

NPL REPORT
DEPC MPR 029

**Preliminary Measurements of
Soldering Flux Residues in
an AC Environment**

**A Brewin, L Zou, M Dusek
and C Hunt**

NOT RESTRICTED

AUGUST 2005

Preliminary Measurements of Soldering Flux Residues in an AC Environment

Alan Brewin, Ling Zou,
Milos Dusek and Christopher Hunt
Engineering and Process Control Division

ABSTRACT

Surface insulation resistance (SIR) techniques are currently used to measure the effects on long-term reliability of electronic assemblies of residues emanating from electronic production processes. This is achieved by monitoring leakage currents under a DC voltage in high temperature and humidity conditions.

This approach has been adopted from the simple expedient that tests run under DC conditions are simpler to implement than those under AC conditions. Historically, however, there has also been a concern that under AC conditions electro-chemical corrosion failures such as dendrite growth will not occur.

This exploratory work has studied the effects of flux residues on the impedance of interdigitated combs under the application of AC voltage. These effects are considered to be more relevant to digital and AC circuit designs currently in use in many applications. The effects of the presence of generic flux types (rosin, water-based, and glutaric acid) on SIR style interdigitated combs have been measured and compared, using the parameters of impedance (real and imaginary) and capacitance.

The study indicates that the use of impedance properties as a tool for differentiating the effects of flux and their potential effect on AC circuits is promising, and warrants further study. Suggestions for further work are included.

© Crown copyright 2005
Reproduced with the permission of the Controller of HMSO
and Queen's Printer for Scotland

ISSN 1744-0270

National Physical Laboratory
Hampton Road, Teddington, Middlesex, TW11 0LW

Extracts from this report may be reproduced provided the source is acknowledged and the extract is not taken out of context.

We gratefully acknowledge the financial support of the UK Department of
Trade and Industry (National Measurement System Policy Unit)

Approved on behalf of the Managing Director, NPL,
by Dr M G Cain, Knowledge Leader, Materials Processing Team
authorised by Director, Engineering and Process Control Division

CONTENTS

1	INTRODUCTION	1
2	EXPERIMENTAL	1
2.1	TEST VEHICLE DESIGN	1
2.2	FLUX APPLICATION	2
2.3	DIRECT ELECTRICAL MEASUREMENT	3
2.4	EVALUATION OF COMB CONDITIONS WITH ON-BOARD CIRCUITRY	4
2.5	ENVIRONMENTAL CONTROL	5
3	RESULTS.....	6
3.1	SYSTEM VERIFICATION.....	6
3.1.1	Effect of bridge	6
3.1.2	Comparison between cable sets	7
3.2	COMB MONITORING AT 100KHZ	8
3.3	FLUX COMPARISONS.....	11
3.3.1	Impedance	12
3.3.2	Resistance (Real component of Impedance).....	13
3.3.3	Reactance	14
3.3.4	Capacitance	14
3.4	TEST METHOD REPEATABILITY.....	15
3.5	ON-BOARD CIRCUIT	17
4	DISCUSSION.....	18
5	CONCLUSIONS.....	19
6	REFERENCES	19
7	ACKNOWLEDGEMENTS.....	19

1 INTRODUCTION

During production processing residues can be left on the surface of circuit assemblies, and some common sources might be solder flux/paste, flux-cleaning chemistries, plating baths, etching chemicals and desmear solutions. The effects of these residues are varied, but they can cause reliability problems for electronic product by increasing the risk of current leakage and electrochemical migration. Surface insulation resistance (SIR) techniques are presently used to measure the effects of such residues on circuit performance, by monitoring leakage currents during the application of DC voltage in high temperature and high humidity conditions [1].

Measuring resistance is currently considered appropriate, since any ionisable residues will be swept to the electrodes and might cause corrosion. With an AC signal it may be argued that the ionised residues do not accumulate at the electrodes, and therefore no corrosion will take place.

This work does not accept this proposition, and the results show that change in impedance can be expected with time. The approach taken here is to continue with the interdigitated comb, and address questions of circuit design, and what circuit parameters to use.

Whilst the SIR technique has a successful record of providing important and valuable information in term of the effects of residues to circuit reliability, it uses a DC voltage as the main accelerating factor in the test. Clearly this is reasonable for electronic circuits operating with DC signals, but the majority of circuits today have at least some functionality utilising an AC or digital designs.

The aim of this exploratory work was therefore to explore the effects of flux residues in an AC voltage environment, in an attempt better to represent digital and AC circuit designs currently in use. The parameters investigated were impedance (real and imaginary) and capacitance.

The loading of an interdigitated comb with samples of a flux, and introducing them into a humidity chamber, was followed since it reflected conventional SIR testing. Two overall approaches were developed. The first was to use a versatile impedance analyser, and the second was to introduce a small circuit adjacent to the comb pattern to produce a low impedance AC voltage proportional to the reciprocal of absolute comb impedance.

2 EXPERIMENTAL

2.1 TEST VEHICLE DESIGN

The test vehicle chosen, TB54, is depicted in Figure 1. The comb has track and gap dimensions of $400\mu\text{m}$ and $200\mu\text{m}$ respectively, and an area of 625mm^2 (as shown in Figure 1). The comb design has 2625 squares. The tracking and pad design allows for connection to the combs at four points with BNC connectors, and also for connection to a measurement circuit on the test vehicle.

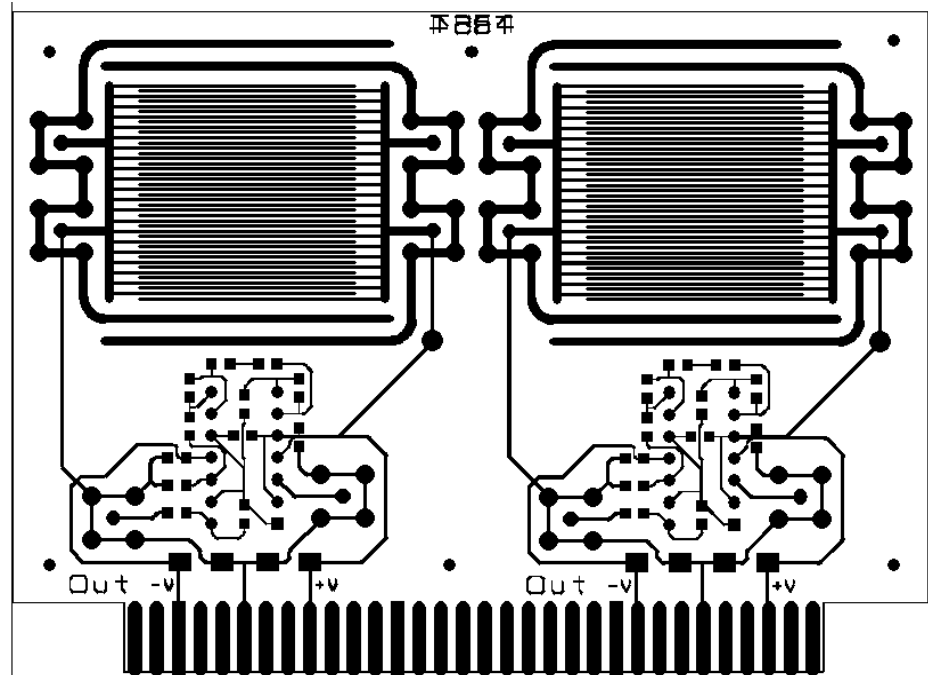


Figure 1. TB54 test vehicle.

The TB54 design allows for the use of an on-board circuit or direct measurement by an impedance analyser. The on-board circuit is described later in Section 2.4.

