

Fig 7. Conduction mechanisms in conductive adhesives

If the conductive filler fraction P of an ICA is slowly increased, the resistance does not change significantly until a critical threshold value Pc at which it drops markedly. This is called the percolation threshold. It can vary dependant on the size and shape distributions of the particles, but typical values are 30 -40% by volume. Clearly practical ICAs are designed to have filler loadings above the threshold value.

Hep experience
It is a matter of observation that checking the resistance of conducting glue joints forming bias connections to silicon wafers using low voltage sources such as DVMs can lead to anomalously high values (and mass panics) Ref[20]
Personal experience of measuring Atlas forward silicon detector bias connections (Traduct 2902) with a Keithley 236 Source measure unit (SMU) was that with a virgin glue joint (i.e. not previously measured) a day after curing an initial voltage of about 1v was needed before significant current was taken. Thereafter, normally resistive behaviour was observed. This would seem consistent with the above the hypothesis that some initial thin film(s), whether oxide or epoxy component are being punched through.

Recall that the breakdown resistance of macroscopic insulating films is typically 40 kv/mil (25kv/mil = 1MV/m = 1 volt/micron)

Voltage effects on atlas glue behaviour are further discussed in Section (9)

8 Failure Modes

A significant problem, particularly amongst the somewhat variable low statistics hep data available is trying to distinguish between failures that are due to essentially random occasional protocol faults such as a badly mixed sample, and failures that are the outliers of a distribution and hence the initial signs of a aging problem.
Once assembled in a detector, the resistivity of a glue joint is often difficult to isolate on its own due to series resistances etc. Slow changes in value are often difficult to detect until quite large effects occur.

8.1 Problems due to glue affecting silicon performance.

When this note was first envisaged it was thought the main area of concern would be the known deteriation in electrical conductivity with aging.
A further significant complication for the Calice detectors has turned out to be the effects on the performance of the detector itself.

Most Hep experience with strip detectors has used conducting glue for bias connections or non conducting glues such as epoxies or silicones for mechanical support , usually sticking the back of the detector to some substrate .The general tendency is to avoid contact to the front face of the detector.

Various tests have been reported of the effects of accidental deposition of glue on the front surface[21]
Almost all epoxies of relevance are 2 component. They can suffer Resin bleed – where unwanted capillary action can separate low viscosity parts of the adhesive which then cannot cure, leaving tacky residues which can cause leakage paths leading to direct shorting of tracks particularly at edges and guard rings, either directly or because they trap dust and water vapour. Whilst epoxy formulations can adjusted to give particular desired cure times, this is strongly temperature dependant, typically 24 hrs at room temperature, 1-2 hours at 70°C, or a few minutes at 120°C. Everything else being equal, any mass production environment will tend to favour high temperature curing to improve through put.
Increased temperatures can decrease the viscosity and increase the vapour pressure of a component, both of which may increasing the chance of component separation before cure. Increased temperature will also favour increased chemical reactivity.
Conversely a quick cure gives less time for materials to separate
For example the data sheet for EPO -TEK E4110 gives the mass loss on curing as 0.33% at 200°C , 0.65% at 250°C and 1.19 % at 300°C.
Ref[22] gives NASA data on outgassing and mass loss for a large no of glues.

Jaffe [23] for instance reports deposition of a thin film of one of the glue components condensing on the detector surface from the vapour during curing in still air, giving rise to unacceptable leakage currents by generating shorting paths. In this case the film could be simply removed by cleaning with a solvent [Propanol]. Condensation was suppressed by curing in a moving air stream.

More subtle effects exist by which surface material can affect leakage currents. Studies by A Chilangarov at Lancaster and C Bakos in our group at Manchester [24] on the effects of humidity on detectors for the Atlas SCT have shown that atmospheric humidity gives rise to a thin adsorbed thin layer of water molecules, on the detector surface. Changes in the high resistivity of this surface layer due to changing humidity can affect buried charges trapped at interface layers close to the surface, affecting internal leakage currents. The important point is that the time constants involved, particularly for low humidity environments (which are where most detectors operate) can be many days - Thus measured leakage currents and detector noise values can reflect previous history. This can cause considerable experimental confusion during short term tests.

For the CALICE prototype, high quality wafers produced were in Prague by On-Semi, acting for the Institute of Physics, Czech Academy of Science. However upon glueing to the pcb, a rising leakage current as a function of time was observed typically occurring over a few days after gluing Fig 8. It is believed that vapour released from the glue during curing at elevated temperatures attacks the polyamide passivation layer on top of the wafer.

A new gluing protocol was tried, using a higher cure temperature to minimise the cure time, with inconclusive results. Affected wafers cannot be recovered.

Further studies are on going ref P.Sicho M.Anduze[25]

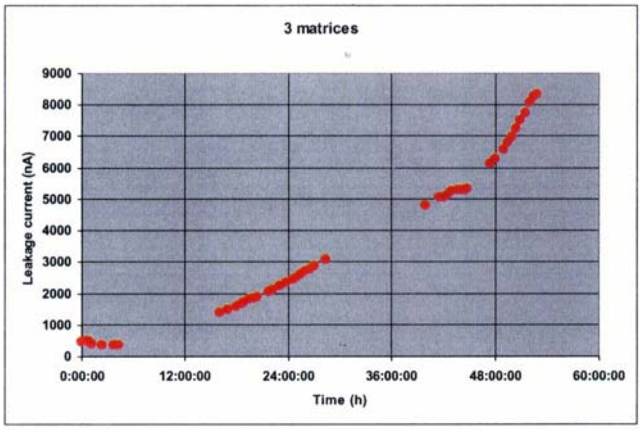


Fig 8a Time increase of leakage current due to glue effects.

