Does the Presence of Components Make a Difference? A New SIR Test Protocol to Characterise a Lead-Free, Electronic Production Process

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Abstract
Surface Insulation Resistance (SIR) Testing, has been used traditionally to characterise process materials, particularly solder fluxes.

Existing Surface Insulation resistance (SIR) test methods are outdated and unrepresentative of modern circuit technology[1]. A recent European research programme found that, using the existing international standards, the SIR value of a typical no-clean flux, could be over estimated by a factor of 10 when compared to a test using parameters representative of today's technology.

This new test method has, in addition, proved easier, cheaper and faster to perform; the equipment required is now readily available and a new IEC (Draft IEC 6-1189) process characterization specification is soon to be available.

Here, in light of these European findings, an updated SIR test method is used to characterise a lead-free and VOC free electronic production process, including board surface finish, solder resist, paste, flux, wire and conformal coating – and using dummy components to more accurately determine their influence on the test protocol.

The SIR test method is very simple in concept, and involves measuring the resistance across two inter-digitated comb patterns, whilst the sample is exposed to artificial ageing conditions of heat and high humidity. If a low SIR is seen on the test sample it is likely that the residues, if left on a PCA, will have a negative effect on the reliability of the circuit in the field. Whilst the principle is simple, the successful implementation of a test is not trivial. Historically the test was implemented simply with a single current metering instrument capable of measuring fractions of a micro-amp.

Modern test equipment allows frequent monitoring of a large number of samples at sensitivities of nano-amps or better. This increased sensitivity has resulted in the European group's ability to study the SIR values over a range of track and pitch widths, using test patterns that are located underneath components.

It was found that the coupons and voltage gradients defined by present standards, again lead to higher SIR values and fewer failure incidents, as compared to the results obtained for a coupon comprising a track width, pitch and voltage gradient combination representative of today's technology.

The Swedish research institute IVF and Delphi-Delco have shown that synergistic interactions with other process chemistries used in the manufacture of PCAs can affect the resulting SIR and hence the resultant reliability of the product.

IPC J-STD-001B Appendix D does tackle the subject of process validation using SIR, but references the same SIR test methods for isolated flux qualification, that need updating.

Examples of the test results will be presented.

The Principle of SIR Testing
The SIR test method is very simple in concept, and involves measuring the resistance across two interdigitated comb patterns whilst the sample is exposed to artificial ageing conditions of heat and high humidity. A simple schematic diagram of a comb pattern is shown in Figure 1.

If a low SIR is seen on the test sample it is likely that the residues if left on a PCA will have a negative effect on the reliability of the circuit in the field. Whilst the principle is simple, the successful implementation of a test is not trivial. Historically the test was implemented simply with a single current metering instrument capable of measuring fractions of a microamp. Modern test equipment allows

frequent monitoring of a large number of samples at sensitivities of nanoamps or better.

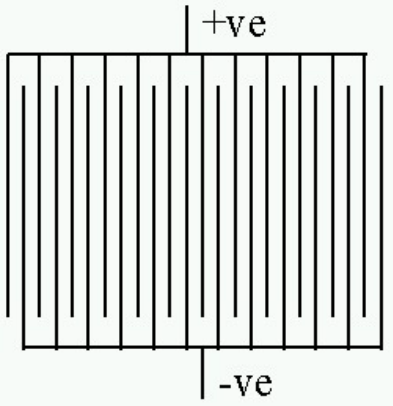

Figure 1 - Schematic Diagram of a Comb Pattern

Current International Standards

At present, several international standards[2,3] are used routinely to characterise *individual* process chemistries in *isolation*. Each test requires a different coupon with different combinations of track width and spacing in the comb pattern, indeed differing environmental conditions are also called up. In addition, each test generally requires a different potential to be applied, and some even require reversing the bias during measurement. Each test method has an arbitrarily assigned pass/fail level, and as few as three SIR measurements are required during the test period.

The differences in the test conditions highlighted above mean that it is very hard to relate the results obtained from each test, both to real life and those from other tests.

Whilst a perfectly processed no-clean flux on bare copper might yield a pass under the present test regimes, the Swedish IVF and Delphi-Delco[4,5] have shown that synergistic interactions with other process chemistries used in the manufacture of PCAs can affect the SIR and the resultant reliability of the product.

