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Development of Surface Insulation Resistance Measurements For Electronic Assemblies

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by

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ABSTRACT

This report summarises the work of a team from National Physical Laboratory (NPL) of the UK, with the following three partners National Microelectronics Research Centre (NMRC) of Ireland, Siemens AG of Germany and Lares Cozzi (LC) of Italy. The authors from NPL are Christopher Hunt & Ling Zou, from NMRC there was John Barton, from Siemens there were Klaus-Peter Galuschki & Hartmut Meier, and from LC there was Ed Seagraves. The project took as the starting point the international standard for SIR testing that existed at the time and developed this to reflect modern soldering processes and instrumentation capability. The work showed very clearly that current methods can frequently falsely predict pass/fail criteria for current fine pitch and fluxes. This work recommends a new SIR pattern and test strategy that reflects today's assembly processes, which is viewed by the authors as being essential if the reliability is going to be correctly identified. Siemens are using the results from this project to qualify all their fluxes.

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1. Introduction

The project took as its starting point the existing ISO standard 9455 part 17 on surface insulation resistance (SIR) measurement. This standard reflects industry practice and instrument capability of the 1970's. Both of these have moved on dramatically since then and this project develops a test method that meets today's electronic assembly standards and can be accomplished with currently available instrumentation. The relevant advances in the assembly industry impacting on the SIR technique are the reduction in pitch of component terminations and a radical change in the flux chemistry used in mass soldering.

In the 1970's flux chemistry was almost exclusively based on a rosin base in a solvent carrier and was always cleaned following soldering. The Montreal Protocol and its updates of the early 1990's, which banned chlorofluorocarbons (CFC), had a major impact on the industry as CFC where used extensively as the cleaning platform for a range of cleaning chemistries. This ban had the surprising result of high volume consumer electronics being assembled without a cleaning stage. This was made possible by the introduction of the so-called "no-clean" and "low residue" fluxes. These fluxes are formulated to leave no harmful residues after soldering, if the correct process is followed. These residues are therefore intrinsically different to earlier residues.

Component termination pitch is now much finer than 30 years ago. Then components tended to be based on wire terminations that were inserted through the board and wave soldered. Today terminations are short flat leads, or even leadless, and are surface mounted using solder paste. Again this change in process as had its effect on the flux residues left on the board following processing.

The other major advance relevant to SIR testing is the improvements in instrumentation. Like everything else today the increased computational power that is available cheaply has had its effect, and coupled with the advances in electronic performance has meant that SIR testers of today are very different to those of the 1970's. The current sensitivity has been improved, and this has been coupled with multiplexing. Hence today's instrument can measure to better sensitivity across a number of channels and repeat this at regular intervals, which can be as frequently as 5 minutes. In fact the next generation of instruments may monitor continuously on a minimum of 128 channels.

The test parameters of the present standard therefore represent old technology, and the parameters of that test are 0.9mm pitch, bias with 50V and measure with -100V, and take 3 measurements during a seven day test. We shall see from this project such an approach is woefully inadequate and produces erroneous data, and the test method that is developed here will use representative parameters of today's industry.

1.1 Project Consortium

The project was supported by the European Commission under Contract Number: SMT4-CR97-2155, and lead by the National Physical Laboratory (NPL) of the UK, with the following three partners National Microelectronics Research Centre (NMRC) of Ireland, Siemens AG of Germany and Lares Cozzi (LC) of Italy. NPL and NMRC were involved in extensive SIR testing; Siemens similarly were involved in much SIR testing and also implemented the production of pseudo industrial fluxed samples for SIR testing. Lares produced all the substrates for the project.

2. Experimental Project Plan For WP2, WP3 & WP4

2.1 The SIR Test Method

The SIR technique was developed to meet the real issue of predicting reliability on circuit boards and assemblies. SIR testing has been used by the industry for more than 30 years and is now inappropriate in a number of aspects. The technique is very simple in concept, measuring the resistance across two interdigitated combs while subjecting the test vehicle to a hot/humid environment in order to accelerate the test. A schematic of a comb is shown in Figure 1. While the concept is simple, the implementation of a successful test set up is not trivial and there are many pitfalls for the unwary. Initially the test was implemented in a very simple fashion with single instrument capable of measuring fractions of a microamp. The operator had to measure a single pattern individually. Today with computer control and improved instrumentation SIR values can be read automatically from up to 256 patterns with sensitivities of a nanoamp, or better.

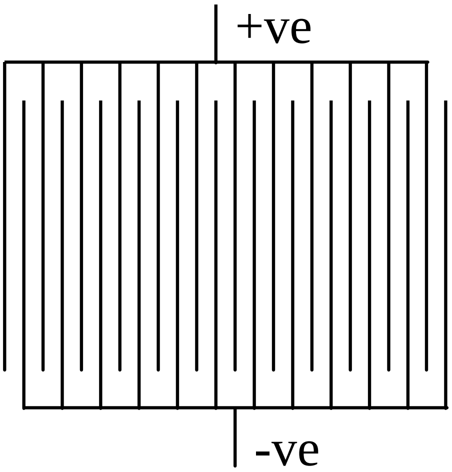

Figure 1: Schematic of SIR pattern

There are number of key issues that are relevant to a successful SIR test, and these are studied in the work here. As discussed above the spacing between the coomb fingers, and known as the gap, is a factor expected to have a major influence on the SIR. The width of each of the coomb fingers, known as the track, along with the gap, combine to form the pitch of the pattern. The bias and the pitch will interact to influence the SIR response. The environment in which the test is implemented is very important. The temperature and humidity both influence the adsorbed water layer thickness, which greatly influences surface conduction across the board surface. The temperature also influences the evaporation of flux residues, and if this too high the harmful influence of the residue will be lost. Another crucial factor in conducting a SIR test is the flux preparation procedure. The heating cycle seen by the flux on the SIR pattern will affect the amount of residue and its composition. All the above factors must be considered if the SIR test is to produce accurate predictive results as to the suitability of any flux. As mentioned earlier the frequency of measurement is an important issue. This is because corrosion products can be deposited at the cathode and start to grow back towards the anode, forming in a dendritic structure. An example is shown in Figure 2. These dendrites are fragile and can grow quite quickly, within the space of an hour.

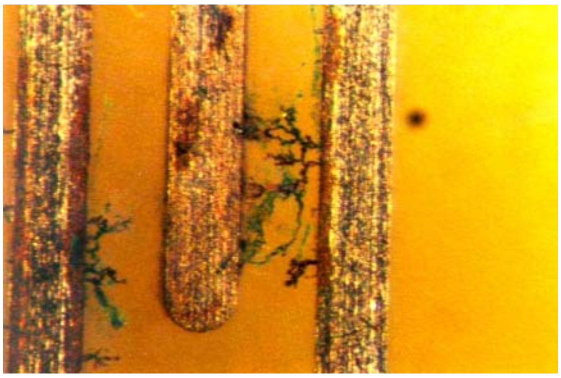

Figure 2: Examples of dendrite growth

2.2 Project Scope

In the first stage of this project the details for the initial phase of experimental work was worked out. The aim of the first phase was to map out under a set of controlled experiments the SIR response to critical parameters such as electrical bias, temperature and humidity and the effect of flux preparation and heating cycle. To accomplish this, the work was divided between the respective partners, NPL, NMRC, and Siemens.

2.3 Selection of Experimental Parameters

A number of experimental parameters were considered and their values selected and these are given below:

Test temperature: 40°C, 65°C, 85°C. This encompasses the current 40 and 85°C and adds the mid temperature of 65°C.

Test humidity: 65%, 85%, 93%. Similarly 85 and 93% are in current standards and 65% was added as an extra parameter.

Field gradient: 10, 50, 100, 200 and 400 V/mm. It was decided to use the concept of field gradient

Flux: Five fluxes, Flux A, Flux B, Flux C, Flux D and Flux E were selected, all activated with either halide or organic acids

Board surface finish: Hot air solder levelled (HASL), electroless nickel with gold (NiAu), electroless nickel with a palladium finish (Pd) and a bare Cu board. These were selected as the most common in the industry today, and being relevant to SIR testing.

Pattern: Four patterns were used with varying track and gap pattern. These were based on the 100/100, 200/200, 350/350 and 400/500 µm track and gap. LC designed and built the necessary boards for testing. The 4 designs are shown in Figure 3, with each board comprising four SIR patterns.

Soldering heat effect: Various soldering methods were assessed as to the effect of the heating cycle on the flux residues. The processes used were reflow with infa-red heating (IR) with air or nitrogen atmospheres (the nitrogen had 100ppm oxygen), reflow with forced convection using air and nitrogen atmospheres, vapour phase heating and finally wave soldering.

The fluxes used for WP2, 3 & 4 are given in Table 1. These fluxes are not industrial available fluxes but are formulated to the relevant ISO standard and represent typical industrial fluxes.

To ensure identical experimental method a detailed procedure was developed that specified all the steps in preparing the fluxed SIR test boards. The approach for the SIR testing itself was also agreed. Common values were set for parameters that were not part of an experiment.

With the strategy and test conditions in place work packages 2, 3 and 4 commenced.

