Surface Insulation Resistance (SIR) Response to Various Processing Parameters

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CONTENTS

1 INTRODUCTION 1

2 EXPERIMENT 1

   2.1 SAMPLE PREPARATION 1
   2.2 FLUXES AND ENVIRONMENT TEST CONDITIONS 2
   2.3 SURFACE INSULATION RESISTANCE MEASUREMENTS 4

3 RESULTS 5

   3.1 PCB FINISH 5
   3.2 EFFECT OF TEST BIAS 6
   3.3 EFFECT OF GLYCOL CHEMISTRY 8
   3.4 IMPACT OF REFLOW AND THE PRESENCE OF COMPONENTS ON SIR 8

4 DISCUSSION 9

5 CONCLUSION 10

ACKNOWLEDGEMENTS 10

REFERENCES 10
Surface Insulation Resistance (SIR) Response to Various Processing Parameters

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ABSTRACT

SIR testing is carried out under a wider range of experimental conditions than those detailed in standards. The work here explores some of the issues when using a range of fluxes with various processing conditions that include the influence of substrate finish, test bias and reflow processing. These results clearly show that care must be exercised when using different test set-ups, and extrapolating between testing and use conditions. In particular the use of 50V test bias can produce anomalous results when compared to a 5V use environment.
1 INTRODUCTION

The Surface Insulation Resistance (SIR) technique has been widely used to assess the effect of contaminants on the reliability of assemblies. Comparison with other methods SIR measurement has the advantage that it can be used to detect the localised contamination and that it can measure the effect of contaminants, both ionic and non-ionic, on the reliability of the printed circuit assembly.

In the production process of the printed circuit assembly, there are several kind of fluxes currently in use. Furthermore, in the preceding board fabrication process of hot air solder levelling (HASL) process the soldering operation uses fluxes, often containing significant amounts of polyethylene (PEG) or polypropylene glycol (PPG). These glycols can be difficult to remove if they have been absorbed into the substrate. If significant traces of these flux residues remain in the board, then the SIR values can be dramatically compromised.

Today the complex nature of flux chemistry requires any test to be sensitive to the composition and detect any synergistic effects between the various components in the flux. Hence, the testing conditions should be sensitive to the flux stability, especially the carboxylic acid residues which are not resistant to high temperature tests.

The aim of this work was to investigate how SIR is effected by the following factors: different fluxes, flux surface load, temperature, humidity and soldering process.

2 EXPERIMENT

2.1 SAMPLE PREPARATION

A NPL designed board with two SIR comb patterns was used. The board is shown in Figure 1. The size of the comb pattern is 25.5 x 26.5 mm with 0.64 mm pitch and 0.32 mm gap, and approximately 1409 squares. Boards with gold on nickel (Au-Ni), hot air soldered levelled (HASL) and plain copper finish were used. The boards were cleaned in an Ionograph 500M with 75% IPA plus 25% de-ionised water solution at 45°C. The cleaned boards were fluxed using four alternative fluxes with three different loads. The boards were placed on a 60°C hot plate during the fluxing operation, to ensure the flux spreading was limited to the SIR pattern. The fluxes were applied by a dropper to the centre of comb pattern and the fluxed area was approximately 1.6 cm². The fluxed boards were then left for 16 hours at room temperature for the flux to dry.

Some of the fluxed boards were subject to a reflow cycle to simulate a full assembly process. In addition on some of these reflowed boards, QFP components were placed over the SIR patterns to simulate the trapping action of components during a reflow process. Figure 2 shows the temperature profile of the reflow oven.
2.2 Fluxes and Environment Test Conditions

The effect of the test bias voltage on SIR was studied at two bias voltages of 5 V and 50 V. The fluxes, flux surface load, test temperature and humidity are listed in Table 1.

Four generic fluxes chemistries were chosen for this work:

1. A rosin based, activated with 0.5% halide, typical of RMA fluxes. This was Actiec5, supplied by Multicore Solders, and is a standard test flux specified in IEC 68-2-20.
2. A weak organic acid (WOA) flux. This was 1.6g/l adipic acid in IPA.

3. A glycol based flux in a IPA solvent (1.6g/l PEG400 in IPA). Glycols are used in SMD fluxes, but more importantly form the major part of hot air solder levelled (HASL) fluxes, during the manufacture of boards.

