The Impact of Temperature and Humidity Conditions on Surface Insulation Resistance (SIR) Values for Various Fluxes

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ABSTRACT

The SIR test has traditionally been performed by taking measurements at certain points during a seven day test under well established environmental conditions. The work reported here explores the influence of test temperature and humidity when using a typical resin flux, a weak organic flux and glycol based fluxes when sampling SIR patterns every 10 minutes. Results indicate that some fluxes are very sensitive to the test temperature, with volatilisation of flux residues an important issue. The frequent monitoring of the results also permitted the detection of dendrites during the SIR test. The results clearly show the importance of selecting the correct testing conditions and the benefit of frequent monitoring.

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 THEORY

SIR is a measurement of an electrochemical process between two metallic conductors on a substrate surface. In order for electrochemical migration to occur between conductive electrodes across an insulating circuit board surface, three conditions must be met simultaneously:

1. Electrical carriers (such as ions) must be present.

2. Water must be present to dissolve the ionic materials and sustain them in their mobile ionic state.

3. An electrical potential between the electrodes is needed to establish an ionic current in the water layer.

In the present work, a 5V bias was applied between the two electrodes. The SIR for a Au-Ni finish comb pattern with different fluxes was measured in the controlled temperature and humidity chamber. The following are some of the possible anode and cathode reactions.

At the anode:

$$2H_2O = O_2 + 4H^+ + 4e$$

$$Au = Au^{+3} + 3e$$

$$Ni = Ni^{+2} + 2e$$

At the Cathode:

$$O_2 + 2\,H_2O + 4e = 4OH^-$$

$$Au^{+3} + 3e = Au$$

$$Ni^{+2} + 2e = Ni$$

In the presence of other anions, such as Cl^- and $C_6H_9O_4^-$, which are present in flux residues from rosin and weak organic acids, metal complex ions may form. The electrolytic metal migration process, as shown in Figure 1, can be considered as a three-step process:

1. Electrodissolution: Oxidation of metal in water which occurs at the anode.

2. Ion transport: Ionic metal species are transported by electrical migration and diffusion.

3. Electrodeposition: Reduction of ionic metal species at a dendritic nucleation site on the cathode.

Clearly in terms of circuit reliability the flow of any stray current is of concern, but the growth of dendritic features can be catastrophic.

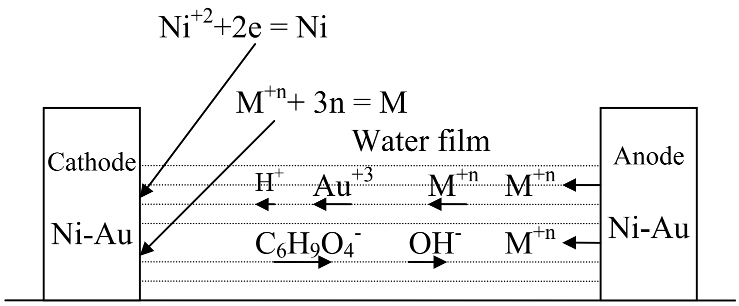

Figure 1 The electrochemical mechanism for SIR measurement

Examining the conditions for dendritic growth the overall flow of ionic species must be considered. Since the above three steps of electrodissolution, ion transport and electrodeposition, are all in series, the current flow is controlled by the slowest step. Under the SIR measurement condition, the water film formed on the test board is thin and the ionic concentration in the water film is low. Even if there are some flux residues dissolved in the water film, the ionic metal transport is usually the controlling step and the SIR value is dependent on the rate of ionic transport of metal ions. Different test temperatures, humidities, fluxes and flux surface loading on the test board, can all affect the ionic metal species transport rate. It has been found that a critical current density of the metal ion current is necessary to initiate dendrite formation(1). Since Ni will oxidise more easily than Au, based on thermodynamic considerations, the main reaction should be the reduction of Ni^{+2} on the cathode, and the formation of Ni dendrites under the requisite test conditions

3 EXPERIMENTS

3.1 SAMPLE PREPARATION

A NPL designed Au-Ni finish board with two SIR comb patterns was used. The board is shown in Figure 2. 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. 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 cm^2. The fluxed boards were then left for 16 hours at room temperature for the flux to dry.

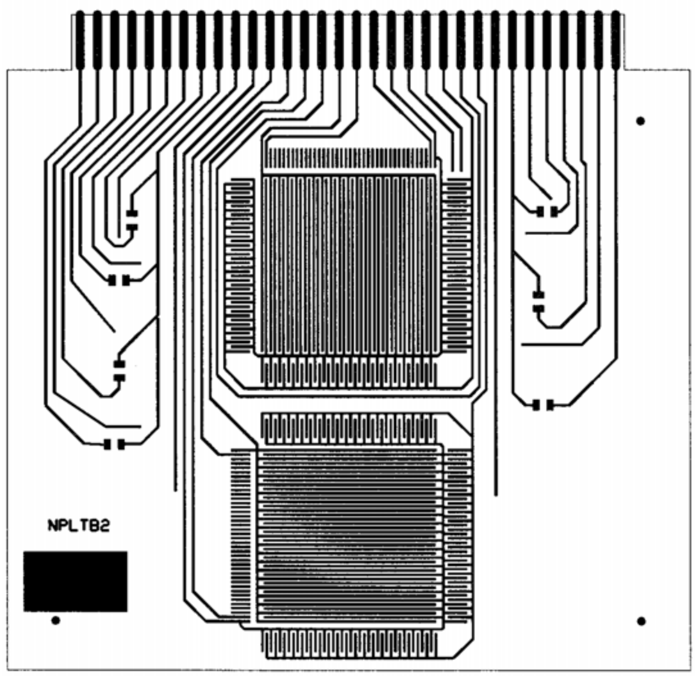

Figure 2 Test board for SIR measurements

3.2 FLUXES AND ENVIRONMENTAL TEST CONDITIONS

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.