As component densities increase, and the size of assemblies' decreases, product reliability will become an increasingly important issue, and unexpected failures may occur in the field or as result of SIR coupon testing (Figure 2) based on SIR results obtained from the current test methods.

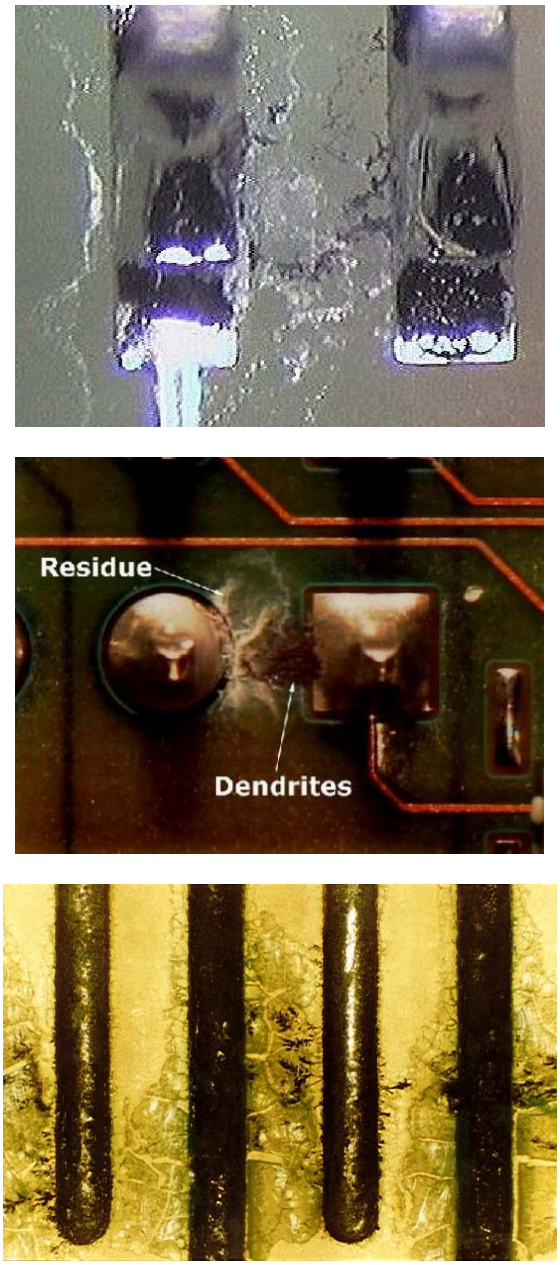

Figure 2 – Field Failures and SIR Test Failures can be Dramatic

Process Validation

First Generation Process Validation

Brewin & Hunt[6] recognized the shortcomings of the existing SIR test methodologies, and in 1998 performed an SIR process validation of a range of process chemistries, using the best test protocol available at that time, to prove that the production chemistry residues in combination, provide an electrically insulating and electrochemically non-corrosive state.

Here SIR measurement was used to test process permutations of a set of no-clean flux based PCA production materials. The work was performed

according to the recommendations made in ISO/PWI 9455-17 and by the NPL.[7,8]

The work performed was designed to represent actual process conditions as much as possible, and a surface mount SIR coupon was utilised (Figure 3). This test substrate has 4 comb patterns on it, but they are arranged to facilitate the mounting of SMT flat pack devices onto the board.