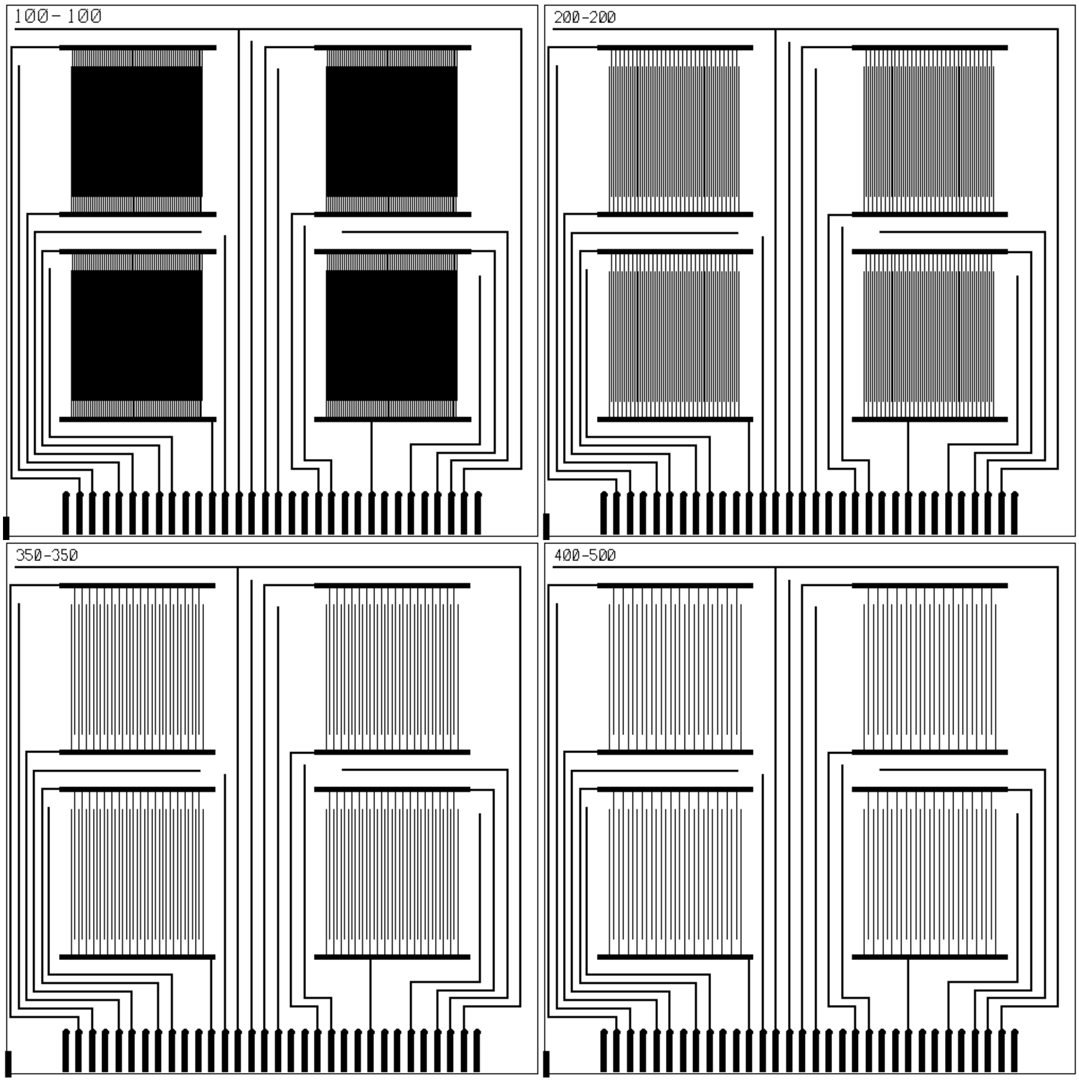

Figure 3: The four board designs used in the first phase of the project

Table 1: Flux Description

Code	Type	Description
A	ISO1.1.2 ; FSW26	Based on natural with activators containing organic halogens. Alcohol based.
B	ISO 2.2.2 ; FSW25	Rosin free with activators containing organic halogens (also added chloride), and polyglycols. Alcohol based.
C	ISO1.2.3 ; FSW33	Synthetic rosin with activators containing organic halogens. Water based.
D	ISO2.1.3 ; FSW23	Rosin free with activators containing organic halogens, and polyglycols. Alcohol based.
E	ISO2.3.3 ; FSW34	Based on halogen free organic acids with natural resins. Alcohol based.

3. Effects of Electrical Biasing on SIR Testing (WP2)

3.1 Introduction

Achieving high reliability is increasing the key issue in today’s electronics manufacture, now that very low defect levels in manufacture are obtainable. Residual contamination remaining on the board that may cause electro-migration issues in service is a concern to high quality manufacturing. The Surface Insulation Resistance (SIR) technique has been widely used to assess the effect of contaminants on the reliability of assemblies. The SIR is a measurement of an electrochemical process between two metallic conductors on a substrate surface. The SIR value is dependent on many test parameters: electrical field strength, the SIR test pattern, board or substrate finish and test environment. The current SIR method described in international standards is based on 30 year old technology. Instrumentation capability, assembly and flux technology, have significantly improved. The time is ripe to update the method and meet the needs of today’s electronic industry.

The work presented here investigates how SIR is affected by field strength in combination with different SIR patterns, fluxes and board finishes. From this study's recommendations for appropriate field strength, track and gap, board finish, sampling rate and test length for the SIR measurement are made.

3.2 Experiment

A wide experimental scope was defined to encompass five fluxes, five field strengths, four SIR patterns, and four board finishes. This gave a total of 400 individual experimental runs, which were reduced to a manageable number by a design of experiment approach. Hence an experimental matrix of 50 combinations of different field strengths, patterns, fluxes and board finishes were defined. All the tests were carried out according to the SIR test board preparation and measuring procedure given in the final report. For these measurements the test temperature and humidity was 65°C/85%.

A further phase of work was carried out just using Flux B and the AuNi finished boards, but with the same range of field strengths and all the SIR test patterns using the Cu finish and the 350/350 and 400/500 Au finished boards.

3.3 Test Results and Discussion

3.3.1 Results from the Design of Experiment

Most SIR values slowly increased with time, and stabilised within three days. Any occurrence of dendrites had also initiated within this period for all test runs. This provides evidence that the test period could be shortened from seven days to three days.

The SIR values for test boards with flux C under 200 and 400 V/mm field strength could not be averaged, due to dendrite formation as seen in Figure 4 and Figure 5. Figure 6 show dendrites that have formed 100 V/mm. Dendrites only formed with the water based flux C. Interestingly dendrite formation was observed at 200 V/mm with the 200/200 track & gap boards, but not with the 400/500 at the same field strength (and hence a high bias). This very important result indicates that the track and gap of the SIR pattern are critical in terms of the measurement response, with the clear implication that the SIR pattern should be representative of the manufactured circuit. Dendrite formation is a very important failure mechanism and can lead to catastrophic failure of circuits.

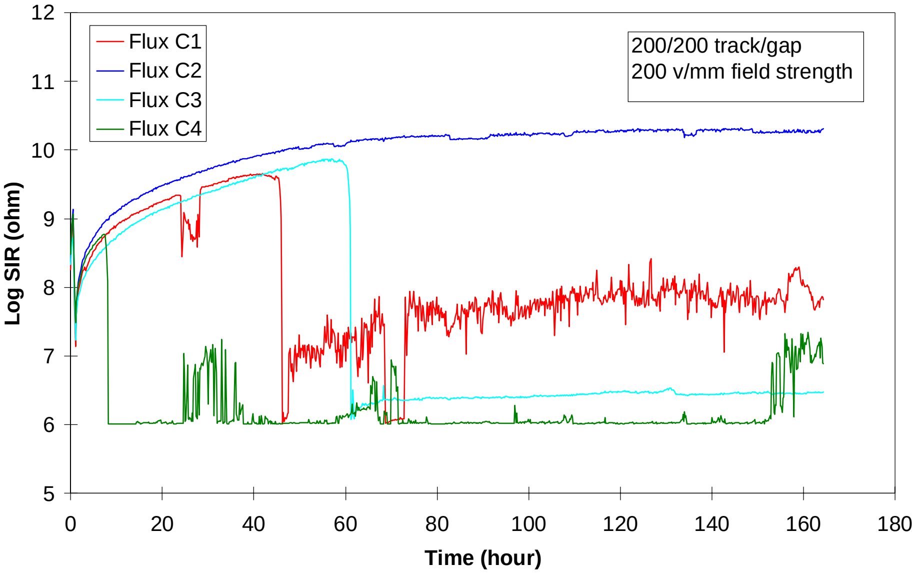

Figure 4: The SIR response with time for dendrite formation (200 V/mm)

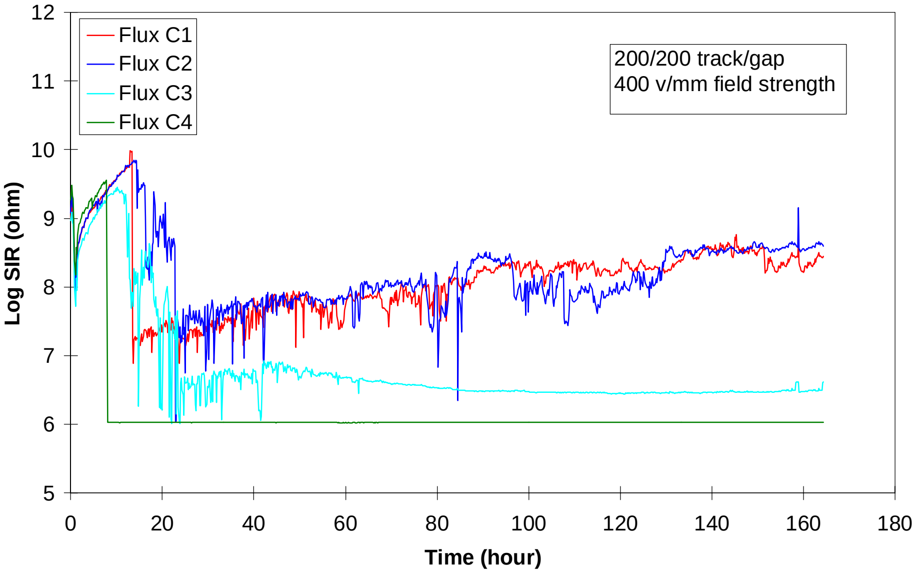

Figure 5: The SIR response with time for dendrite formation (400 V/mm)