4. A combination of the above adipic and PEG fluxes.

The different fluxes, flux surface load, test temperatures and humidities are shown in Table 1. (These boards were not reflowed.)

<table>
<thead>
<tr>
<th>Flux</th>
<th>RMA</th>
<th>WOA</th>
<th>PEG</th>
<th>WOA + PEG</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flux chemistry</td>
<td>Actic 5</td>
<td>1.6g/l adipic acid in IPA</td>
<td>1.6g/l PEG400 in IPA</td>
<td>0.8g/l adipic acid + 0.8g/l PEG400 in IPA</td>
</tr>
<tr>
<td>Flux surface load (µl)</td>
<td>100</td>
<td>30</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test temperature and humidity conditions</td>
<td>65°C/85% RH</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Flux surface load (µl)</td>
<td>100</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test bias voltage</td>
<td>5 V (D C)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The loading for the WOA plus PEG flux were different since with this flux the SIR was dramatically affected, consequently a different range of loading was selected.

In addition the influence of various glycols were investigated. So, in addition to polyethylene glycol 400 (PEG400) in IPA, polyethylene glycol 2000 (PEG2000) in IPA and polypropylene glycol 1025 (PPG1025) in IPA were investigated. A 100 µl surface load was used to investigate the effect of the different glycol on SIR. The different glycol Fluxes, flux surface load, test temperature and humidity are shown in Table2. (These boards were not reflowed.)
Table 2:
Effect of Different Glycol Fluxes, Flux Surface Load, Test Temperature and Humidity

<table>
<thead>
<tr>
<th>Flux</th>
<th>PEG Chemistry</th>
<th>PEG Chemistry</th>
<th>PEG Chemistry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flux surface load</td>
<td>1.6g/l PEG400</td>
<td>1.6g/l PEG2000</td>
<td>1.6g/l PPG1025</td>
</tr>
<tr>
<td>(µl)</td>
<td>in IPA</td>
<td>in IPA</td>
<td>in IPA</td>
</tr>
<tr>
<td>Test temperature and humidity</td>
<td>65°C/85% RH</td>
<td>65°C/85% RH</td>
<td>65°C/85% RH</td>
</tr>
<tr>
<td>Test bias voltage</td>
<td>5 V (D C)</td>
<td>5 V (D C)</td>
<td>5 V (D C)</td>
</tr>
</tbody>
</table>

The influence of the reflow process on SIR was investigated at 65°C/85% RH with a single flux loading. This experiment was further modified by manual placing of a QFP over the SIR pattern just for the duration of the reflow, i.e. the part was not soldered. The conditions are shown in Table 3.

Table 3:
SIR Experimental Conditions Used for the Reflow Boards:
With and Without QFP Component

<table>
<thead>
<tr>
<th>Flux</th>
<th>RMA (Actiec 5)</th>
<th>WOA</th>
<th>PEG</th>
<th>WOA + PEG</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flux surface load</td>
<td></td>
<td>100</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(µl)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test temperature and humidity</td>
<td>65°C / 85% RH</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test bias voltage</td>
<td></td>
<td>5 V</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2.3 SURFACE INSULATION RESISTANCE MEASUREMENTS

The SIR measurements were performed using a bias voltage of 5 V DC. The test period was 48 hours. A Concoat AutoSIR was used to monitor SIR values on 16 channels every 10 minutes in an automatic fashion, the current sensitivity of the instrument is $2 \times 10^{-12} \text{A}$, and a $10^6 \Omega$ limiting resistor is included in each measurement channel. All SIR values are those for the whole pattern and not ohm squares. Two boards for each condition were measured, hence the results are the average of four measurements and the standard deviation of the log SIR is $< 0.3$ log ohms. The SIR value of a control board was measured during each test and the results are given in each Figure.
3 RESULTS

3.1 PCB FINISH

SIR testing is commonly carried on any number of different PCB finishes. Here we consider the influence on SIR of PCB finishes, specifically bare copper, HASL and AuNi. The same procedure as reported earlier\(^1\), and the conditions and fluxes given in Table 1 were used. The results are presented in Figure 3.