2.2 FLUX APPLICATION

The combs were cleaned with a 50/50 mixture of Propan-2-ol and deionised water and dried prior to flux application. The flux was applied using a calibrated micropipette directly onto the comb, ensuring even distribution across the comb. The fluxed combs were then baked in a pre-heated convection oven for 5 minutes at 100°C.

The fluxes used were:

Water-based Flux

- 90.1% De-ionised water
- 5% Glycol ester (CAS no 34590-94-8)
- 1.6% Adipic acid
- 1.6% Succinic acid
- 1.6% Glutaric acid
- 0.1% alcohol ethoxylate surfactant (CAS no 68131 - 39 - 5)

Glutaric Acid Flux

- 0.1% Glutaric acid
- 99.9 % Propan-2-ol

Rosin-based Flux

- Commercially available high solids rosin flux

2.3 DIRECT ELECTRICAL MEASUREMENT

The impedance and capacitance measurements were taken using an Agilent 4294A Precision Impedance Analyser.

Each comb pattern was connected for the four-point measurements as shown in Figure 2.

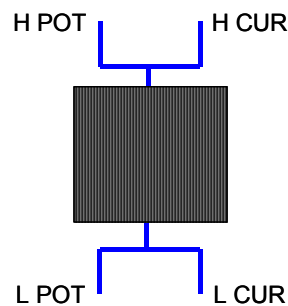


Figure 2. Four point measurement connections to comb pattern

For the connections to the combs shielded co-axial cabling was used with SMB connectors, and all the cables used were identical (2m long). Four combs were monitored at a time, and were held in a humidity chamber in a rack system, which provided mechanical support but no electrical connection. Four sets of cables (four for each comb) were routed through a switching bridge unit (Agilent 34970A) allowing the impedance analyser to be connected selectively to each of four combs and hence measurements to be made. Note that when the analyser was not looking at a pattern, the comb was not connected to any supply.

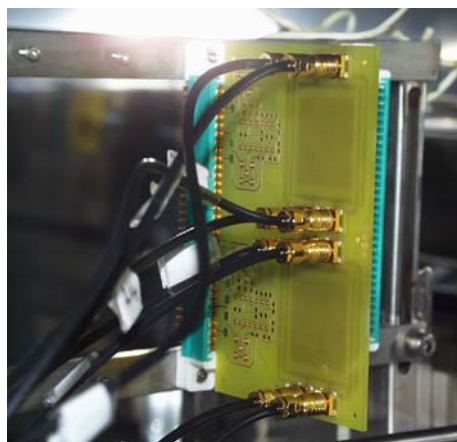


Figure 3. Connection to combs using SMB connectors

A PC running a Visual Basic Macro was used to log data from the impedance analyser and control the impedance analyser and bridge.

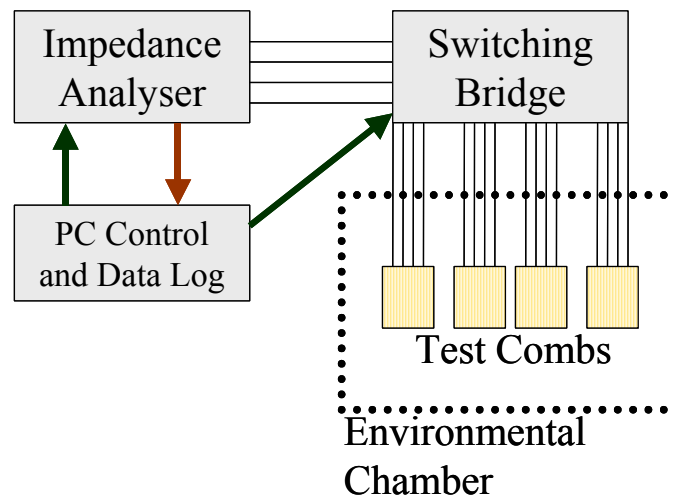


Figure 4. Schematic of system set up

The switch arrangement could only apply bias to a comb when the impedance analyser was switched to that pattern. This loss of bias to patterns while not being measured is a drawback to this particular experimental arrangement.

The parameters logged by the impedance analyser are given in Table 1. The system was configured to perform a frequency sweep, and log these properties for each comb at 1, 2, 5, 10, 20, 50 and 100 kHz. The applied bias was 500mV, and the properties for each comb were measured every 3 minutes throughout the test.

Table 1. Parameters logged from impedance analyser

[Z]	Magnitude of Impedance
C	Capacitance
X	Reactance (Imaginary component of [Z])
R	Resistance (Real component of [Z])

The system was calibrated to compensate for the effects of the bridge, cables and connectors by the use of a fixed resistor connected in place of the comb pattern. Agilent procedures describing compensation are available in the manual for the impedance analyser.

2.4 EVALUATION OF COMB CONDITIONS WITH ON-BOARD CIRCUITRY

A measurement circuit was incorporated into the test vehicle design to allow monitoring of the properties of the comb patterns, as an alternative to the use of the impedance analyser. The Electronics Systems Design Centre at University of Canterbury, Kent designed the circuit, which is illustrated in Figure 5. The circuit is intended as an option

to the impedance analyser, and produces a simple DC level that is proportional to the impedance of the comb pattern.

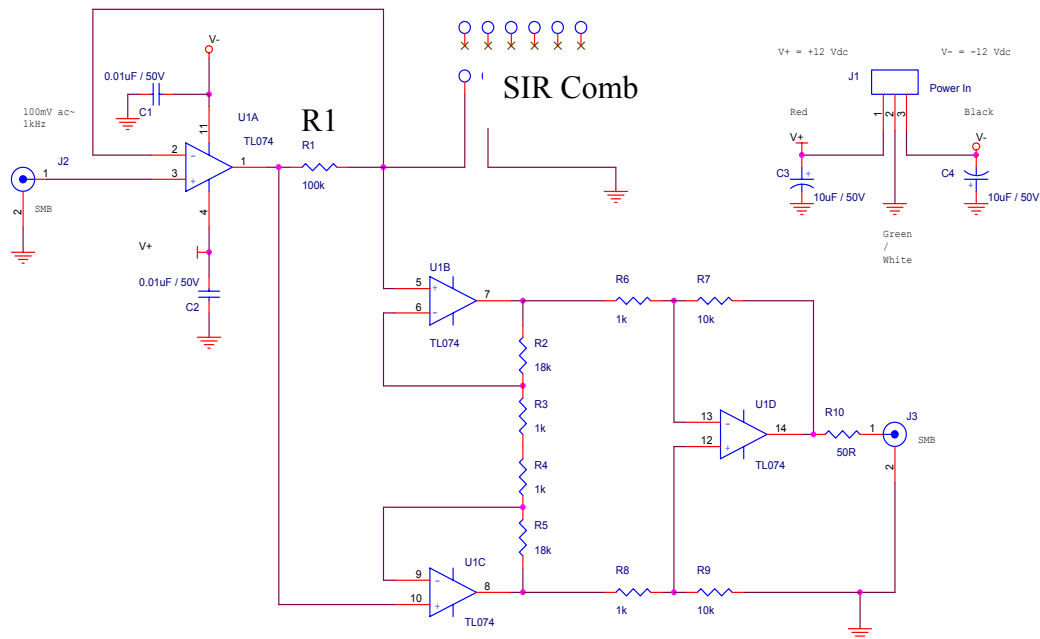


Figure 5. On-board comb monitoring circuit

The circuit is designed as a two-stage amplifier with an interdigitated comb in the feedback of the first stage. The first stage amplifier comprises a non-inverting operational amplifier (U1A). The comb is connected as feedback impedance and is supplied with the same potential (V_1) as the input of U1A amplifier. The voltage amplitude at the resistor R1 is $V_1 \cdot (R_1/Z_2)$, where Z_2 is the comb impedance. This voltage drop across resistor R1 is sensed by the stage 2 - instrumental amplifier (U1B, U1C, & U1D). Hence the coupon is monitored without loading. The second stage amplifier has fixed gain proportional to resistors ($R_3 + R_4$).