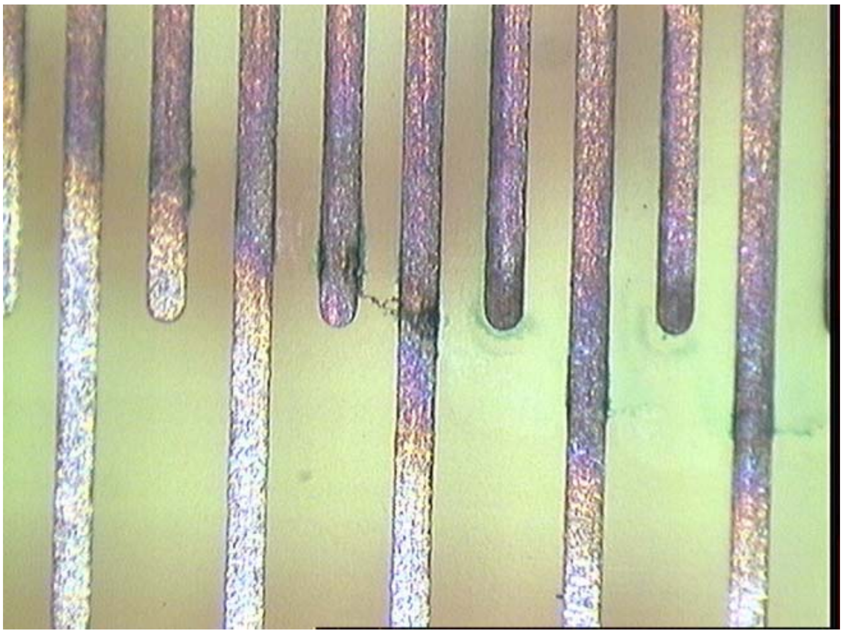

Figure 6: The dendrites formed on 200/200 Cu boards with flux C under 100 V/mm field strength

3.3.2 Discussion of the DOE Results

Figure 7 shows an analysis of the means and medians of the final SIR values from the statistical analysis. It is clear from Figure 7 all the parameters influence the SIR measurement.

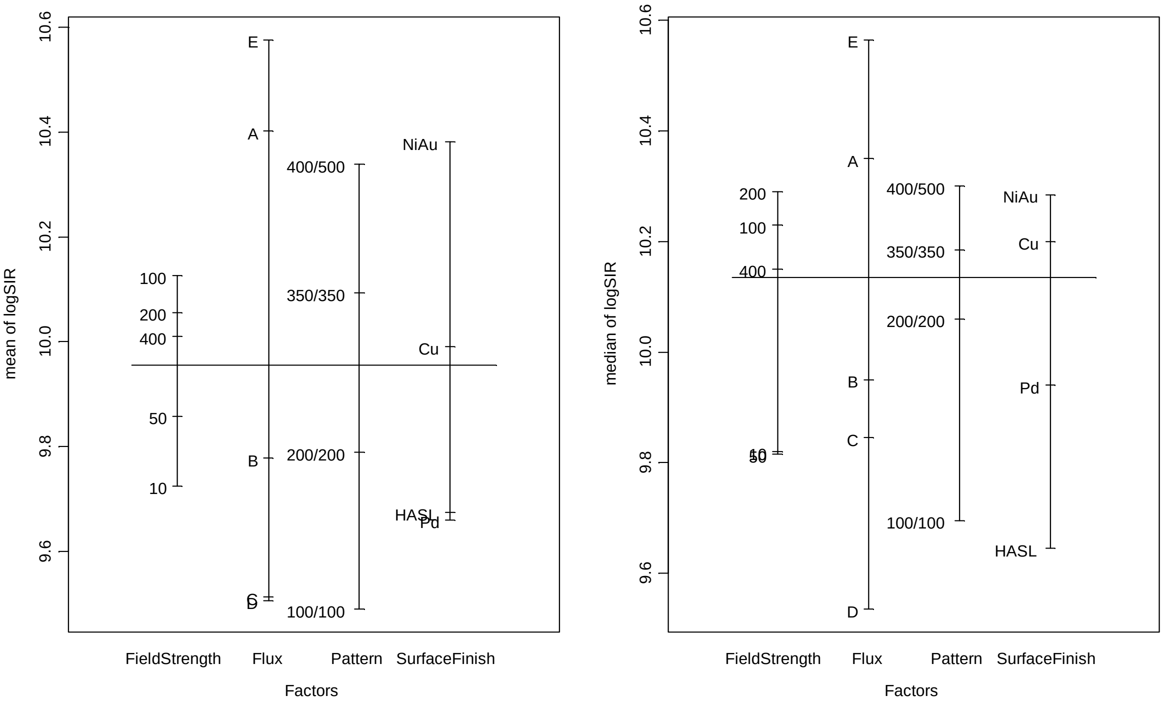

Figure 7: Analysis of Means and of Medians for the final Log SIR values

3.3.2.1 Surface Finish

Surface finish clearly has an effect, and the HASL finish has a significantly poorer performance. The low SIR of the HASL finish is due to the manufacturing process, which impregnates the board with polglcol flux residues. The Cu and Au finishes offer the highest SIR, and Au is probably the preferred finish as it is more tarnish resistant.

3.3.2.2 Flux

Flux clearly has a strong effect, and reflects the wide range in flux performance that is available today. Generally the higher activity fluxes will leave more harmful residues and hence lower SIR.

3.3.2.3 SIR Pattern

Here we see as the pattern becomes finer the SIR drops. This trend is expected since the finer patterns effectively have longer electrodes and so a lower SIR is expected.

3.3.2.4 Field Strength

This shows a more complicated behaviour with the SIR rising with field strength and then dropping again at the very highest field strength values. If the flux residues where simply resistive we would expect a constant SIR with changing field strength. The reduction in SIR for the highest filed strength reflects the occurrence of dendrites during the test.

3.3.3 Summary of the DOE Results.

This work has shown that in the absence of dendrites the SIR is not simply resistive. It behaves in a complex way, which is dependent on the field strength and the SIR pattern itself. Certain fluxes are more likely to cause dendrites and corrosion, than others and this is seen in the SIR response. The choice of board finish is important, and copper and AuNi are the preferred choice. In terms of the robustness of the finish during the flux preparation procedure, the AuNi is superior over the copper.

3.3.4 SIR Response with Field Strength and Pattern

The main issue to come out of section 3.3.1 is that the pattern and the field strength should be studied in more detail, so that the response can be more clearly identified. For this next phase the complete range of SIR pattern and field strength were studied using the AuNi and Cu boards and Flux B. The results for the Cu boards are shown in Figure 8 and Figure 9 for the final SIR values at 48 hours against field strength and different SIR patterns.

The results in Figure 8 show the effect of increasing field strength gradient as the pattern pitch diminishes. This is very significant in terms of today’s circuitry where fine pitch and low voltages are becoming more prevalent. The current international standards for SIR testing use the 400/500 pattern and 100 V/mm field gradient, this would over estimate the SIR by 1.5 log ohm decade, when comparing with the 10 V/mm and the 100/100 pattern. This is a very large margin and would be extremely significant in predicting reliability.