The results show that for three of the fluxes, RMA, WOA, and PEG there is little difference in the SIR response between the different PCB finishes. There is however a considerable difference in the SIR response when using the WOA+PEG flux. With this flux the SIR is dramatically reduced on the AuNi finish, which is due to dendrite formation. Previously it was shown that the dendrites on the AuNi boards were nickel. The susceptibility of the AuNi finish to dendrite formation can be understood with reference to the standard reduction potentials of the metals and their relative position to the hydrogen potential. The standard reduction potentials are given in Table 4.
### Table 4: Standard Reduction Potentials

<table>
<thead>
<tr>
<th>Element</th>
<th>Ion considered</th>
<th>Standard Reduction Potential (volts)</th>
<th>Solubility Products ($K_{sp}$, $M(OH)_n$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gold</td>
<td>Au$^{3+}$</td>
<td>+1.500</td>
<td>2.7x10$^{-27}$</td>
</tr>
<tr>
<td>Copper</td>
<td>Cu$^{2+}$</td>
<td>+0.337</td>
<td>1.2x10$^{-15}$</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>H$^+$</td>
<td>0.0</td>
<td>1x10$^{-14}$</td>
</tr>
<tr>
<td>Lead</td>
<td>Pb$^{2+}$</td>
<td>-0.126</td>
<td>1.2x10$^{-15}$</td>
</tr>
<tr>
<td>Tin</td>
<td>Sn$^{2+}$</td>
<td>-0.136</td>
<td>8.5x10$^{-30}$</td>
</tr>
<tr>
<td>Nickel</td>
<td>Ni$^{2+}$</td>
<td>-0.250</td>
<td>1.5x10$^{-16}$</td>
</tr>
</tbody>
</table>

From Table 4 it is clear that nickel will be of the greatest concern in terms of its propensity for dissolution at the anode, and that copper should be of the least concern, apart from gold. For dendrites to form a number of key conditions must be met, but principally there must be enough ions in solution. Metal ion concentration will in turn be dependent on the total volume of the electrolyte and the pH of that electrolyte. For a given electrolyte a metal ion with a lower standard potential will contribute a higher concentration of metal ions in solution, and hence be more harmful in terms of dendrite formation.

### 3.2 EFFECT OF TEST BIAS

The influence of test bias is considered next, and the results are shown in Figures 4 and 5, for the 5 and 50 volt conditions respectively. The plotted results are individual SIR curves. It is immediately clear that with the 5V data the SIR response is more stable when compared with the 50V data. With the RMA flux which tends to produce the most stable SIR response, in terms of sensitivity to experimental parameters, the 5V data reveal very repeatable results, whereas at 50V two results reveal the likely occurrence of dendrites, which was confirmed by visual inspection. With the RMA flux the final SIR values with the two voltages are very similar, approximately $10^9$Ω, indicating the electrolyte behaves in a very simple resistive manner. However, with the PEG flux this is not the case. No dendrites were formed but the final SIR values are different, the SIR value for the 5V experiment being approximately one decade lower. Hence the current flow in the electrolyte is broadly constant and independent of the applied voltage.

With the WOA flux there is a marked difference between the two applied voltages. At 5V no dendrites occurred, and the repeatability of the results was fairly good. However at 50V there is a much larger scatter in the results and dendrite formation was noted. For the WOA+PEG flux dendrite formation was noted for both bias conditions. However, it is interesting to note that with the 50V condition there was a recovery in the SIR values for some results. It is interesting to speculate that a high bias has a beneficial influence in destroying, or even preventing, the formation of dendrites.
Figure 4: SIR values at 5V test voltage and 65°C/85% RH for different fluxes

Figure 5: SIR values at 50V test voltage and 65°C/85% RH for different fluxes
3.3 EFFECT OF GLYCOL CHEMISTRY

The SIR values with the different glycol fluxes at a 100 µl surface loading and at 65°C/85% are shown in Figure 6.

![Figure 6: The SIR value with time for boards with different glycol with 100 µl surface load at 65°C/85%](image)

It can be seen from Figure 6 that polypropylene glycol does not reduce the SIR as much as polyethylene glycol, and that there is no effect on SIR by increasing the polyethylene glycol molecular weight. The difference between the polyglycols depends on the different hydroscopic property and the structure of the molecules and hence we can see that the polyethylene glycol is more hydroscopic than polypropylene glycol. This behaviour can be attributed to the steric branched chain molecular structure of polypropylene glycol, which inhibits the easy water adsorption along the chain.