If the absolute value of impedance of the comb falls below the resistance R1, the monitored output will approach the maximum supply voltage i.e. output is saturated. Conversely, if the impedance of the comb is higher than R1, the gain of the first stage amplifier will approach zero, and the measured output of second stage of the amplifier will be only the residual noise. Typically this type of circuit provides a dynamic range of three or more orders of magnitude, depending on the sensitivity of the measuring device.

2.5 ENVIRONMENTAL CONTROL

A Sanyo/Gallenkamp HCC031 environmental chamber was used to expose the test combs to high temperature and high humidity environment. Conditions of 40°C and 93%RH were used as the typical environmental condition, consistent with those used for no-clean flux technologies in traditional DC SIR testing. The relatively low

temperature avoids volatilisation of flux materials, and the high humidity promotes surface leakages and moisture absorbance.

3 RESULTS

3.1 SYSTEM VERIFICATION

The effects of cables, switches and connectors can be significant under AC conditions, especially at higher frequencies. In order to ascertain the extent of these capacitance effects for the measurement system and ensure correct compensation, measurements on a blank control comb were made for different connection methods.

3.1.1 Effect of bridge

Measurements of AC parameters were made on a bare comb under ambient conditions, connecting directly with cables between the impedance analyser and comb pattern, and then connecting through the switching bridge. The system was calibrated without the bridge in place. The results are presented in Figure 6:

Blue – Connection via 4cm copper flying leads, with the impedance analyser compensating for cables

Purple – Connection via SMB coaxial cables, including switching bridge, but without compensation for bridge

Green – Connection via SMB coaxial cables and including switching bridge, and with the impedance analyser compensating for the bridge

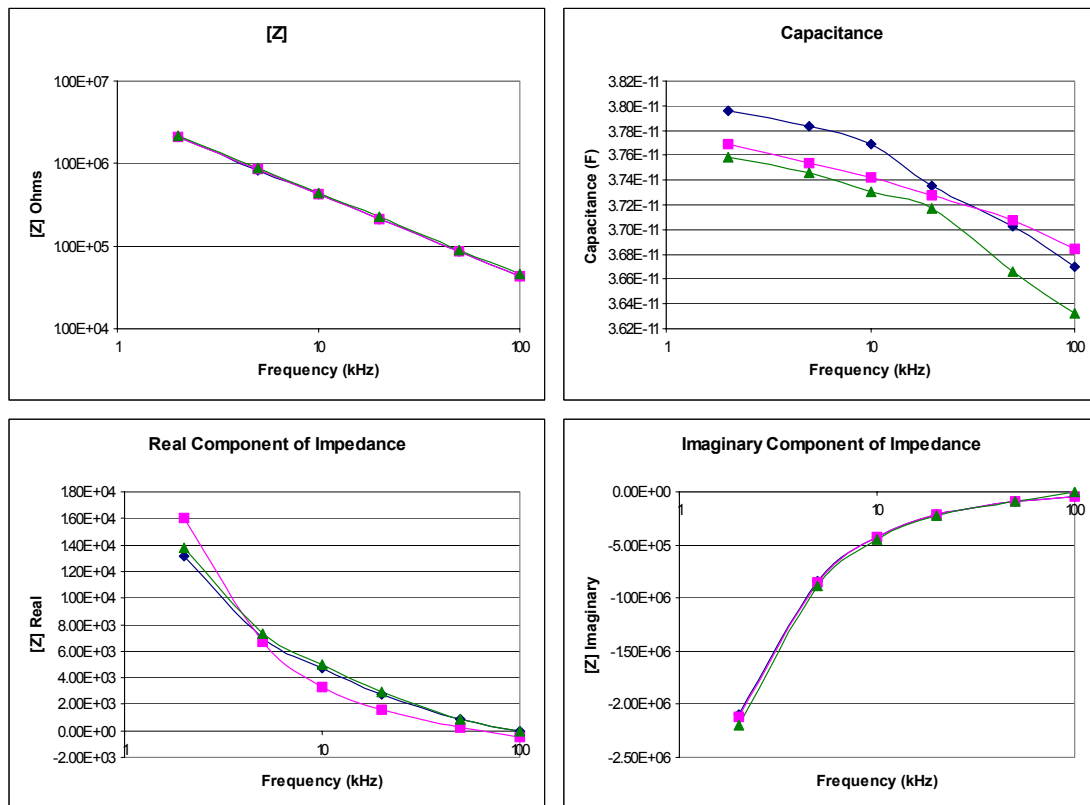


Figure 6. Measurements on a blank comb at ambient conditions

The results confirm that without compensation, capacitance effects do occur, but the impedance analyser does apply adequate compensation.

3.1.2 Comparison between cable sets

The impedance analyser (IA) system was able to compensate for the resistance and inductance effects of the switching bridge, cables and connectors, this compensation was performed for each set of cables. When the switching bridge changes channels, so that the properties of another comb can be measured, another set of cables come into use. However only one compensation could be set up on the impedance analyser. Therefore efforts were made to ensure that the cables were identical in design, and the data presented here helps to establish the effect of switching between cable sets - ideally this difference should be negligible.

Figure 7 gives data for measurements on a resistor and a capacitor component (10M Ω and 100pF) in parallel placed inside the environmental chamber and connected with four different sets of cables through the switching bridge. The values plotted are average values for the four measurements, with the error bars showing the maximum and minimum values measured, not the standard deviation. The agreement between data collected with each cable system is good. The differences are $\pm 3\%$ in all cases, except for [Z] Real at high frequencies, which highlights that the differences due to the cabling become more apparent as [Z] real decreases.