A new processing run with a different passivation layer (Silox) was then tried Initial wafers, perfectly in specification, before gluing showed a x10 increase in current after gluing however this seems to relax.

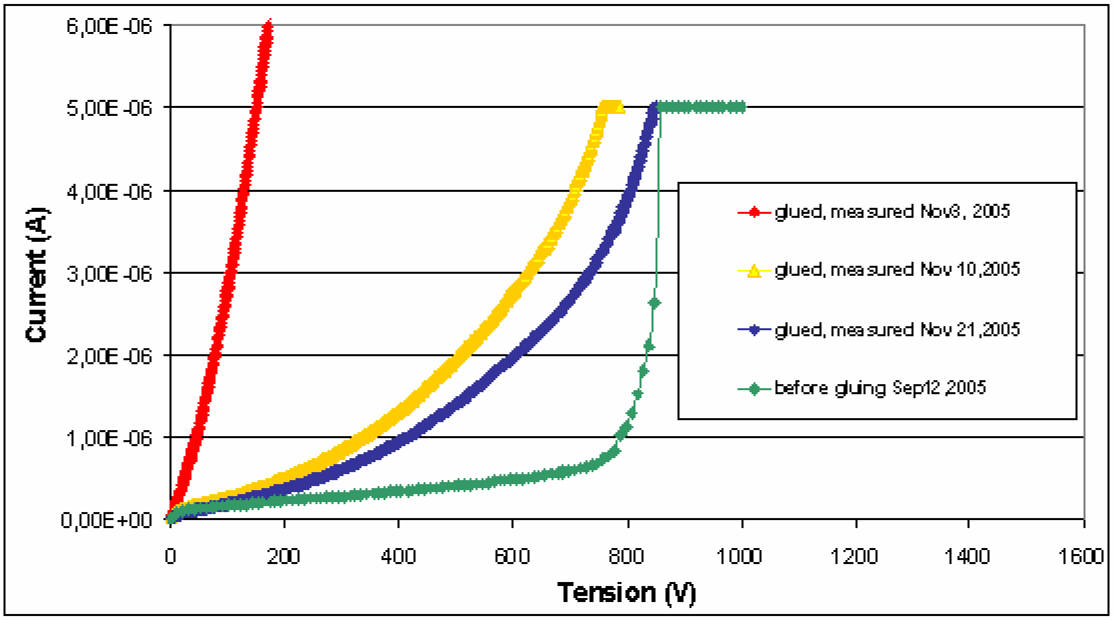


Fig 8b Relaxation of initial high current with time

8.2 Aging

A major performance issue for current adhesives is the observed degradation in electrical conductivity during aging. Electronics industry testing concentrating on SnPb surfaces has shown resistivity increases of up to 3 orders of magnitude after thermal cycling, but this does seem to plateau.

For typical hep connections (see section) 1-10 milliohm connections aging to a few ohms would still be perfectly viable.

Several factors have been implicated or hypothesized.

Build up of oxide layers at component connection surfaces – it is known the problem is much worse with standard PCB finishes such as SnPb alloy, Cu or Ni by comparison with noble metals such as gold and platinum.

Electrochemical corrosion between the silver filler particles and the connection surface driven by the difference in electro chemical potential between silver (-0.79v) and SnPb (+0.13v). By comparison silver and the noble metals have very similar electrochemical potentials. Electrochemical cells of course require the presence of oxygen and water - however most epoxies are permeable to some degree, increasingly so above Tg

This problem is exacerbated in hep applications in that the silicon metallization is aluminium with a potential of + 1.66v
ICAs have been used for many years in semiconductor die attachment.
Classically problems with aluminium corrosion have been attributed to the leaching of mobile ions Na^+ K^+ Cl^- $NH4^+$ from cured epoxies in presence of water. Much work has been done to reduce ion concentrations in glue formulations. Typically these ions exist at concentrations of 0-100 ppm.

Corrosion can cause problems both a) by the increased resistivity and mechanical weakness of the corrosion products and b) by metal transport in effect electrolysis. In principle this should not be a problem if water and oxygen exposure are reduced.

True electromigration [26] (as opposed to electrolysis) is a solid state effect which occurs at high current densities. It is due to direct momentum exchange between the flowing electrons and the metal atoms, tending to “push” metal atoms in the current direction. Material movement can create voids or distortions. This is a well known serious problem in semiconductor design as line widths of microns can easily create current densities $>10^7$ amps/cm^2 . Pb migration in bump bonds has been observed at current densities of 10^4 amps/cm^2.
A standard ic design rule to avoid this is to allow 1 micron of track width per mA of current.

The standard industry test involves exposure to damp heat conditions – 85C/85% Relative humidity usually for 168 hours (EIA JED22-A1).

Effects of water on the adhesive

Water absorption in the polymer has a range of reversible and irreversible effects.

Absorption into the polymer causes swelling and plasticization, both reversible processes. By acting as a plasticizer it can depress the Tg, and reduce the glues strength and modulus. Water can attack the matrix /filler interface and cause debonding there [27]

Water can also weaken the joint strength, attacking the glue/substrate interface. It is believed[18] that there is a critical water concentration below which attack does not take place. This is thought to be about 1.35-1.45% for an epoxy system, corresponding to a critical humidity of 50- 65 %. Various mechanisms have been proposed, one of which is reactions with polymer OH groups, causing the breaking of hydrogen bonds and displacement of adsorbed OH groups from the substrate surface.