Table 1:
Test Parameters: Fluxes, Flux Surface Load, Test Temperature And Humidity

Flux	RMA	WOA	PEG	WOA + PEG
Flux chemistry	Actiec 5	1.6g/l adipic acid in IPA	1.6g/l PEG400 in IPA	0.8g/l adipic acid + 0.8g/l PEG400 in IPA
Flux surface load (µl)	30, 100, 300			6, 15, 30
Test temperature and humidity conditions	65°C/85% RH, 85°C/85% RH, 65°C/65% RH, 50°C/85% RH			

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 were selected.

3.3 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 $2x10^{-12}$A, and a 10^6 Ω 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.

3.4 SEM-EDX ANALYSIS

The boards which exhibited intermittent electrical failure during SIR measurement, were visually inspected to detect the presence of dendrite formation. The dendrites were analysed with SEM-EDX.

4 RESULTS

The SIR values with the RMA flux as a function of surface load, temperature and humidity are presented in Figure 3. Similarly, the results for WOA flux are presented in Figure 4, the PEG flux in Figure 5, and the WOA plus PEG flux combination in Figure 6. There are some salient points to note from these four Figures. For the RMA, WOA and PEG fluxes the SIR values slowly increased with time, following an initial settling in period. The flux combination of WOA + PEG does not follow this trend, under certain conditions rapid changes in SIR were observed. The response using the RMA flux is very similar for all four test temperature and humidity conditions, as is also seen with the PEG flux. The WOA reveals that the SIR value is dependent on the combination of flux loading and test condition. With low WOA flux loading and increasing temperature the SIR value increases. This can be attributed to volatilisation of the flux and the board cleaning up[2]. With the PEG flux there is a clear decrement in SIR with increasing flux under all environmental conditions. This is also observed with the RMA flux with the more benign environment conditions, but as the conditions become more severe the SIR values tend to collapse to a single value of approximately 10^8 Ω, independent of flux concentration.

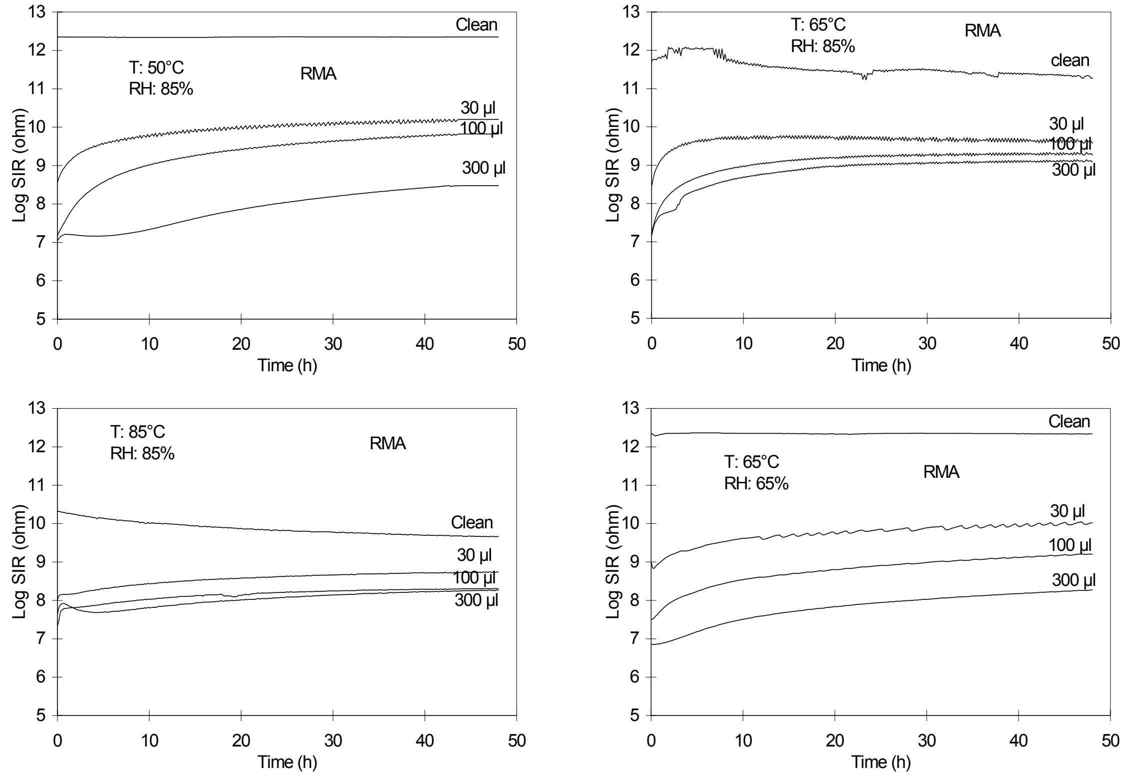

Figure 3: SIR values with Actiec5 for different surface loads, and different temperature and humidity conditions.