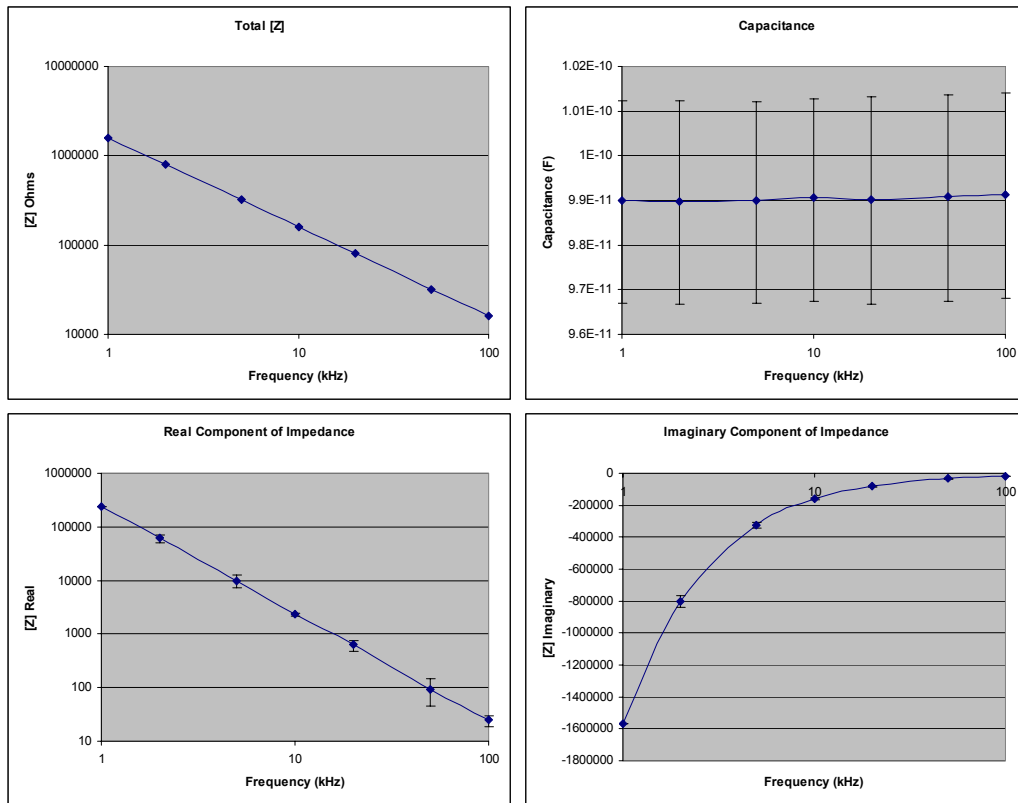


Figure 7. Effects of different cable sets on measured parameters

3.2 COMB MONITORING AT 100KHZ

Data for Z (Impedance), R (Resistance), X (Reactance) and C (Capacitance) are presented in Figure 8 for four different conditions (bare board, water based flux, glutaric acid and rosin). The capacitance was calculated using an assumed model of resistor and capacitor in parallel. 50 μ l of each flux was applied to the test patterns, which were then heated for 5 minutes at 100°C. These typical data reveal whether or not time-dependent behaviour can be observed at this frequency (100kHz) for the different flux loadings on the comb.

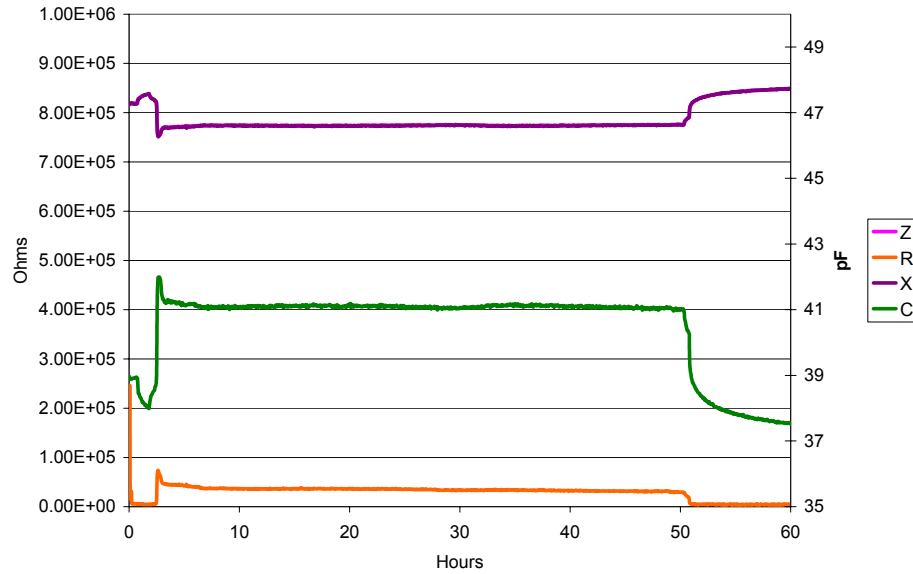


Figure 8. Plot of Z (Impedance), R (Resistance), X (Reactance) and C (Capacitance) against time for a bare comb

The results in Figure 8 indicate that after the introduction of humidity, there was little variation in the values of Z, X, C and R with time. Moreover, the original values were recovered when the temperature and humidity were removed. This demonstrates that without flux this system behaves in a very straightforward way. However, in Figure 9, with the water-based flux present there is a time-dependent behaviour for these interrelated values, but at the end of the test the initial values were again recovered. There was a large change in capacitance as the temperature and humidity were introduced and this drove a large change in the reactance and impedance. There was a slower change in the resistance, revealing a longer term electrochemical behaviour, a fact also inferred from the impedance data.

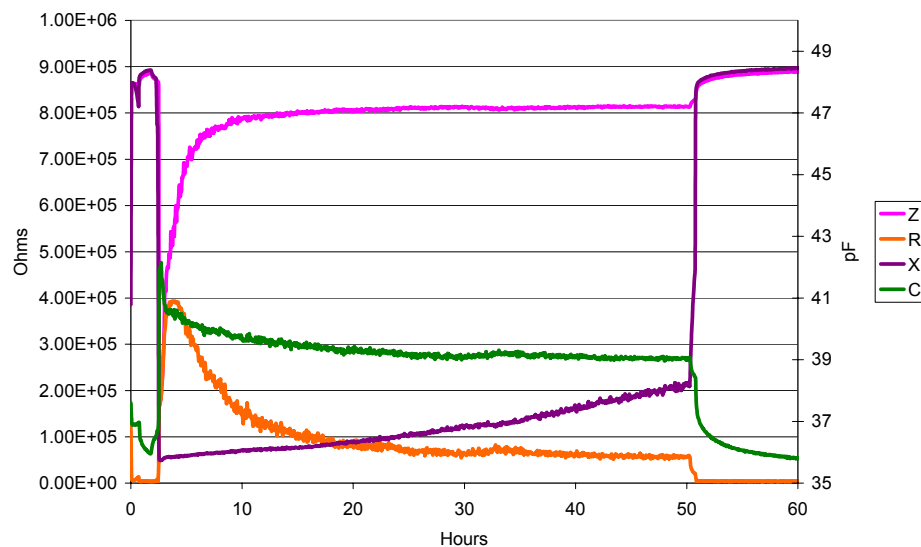


Figure 9. Plot of Z (Impedance), R (Resistance), X (Reactance) and C (Capacitance) against time for a water-based flux prepared comb

The effect of glutaric acid on the Z, X C and R parameters appears to be stronger than that of the water-based flux, with continuous changes in capacitance and resistance (see Figure 10). Hence neither impedance or reactance were constant throughout the test.

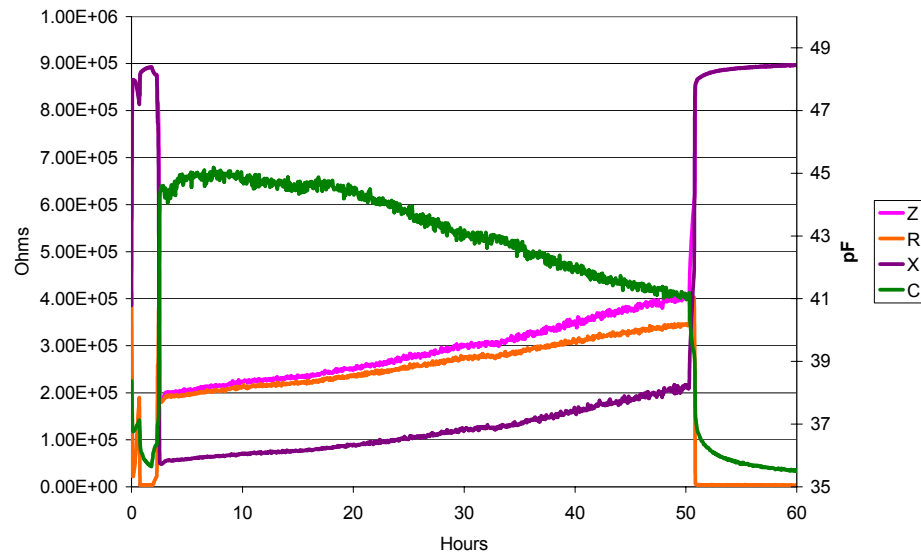


Figure 10. Plot of Z (Impedance), R (Resistance), X (Reactance) and C (Capacitance) against time for a glutaric acid-based flux prepared comb

Finally, the results for rosin flux are presented in Figure 11, and these show even less response than did the bare comb. This is consistent with the known ability of rosin to act as an encapsulant, and protect the comb from the effects of increasing the temperature and humidity.