It is well known that water can hydrolyse aluminium oxide, giving rise to a mechanically week hydrated oxide layer at the detector surface.

The standard industry test involves exposure to damp heat conditions – 85C/85% Relative humidity usually for 168 hours (EIA JED22-A1).

Mechanical Stress

Sticking silicon with a coefficient of thermal expansion (CTE) of 4 ppm/^{o}C to PCB substrates with CTEs 15-20 ppm/^{o}C clearly gives rise to a mismatch, creating stress due to thermal expansion.
Mechanical stress due to thermal cycling can affect the particle connectivity in the bulk adhesive. Stress can give rise to micro cracks, which can increase water and oxygen penetration.
Ref [28] describes detailed stress calculations and thermal cycling tests to validate the silicon glues used in GLAST, a satellite based experiment, together with vibration tests. EPO-tek 4110 is used for hv bias connections. Temperature swings of $\Delta T=80^oC$ are encountered.

Under stress polymers will undergo creep as a function of time, with both viscous and elastic components (The elastic component recovering when the stress is removed) Below Tg the polymer is rigid – low elongation, high modulus and only limited creep will occur even after long time intervals. Conversely above Tg the polymer softens and the creep rate will increase. Creep rates can be shown to be highly temperature dependant, obeying a standard Arrhenius type exponential form. Hence knowledge of time to fracture at elevated temperatures , can be used to predict behavour at lower temperatures. Note that as the creep mechanisms are different either side of Tg, there are different activation energies and hence different slopes to the equation.

Choosing a lower Tg imparts a non–brittle or resilient character to the adhesive allowing thermal cycling without micro cracks developing, however above Tg the rubbery nature is more prone to water diffusion.

On balance, in a dry environment it seems desirable to choose a lowish Tg to minimise stress.

Relevance to calice

Gold plated PCB board should be used
Problems are likely to be focused at the aluminium /silver interface.

Cleaning of Aluminium surfaces
In a Hep production environment wafers can be several years old, and despite being stored in dry nitrogen environments slow build up of aluminium oxide films can occur.

In general the Calice environmental requirements are less demanding than most electronics usage.
The overall temperature is relatively stable, due to the large thermal mass of tungsten. Initial simulations by S.Snow [29] suggest typical temperature variations across the detector of 14°C assuming cooling of the tungsten plank at the far end.

Important that the detector array is kept flushed with dry nitrogen - conventional and easily achieved

9.1 Tests

NPL report 1999 [11]
"There is no consensus as to how to test the quality and performance of adhesive joints. Typical measures of in service performance are either a drop tests or some combination of thermal and humidity cycling relying on the mismatch in thermal expansion coefficients to induce failure through shear fatigue .Drop tests rely on the inertial shear force that results from rapid deceleration to cause delamination "

Some tests developed for electronics industry use do not necessarily translate well to the HEP environment.

Destructive mechanical strength tests and impact tests do not seem do not seem all that relevant. – There appears little observed correlation between mechanical and electrical failures.

We choose to concentrate on resistance changes as these are capable of (semi) continous monitoring.

Resistance measurement

The EPO-TEK 4100 s data sheet quotes a volume resistivity of 0.007 ohm-cm typical for these ICAs.
For a dot say 1 to 3 mm diameter, 100 micron thick, this gives a typical resistance of the order 1 to 10 milliohm. With appropriate care, using 4 terminal probes and a good source measure unit such as a Keithley 236 such values can be accurately determined.

Hep tests

The recent construction of the Alas and CMS silicon trackers represent possibly the largest current silicon detector programs, providing approximately 20k modules, made in a large number of institutions. The Hv bias connections to the silicon in each case are made using conducting glues. In both cases problems have been observed with resistance aging.

9.2. CMS Silicon Tracker

A large no (4000) of the silicon tracker detectors for CMS were assembled at UCSB (information courtesy of Tony Affolder, University of Califonia Santa Barbara)[30],[31]

Here the bias connection is made using Tra-Duct 2902 from the silicon backplane to a gold plated kapton flex. Adhesive was deposited by robot.
Significant resistance changes were observed over a period of 1 month (May-June 05) in many detectors. Of 144 samples, 29 samples having resistances changes of > 1k to 40Meg ohm + 1 open circuit. Tests in cases where the glue is accessible show the problem is the glue/aluminium backplane transition rather than the glue/kapton interface. See Figs 9 and 10.
Similar results were obtained with changing to glue (EP0-TEK 129-4, a faster curing variant of EPO-TEK E4110).