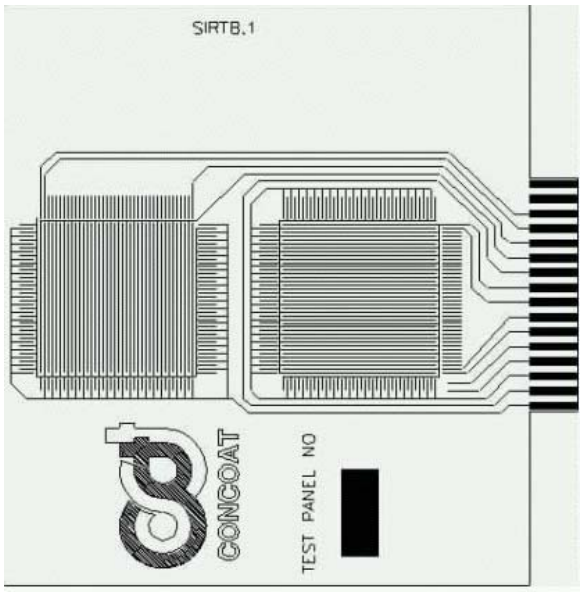

Figure 3 – Test Substrate – Surface Mount SIR Coupon

This allowed the coupons to be prepared for test using actual process settings, and with the inclusion of solder resist and components, the effects of flux entrapment, and thermal mass are also present. This is not the case for a flat test substrate. Some process materials, themselves giving good reliability, individually can cause low SIR levels by synergistic interactions.[9] To detect such occurrences SIR testing must be performed with all process chemistries present. Also in line with this philosophy both HASL and NiAu finishes were used in the testing, and the effects of a rework wire investigated. The aim of the project was to provide a set of PCB production products with known compatibility under typical process conditions.

The test-regime was performed using a test bias of 50V continuous bias and 50V measurement bias, under conditions of 40°C and 93%RH. The coupon used had a track spacing of 400µm, giving a voltage gradient or field strength of 125V/mm. The coupons were prepared as below and handled with gloves at all times to prevent contamination.

Sample Preparation – HASL Finish
Submergence for 5 seconds at 255°c, standard proprietary flux used.

Sample Preparation – NiAu Finish
ATOTECH Palladium activated NiAu. Bath temperature 88°c, 6 g/litre Ni. Submergence for 25 minutes (4-6µm Ni). The gold plating bath was controlled at 84°c, and submergence was for 12 minutes (0.06µm Au).

Sample Preparation – Permanent Solder Resist
The samples were pre cleaned with a pumice scrub, and the mixed resist was applied through a 32.82 mesh screen. The resist is applied to the whole surface of the coupon. The samples were pre dried at 85°C for 30 minutes before exposure to 450mJ/cm^2. The exposure pattern ensured that half the sample remained free of mask after development

The un-exposed mask was removed using 1% anhydrous K_2CO_3 at 35°C under 2.5 Bar of pressure. With a post bake of one hour at 150°C, the cured resist was 26µm thick on the open board.

Sample Preparation - Flux
The flux was liberally applied to the test samples using a hand spray gun. The samples were then held vertically for 30 seconds on absorbent material to drain excess flux. The alloy used in the wave was 63% Sn, 37% Pb at 250°c, and the samples were soldered in an air atmosphere. The boards passed flux side up as a worst-case scenario (lower de-activation) (Figure 4).

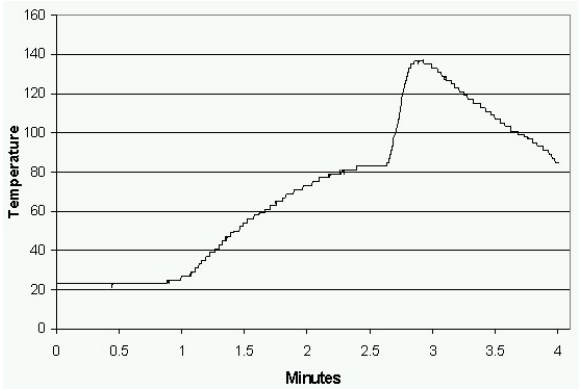

Figure 4 - Wave Temperature Profile

Sample Preparation - Paste
The paste was applied using a DEK 260 screen printer with a ProFlo™ solder cartridge system. The 150µm (0.006") stainless steel stencil was designed to apply paste to the outer comb patterns to facilitate mounting of QFPs (Figure5).

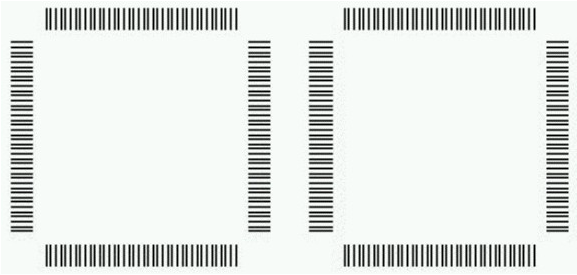

Figure 5 - Paste Stencil Design

Although paste was deposited on both sites a QFP device was mounted only on the SIR comb surrounded by solder resist, and was placed by hand. Reflow was at a peak of 222°C (Figure 6).