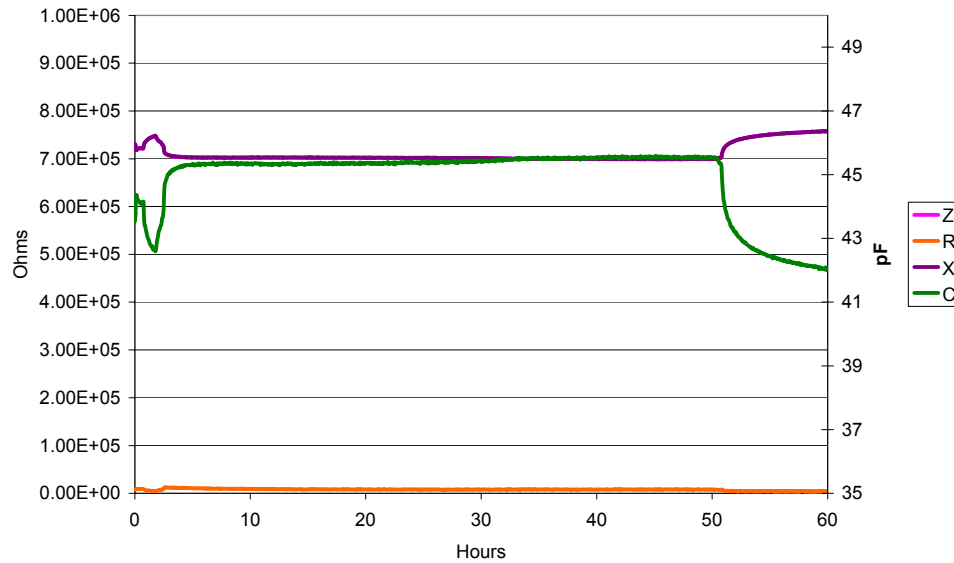


Figure 11. Plot of Z (Impedance), R (Resistance), X (Reactance) and C (Capacitance) against time for a rosin-based flux prepared comb

The differences in the results for these fluxes were quite marked, particularly when comparing the results of rosin with those of the organic acid fluxes. The rosin result is similar to the response in a DC measurement of SIR. The results are important in that they confirm that time-dependent behaviour can occur with an applied AC signal. The extent of the impedance changes was driven by both capacitance and resistance changes. The increase of temperature and humidity did have a big effect on the measured parameters.

3.3 FLUX COMPARISONS

This Section presents impedance and capacitance data collected with the impedance analyser for a range of fluxes, over a range of frequencies during 48 hr exposure tests to high temperature and humidity. The data are shown using 3 axis plots, see (Figure 12), allowing any changes in the AC electrical parameter over time throughout the test to be displayed for a range of frequencies. The introduction and removal of heat and humidity in the chamber at the beginning and end of each test, are included in the plots.

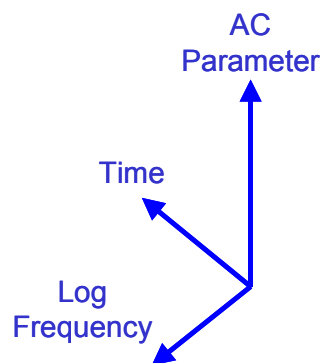


Figure 12. Key for 3D plots.

3.3.1 Impedance

Figure 13 presents impedance data for combs prepared with the rosin, water-based and glutaric acid-based fluxes, and a control blank comb.

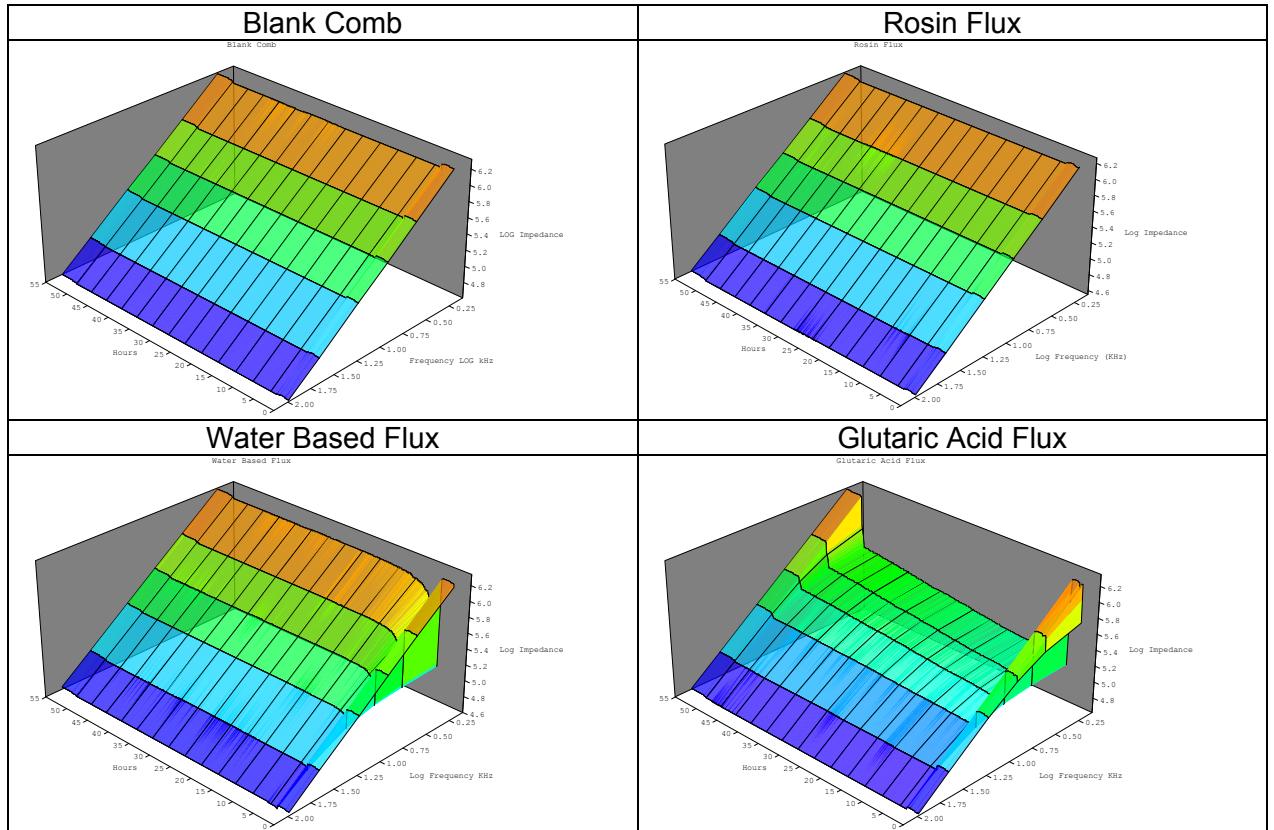


Figure 13. Impedance plots for a blank comb and three flux types.

The following points can be deduced from Figure 13:

- The impedance of the combs generally decreased with higher frequency
- For the blank and rosin combs the impedance was stable throughout the constant condition exposure
- For the water-based, and glutaric acid flux prepared combs there was a decrease in impedance as a result of the addition of humidity to the environment, which was more evident at lower frequencies
- This effect dissipated within 5-10 hours for the water-based comb, but much more slowly for the glutaric acid-based comb

The plot also displays the effect of adding and removing humidity from the chamber for all combs at all frequencies (at 5 and 50 hours)

3.3.2 Resistance (Real component of Impedance)

Figure 14 presents resistance data for combs prepared with the rosin, water-based and glutaric acid-based fluxes, and a control blank comb.