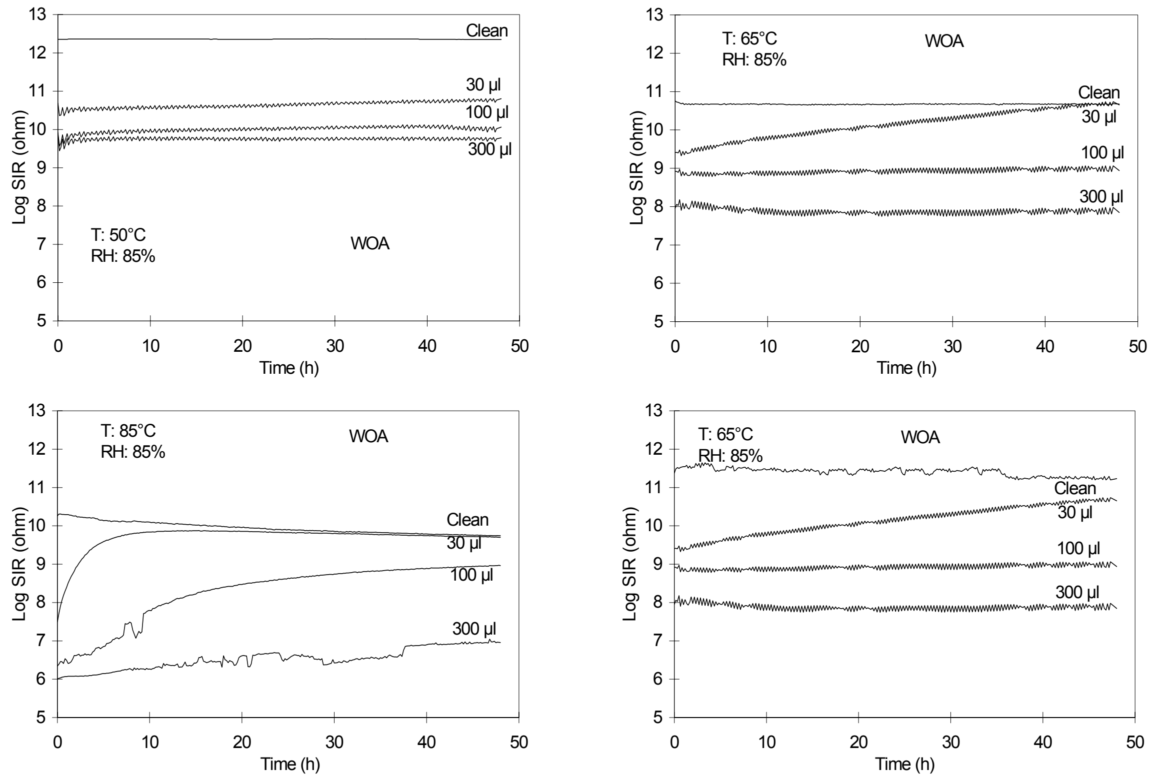

Figure 4: SIR values with 1.6g/l adipic acid in IPA for different surface loads, and different temperature and humidity conditions.

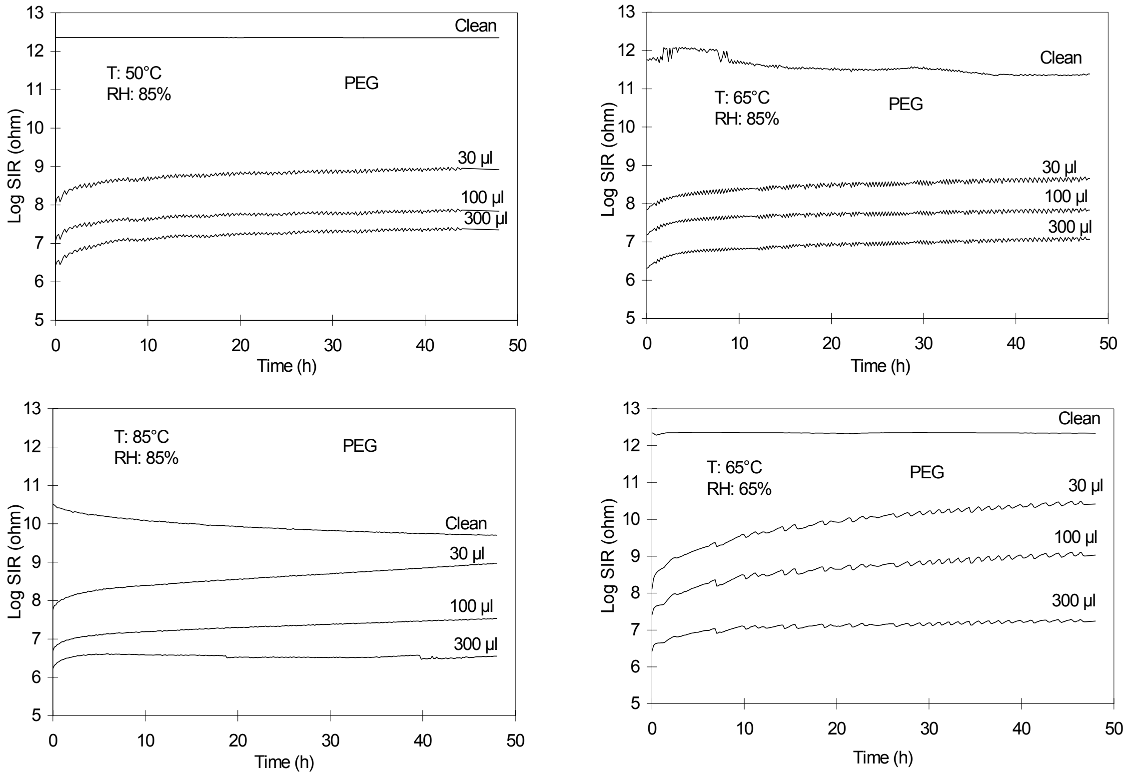

Figure 5: SIR values with 1.6g/l PEG400 in IPA for different surface loads, and different temperature and humidity conditions.