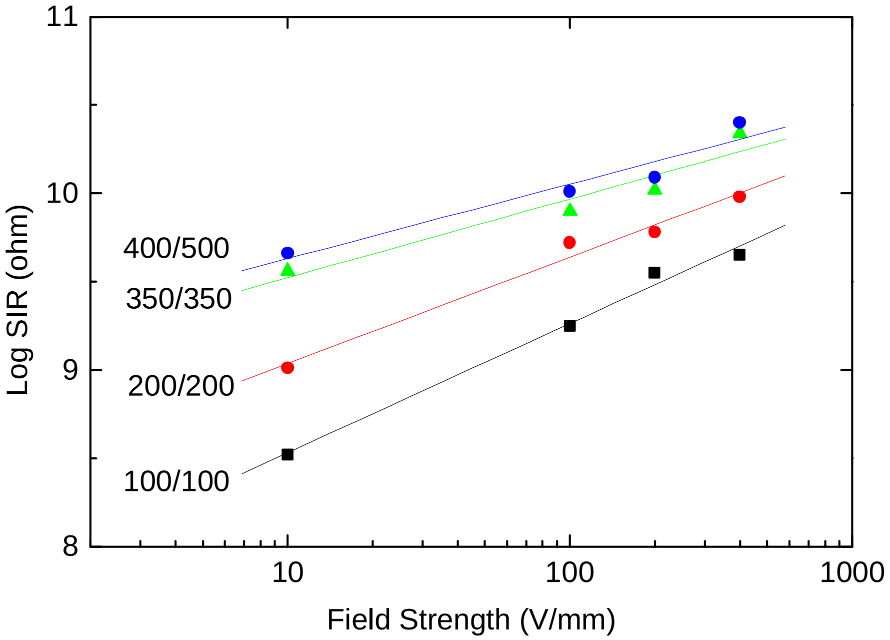

Figure 8: The SIR value with field strength for different patterns of Cu boards

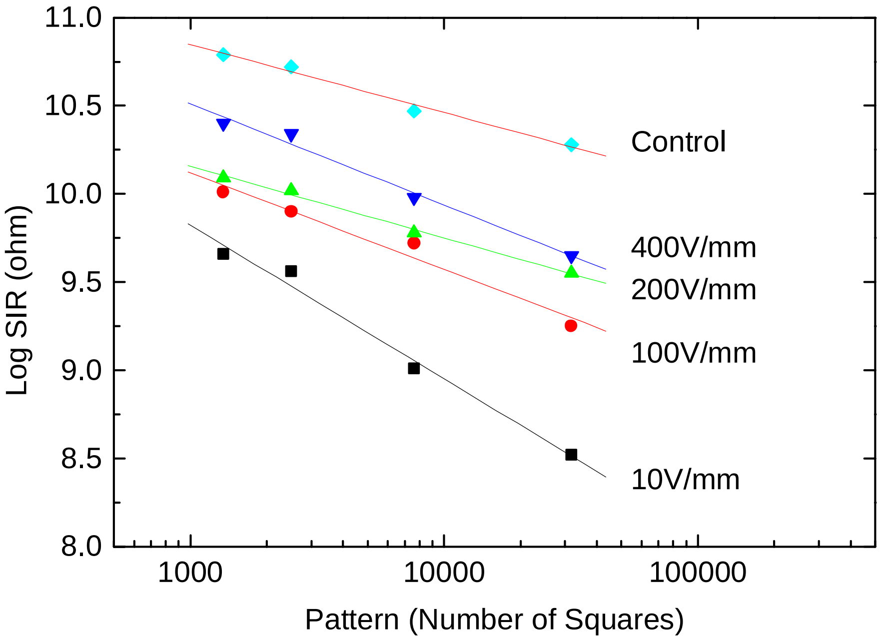

Figure 9: The SIR value with patterns for different field strength of Cu boards

In Figure 9 the SIR values are replotted as a function of test pattern. The slope of the trendlines would be -1 if the SIR was simply resistive. It is clear from this figure that as the pattern pitch decreases the SIR value is becoming more resistive, i.e. the slope decrease closer to -1. At 100 V/mm and higher filed strengths the gradient is typically around 0.5. The change in SIR is due only to the change in available length of the electrodes in the SIR pattern and not the number of squares. This conjecture is consistent with a highly resistive barrier being created at the electrodes, and dominating the resistance across the central part of the gap. The 10 V/mm data is different, and this maybe due to the absence, or limited degree, of a barrier forming. Hence, at this field strength the resistance is better correlated with the gap value.

3.4 Conclusions and recommendations for draft SIR method

3.4.1 Field strength

Surface insulation resistance (SIR) is strongly dependent on the test field strength. Generally, the SIR increases with field strength. Increasing the field strength also increase the risk of dendrite formation. Hence, high field strength is bad, and usually unrealistic. Therefore, in using SIR measurements to assess the reliability of electronic circuits, the test field strength should be close to the realistic working field strength.

3.4.2 Test pattern

The test board pattern affects the SIR. The concept of ohm squares cannot be used, it does not permit a useful comparison of SIR patterns. The SIR response to various patters can be useful in qualifying behaviour, and hence reliability. However, in formulating a test method the test pattern should be fixed. This pattern should be representative of fine pitch. We recommend that a pattern with a 400µm track and a 200µm gap be used. This is a fine pitch pattern and hence will be representative of today’s industry. The track is 400µm to facilitate stencil printing onto the pattern, a finer track maybe too demanding in general for printing. The fine pitch or small gap will also be more efficient in capturing potential problems with dendrite formation.

3.4.3 Board finish

The board finish dramatically affects the SIR values. The HASL finished boards always give the lower SIR value due to the residual contamination from the manufacturing process. This was in spite of all the different finishes boards going though the same cleaning process prior to fluxing. Hence, the HASL board should not be used in SIR flux qualification. The AuNi or copper finish is recommended for the SIR flux qualification. (AuNi is preferred as it is more robust during flux preparation).

3.4.4 Test time and sampling frequency

The test results show that the SIR values for all the combinations of different field strengths, track/gaps, fluxes and board finishes became stable after 72 hours measurement. When dendrite formation did occurred, it always started before 72 hours. The data after 72 hours did not provide any further useful information. The 72 hours test period is recommended, but this is conditional on frequent monitoring of the data, at least every 10 minutes and preferably every 10 minutes. Frequent monitoring should be mandatory.

4. Effects of Temperature and Humidity (WP3)

4.1 Test Design

The aim of WP3 was to determine the influence of temperature and humidity ambient conditions on the SIR performance of a range of board materials, SIR test structures and fluxes. There were a large number of experimental variables to be included in this examination of the influences of environment on SIR. These variables and conditions were as follows:

Table 2: Experimental Parameters and levels

Temperature/°C	Humidity/%	Flux	Surface Finish
40	65	Flux A	HAL
65	85	Flux B	NiAu
85	93	Flux C	Pd
--	--	Flux D	Cu
		Flux E	
--	--	Clean (Or Control)	--

These factors and levels generated around 216 runs so design of experiment (DOE) techniques were used to reduce the number of experimental runs to 47. Additional runs were added to the matrix as follows:

- A series of runs were included at 85°C/85%RH as this is the industry standard
- The 65°C/93%RH sequence was changed to 65°C/85%RH to reflect the common conditions used by the other partners in their work packages and more runs were added.

This matrix gives 6 temperature/humidity runs and it was decided to repeat each run once to give a total of 12 runs. Two test boards and one control board were measured in each run. The track/gap spacing and field strength were kept constant at 350/350μm and 100V/mm respectively.

4.2 Test Procedure

The boards were cleaned using IPA and DI water and dried in an oven. Initial SIR measurements were taken and then the boards were placed in the chamber for 16 hours at 65°C/85%. The boards were then fluxed and dried at 100°C for 5 minutes. The boards were then placed in the test fixture for the start of environmental testing. On commencement of testing, measurements were taken every 10 minutes. For temperatures below 65°C, i.e. the 40°C/65%RH and 40°C/93%RH runs, the test duration was 168 hours while for the second set of all the other combinations a test duration of 72 hours was used. Observations from the first set of test runs, which were of duration 168 hours, as well as similar observations from

NPL and Siemens indicated that test results stabilised within 72 hours. Dendritic formation also initiated within this time period. The results were presented as an average value from two boards for fluxed boards and one board for control (clean) boards. Hence, the results are an average from 8 and 4 comb patterns respectively. A representative SIR graph is shown in Figure 10. Control board values are also included.

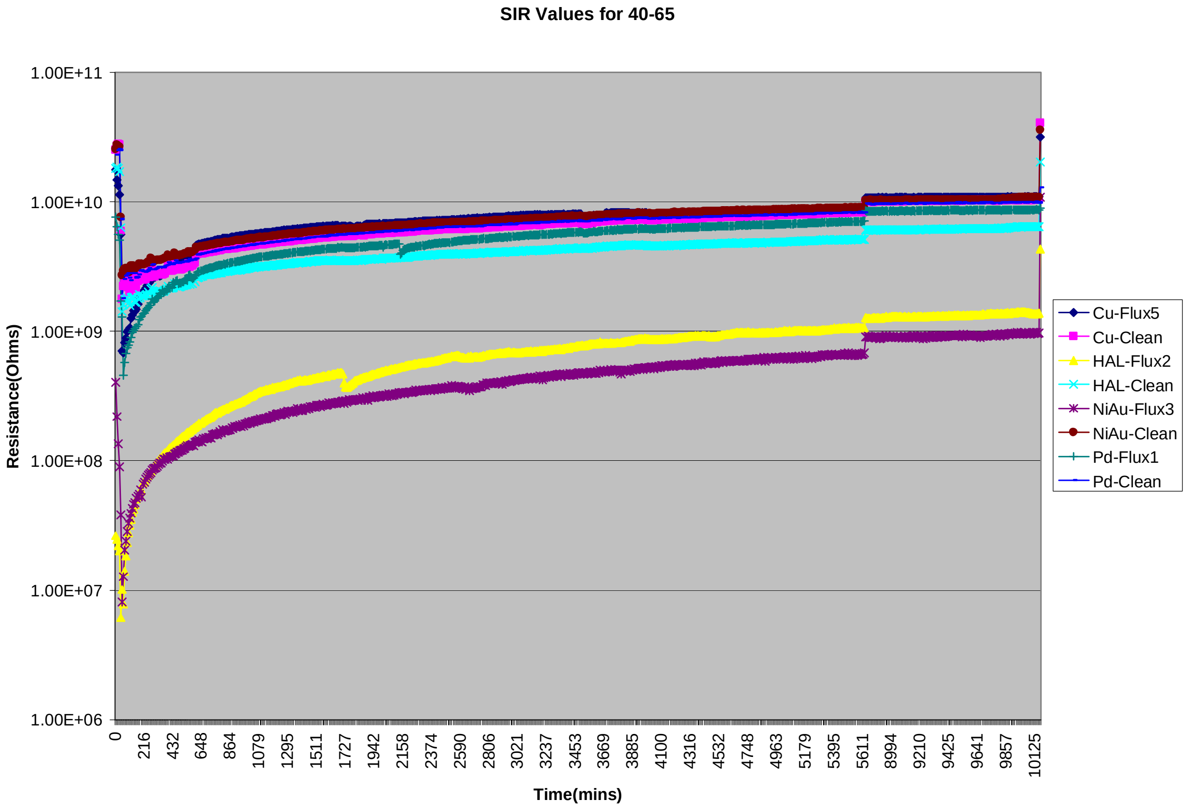

Figure 10: Representative SIR curves of fluxed and control boards

Table 3: List of parameters under which dendrites were observed

Temperature	Humidity	Flux	Surface Finish
40	93	Flux A	NiAu
		Flux B	Pd
		Flux D	HAL
65	85	Flux B	NiAu
		Flux C	HAL
85	85	Flux C	Pd
85	93	Flux C	Cu
		Flux C	HAL
		Flux E	NiAu
		Flux E	Pd

Dendrite formation was observed on many of the runs, and Table 3 summarises the conditions under which dendrites were found. All fluxes and all board finishes showed dendrites at some stage. The main conclusion from these results is that humidity appears to be the main driver for dendrite formation. No dendrites appeared on any flux/finish combination at a relative humidity of 65%. When the humidity was increased to 85% and beyond, dendrites start to appear. Figure 11 shows a typical dendrite found.