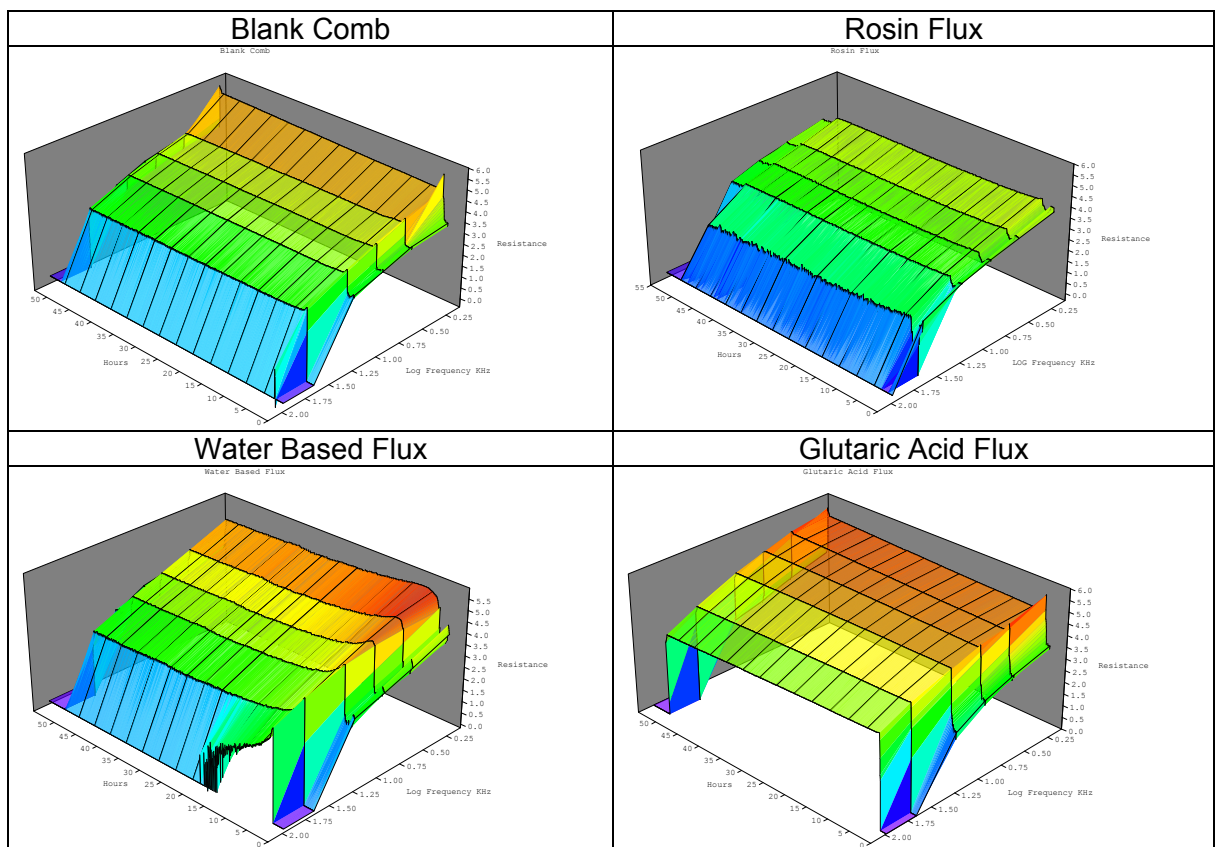


Figure 14. Resistance plots for a blank comb and three flux types.

The following points can be deduced from Figure 14.

- The resistance of the combs generally decreased with higher frequency
- For the glutaric acid comb the resistance remains relatively stable even at 100kHz at around $10^3 \Omega$.

- At low frequency there were only subtle changes in resistance during the constant condition period, but at 100kHz there was a dramatic drop in resistance (sometime negative) indicating the standard R-C model is breaking down
- The plots also display the effect of adding/removing humidity from the chamber for all combs and frequencies (at 5, 50 hrs), with glutaric acid exhibiting the least dependence.

3.3.3 Reactance

Figure 15 presents resistance data for combs prepared with the rosin, water-based and glutaric acid-based fluxes, and a control blank comb.

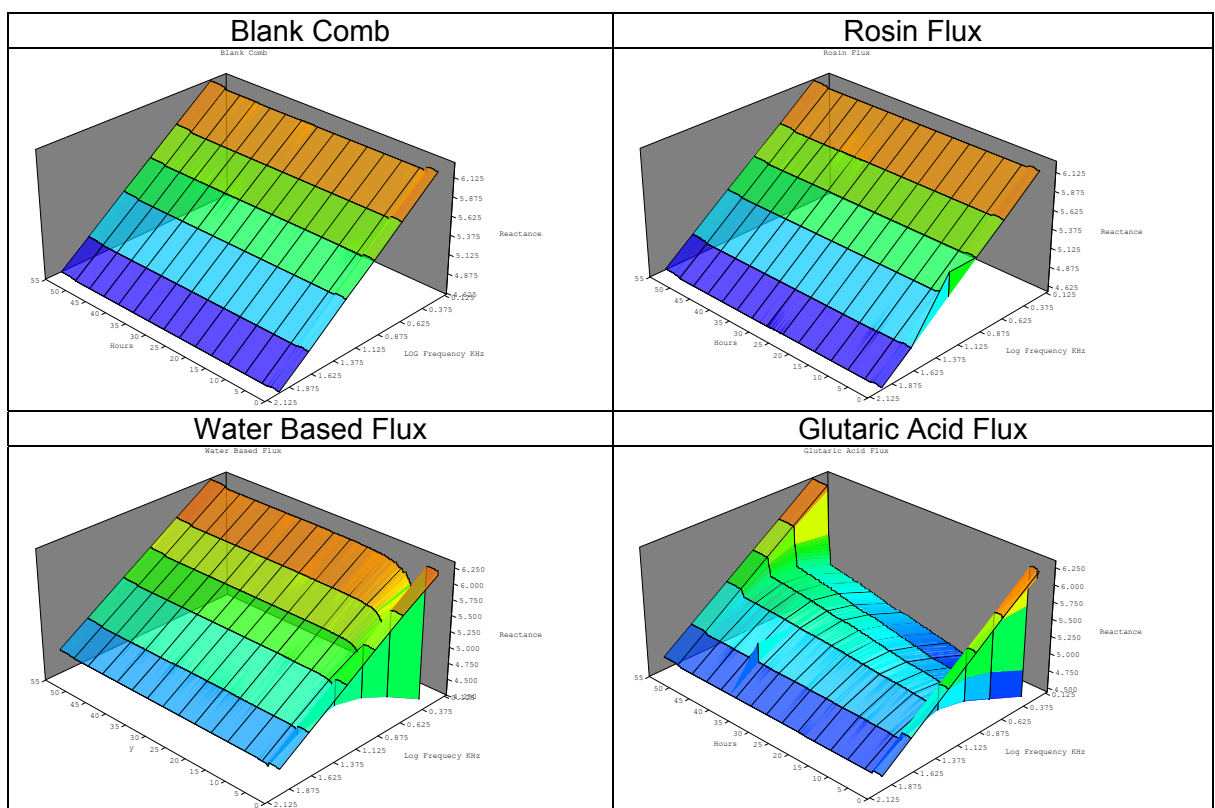


Figure 15. Reactance plots for a blank comb and three flux types.