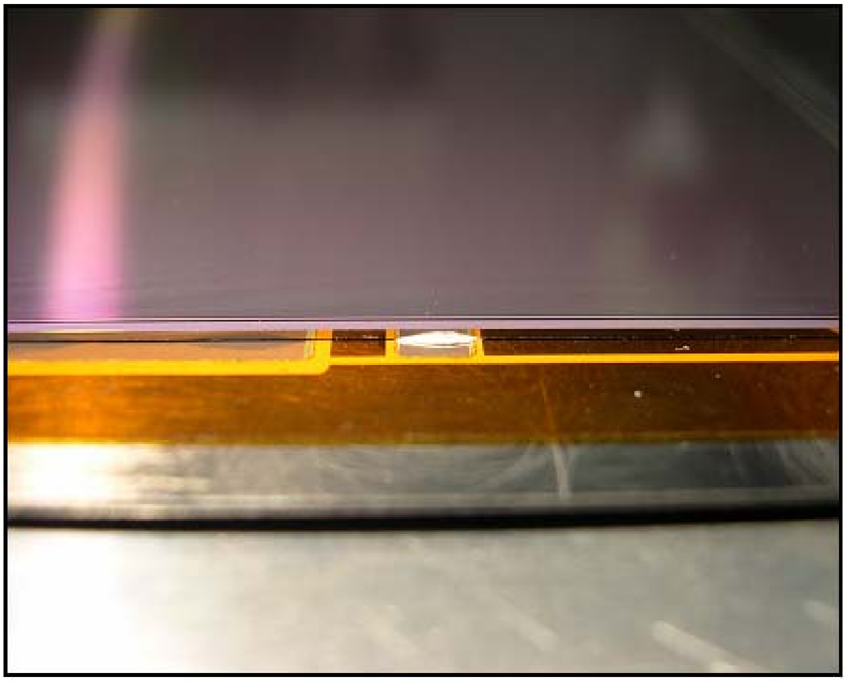


Fig 9 CMS sensor showing glue connection to Backplane

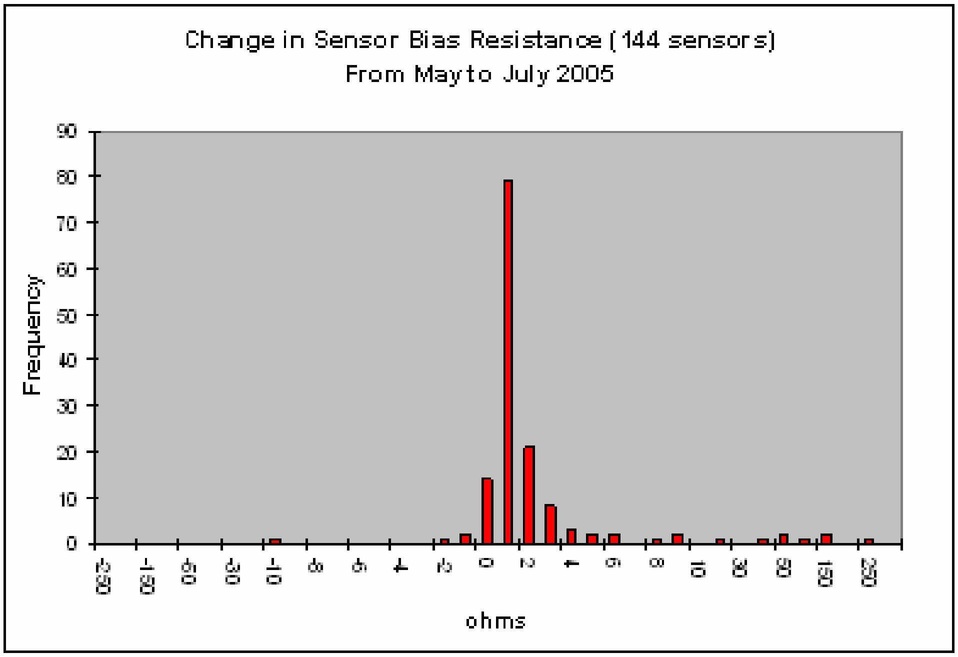


Fig 10 Change to CMS Sensor Bias Resistance over one month

Attempts to replicate the problem with hand glued samples proved totally fruitless. However, using a precision movement stage to deposit the glue (similar to the robot motion) the problem reappeared. The hypothesis is that the resistance increase is caused by some oxide layer on the aluminium surface of the silicon. Glue deposition by hand held applicators, however careful, breaks through this layer, whereas machine dispensation does not touch the surface.

Having detectors to build, the CMS solution was to literally bypass the problem, by also making parallel wire bond connections. So unfortunately further tracking of the resistance changes cannot be done.

9.3. Alas SCT

Tests on the first assembled forward tracker (end cap C) after delivery to CERN preparatory to installation revealed changes in the resistance of the HV bias line. Normal resistance for HV bias to Hv return is ~35Kohm (dominated by protection resistors etc). A significant no 5% (50/988) modules showed R> 50 kohms. See T.Jones [32]

In the forward tracker Hv bias is supplied via joints from a conducting pyrolytic graphite) support to the aluminised silicon backplane. The glue is Tracon 2902 cured at room temperature. After a 24 h resistances were checked using a Keithley Source Measure unit. At this point is possible to directly measure the backplane to TPG resistance directly. As mentioned in Section (7) a first measurement on a “virgin” joint required a voltage of about 1-2v before current was taken, but further measurements were straightforwardly resistive. The resistance for this measurement is dominated by the TPG resistance of about 150 ohm.

After about 20 to 30 days the now wire bonded detector is thermally cycled from -20°C to +50°C 10 times (staying below Tg). Note that the conventional gluing of the silicon to the tpg reduces stresses on the joint akin to partially under filling. No significant increases were noticed.

Modules from various sites made over a period of 18 months were assembled into discs, which were then put together to form the tracker. 4 Sets of resistance measurements have been made. During this time detectors have only been sporadically powered for relatively short periods.

Nov 05	0	cases R >50Kohm
Feb 06	4	
Mar 06	28	(after shipment to CERN)
May 06	52	

Suspect resistances range from just >50kohms to Megohms

Further tests on End Cap A and the Barrel (4000 modules) + spare detectors showed similar effects on the Barrel - 20% of modules tested were affected.