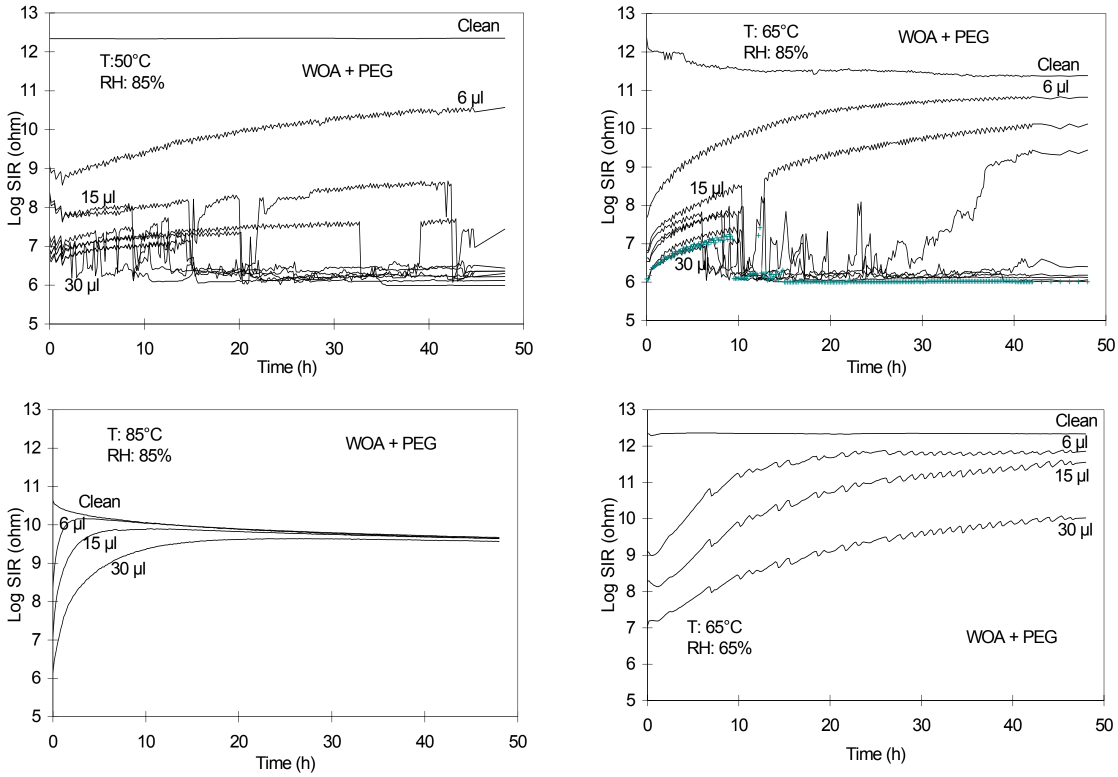

Figure 6: SIR values with 0.8g/l adipic acid +0.8g/l PEG400 in IPA for different surface loads, and different temperature and humidity conditions.

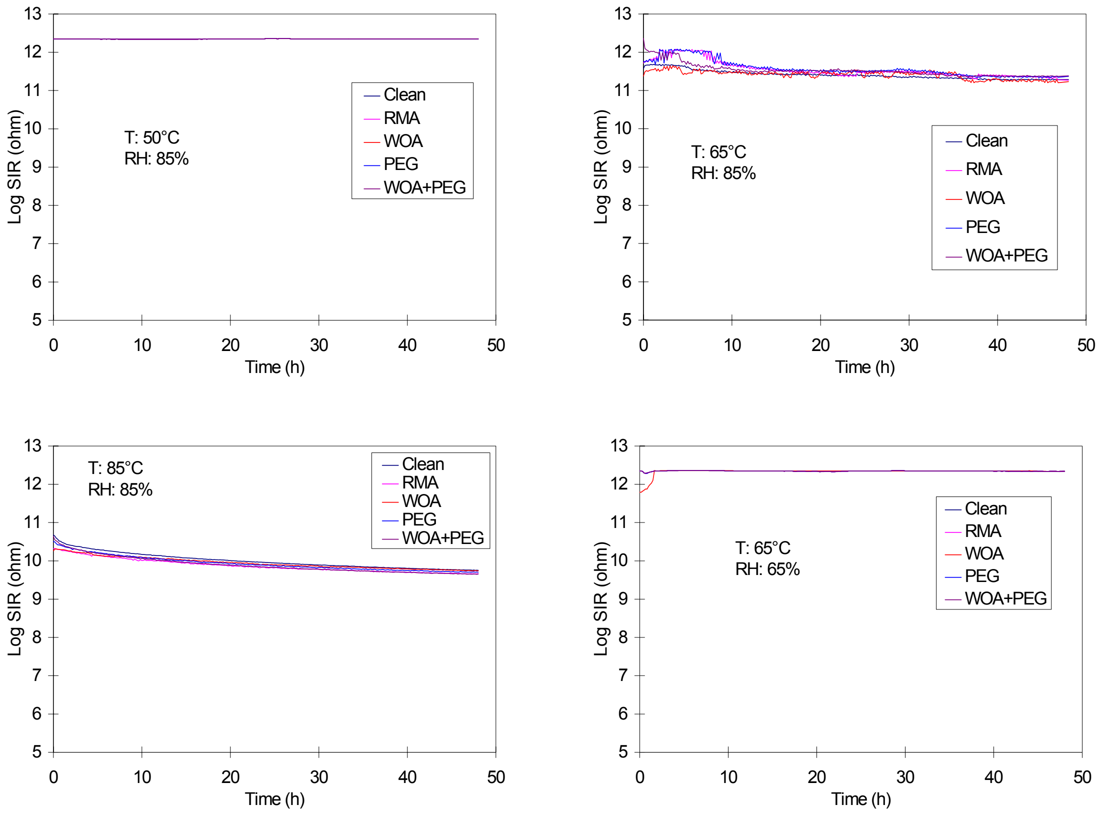

Figure 7: The SIR value for the clean board when tested with different fluxed boards, in the different temperature and humidity conditions