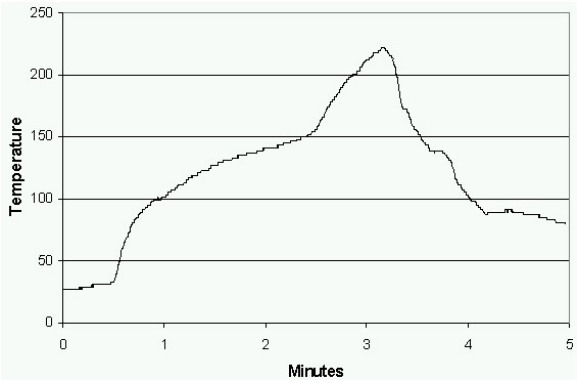

Figure 6 - Reflow Temperature Profile

Sample Preparation – Mixed Technology

To model production of a mixed technology board the samples were spray fluxed and passed over the wave comb side up. Then the procedure for paste application was followed as above.

Sample Preparation – Rework Solder Wire

Each central comb pattern was soldered in four places with a small amount of solder wire (Figure 7). Solder was flowed onto tracks avoiding bridging. The tip temperature of the soldering iron was held at 350°C.

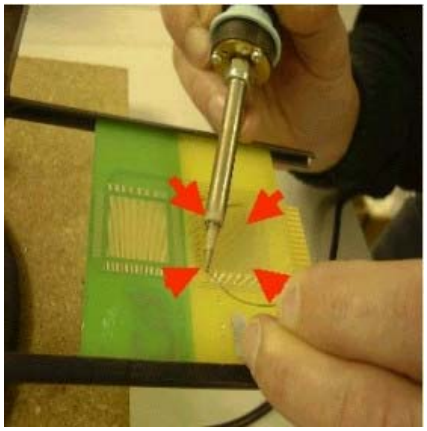

Figure 7 – Solder Rework to Prepare the Sample

SIR Testing

The sample coupons were mounted into a 256-channel test rack, and connected to an Auto-SIR™ automatic surface insulation resistance tester. The coupons were all labelled using a metal stamp so as not to introduce contamination onto the samples. The samples were handled with gloves at all times.

The rack system holds 64 coupons, which require 256 (4 x 64) cables for connection (Figure 8). This was achieved with the Auto-SIR™ system via a series of internally and externally grounded 34-way ribbon cables, in this case 16 were required each carrying 16 signals and 16 grounding wires.

Figure 8 - Test Rack Shown in Environmental Chamber

These precautions eliminate tribo-electric currents affecting the low current readings.[9] The samples were mounted into the test chamber to ensure even airflow over their surface, as recommended in ISO/PWI 9455-17.[11]

The chamber was set to ramp slowly to 40°C, and then a relative humidity of 93% (according to IEC 68-2-20). Using this lower temperature condition (as opposed to the more traditional 85°C/85%RH condition) means, importantly, that any temperature sensitive components of no-clean fluxes (mainly organic acids) remain present throughout the test[10]. This is becoming the recognised test condition for no-clean flux formulations. These conditions were held for 7-days. During this time a bias of 50V was applied to the coupons (relating to a voltage gradient of 125 V/mm on a track spacing of 0.4mm). The leakage current was measured and the Log Resistance value calculated and logged for each sample every ten minutes.

At the end of the 7-day period the humidity was removed from the chamber before cooling so that at no point during the testing was condensation formed on the samples.

European Work Highlights Issues with Current Test Methods[12]

There has been argumentation about the significance of each of the individual test parameters: temperature, bias, coupon design, field strength and frequency of measurement. Consequently, a recent European Collaborative Research Programme, built on the initial work performed by the NPL[13] and determined the experimental parameters that are the key to a successful SIR test.

The width of the comb fingers (tracks) and the spacing between them (gap) were factors expected to have a significant effect on the SIR obtained. The width of the tracks and the gap combine to form the pitch of the pattern.