The following points can be deduced from Figure 15

- All the statements relating to overall impedance (Section 3.3.1) can be applied since reactance dominates impedance in these cases
- Early in the test, at low frequency, the magnitude of reactance was actually lower than resistance (compare to Section 3.3.2), but at all other times reactance tended to dominate.

3.3.4 Capacitance

Figure 16 presents resistance data for combs prepared with the rosin, water-based and glutaric acid-based fluxes, and a control blank comb.

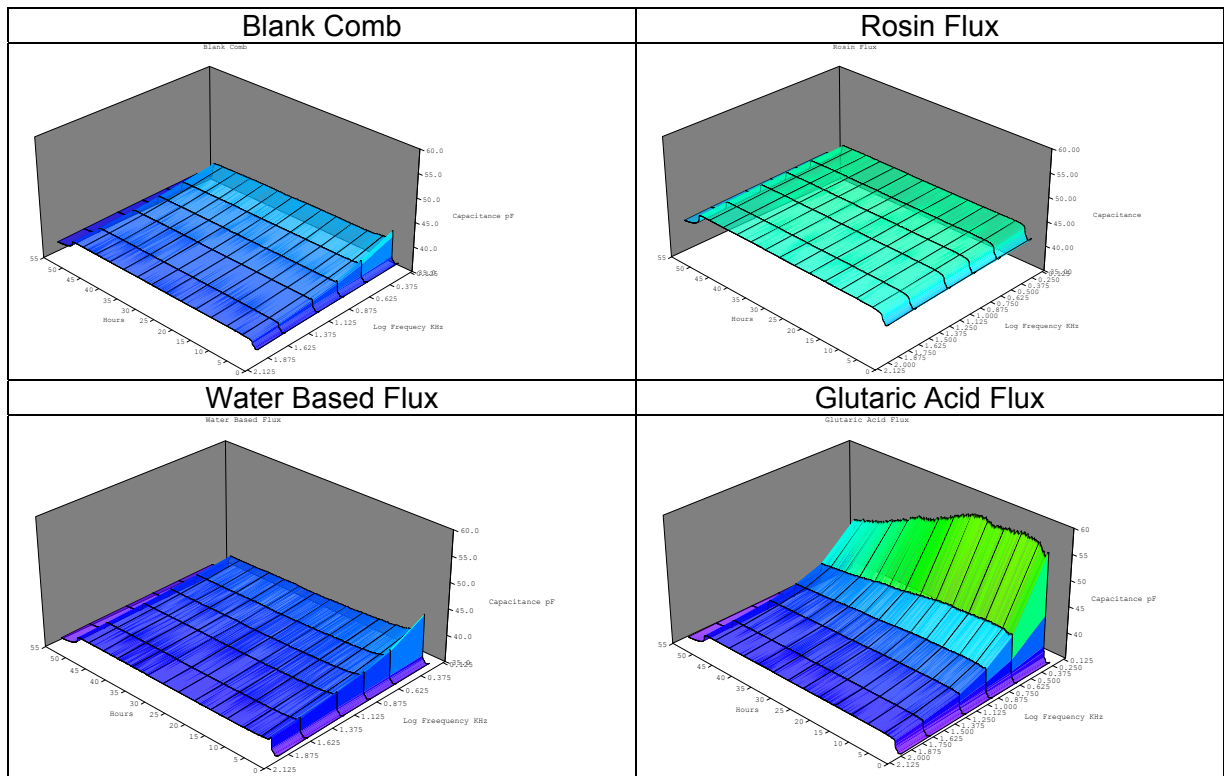


Figure 16. Capacitance plots for a blank comb and three flux types.

The following points can be deduced from Figure 16.

- The capacitance of the rosin fluxed comb was the most stable across all frequencies and with time. In an ideal system the capacitance will be independent of frequency.
- For other combs the capacitance was higher at lower frequencies, and was at a peak early in the test (i.e. low frequencies).
- The capacitance values for the glutaric acid exhibited an interesting increase with decreasing frequency. This effect was probably due to a dielectric effect, and as the frequency drops more (or different) molecules start to acquire a polar nature, changing the dielectric constant.
- Capacitance increased in the order rosin < blank < water-based < glutaric acid.

The effect of glutaric acid on changing the capacitance is an interesting effect which is easily detectable and could have significant implications for circuit performance.

3.4 TEST METHOD REPEATABILITY

Test method repeatability was investigated in those cases where the effect on impedance parameters was greatest, i.e. at 2kHz, and using the two fluxes whose behaviours most varied with frequency, water-based weak organic acid flux (WOA), and the glutaric acid (GA) in IPA.

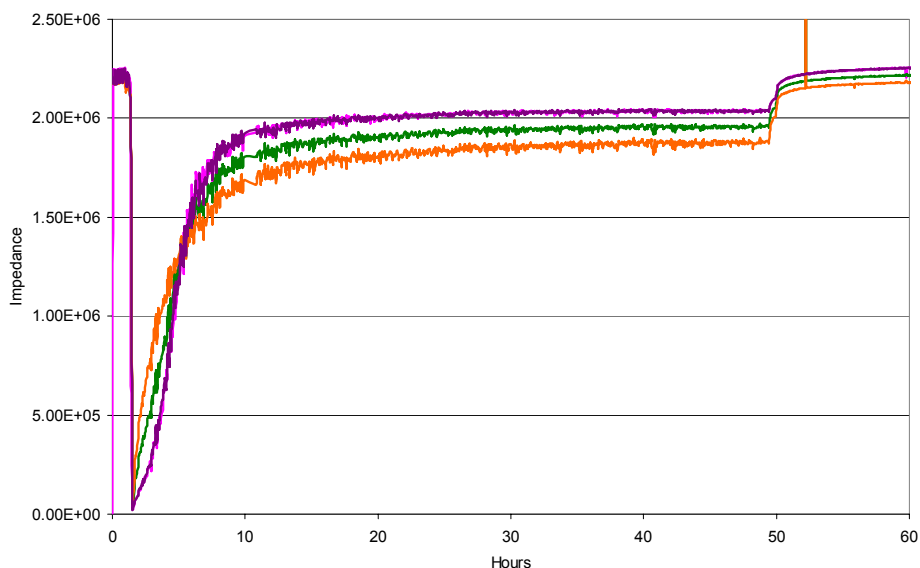


Figure 17. Impedance data for four repeats of water-based fluxed combs (2kHz).

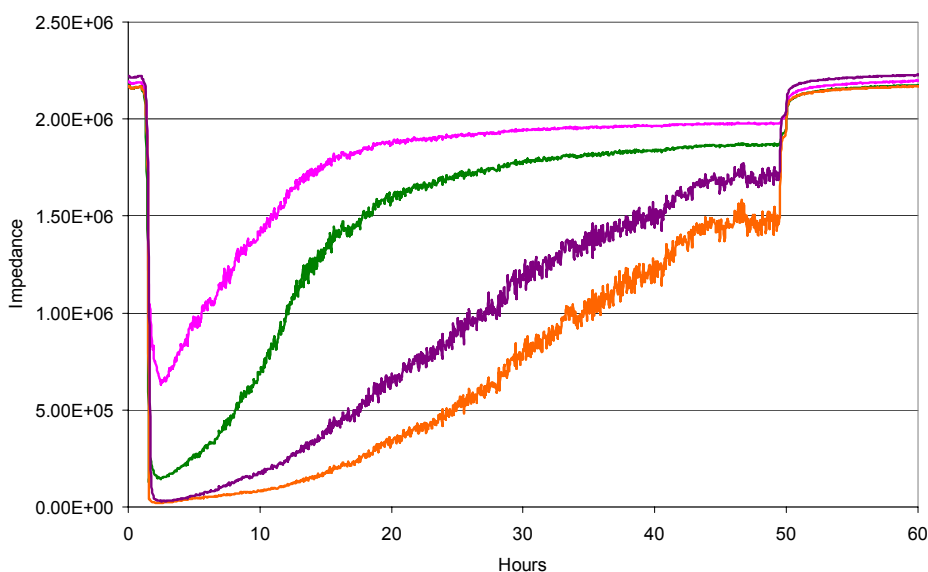


Figure 18. Impedance data for four repeats of glutaric acid fluxed combs (2kHz).