The clean board SIR values from each test are plotted in Figure 7 from each test. These values are independent of the fluxed conditions and are a function of the various temperature and humidity conditions, as would be expected. These plots also contain SIR values for clean boards tested without any fluxed boards being present. This was performed to confirm that there were no effects due to cross contamination from the adjacent fluxed boards. The clean board SIR values for 50°C/85% RH and 65°C/65% RH were both very high and at the measurement limit of the instrument. The 65°C/85% RH SIR values were intermediate with the 85°C/85% RH the lowest. The change in the clean board values reflect water absorption on to the board, in combination with a possible response of the board to the presence of water. The 85°C/85% RH shows a long settling in period, which is indicative of the water layer absorbing ions, either from the substrate or the atmosphere.

Rapid changes in SIR values were observed with the WOA and PEG flux combination, as shown in Figure 6. These rapid SIR fluctuations principally occurred with the 50°C/85% RH and 65°C/85% RH conditions, and with flux loading's of 15 and 30 µl. For these four testing conditions the four individual SIR traces are shown, illustrating the rapid change in resistance that can be observed in SIR testing. These rapid changes in current are associated with the formation of dendrites, fragile filamentary structures that grow from the cathode. An example of dendrite formation between the electrodes of the SIR pattern is shown in Figure 8, when 30µl of WOA plus PEG flux was tested at 65°C/85% RH. Figure 8 clearly shows the dendrites growing from both sides of a cathode.

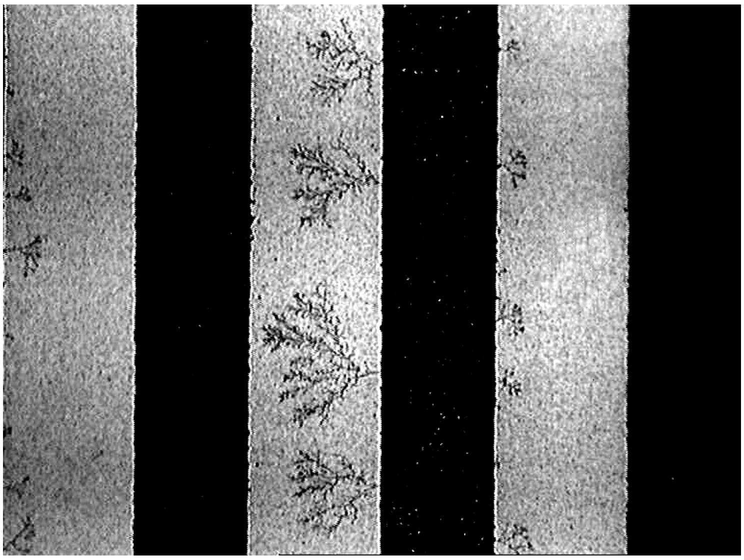

Figure 8: Dendrites formed when testing with 30 µl Adipic acid + PEG400, at 65°C/85% RH.

The increasing trend in SIR values through the test for the RMA and PEG flux are typical for SIR tests where the free charge carriers are swept to the electrodes and no longer contribute to the measured current. With the WOA flux and the combined WOA and PEG flux a much faster rise in SIR value is observed for the 85°C and to a lesser degree the 65°C results. This large increase in SIR can be attributed to evaporation of the WOA(2). Considering the WOA data at 85°C/85% RH different rates of increase in SIR were observed, see Figure 4, where after 15 hours with 30µl of flux the SIR value was the same as the clean board. The 100µl data also show a large increase in SIR, but the 300µl data only increase slowly. It is clear that if a WOA is present in a flux on its own or in combination with PEG, evaporation effects and increasing SIR are observed for high temperature testing.

The detrimental effect on SIR and concomitant formation of dendrites by the WOA plus PEG flux can be seen for the 50 and 65°C data at 85%RH. With just 15µl of this flux the SIR value dropped to the limiting value of the machine, $10^6\Omega$. The propensity of dendrites was observed to increase with the flux load form 15 to 30µl. None of the dendrites bridged the gap to the anode. SEM-EDX analysis of the dendrites revealed them to be nickel, with only a trace of gold detected. This is consistent from an electrochemical understanding with preferentially dissolution of the Ni^{2+} to any Au ion. At 85°C no dendrites were formed due to the high evaporation rate of the WOA. These results confirm a synergistic effect between PEG400 and adipic acid, leading to dendrite formation. The occurrence of dendrites is critically dependent on the test conditions: flux surface load, test temperature and humidity. At 65°C and 65% RH the erratic SIR behaviour and dendrite formation were not observed, illustrating the necessity for high humidity.