Bias

The bias applied during the test and the pitch were expected to interact and influence the SIR, as was the combination of temperature and humidity under which the test was performed (Figures 9 and 10).

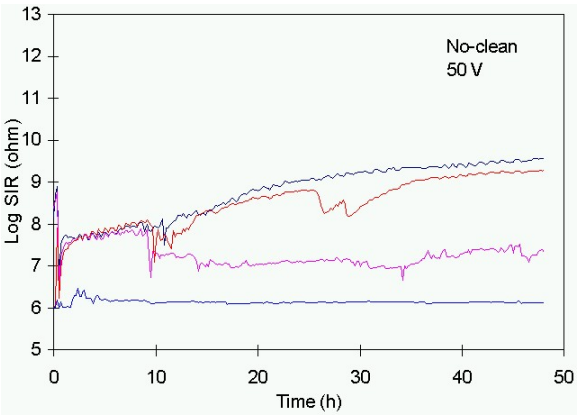

Figure 9

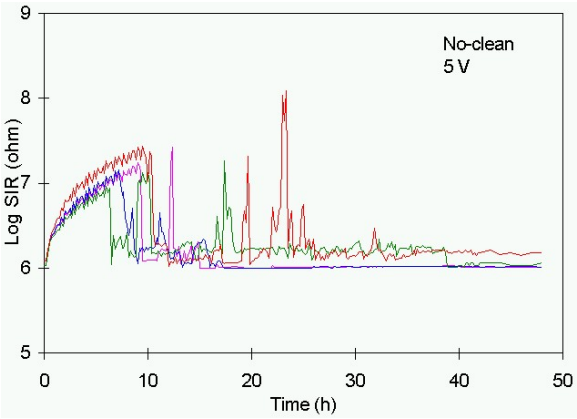

Figure 10

Effect of Temperature

The effect of high temperature for traditional RMA fluxes is to reduce SIR; most likely due to increased water adsorption (Figure 11). If the temperature was too high, no-clean flux residues could sublime and thus potentially harmful residues could be lost.[9] The temperature and humidity influence the adsorbed water layer thickness across the surface of the coupon, and this greatly influences the surface conduction.

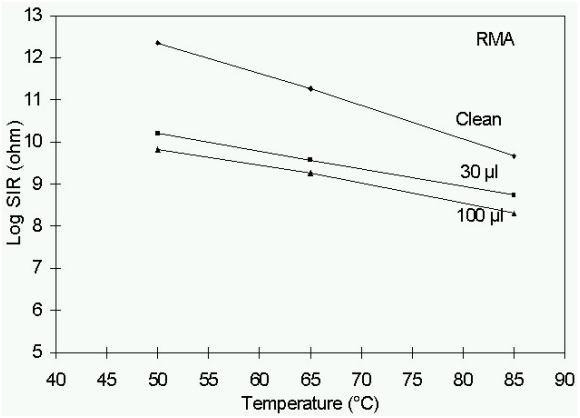

Figure 11 - Effect of High Temperature for Traditional RMA Fluxes is to Reduce SIR

Figure 12 shows that below 65°C, with 15 & 30 µl applied, the no-clean flux gives a very low SIR reading, but above this temperature the residues are dissociated. It is important to note how the results are dependent on flux loading. The amount of flux used for testing should reflect the amount used on actual boards.

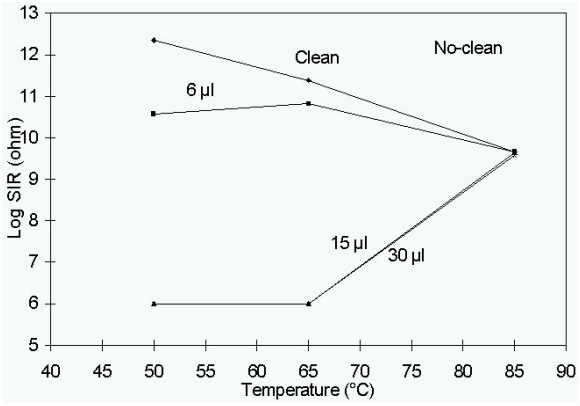

Figure 12 - Below 65°C, with 15 & 30 µl Applied, the No-Clean Flux Gives a Very Low SIR Reading

Frequency of Measurement

The frequency of measurement was also identified, since corrosion products can be deposited at the cathode, and begin to grow back towards the anode. This can result in the formation of dendritic structures, which are sensitive and can grow in the

space of an hour. They can also disappear, and can be missed by existing experiments.