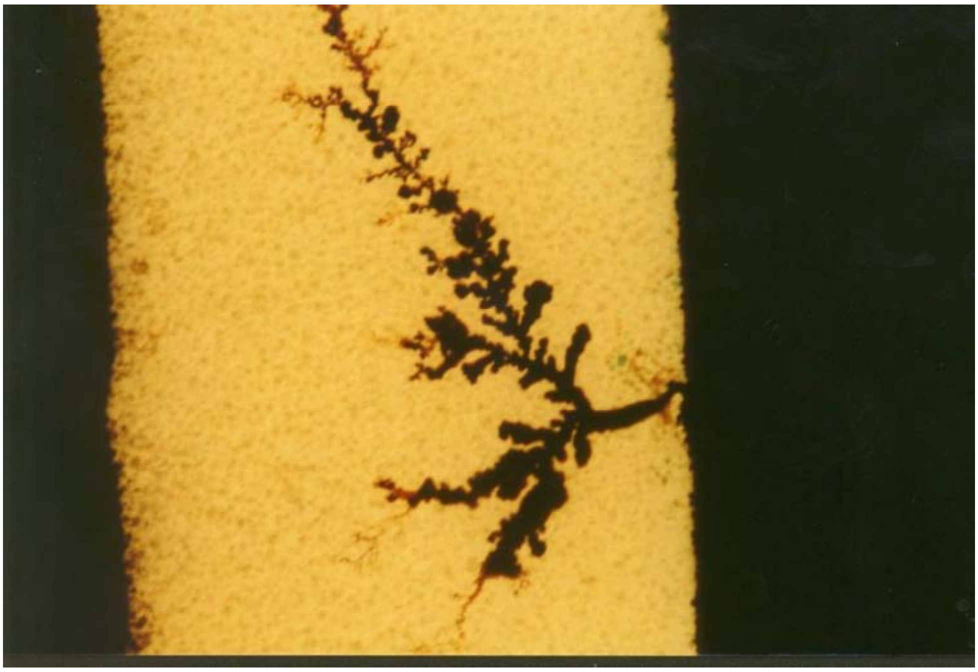

Figure 11: Dendrite on Pd - Flux C - 85°C/85%RH. Mag x200

4.3 DOE Results

The SIR results were then inputted into the Design of Experiments. The objective of the DOE was Response Surface Methodology (RSM). The design type was D-Optimal and the model type was quadratic. This model type included all linear terms, all interaction terms as well as quadratic terms. This produced a DOE with 50 runs from the 216 candidates and these were the runs performed. The results are summarised in Figure 3. These graphs show that the SIR value has a quadratic relationship with both Temperature and Humidity. Of the fluxes, Flux A gives the highest SIR values while Flux C has the lowest. The NiAu boards give the highest SIR values followed closely by Pd.

4.4 Conclusions

Analysing the design and model gives some interesting conclusions. If we examine the components ANOVA table, we see that the most significant parameters are the humidity and the flux type. The temperature term would be removed from the model if not for the significant interaction term with humidity and the significant quadratic term. Model 2 shows what happens when a reduced model with less runs is used. Eventually, temperature is removed from the model and the SIR value is totally dependent on humidity when the flux type and surface finish are kept constant. However, of necessity, these runs involved control boards and none of the other significant interaction terms were included.

From Model 1, we see that Surface Insulation Resistance is strongly dependent on humidity and has a quadratic relationship with the parameter. There is a very strong interaction between temperature and humidity. In general, as the humidity increases from 65% up to about 70% there is a slight increase in SIR values followed by a slow decrease as the relative humidity is increased from 70% to 79%. Above a relative humidity of 79% there is a very sharp decrease in SIR values. This behaviour is depicted in Figure 12.

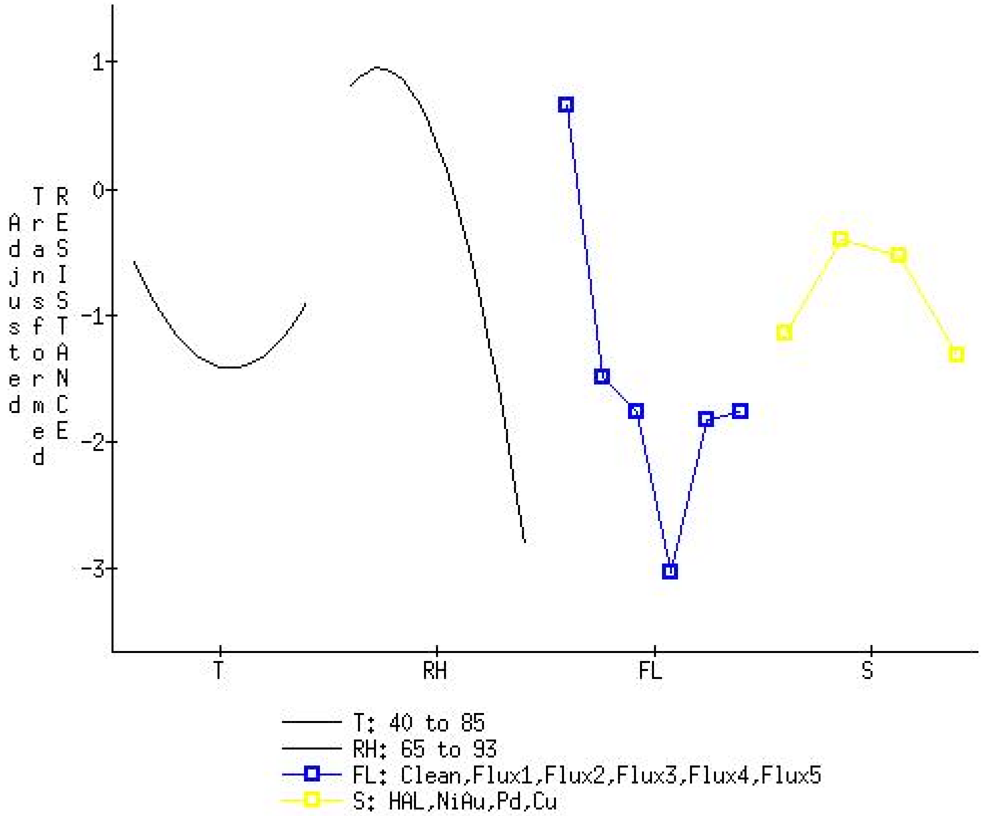

Figure 12: Parameter response curves

As the temperature increases, the SIR value slowly decreases reaching a minimum between 60-65°C. The SIR value then slowly increases as the temperature is increased from 65°C up to 85°C. The actual SIR value is strongly dependent on the settings of both parameters.

The flux type also has a significant effect on the SIR values. Flux A gave the highest SIR values while Flux C gave the lowest SIR values. The flux type also has an interaction term with the humidity. Varying the humidity has the greatest effect on Flux E. This curve is sharply different from the other flux types. The temperature/flux type interaction was deemed to be insignificant.

Changing the surface finish also affects the SIR values. NiAu gave the highest SIR values while Cu gave the lowest SIR values. Surface finish also has an interaction term with the flux type. Certain flux/finish combinations give higher SIR values than others.

All fluxes and all board finishes showed dendrites at some stage. The main conclusion from these results is that humidity appears to be the main driver for dendrite formation. No dendrites appeared on any flux/finish combination at a relative humidity of 65%. When the humidity was increased to 85% and beyond, dendrites start to appear.

The SIR values stabilised after about 48 hours measurement - provided there was no dendritic formation. When dendrites did form, they generally formed before 48 hours. However, there were a small number of dendrites that formed between 48-72 hours. No dendrites formed after 72 hours measurement.

5. Assessment of Soldering Heat on Flux Stability (WP4)

5.1 Assessment of Soldering Heat on Flux Stability

SIE.ZTME determined the following factors on SIR: thermodynamical stability of fluxes and their residues, the influence of preheat, temperature and dwell time on flux reaction. This work will be carried out on generic formulations of flux. In general SIR is used to qualify commercial fluxes. To avoid any spurious effects due to using specific flux from anyone supplier SIE.ZTME selected generic based fluxes. These generic fluxes are available for all project partners.

Table 4 summarizes the physical properties of the generic fluxes by different analyse procedures.

Table 4: Physical properties of the generic fluxes

typ	solid content g/l	solid content %	specific gravity g/cm³	moisture absorption %	acid value mgKOH/cm³	water content %	resistivity µS/cm
A	26,3	3,25	0,807	<1	12,9	4,06	0,53
B	215,2	24,82	0,867	15	56,7	4,8	125
C	23,6	2,4	1,006	6	16,1	97,2	130
D	220,8	25	0,883	<1/8	33	5,58	1,95
E	21,5	2,67	0,806	1,5	11,6	1,59	0,15

After first tests the fluxes were diluted following work with the NPL.