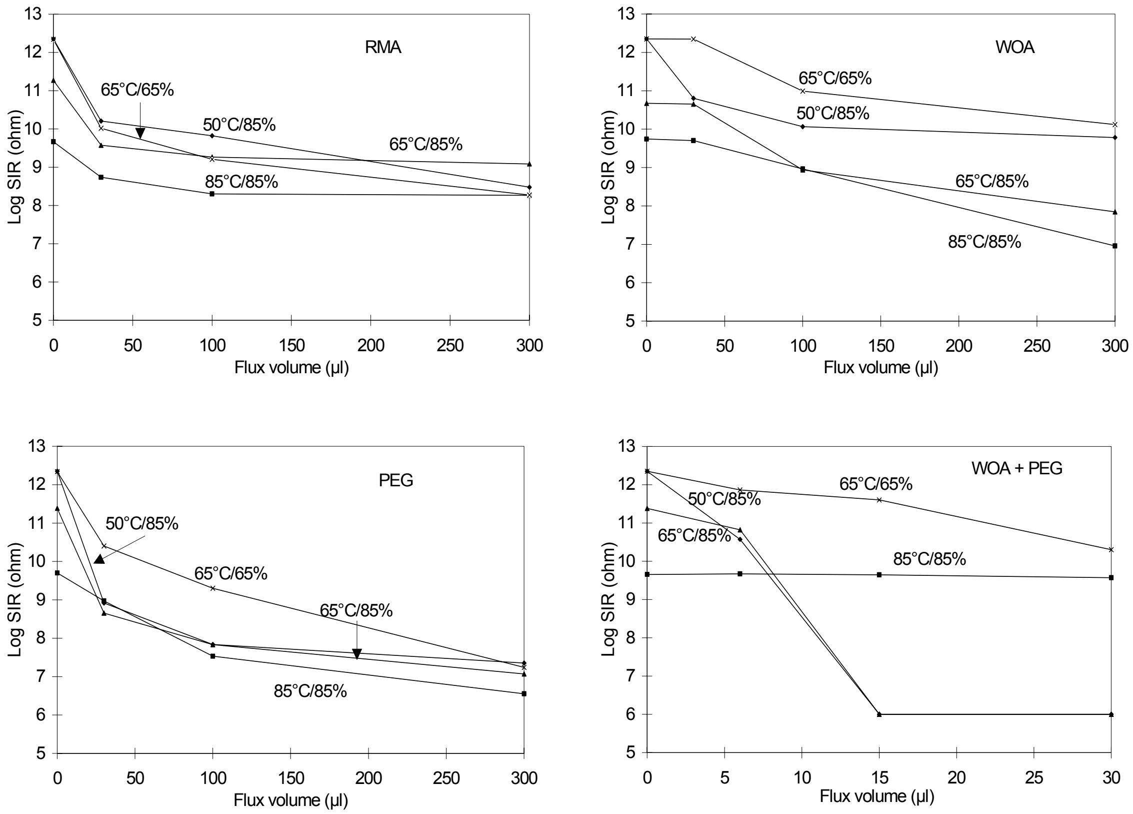

Figure 9: Effect of flux volume on SIR values at 48 hours for the different fluxes.

The effect of flux loading was seen to decrease the SIR value. For the RMA and WOA this clearly reflected an increase in the available ions. With the PEG flux no extra ions were introduced by adding more PEG, but since PEG is hydroscopic, it enhanced the water layer thickness, and hence decreased the SIR. With increasing WOA+PEG flux, both the ionic and hydroscopic components increased, however the previously noted synergistic effect, of a dramatic reduction in SIR for relatively small flux loads was observed. Note that the adverse effects were not observed with the 6µl.

Since the SIR response was generally a smooth function, with the notable exceptions described above, and with no marked variation during the test following the stabilising of the SIR value after 30 hours, the final SIR value at 48 hours was taken for each test and plotted against the various test parameters. These results are shown in Figures 9 to 11.

4.1 FLUX LOADING

The influence of flux loading on the SIR appears dependent on the flux. The drop in SIR with flux loading is a minimum for the RMA flux, and indeed the majority of the decrease in SIR occurs within the first 25µl of flux. For this flux this behaviour is easily understood in terms of the encapsulating properties of the resin. Increasing the resin beyond a certain point has little further influence in the drop of SIR. A thicker film clearly does not provide any additional ionic carriers and as we see later increasing the humidity does not reduce the SIR, indicating the barrier nature of the flux residue.

For the PEG flux there is clearly no saturation effect, with SIR dropping for any of the four environment conditions used. However, the reduction in SIR is significantly less above 100µl than below, except for the 65% humidity condition. The results for 85% RH show little dependence on temperature, whereas the 65%RH data is significantly different 85% RH data. The results indicate that for low to moderate PEG flux loads, the impact on SIR values are very dependent on RH and relatively insensitive to temperature.