Figure 13 shows clearly that current standards will only detect dendritic growths if they occur at exactly the same time as the measurement. Even then, the reversing of the bias required by many standards can destroy the dendrite and thus the dendritic event will be missed.

The group then set out to determine the SIR response to these parameters, under a controlled set of experimental conditions, and used Design of Experiment (DOE) methods, to increase the efficacy of the experiments.

Current SIR test methods are based on technology that is thirty years old. The European group started by investigating the effect of field strength on SIR, in combination with different SIR patterns, fluxes and board finishes, using technology representative of that used in production today. All experiments were performed at 65°C and 85%RH in this preliminary work.

Most of the SIR values had stabilized within three days and any dendritic growth was initiated within this time. Thus it seemed reasonable to shorten the test from seven to three days.

The SIR values were averaged wherever possible. However, the water-based flux formulation showed dendrite formation. Interestingly, dendrite formation was seen at 200V/mm with the 200/200 track and gap coupons, but not with the 400/500 coupons at the same field strength (much higher bias). This result highlights the importance of the track-width and gap of the coupon in determining the SIR obtained, and that the track and gap widths should be representative of those in a manufactured circuit. Dendrite formation can lead to catastrophic failures of circuits in the field.

Figure 14 shows an analysis of the means and medians of the values obtained from the statistical analysis.

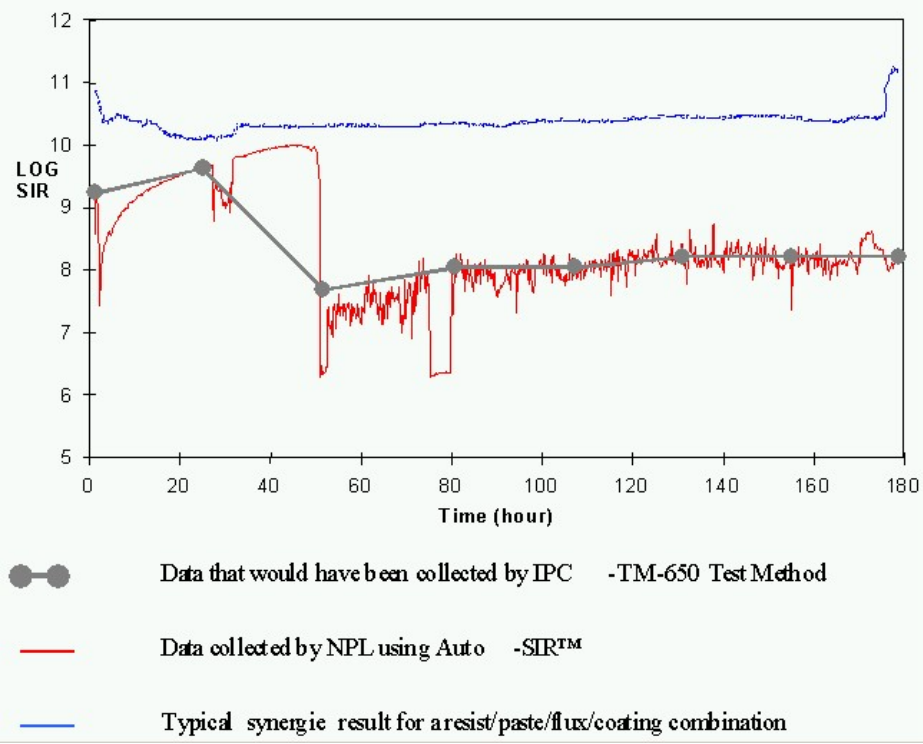

Figure 13 - Current Standards will only Detect Dendritic Growths if they Occur at Exactly the Same Time as the Measurement