3.4 IMPACT OF REFLOW AND THE PRESENCE OF COMPONENTS ON SIR

The tests discussed so far are for flux residues dispensed onto boards at 60°C. This is clearly not representative of a production process, but may well be typical of residues that are trapped on boards following processing. To simulate the soldering process, boards were prepared in an identical method of flux dispensing, but then given a reflow cycle, as described in 2.1. To replicate possible flux entrapment, on some of the boards a QFP component was manually placed over the SIR pattern for the duration of the reflow cycle. These components were not actually soldered to the board, and were removed for SIR testing. The effect of the reflow processes, with and without the QFP components, on the SIR values are shown in Figure 7.
Figure 7: The effect of reflow process on SIR for the board with different fluxes with 100µl surface load at 65°C/85% RH

It is immediately clear from Figure 7 that reflowing the boards has a big impact on the flux residues and the SIR. Generally, the SIR values for the boards that are not reflowed are low, whereas the reflowed values are close to the clean board values. Clearly volatilisation of the flux residues during reflow causing the SIR values to be high. The impact of the QFP can also be seen, and the effect of flux entrapment. For the RMA flux there is a steady drop in SIR with the applied conditions, and the effect of the QFP component is to cause a drop of half a decade in SIR. With the PEG flux there is no effect of reflowing with or without the QFP. For the WOA flux the presence of the QFP is significant, the simple reflowed data being the same as the clean board, indicating complete volatilisation of the flux, whereas with the QFP component the SIR drops by over a decade. With the flux combination of WOA+PEG the effect of the reflow is to remove almost completely the flux residues and lift the SIR values to that of the clean boards. The presence of the QFP has very little effect. These are surprising results considering the PEG flux SIR values and the effect of the QFP with the WOA flux.

4 DISCUSSION

This paper addresses a number of key issues in SIR testing that are often assumed to be constants. It is clear for example that the preparation of the samples is key to the outcome of the result. If the SIR test is being used to assure product reliability then the samples should resemble the product closely. It is clear from Figure 7 that reflowing the boards does have a considerable effect on removing the flux residues. Consequently in a properly optimised process, which may contain a cleaning stage, flux residues will be non-existent. However, as
shown in the simple example with a QFP component, with its relatively high stand-off, flux residues can be screened from the full reflow and hence result in residual flux deposits being present after processing. This is clearly of more concern in a no-clean process. Hence, careful SIR testing will be required if the future reliability of an assembly is to be assessed accurately.

The experiments to investigate the effect of bias showed that it did indeed have an effect. The major standards, ISO, ANSI-J-Stds and Bellcore, call for testing at 50V. This is somewhat concerning with today’s fine pitch components that typically use 5V or less, and certainly a test voltage of 50V is unrepresentative for these devices. There is concern that testing at 50V may well produce spurious results that are not typical of normal failure modes. This concern is vindicated by the results presented in Figures 4 and 5 where different SIR responses are noted for the different bias conditions. The track and gap used here, 0.32mm, for the SIR test pattern is typical of the spacings for fine pitch components.

5 CONCLUSION

1. PCB finish can have an influence on SIR values. Specifically it was noted in this work with a flux combination of WOA+PEG that there was a significant increase in the formation of dendrites. The propensity for dendrites to form with this flux appears to correlate with the standard reduction potential of the metals in the PCB finish. The bare copper finish generally had the highest SIR values.

2. Increasing the electrical test bias influences the mode of the SIR response. Dendrites were observed to form with the WOA flux at 50V but not at 5V. With the PEG flux the SIR value was not independent of bias, the current remaining constant with changing bias. Hence testing at higher biases give lower SIR values.

3. The use of 50V bias is common in all major SIR tests. Careful consideration needs to be given to testing at high voltages that are not realistic of current day use environments.

4. Subjecting flux residues to a soldering process increases the SIR, but this can compromised by the introduction of components which screen the residues from the full heat and permit their survival of the soldering process.

5. The effect of polyethylene glycol on SIR values was greater than that of polypropylene glycol. There was no effect on SIR by increasing the polyethylene glycol molecular weight.

ACKNOWLEDGEMENTS

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