All the diluted fluxes show a specific spreading behaviour. Results of measuring the spreading of the different fluxes following the project procedure show the following behaviour.

The fluxes B, D, E show the same order of magnitude for spreading. The flux C is characterised be general less spreading because of water content.

The flux A shows in some cases much greater spreading that means there is a dependence from structure (line and gap). From 0.1->0.35mm the spread increases by factor 3. From 0.35->0.5mm spread decreases by 10%. Flux B shows a 33% decreasing spread when changing from 0.35->0.5mm.

For evaluating the undiluted fluxes we had only a limited number of results because of not enough samples. The figure shows decreased spreading for fluxes A, E and increased spreading for fluxes B, C, and D.

5.2 Determination of Soldering Heat Effects

The heating cycle influences the volatilisation and polymerisation of flux residues. Different assembly techniques will induce various effects due to their heating methods. The most common soldering techniques reflow and wave soldering will be varied to investigate their effect on SIR. The following test combinations, given in Table 5, were carried out.

Table 5: Experimental matrix for determination of heat effects

Heating Method	Temperature Distribution	Heating Energy
soldering bath	top side	contact time 2 s
soldering bath	top side	contact time 10s
soldering bath	bottom side	contact time 2 s
soldering bath	bottom side	contact time 10s
reflow with forced conv.	Profile with Tmax=210°C	
reflow with forced conv.	Profile with Tmax=235°C	
vapour phase soldering		
furnace with forced conv. 100°C/5min		

This set of experiments were carried out with all 5 types of generic fluxes, on line space structure 0.35mm/0.35mm with the NiAu board finish layer. Each situation was analysed on 3 samples.

The determination of the SIR value has been done under the following measuring and experimental conditions:

- measuring voltage: 35V DC
- bias: 35V DC (100V/mm for the 0,35 line/space structure
- test structure: line/space = 0,35mm/0,35mm; AuNi-surface
- test environment: 65°C/85%RH.

The sample preparation and measuring procedure was the same as for the other work packages.

The Siemens SIR procedure includes all the preparation of the test structure like the project procedure. However the experimental conditions were different from those defined for the project:

- measuring voltage: 100V DC
- bias: 100V DC
- test structure: line/space = 0,3mm/0,3mm; Cu-surface
- test environment: 40°C/93%RH.

There were a few examples of corrosion effects for both test methods. In the case of diluted fluxes there were no corrosion effects following the Siemens procedure. The corrosion effects in the case of the project procedure are described later.

Summarising these initial trials the results are shown in Table 6

Table 6: Summary of the initial tests

measuring voltage: 35V DC (Project Procedure)
bias: 35V DC
test structure: line / space = 0,35mm / 0,35mm; NiAu-surface
test environment: 65°C / 85% R.H.

Flux	undiluted					diluted *				
	SIR (Ohm)	increasing	decreasing	fail	corrosin	SIR (Ohm)	increasing	decreasing	fail	corrosin
A	>1E+8		X		no	>1E+8	X			no
B	<1E+8		X	X	yes	<1E+8	X			yes
C	<1E+8	X			yes	<1E+8	X			yes
D	<1E+8		X	X	yes	<1E+8	X			no
E	<1E+8	X			no	>1E+8	X			no

measuring voltage: 100V DC (Siemens
bias: 100V DC
test structure: line / space = 0,3 mm / 0,3mm ; Cu-surface
test environment: 40°C / 93% R.H.

Flux	undiluted					diluted *				
	SIR (Ohm)	increasing	decreasing	fail	corrosin	SIR (Ohm)	increasing	decreasing	fail	corrosin
A	>1E+8	X			no	>1E+8	X			no
B	<1E+8		X	X	yes	<1E+8	X			no
C	<1E+8	X			yes	<1E+8	X			no
D	<1E+8		X	X	yes	>1E+8	X			no
E	<1E+8	X			no	>1E+8	X			no

*: A (4xIPA)
B (60xIPA)
C (10xDI-w
D (200xIPA
E (4xIPA)

The goal of this initial investigation was to identify the appropriate amount of flux dilution. Therefore we made a comparison of the project and the Siemens test procedure. Finally the results show that only the diluted fluxes pass both test conditions. A disadvantage could be the fact, that during all future test no flux fails and all results will have no dependence from the different experimental conditions (bias, climate, heat;...). The advantage is to have greater sensitivity in detecting flux failures. This was the reason to work with diluted fluxes

All 5 diluted fluxes have been investigated with regard to heat effects on SIR. The results are summarised in the following chapter.

5.3 Analysis of Flux Type and Effect of Soldering Heat

The application of direct contact heat from top onto the SIR pattern proved impossible due to the creation of short circuits.

The only significant influence of heat effects on SIR was observed with the condition of 5min at 100°C in forced convection furnace**.** Fluxes B,C failed because of corrosion and had SIR values of approximately 10^8 Ohm or less.

All other heat effects show no significant influence on SIR for the diluted fluxes. The conclusion is that the soldering operation is sufficient to remove the flux residues and hence increase the SIR. Therefore the effects of soldering heat with more flux (undiluted fluxes) and with lower soldering peak temperature were tested. This was done with the undiluted fluxes for the case of forced convection reflow at the lower peak temperature of 185°C and 210°C for comparison purposes.

5.4 Conclusions

The main goal of this work package was to determine the effects of flux chemistry and various heating cycles on SIR. This was done with generic fluxes.

Five generic fluxes have been chosen having different chemical constitutions. They are described in the Technical Report ”Development of SIR Measurements in Electronic Assemblies” in April 1998. Their soldering properties correspond to the ISO-specification: ISO1.1.2., ISO 2.2.2., ISO 1.2.3., ISO 2.1.3. and ISO 2.2.3..

The rationale behind this choice was to have fluxes that fail in the SIR-test and to have fluxes that are expected to pass the SIR-test. Also a water-based flux was included, so that we could study the special effects of spreading and vaporisation during heating.

In the first step the most important properties of all the 5 fluxes have been determined, analysing the different chemical and physical properties influencing wetting and spreading behaviours, corrosiveness and moisture absorption.

The following properties have been measured and documented: solid content, specific gravity, moisture absorption, acid value, water content, resistivity, spreading behaviour.

After the first tests the fluxes have been diluted following the NPL-recommendation. The spreading behaviour of the diluted fluxes has been measured and documented too.

The spreading was tested in dependence of line structure width. Three diluted fluxes show always the same order of magnitude for spreading. One flux has general less spreading because of the water content. The fifth flux show different spreading in dependence from the line structures.

To avoid spreading effects on SIR measurement it is therefore recommended to ensure an equal flux distribution on SIR-test patterns during flux application.

The heating cycle influences volatilisation and polymerisation of flux residues. Different soldering techniques induce various effects due to their heating method. Most common soldering techniques are reflow and wave soldering. So the following heating methods have been used:

- Top and bottom side on a soldering bath with different contact time
- Reflow with forced convection and different peak temperatures
- Vapour phase soldering
- Furnace with forced convection 100°C/5min.

The SIR measurement was done with only one set of test parameters, in a comparable way to the other project partners. The diluted and the undiluted fluxes have been tested with regard to SIR and corrosivity.

For two of the five diluted fluxes the only significant influence of heat effect on SIR was observed at condition 5min/100°C in forced convection furnace. In all other cases the heat effecting seems to remove the flux residues and hence increases the SIR.

The undiluted fluxes have been tested in addition for the case of forced convection at two peak temperatures, so as to have more flux. Again the same two fluxes and a third one show the same effect on SIR and corrosivity. All three fluxes fail in both cases of peak temperature.

Soldering heat <u>can</u> remove flux residues from the test pattern. The only sensitive heating method for SIR measurement is the 5min/100°C storage of the test samples in the forced convection furnace.

6. Development and Test of New Draft SIR Test (WP5)

The previous three sections:

Effects of Electrical Biasing on SIR Testing (WP2), Effects of Temperature and Humidity (WP3), and Assessment of Soldering Heat on Flux Stability (WP4) all made recommendations for a new test method. These results predicted how the SIR is effected by different field strength, test patterns, test temperature and humidity and soldering heat in combination with fluxes and board finishes. Recommendations for these parameters were made and a new draft SIR method has been developed and is detailed below

A SIR testing method was formulated detailing the sample preparation and SIR measurement procedure the details of which are given in the final report.

To validate the above method three laboratories conducted a joint set of tests. These were NPL, Siemens and NMRC. The intercomparison test was based around two experiments:

1. Test boards prepared by Siemens and sent to all the partners by post for testing at their laboratories.
2. The samples prepared and tested by each partner at their laboratory.

The test board, fluxes and test conditions are listed in Table 7. The preparation and testing of samples were carried out according to the developed test method.

Table 7 : The test board, fluxes and test conditions

Flux	Flux A (ISO1.1.2)	Flux D (ISO2.3.3)
Dilution with IPA (times)	4	200
Test condition	40°C/93% RH	65°C/85% RH
Number of test boards	4 test boards and two control boards	
Board pattern	200µm gap and 400µ m track with NiAu finish	

6.1 Results of Intercomparison

The results in the following figure are an average value from all test boards for each condition. The standard deviation of the log SIR was less than 0.2 decade ohms. The SIR value with time on the ***control boards*** prepared by Siemens and each partner showed excellent agreement for the two environment conditions. Similarly the SIR value with time on the fluxed test boards, shown in Figure 13, demonstrate an excellent agreement. The nomenclature in the figure refers firstly to where the boards were prepared and secondly to where they were tested.