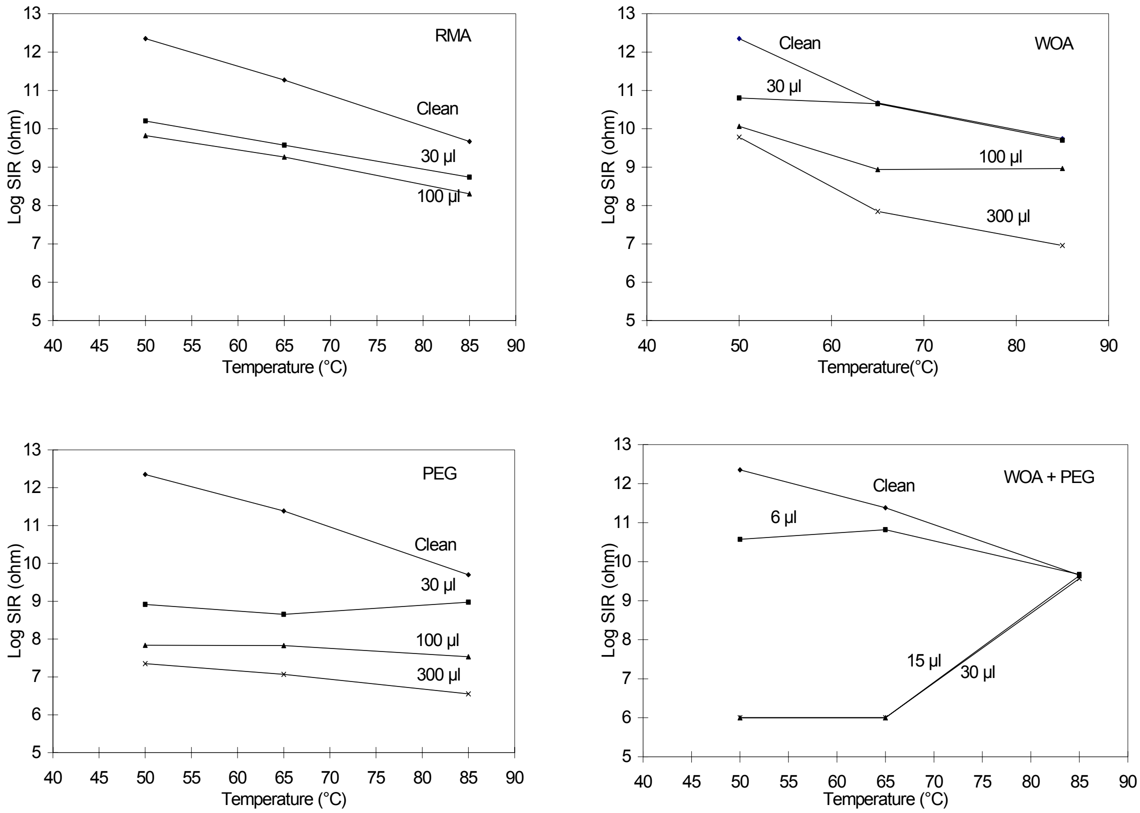

Figure 10: Effect of temperature on SIR values at 48 hours for the different fluxes (humidity 85%).

With the WOA flux the response with flux loading becomes more complex, which is a result of the volatilisation of the flux at elevated temperatures. The 65 and 85°C data show no drop in SIR with 30µl of flux. This is because the flux has volatilised as shown by the increasing SIR in Figure 4. Only the 50°C data show the effect of flux loading with 30µl of flux, again confirming that testing for these fluxes should be carried out at temperatures no higher than 50°C. Both 65°C data show a similar response to increasing flux load above 30µl, whereas with the 85°C the increase is more tempered. This confirms that humidity is not important in the volatilisation rate. Clearly at higher flux loading of 100µl and above the level of flux load is sufficient to influence SIR and overcome the effects of volatilisation.

The flux mixture of WOA+PEG is extremely sensitive to even small flux volumes, note the axis on this graph compared to the others. As noted above the formation of dendrites have a significant affect on the SIR, as evidenced by the rapid changes in Figure 6. The 85°C data are independent of flux load, and it is clear that the flux has completely volatilised by the end of the test. At 65%RH no dendrite formation was observed at these flux loadings, and the results show a drop in SIR that follows the PEG flux response. The 50 and 65°C data behave almost identically, indicating that if the correct chemical conditions are met, then the dendrite formation is driven by the supplied electrical bias.

4.2 TEMPERATURE

The impact of temperature is shown in Figure 10, where a range of responses is observed. With the RMA flux there is a steady drop in SIR with increasing temperature, that probably reflects increasing ionic mobility and possibly ionic concentration in the electrolyte.

The PEG flux is not as temperature sensitive, the conductivity rather following the humidity as seen in Figure 11. Therefore, water absorption with the PEG flux only weakly follows the temperature. The PEG 30µl data reveal that there could be an evaporation effect of the PEG at high temperatures, there is an increase in SIR from 65 to 85°C.

The WOA flux shows a clear temperature effect. The 30µl data shows hows the SIR does not increase between 50 and 65°C, and then follows the clean board SIR values as the temperature is increased to 85°C. As described above this is rationalised with the evaporation of the WOA flux. With the 100µl data at 85°C there is a similar effect on SIR.

With the WOA+PEG flux the effect of temperature is even more apparent. For the 15 and 30µl loads all the flux is evaporated at 85°C, with an intermediate effect with 6µl.

4.3 HUMIDITY

Generally increasing the humidity reduces the SIR. However with the RMA flux this does not occur, and can be attributed to the encapsulating nature of the rosin residues. With the PEG flux there is a clear trend of reducing SIR with increasing RH, except at 300µl where some saturation effect is observed. Similarly with the WOA flux there is again a trend of reducing SIR with increasing RH. With the WOA+PEG flux the SIR response to different humidities reveals a critical dependence on flux load. With the 6µl of flux the SIR value follows the clean board closely with a small drop in SIR from 65 to 85%RH, but with the 15 and 30µl of flux the SIR drops dramatically as the humidity increases from 65 to 85%RH. This drop in SIR clearly illustrates the synergistic effects described earlier and the formation of dendrites. It is clear that high humidity and the concomitant thicker water film that forms, is critical for dendrite formation.