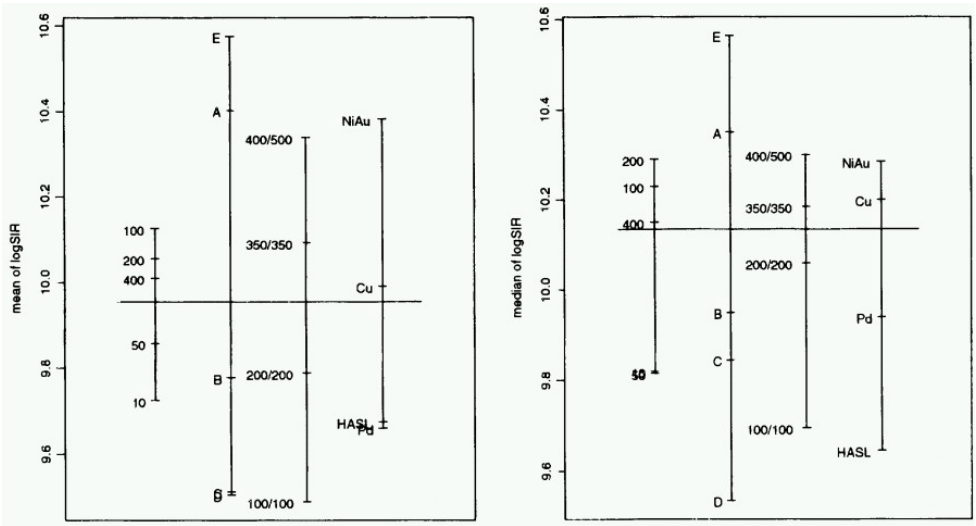

Figure 14 – Analysis of the Means and Medians of the Values Obtained from the Statistical Analysis

The main point to emerge from this early work was that the pattern and field strength should be studied in more detail.

Figure 15 shows the effect of field strength on SIR increases as the pitch of the coupon decreases. This is critical in today's applications where fine pitch and low voltages are used in most circuit assemblies. Current international SIR standards use the 400/500 coupon as standard and greater than 100 V/mm field strength. It can be seen that this combination overestimates the SIR by 1.5 log Ω decades as compared to the 10V/mm and 100/100 pattern combination. *Thus a comfortable SIR pass in a current standard test could produce a failure when tested at combinations representative of circuit technology*.

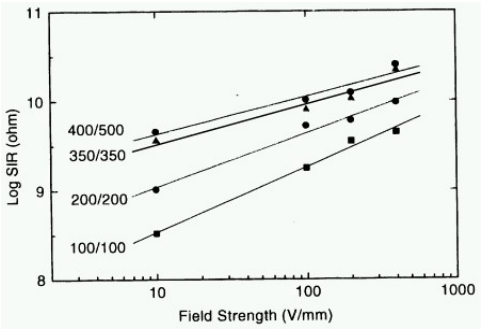

Figure 15 - Effect of Field Strength on SIR Increases as the Pitch of the Coupon Decreases

The main aim of the European's work was to improve the SIR test methods used to *qualify fluxes and pastes*, by making the conditions more representative of today's technology and hence give the user more confidence in the SIR values being quoted to them. However, these methods are used singularly to qualify individual materials, whereas in production, many different chemistries are used, and can lead to unpredicted synergistic interactions which can lower SIR. The European Group's methodology can be extended to process validation work, where all the materials used in a production process are tested singularly and in combination to ensure compatibility and reliability.

Second Generation Process Validation

In light of the findings of the European Group and with the impending drive to more environmentally acceptable process chemistries, it was decided to update the work of Brewin using a mixed-technology SIR coupon to better model production processes, and to use Lead-Free Paste and Wire, water-based no-clean Flux and water-based Conformal Coatings, to investigate any synergistic interactions between these new chemistries. Ni/Au and Immersion tin were identified as the most common lead-free solderability finishes and were the finishes used in test.

The recommendations of the European Group were taken into account when designing the coupon and choosing the test conditions (bias, temperature and humidity, frequent measurements and duration of test). It was desirable to see whether the process validation work could be extended to a practical quality assurance test method to ensure that a manufacturing process is under control.