Comparing the impedance results when using these two fluxes (see Figures 17 and 18), emphasises that the repeatability differed markedly. For the WOA flux the repeatability was comparable to that expected for SIR measurements, but for the GA flux there was a marked variability. From the WOA results it might be reasonable to conclude that the experimental approach is reasonably robust. A possible variable between the two sets of results lies in the difficulty of achieving an even layer of flux over the comb, this may well be dependent on the spreading of the flux. In dispensing the 50 μ l of flux from the micropipette, a series of drops was placed on the comb and the flux was expected to coalesce to form a uniform film. Visual inspection can be used to assess if this does not happen at all, but when a film does form it is not possible to determine the level of

evenness of the flux film. The GA flux appeared to spread out evenly, but it is possible that at the molecular level agglomeration had taken place leading to significant variability, and hence to an on effect on impedance. This variability has not been observed in SIR results for GA. However, conduction occurs by different mechanisms and clearly capacitance is significant in impedance measurements. Analysis of the capacitance values showed that higher values were obtained for the GA flux when compared to the WOA flux. A further question on these data is that the impedance analyser only applies signal when interrogating a comb through the switch system. Hence when it is not being measured, the comb is open circuit. Future work will study the effects of applying the voltage continuously.

3.5 ON-BOARD CIRCUIT

The on-board circuit was evaluated with two fluxes, and the results are presented in Figure 19.

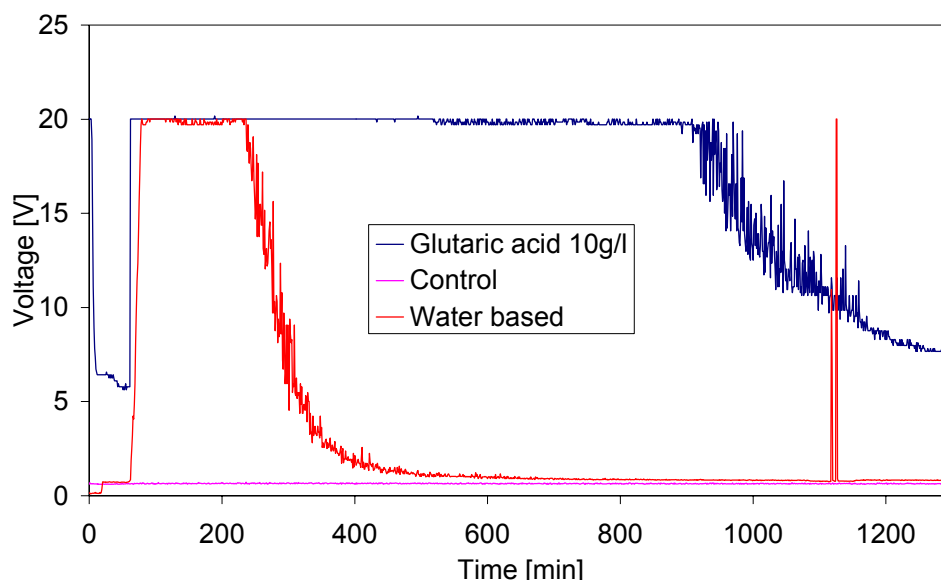


Figure 19: Response from on-board circuit at 2kHz for glutaric acid and water-based fluxes

These results appear quite different to the data from the impedance analyser discussed previously. The circuit produces an AC voltage (peak to peak value) that is related to R1 in Figure 5. A low voltage corresponds to a high impedance, and vice-versa. The circuit response clearly lacked dynamic range but the response from this circuit did follow that obtained from the impedance analyser. For future work, either the set up for the circuit needs improving to match the loading introduced by the presence of the flux, or a more fundamental change in the design is required.

4 DISCUSSION

These exploratory measurements set out to establish the conditions under which impedance measurements can be taken with an interdigitated comb, similar to that used in conventional SIR testing with DC voltages. Two approaches were developed here, the first was to use a very versatile impedance analyser and the second was to introduce a small circuit adjacent to the comb pattern that a DC level proportional to the impedance.

Testing was carried out in a humidity chamber using three fluxes deposited on the comb pattern. These flux chemistries produced difference changes in the AC parameters tested, and hence it was possible to differentiate between the different fluxes. The encapsulating properties of the rosin flux were minimal. Changes in impedance were observed, but the glutaric acid flux produced much larger changes in impedance. That impedance changes occurred at all is significant as there is scepticism in some parts of the industry that AC conditions will not produce any changes in impedance.

Humidity had a significant effect on impedance values, even on the bare comb, but more so when flux residues were present; in dry conditions minimal effects of flux were measured. Breathing on the comb at ambient produced significant changes in impedance. Although large impedance changes were observed with the WOA and GA fluxes, the blank, and particularly the rosin-fluxed boards, produced stable values.

Impedance values generally dropped with increasing frequency and hence the greatest sensitivity to changes on the combs was observed at low frequencies. In the experimental set up here the lowest stable frequency was 2kHz. Hence more detailed studies were undertaken around this frequency.

Reactance is the dominant contributor to overall impedance in these tests (and this is largely influenced by capacitance), the exception being early in the test, at low frequency. This was the point at which humidity was introduced into the chamber, so it is possible that slight condensation was occurring, or perhaps moisture was on the surface rather than in the bulk of the sample.

The repeatability of the test needs to be improved, possibly in the pre-treatment of the samples in terms of flux loading and distribution and heating. Volatilisation or ageing of the flux may also contribute to changing impedance values. Difference in pre-treatment heating may result in variation in flux ageing on the samples.

In the experimental set up used here, the switch bridge did not maintain the applied bias across all four channels when the impedance analyser was interrogating a specific channel. This means that signal source was only applied periodically. Further work should investigate connecting the impedance analyser directly to a comb pattern.

Although this exploratory work has shown that impedance effects were observed, further work is required to characterise the conditions for testing with the impedance analyser, and the on-board circuit. Frequency and supply voltage need to be further explored, and parameter values within the on-board circuit need to be optimised to improve the robustness of the approach.

5 CONCLUSIONS

- This work has highlighted the promise of impedance properties as a tool for differentiating the effects of flux and their potential effects on AC circuits
- The use of an applied AC bias to SIR style combs has shown that impedance changes with frequency do occur. The effects of flux residues have been shown to be different for generic flux types, and very dependent on humidity levels.
- Whilst improvements in repeatability are necessary the technique warrants further study with the aim of a more product representative SIR technique.
- Future work should include further development of the on-board circuit approach, as an alternative approach to using impedance analysers. In addition, the effects of the switching system may need to be understood in more detail perhaps by the direct and permanent connection of the impedance analyser to single combs so that AC conditions are applied throughout testing.

6 REFERENCES

- [1] HUNT, C.P. *Development of Surface Insulation Resistance Measurements For Electronic Assemblies*. NPL Report MATC(A)70, October 2001.

7 ACKNOWLEDGEMENTS

The work was carried out as part of a project in the Materials Processing Metrology Programme of the UK Department of Trade and Industry.