Intriguingly the high resistance can be removed by cycling to high voltage (5 minutes at 350v) or by sourcing currents ~100 micro amp.

Fig 11 shows results of forward biasing 49 problem modules to source high currents.

A plausible hypothesis is that oxide films are disrupted by voltage, or very local heating effects of high currents densities causing differential expansion of the silver flakes. How permanent a cure this is remains to be seen – in at least one instance the high resistance behaviour returned but could again be cured by current sourcing.

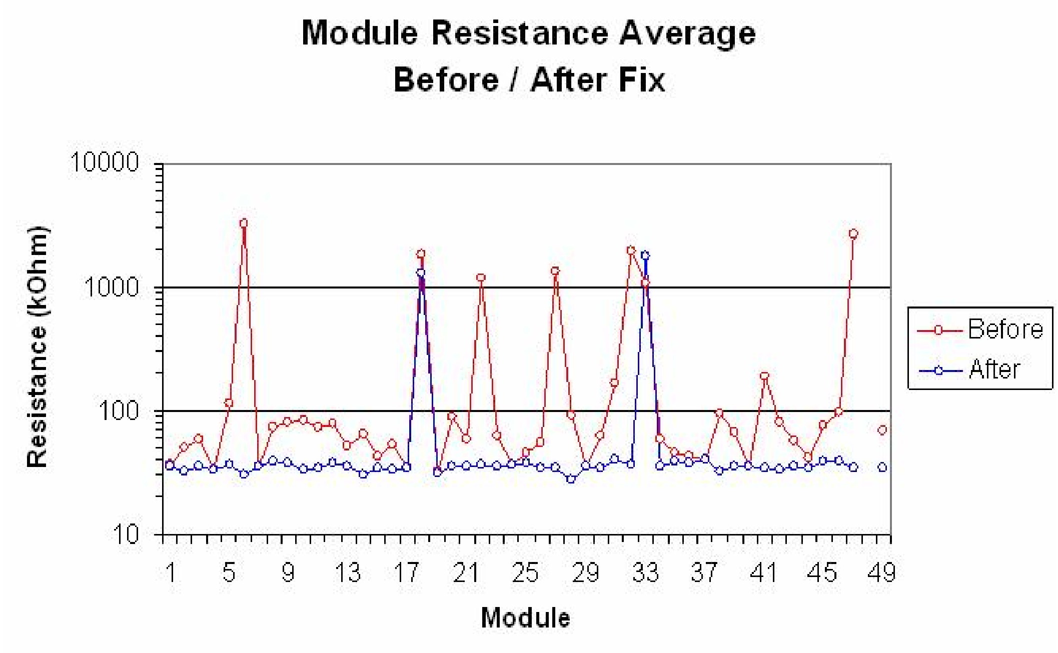


Fig 11 Module Resistance after high current sourcing.

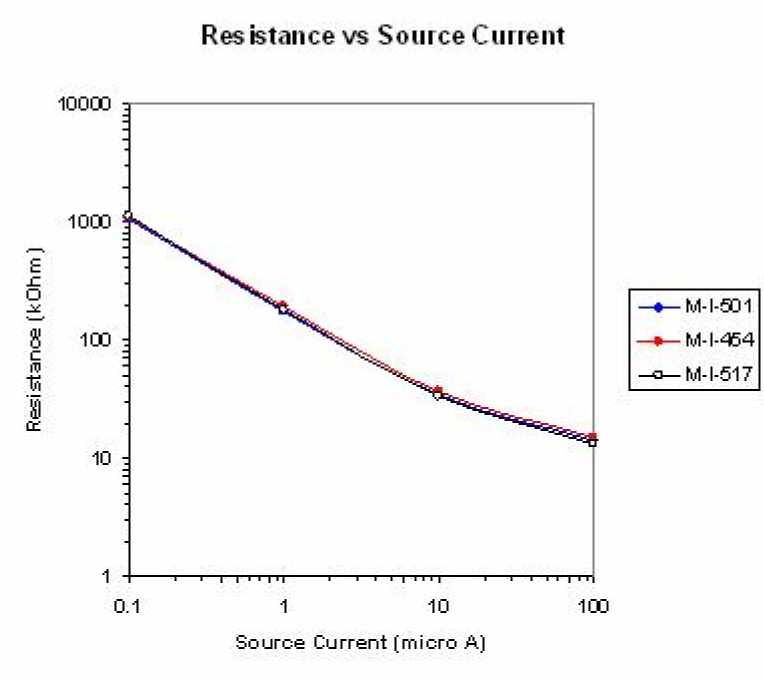


Fig 12 Resistance vs Source Current for 3 Modules

9.4 Calice protype detector

As described above the prototype consists of about 10 k pixels (300 wafers x 36 pixels), glued with EPO-TEK 4110 .This has now existed for >1 year. Despite problems described in 8.1, this is clearly, an existence proof of the general mechanical and electrical stability of the glue joints, although since the electronics are entirely outside the detector it remains at ambient temperature, and its large thermal mass will protect it from local transients. Although the detector prototype is not flushed with gas, the aluminium foil screening will provide some degree of protection from normal humidity. We need to define an acceptable limit to the joint resistance - from electronics considerations

We should keep careful track of dead channels from beam/cosmic data.