Once the chemistries to be investigated were determined, a process validation matrix was determined. This is composed of two parts. Firstly, the individual chemistries are tested in isolation, and then sequentially, in the various combinations in which they are used in a production process. The reason for this is simply, that if a low SIR is recorded, it can easily be traced to the stage of manufacture. The isolated tests are performed to ensure that the individual chemistries are behaving as expected in isolation, and should be repeatable from

batch to batch of material. In essence, it is an incoming quality inspection process.

The matrix determined for the immersion tin finish is shown below in Table 1. 2 Coupons of each combination are tested to give some sort of repeatability. A statistical analysis would require about 16 coupons at each stage, but this is clearly impractical for a process validation and quality assurance regime. A bare copper finish is included to identify the effect (if any) of the chemistries involved in the immersion tin or Nickel Gold processes. The matrix is repeated for the NiAu finish.

The process validation work was still being undertaken at the time of submission, but the results will be presented at the Conference in December, and a CD will be available containing the experimental results in a paper dedicated to process validation.

Conclusion

Current test methods are outdated and unrepresentative for today's technology. A European Research group has established the key SIR parameters and shown that current SIR test methods may over estimate the true result by a factor of ten. Frequent Monitoring has been shown to be of key importance in the detection of dendrites during the course of an experiment.

The findings of this research group have been taken into account, and a new coupon designed to better model today's technology has been used to apply this group's work to process validation. With the increased adoption of lead-free production, a range of more environmentally acceptable chemistries has been tested individually and in sequential combination, to validate the use of these new chemistries in an assembly process, using the new test protocol.

The methodology has been extended to include practical process monitoring of a production line.

Note

Pictures courtesy of CSL Inc., and NPL

References

1. Richards et al, "SIR Testing: Are we doing it correctly?", OnBoard Presentation, London, UK Sept 2000
2. IPC-TM-650 2.6.3.3
3. Bellcore TR-NWT-000078 Issue 3, Telecordia Technologies
4. "Relationship between Surface Insulation Resistance and contamination of polyglycols and Dicarboxylic acids on Printed Circuit Boards". IVF Report 95052
5. Witmer et Al, "Assembly Materials Interaction Study" IPC/SMTA Fall '98 Conference, S23-2
6. Brewin & Hunt, "SIR Testing Comres of Age", Brasage Conference on Soldering Printed Circuit Boards Interconnection Technologies 1999 Proceedings, 7.
7. Hunt et al, "The Impact of Temperature and Humidity Conditions on Surface Insulation Resistance Values for Various Fluxes" NPL Reports CMMT(A) 119 National Physical Laboratory, London, UK.
8. Hunt et al, "SIR Response to various processing paramaters", NPL Reports CMMT(A)120, National Physical Laboratory, London, UK.
9. IPC 9201 Surface Insulation Handbook
10. Tellefsen & Stromgen, "The dissapearance of Dicarboxylic Acid No-Clean Flux Activators with Time and Temperature". IPC Expo Conference, San Jose, March 97
11. Draft ISO/PWI 9455-17
12. 'Development of Surface Insulation Resistance Measurements in Electronic Assemblies' - SMT4-CR97-2155, EU Project Co-Ordinator – National Physical Laboratory, www.npl.co.uk/npl/ei.
13. Hunt et al, "The effect of test voltage, test pattern and board finish on SIR measurements of various fluxes", NPL Reports CMMT(222), Sept 1999, National Physical Laboratory, London, UK.

Table 1 - Experimental Matrix for Immersion Tin Process Validation

Finish	Resist	Paste	Flux	Wire	Coating	Quantity
Cu						2
Im Sn						2
Im Sn	PSR					2
Im Sn		LF N/C				2
Im Sn			WB N/C			2
Im Sn				LF N/C		2
Im Sn					WB AR	2
Im Sn					WB UR	2
Im Sn	PSR	LF N/C				2
Im Sn	PSR	LF N/C	WB N/C			2
Im Sn	PSR	LF N/C	WB N/C	LF N/C		2
Im Sn	PSR	LF N/C	WB N/C	LF N/C	WB AR	2
Im Sn	PSR	LF N/C	WB N/C	LF N/C	WB UR	2