These results verified the technique, and was achieved for samples being prepared at each laboratory and for samples being prepared only at Siemens. Each laboratory testing both sample sets achieved the same results. This indicated that the reproducibility for individual sample preparation and SIR measurement according to the procedure at different laboratory is very good, ***and demonstrates that the draft SIR method is feasible.***

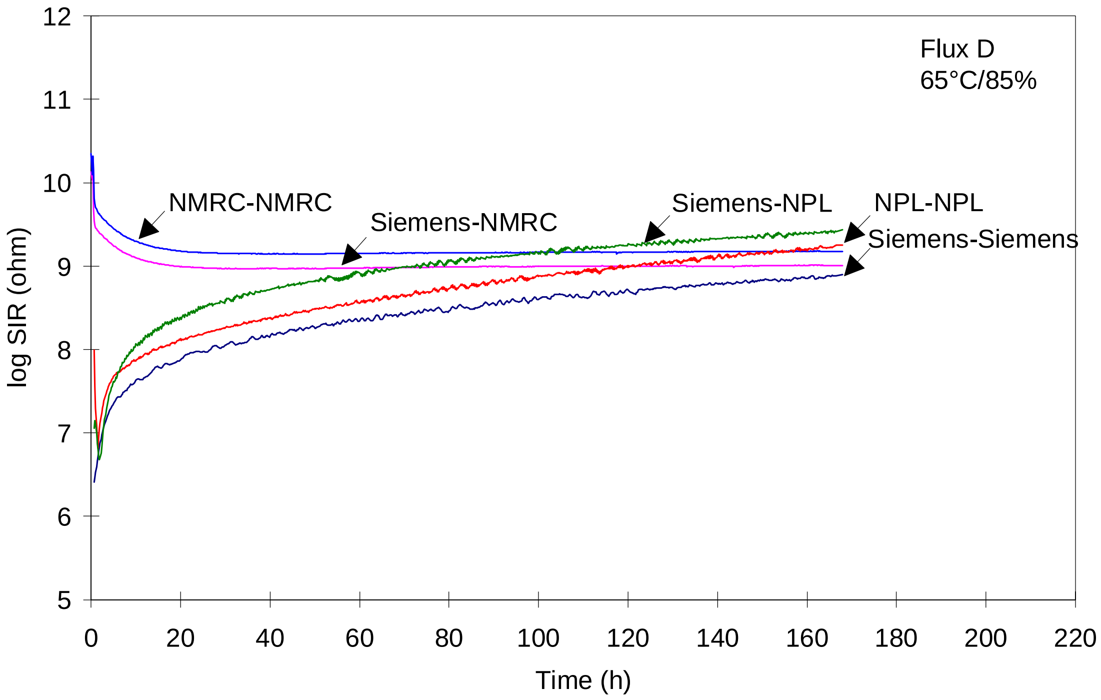

Figure 13: The SIR value with time on the test boards tested at 65°C/85%

6.2 Conclusions

- A new detailed method for SIR testing has been developed here, and a complete procedure given.
- This differs significantly from previous SIR methods in calling for frequent monitoring, low test voltage, a new test pattern with fine pitch, and reinforcing the use of 40°C/93%RH as appropriate for volatile fluxes.
- The new method proposed here has been verified by different laboratories, with good agreement demonstrated with the flux D results. However, the results for flux A and test condition 40°C/93%RH, gave some cause for concern, and these results will be checked in late work.

7. Application of SIR Method to Industrial Context (WP6)

7.1 Introduction

This work transformed the previous laboratory work on generic fluxes to conventional fluxes and solder pastes in a full production environment. The technical approach is to assess the SIR method in an industrial context by using commercial materials in a standard manufacturing production line. All the experimental work was highly influenced by the knowledge of high volume assembly lines for single and double sided printed circuit board assemblies.

7.2 Experimental conditions

7.2.1 Analysis of commercial fluxes and pastes

Fluxes are mostly used in an industrial environment for wave soldering processes. Solder pastes are required for reflow soldering techniques. These processes are quite different.

Two fluxes and two solder pastes were chosen and are qualified to be no-clean systems by existing standards, and are widely used in the electronics' industry. To be free of any bias these products are not used by any partners in production. All partners agreed, that the product names and the supplier remain confidential.

Table 8 describes the fluxes and pastes for the following experiments, the data was provided by the supplier.

Table 8: Flux and Paste Description

	Solder Paste A	Solder Paste B	Flux C	Flux D
ISO CLASS	1.1.2.C	1.2.2.C	1.1.2.A	1.2.3.A
Halides	< 0,1%	< 0,1%	< 0,1%	none
Amines	none	none	none	none
Carboxyl Acids	Yes	Yes	Yes	Yes
Acid number	121 mg / KOH	122 mg / KOH	22,2 mg KOH/g	15,4 mg KOH/g
Density at 20°C			0,820 g/cm³	1,004 g/cm³
Solvents	high BP mat.	high BP mat.	IPA	Water
Metal content	90,6 %	90,6 %		
Alloy	Sn62Pb36Ag2	Sn62Pb36Ag2		
Particle size	20-45µm	20-45µm		

7.2.2 Test Parameters

For reflow soldering the solder and flux volume is transferred in paste form. Soldering reflow can take place under normal and nitrogen atmospheres with different temperature profiles and different reflow processes, forced convection , vapour phase. The influences on the quality and quantity of residues and their distribution corresponding to the SIR test will be evaluated .

The test-structure and process parameters for ***reflow soldering*** are the following: The laminate type was FR4 and used the SIR patterns with 200µm gap/400µm track, and a NiAu finish on Cu. The stencil thickness for printing was 150 µm. Reflow forced convection was with atmospheres of air or N_2 (O_2 500ppm). Various temperature profiles were used: high (1); low (2) and vapour-phase (5). Vapour phase soldering is an existing and growing type of reflow soldering that is very different from forced convection reflowing. To ensure that also this technology is recognised a third profile was used in a vapour phase environment.

The test-structure and process parameters for ***wave soldering*** are the following. The laminate type was FR4 and used the SIR patterns with 200µm gap/400µm track, and a NiAu finish on Cu. In addition a copper finished board with 500µm gap/400µm track was used. The flux application was by spraying, and the amount 200 µl for every board (50 µl / comb). Soldering was in air, or N_2 (O_2 500ppm), or vapour-phase. Various temperature profiles were used: bottom side (3) ; top side (4). Note: Bottom side is with comb patterns face down on the wave at 240°C and a conveyer speed with a contact time of 2 sec. Top side is with comb patterns face up at 240°C and a conveyer speed with a contact time of 2 sec

7.2.3 Experimental Matrix

In the design of experiment approach 172 test combinations with 34 test runs were performed. Two test boards for each combination and two control boards have been used for each run. All together 412 tests runs have been evaluated.

All test samples were prepared by Partner 3 (Siemens AG) in an industrial environment. All the reference data were also provided by Partner 3. After preparing and monitoring, the test boards have been shipped to the other partners. Different colours mark the measuring activities of all the project partners.

7.3 Determination of the SIR-values and analysis of the results

The SIR measurement method was based mainly on work package 5, some influencing-factors have been recognised within the testing. For the SIR evaluation all partners provided the final and the minimum SIR value as well appearance of dendrites.

From the experimental matrices and with support of STATGRAPHICS software, four statistical designs of experiments (SDOE) were created to find the critical cause variables. There are DOE with 2-Level designs (low or high, for example –1, +1). Only the boards had 3-Levels (-1, +1, 0). The evaluation was realised with a probability of 95 %. The F-value is defined as a ratio of 2 variances . At the SDOE the cause variables with **F-ratios > 4 are significant different**.

7.3.1 Results for the Reflow-soldering with forced convection

Critical variables with regard to SIR as a result of main effects are: soldering atmosphere, board design, and humidity conditions. The SIR results revealed that the air atmosphere is more critical than the nitrogen one. The board design has only a significant influence on the final SIR value. The more critical is the finer test pattern (400/200µm). The more critical test condition is the 65°C/85%RH, but only at the minimum value with a F-ratio 4,9.

Interactions were founded between: humidity conditions and soldering atmosphere; and measuring voltage and soldering atmosphere. The more critical combination at 40°C/93% RH is with atmosphere. The more critical combination at 5V is also with atmosphere.

7.3.2 Results for the Reflow soldering with vapour phase

The critical variable with regard to the SIR response, resulting in a main effect was board design. The board design has only a significant influence on the final SIR value, and is more critical with the finer test pattern (400/200µm).

Interactions were founded between paste type and measuring voltage. Paste A is more critical in combination with 50V than with 5V, whereas with paste B 5V is more critical.

7.3.3 Results for the wave soldering

The main effects on SIR response with board design were top and bottom side profiles. The board design has a significant influence on the final SIR value. The more critical is the finer test pattern (400/200µm). The top and bottom effects are due to variation in flux residues. On top of the board is the more critical condition, since there is less heat effect and less cleaning by the wave itself.

Interactions were founded between humidity conditions and measuring voltage. Using 5V bias the 65°C /85%RH is more critical. Using 50V bias the 40/93 condition gives smaller SIR values.

Critical variables with regard to appearance ***dendrites*** as a result of main effects are: board design, soldering environment, humidity conditions, top and bottom side, and voltage. The board design has a significant influence on the appearance of dendrites. The more critical is the finer test pattern (400/200µm). Wave soldering under oxygen atmosphere produces more dendrites in our tests. The humidity condition 40°C/93%RH is the more critical condition with a F-ratio 21,1! Flux on the top of the board is the more critical condition, since there is less heat effect and less cleaning by the wave itself. Using 5V bias is more critical test condition since the higher voltage easily destroys dendrites.