10.1 Initial aging test set up in Manchester.

It was decided to concentrate on the change in joint resistance as a result of thermal cycling.
A sequence of initial measurements was made to commission the test system.
The initial test piece consisted of 30 1 cm x 2cm pieces of aluminised silicon cut from old Atlas wafers glued on to a gold plated pcb patterned to give a serial connection usually called a snake test. This enabled continuous monitoring of the resistance of 60 glue joints.

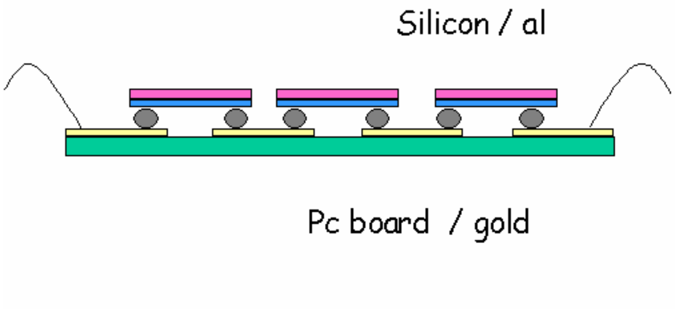


Fig 13 Snake Test concept

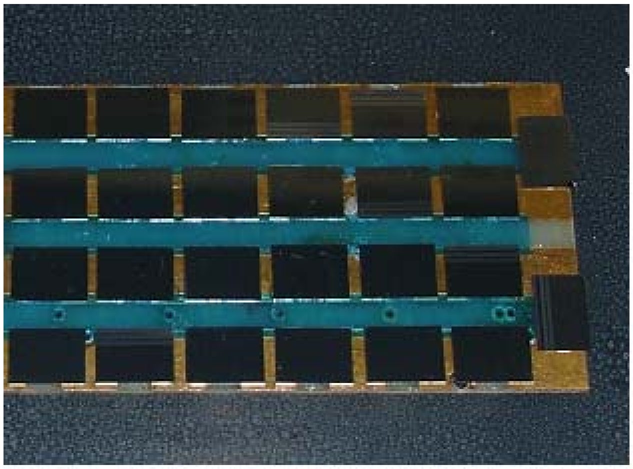


Fig 14 Snake test realisation

Fig 15 shows the test set up.
Glue was deposited by a pressure controlled dispensing system mounted on a Sony positioning Robot.
Glues were cured in a small oven to manufacturers protocols.
Temperature cycling is achieved by an Environmental control cabinet capable of –40^{o}C to +100^{o}C. A typical temperature cycle would be -20^{o}C to +50^{o}C at a ramp rate of 1^{o}c, with 1 hour dwell times at top and bottom.
The environmental chamber can be flushed with N_2 to maintain a dry atmosphere. Alternatively a high humidity atmosphere can be achieved using water/salt solutions.

Resistance measurements can be done using a couple of Keithley auto ranging DVMs Keithley 2 wire measurements) A Keithley 236 Precision Source measure unit can be used to source particular current or voltages, enabling the measurement of I-v curves, and measurement of low resistances to high accuracy.

Dataloging and control is performed by a LabView program.

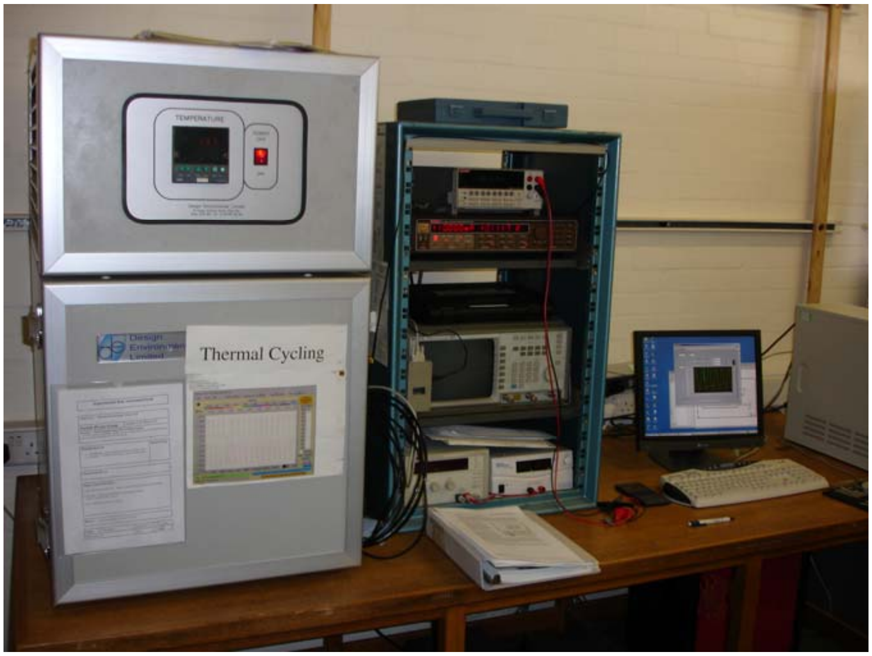
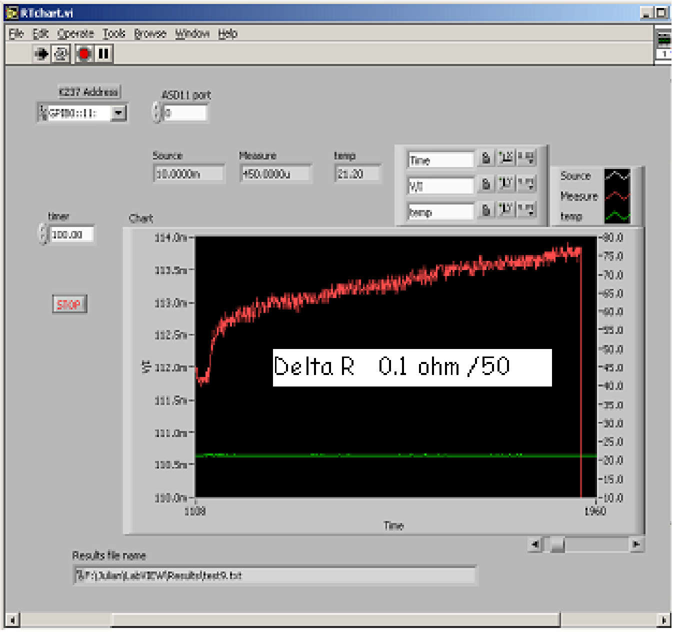