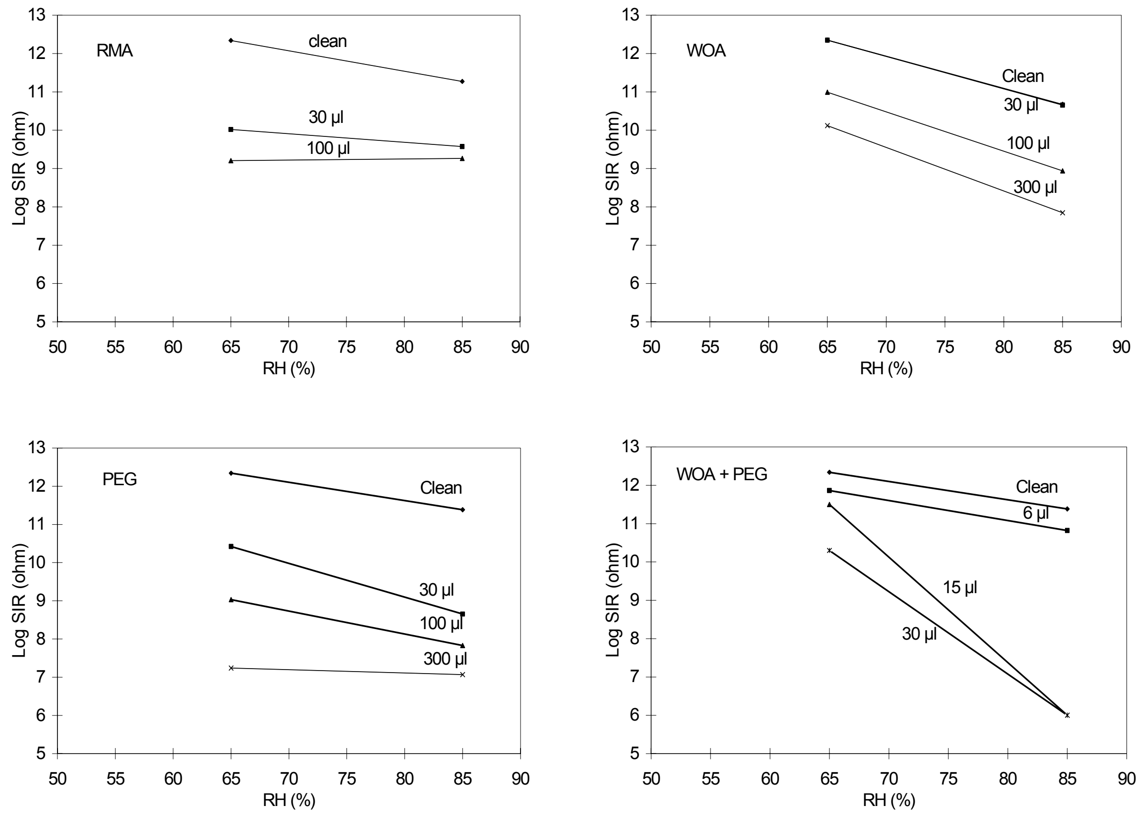

Figure 11: SIR value at 48 hours as a function of humidity for boards with different fluxes with different surface loads at 85°C

5 DISCUSSION

The work set out to explore how certain key components in modern fluxes behave during SIR testing. Hence, while the results do not apply to specific commercially available fluxes they are indicative of the behaviour that may be observed if these flux residues are found on processed boards.

The results show that the choice of testing environment can be influential in the outcome. In particular the WOA was found to be sensitive to the test temperature, as reported elsewhere(2). It is apparent that with the WOA flux the result is also dependent on the flux quantity, so that a simple relationship between flux volume and volatilisation rate can be established.

The PEG and the RMA flux proved to be robust during SIR testing with different test environments, with SIR values varying incrementally with changing conditions. Clearly, this was not the case with the WOA flux, and even more so when the WOA flux was mixed with the PEG, when the behaviour became even more unstable. Although this simple flux combination is unusual in terms of commercial fluxes it is useful in permitting the characterisation of this family of fluxes, and specifically fluxes which show a propensity for dendrite formation.

Monitoring the SIR values every 10 minutes permitted tracking of the formation of dendrites very closely. The data in Figure 6 reveals the rapid changes in SIR that can occur as the fragile structures form. Clearly monitoring at 1, 4 and 7 days as called for in current international standards would miss all the information contained within the curves. If the occurrence of dendrites during testing constitutes a failure, then a rapid change in SIR should be considered as the failure criteria.

It is interesting to note the impact of humidity, especially for the WOA flux. The results show that SIR values change significantly with RH, which implies that RH should be carefully controlled. Since most chambers use a wet and dry bulb method, the small difference in temperature at high RH, will cause there to be a greater error in the RH. As the results in Figure 11 show this will be significant and it is inadvisable to operate above 85%RH.

6 CONCLUSION

1. Surface insulation resistance (SIR) is strongly dependent on the humidity and temperature. Increased humidity and temperature reduce the SIR. However, for fluxes containing adipic acid, higher temperatures accelerate volatilisation of the flux, and consequently the SIR increases. A test temperature of 85°C is too high for fluxes which contain adipic acid.

2. Frequent monitoring proved very effective at monitoring dendrite formation electrically. Full consideration should be given to incorporating into the standards as a pass/fail criteria the rapid changes in SIR as constituting a failure in the standards.

3. Dendrites were formed on boards fluxed with adipic acid + PEG400 at 15µl and 30µl surface loading, and tested at 65°C/85% RH and 50°C/85% RH. The propensity of dendrite formation was also dependent on flux volume. These result are significant, since if flux residues from flux formulations containing WOA used in the assembly process and flux residues from the HASL process, combine, then SIR values can be dramatically compromised.

4. SIR values are strongly dependent on RH. In view of instrumental issues, caution should be exercised when operating above 85%RH.

ACKNOWLEDGEMENTS

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