Interactions with regard to the dendrites have been founded between flux and top and bottom side. The appearance of dendrites for Flux C depended on the board side, with a higher probability of dendrites on the topside. Flux D shows no differences.

7.3.4 Results for the forced convection furnace

The main effects on SIR response with forced convection were board design and humidity condition. The board design has a significant influence on the final SIR value with finer test pattern (400/200µm) the more critical. The more critical test condition is 40°C/93% RH environment and the minimum SIR value with a F-ratio 11,5 and at the final value with a F-ratio 10,6.

Interactions were found between humidity conditions and measuring voltage. Using 50V bias the 40°C/93%RH is more critical. Using 5V bias there is no interaction with the humidity conditions.

7.4 Final Conclusion for the Proposal for the SIR Test in WP7

Based on the results from WP6 and the results from earlier work packages the following test procedures are proposed. The recommendations take into account different manufacturing environment for wave and reflow soldering.

Solder paste		Wave soldering	
Board track and gap (µm)	400/200	Board (µm)	400/200
Finish	NiAu or Cu	Finish	NiAu or Cu
Stencil (µm)	150	Flux amount (µl) per comb pattern	50
Soldering profile	According to paste requirement	Applied method	pipette
Soldering condition	Air	Heating condition	100°C for 5 minutes
Test voltage (V)	5	Test voltage (V)	5
Test condition	40°C/93%RH	Test condition	40°C/93%RH
Pass / fail	Minimum SIR value over whole test ≥ 10^8 Ω no dendrites	Pass / fail	Minimum SIR value over whole test ≥ 10^8 Ω no dendrites

8. Validation and Production of Final SIR Method (WP7)

8.1 Introduction

Following all the previous work a method was agreed as the final method for SIR testing. In WP7 a commercial flux and solder paste were used to finally validate the SIR method by round robin tests.

8.2 Experiment

The round-robin test was based around two experiments:

1. The samples prepared centrally by Siemens and sent to all the partners by post for testing at their laboratories.
2. The samples prepared and tested by each partner at their laboratory.

The test board, flux, paste and test conditions are listed in Table 9. The preparation and testing of samples were carried out according to the Final SIR Test Method.

Table 9: The test board, fluxes, paste and test conditions

Test flux and paste	Flux 1 (ISO2.2.3.A)	Paste1 (62/36/2Ag, No-clean)
Board finish	Cu	NiAu
Test condition	3 test boards and 1 control board	3 test boards and 1 control board
Number of boards	40°C/93% RH	
Board pattern	200µm gap and 400µm track	

8.3 Results and Discussion

The intercomparison proved very successful with excellent agreement between all the partners. The results of the fluxed boards are shown in Figure 14. The results nomenclature in the figures refers firstly to where the boards were prepared and secondly to where they were tested. These results verified the technique and the ability of multiple testers to achieve the same results. This was achieved for samples prepared at each test laboratory and for samples being centrally prepared at Siemens. Each laboratory testing both types of sample got the same results. This indicates that the reproducibility for individual sample preparation and SIR measurement according to the procedure at different laboratory is very good. Both the flux and the paste results were better than the pass/fail criteria. Therefore the new SIR measurement method is very successful at evaluating the reliability of modern electronic board.

8.4 Final SIR Method

A detailed method has been produced giving complete details from the initial sample preparation through to processing the results. The method as well as including experimental parameters also describes best practice in performing the test and on the calibration of the equipment.

The results from WP7 for a commercial flux and solder paste have completely verified the test method and the approach adopted in the project. These results are for intercomparisons performed where the sample preparation was carried out at more than one location and with samples at various ages over a few weeks.

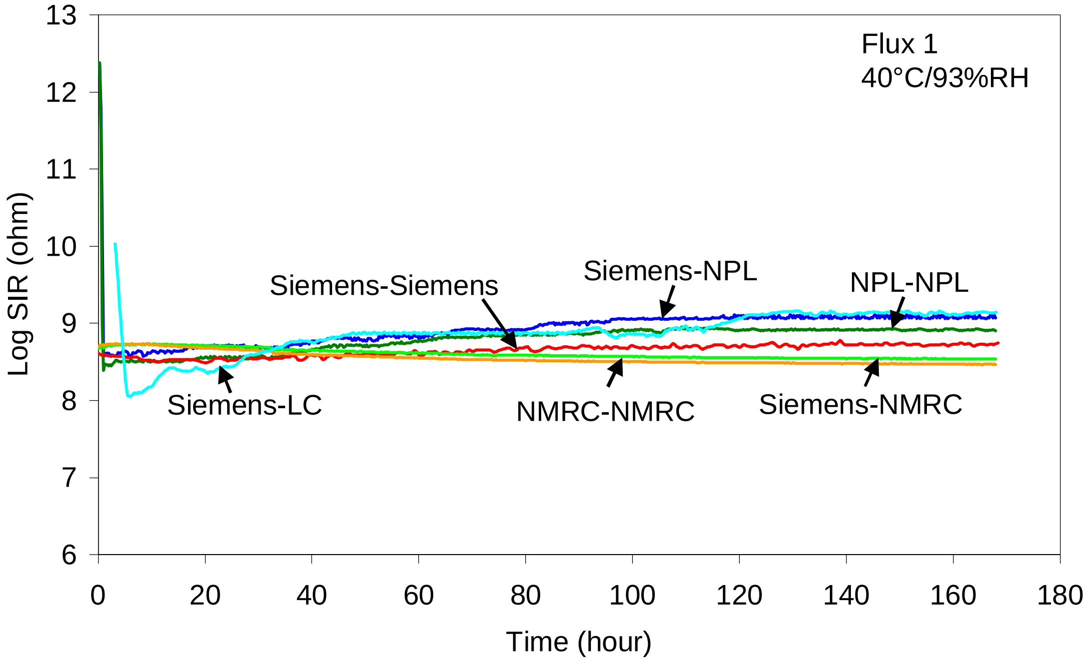

Figure 14: The SIR value with time on the fluxed boards tested at 40°C/93%

9. Conclusions

The electronics assembly market in the EU is estimated to be 160 billion Ecu. All of this will have been assembled with fluxes that will have been qualified by an SIR method. Even with the occurrence of only a small % of problems, this will represent a significant cost given the sector size.

This three-year project has been innovative and has successfully developed a test method for evaluating modern electronic fluxes. The project has revealed a number of shortcomings in the existing method. The project method has looked carefully at the experimental parameters and has selected values that have high discrimination for to days flux chemistries. The test method has utilised current instrumentation technology to facilitate rapid data acquisition that allows accurate prediction of reliability. Specifically the detection of electrochemical corrosion and the occurrence of dendrites are readily detected electronically, significantly enhancing reliability prediction.

Some of the key features of the project have been to demonstrate the requirements for the pitch of the SIR pattern and the test voltage. Both of these need to reflect to days assemblies, and the work has shown that fine pitch and low voltages dramatically reduce the SIR values from current SIR standards. Dendrite formation during a test can be rapid, with their existence lasting a short while. Current instrumentation permits multiplexed data acquisition over 128 channels every 10 minutes, thus making it feasible to monitor dendrite formation and hence capture potentially catastrophic causes of failure. The use of weak carboxylic acids as the activators and the use of synthetic resins in fluxes makes the choice of environmental test conditions more critical and the work here shows the advantage of using 40 °C / 93% RH as the standard test condition. Therefore this new test will result in faster testing of a higher quality and hence a more reliable product and will hence have a very beneficial economic impact.

Dissemination has been very strong throughout the project. Most importantly the output from the project has been fed into the standards arena, and hence has been made available to all. The project coordinator chairs the ISO working group. The project is fully committed to influencing the relevant standard. During the working group meetings every opportunity has been taken to influence the direction of the new standard. The working group held its last full meeting in April 98 and before that in November 97. At the April 98 meeting the group was fully updated with the project progress. In July 98 the working group forwarded its draft standard to the ISO secretariat. ISO circulated the document and held a full ISO meeting in December 1999, where again the project coordinator was present to expound the methods developed during the project.

The influence of the project coordinator was unfortunately limited by two factors. Firstly, other working group members proved reluctant to embrace new ideas that would significantly impact on test house abilities to carry any new method. Secondly, the time scales of the standards committee didn't overlap perfectly with the project. With respect to both of these the project leader has an interest in pursuing the uptake of this work in the ISO standards.

The biggest impact of this work will be on the flux vendors. So as to maximise the impact of this work two workshops were organised. The first workshop was held in Berlin, Germany in May 2000, and the second was held in Brighton, England in November 2000. The meetings were attended by material and equipment suppliers to the industry, which are typically SME’s. The project partners shared their knowledge and experience from the project with the manufacturers of fluxes, pastes and instruments. Both meetings were a great success with the attendees keen to see the output of the work implemented in international standards.

Other dissemination has been via publications, presentations and workshops. So far the only publication has been in the European conference held in Brest in October 1999. Presentations have been made at this conference and at the Soldering Science conference of the NPL in May 2000, and the Apex conference in Long Beach in April 2000. A workshop organised at the NPL has also been carried out. Publications are being prepared on the project.

Siemens are adopting the method in all 27 manufacturing sites in Germany, and their sites around the world, more than 50 in total. The method developed in this project will be used, and the Siemens internal method of qualifying solder creams has been modified. The new Siemens method will use the SIR test vehicle developed in this project. A paper on the work is being written in German.

The work will also have a beneficial impact on health and safety by facilitating the qualification of flux families that are no-clean or VOC free. These fluxes are more beneficial to the operator and the environment. This influence is infrastructrual and will have a pervasive effect across the industry.

10. Acknowledgements

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