Fig 15 Manchester Test Rig Showing Environmental Chamber



K237 Address GPIB0::11:

ASD11 port 0

Source 10.0000m

Measure 450.0000u

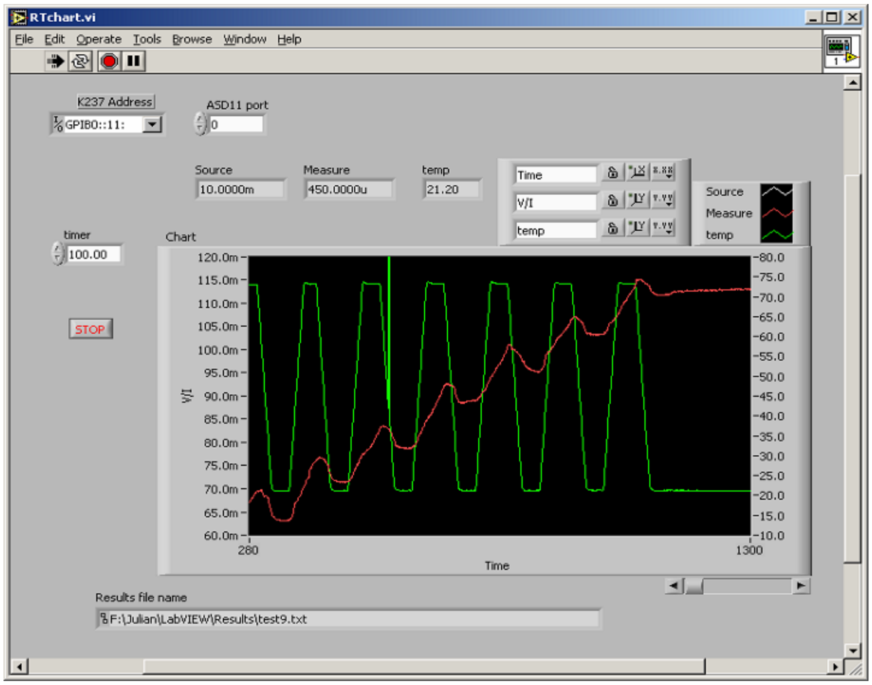
temp 21.20

timer 100.00

Delta R 0.1 ohm /50

Results file name F:\Julian\LabVIEW\Results\test9.txt

Fig 16   Snake test Slow increase of resistance with time -



K237 Address: GPIB0::11:

ASD11 port: 0

Source: 10.0000m

Measure: 450.0000u

temp: 21.20

timer: 100.00

Results file name: F:\Julian\LabVIEW\Results\test9.txt

Fig 17 Resistance changes with thermal cycling

Fig 17 shows a typical temperature cycle here 20°C to 70°C. The resistance behaviour is interesting – On the rising edge of the temperature cycle the resistance increases in line with the estimated increase due to the silver coefficient of expansion, and drops on the falling edge. However each time it does not return to the initial value – each cycle is worsening the resistance. Note that here we are cycling though Tg.

10.2 Further tests

As the detector/aluminium surface and its oxide layer/ cleanliness history appear pivotal, any detailed tests need to use production wafers. We have recently obtained from Prague 10 wafers with various electrical defects – This provides 10 full size 6 cm square detectors (36 1 cm square pixels) and 40 1 cm square test detectors (9 3mm square pixels)

Double sided pcbs, with matching pad pattern arrays on both sides connected by vias have been fabricated. By cutting appropriate tracks, snake test arrays or individual pixel joints can be selected for measurement.

Tests will concentrate on the prototype glue EPO-tek 4110 and variants, together with Tracon 2902 in view of the Atlas results.
These will include biasing to see the effects of current sourcing.

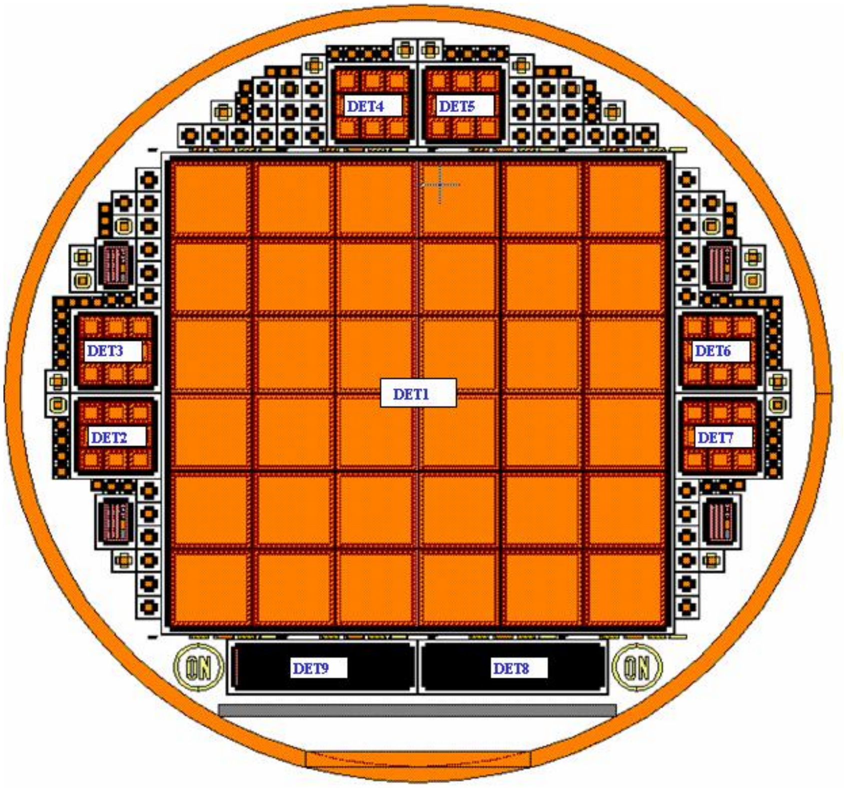


Fig 18 Calice prototype wafer

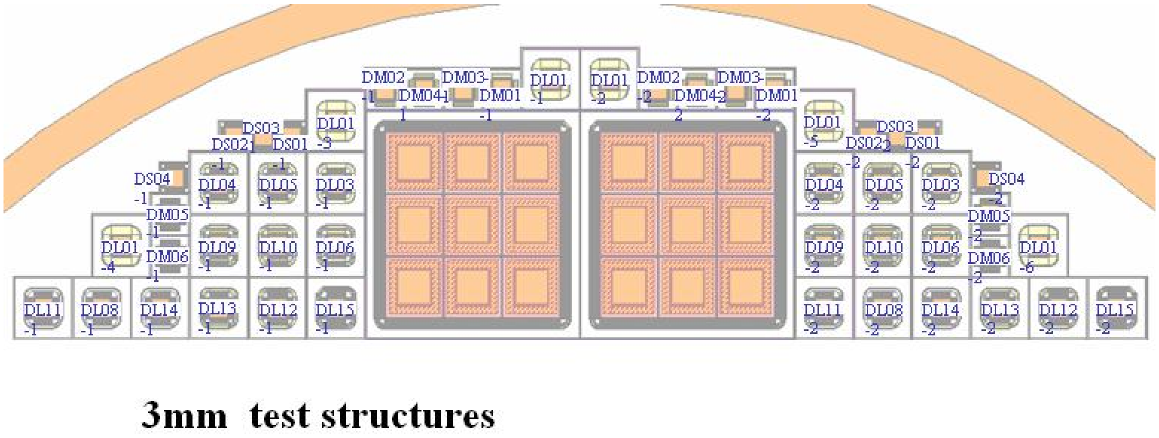


Fig 19 CALICE test Structures - 3x3 arrays 3mm square pixels

11 Conclusions and proposals

Silver loaded conducting glues have been successfully used for bias connections to silicon detectors in HEP applications for some years. Recent detectors represent a considerable increase in the number of joints.
Detectors for space applications have survived rigorous mechanical test regimes.

However both CMS and Atlas experiments have seen evidence of significant resistance ageing of the glue joints.

The behaviour of the joint has to be considered as an entity, it's not a matter of a 'correct' glue.

Problems in hep applications seem related to interface layers, predominantly the Aluminium/glue interface to the detector.
The glue/gold on PCB interface seems much less of a problem[33]. Hence tests should use gold/pcb connections

Possible aging mechanisms are oxide growth, metal migration, possibly caused by water and oxygen access

Mechanical, thermal and environmental stresses for the Calice detector are markedly less than most electronics uses. Ie low temp change, no impact stress, controllable atmosphere.

Some tests developed for electronics industry use do not necessarily translate well. Destructive mechanical strength tests and impact tests do not seem do not seem all that relevant. – There appears little observed correlation between mechanical and electrical failures.

Water exposure is a major source of trouble via several mechanisms, from corrosion effects to direct mechanical disruption by absorption.
Care should be taken to maintain a dry atmosphere through out the experimental life of the calorimeter I.e. Store processed wafers under dry N2, Control RH of assembly areas. Flush the assembled calorimeter with dry N2.

As the detector/aluminium surface and its oxide layer/ cleanliness history appear pivotal, any detailed tests need to use production wafers.
An important point is the storage history of such production wafers, which can be several years.

Tests need to ensure the defined cure protocol is followed, giving a known Tg
Thermal cycling should not pass Tg

Concentrate on electrical resistance tests capable of (semi) continuous measurement
Continue with Manchester thermal cycling tests using Prague wafers

Decide on an acceptable resistance end point criteria for the Calice front end.
Consult front end electronics designers.

It would appear from the current Atlas investigations that some electrical ageing effects can be reversed by cycling to high volts or sourcing currents ~ 100uA.

Try this on test samples. Keep track of further developments here.
Keep track of behavour of Calice Protoype – number of dead channels from beam and cosmic tests.

Appendix 1. Glue data sheets

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