

Evaluation of fluorinated protective printed circuit board coatings and non-resinated, non-volatile organic compounds (NVOC), water based liquid fluxes

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1. Background

A study was conducted evaluating the performance and compatibility of a commercially available fluorinated coating for protection of printed circuit boards (PCBs) with six commercially available liquid flux formulations. The study was designed to be used as a general guideline for the use and capabilities of these materials.

The fluorinated polymer protective coating evaluated was 3M™ Novec™ 1700 Electronic Grade Coating. Novec 1700 is a low viscosity, low solids coating solution that, when coated, leaves a protective thin film on the PCB. Traditional, high viscosity conformal coatings were not included in the study as they can exhibit problems with many modern electronics structures such as incomplete coverage of low standoff structures. This limitation can result in gas cavities and unprotected areas, air pressure or moisture build-up within cavities or drilled vias and incompatible thermal expansion rates in critical structures.

The six liquid fluxes were chosen from Non-Volatile Organic Compound (NVOC) based and iso-propanol (IPA) based formulations. NVOC liquid fluxes for wave soldering have been of interest in the industry for the last several years to replace IPA-based fluxes. NVOC liquid fluxes offer benefits over IPA-based liquid fluxes such as non-flammability (making it easy for logistic systems and safe to use), environmental compatibility, and low cost since they use water as their solvent. NVOC fluxes also have improved chemical performance. To highlight these benefits, part of this evaluation was to compare the latest NVOC liquid fluxes against well known IPA-based fluxes.

2. Experiment

Test boards

The test board designed by FMI (Figure 1) was used for most evaluations. The test board was a combination of:

- Surface Insulation Resistance (SIR), (0.2 mm gap and conductor width, 10 mm × 10 mm area) and *bulk resistance* (6 × 6 matrix of drilled vias) test patterns for evaluation of leakage currents in drip water/soak and damp heat tests
- Surface insulative resistance, wave soldering *process capability* test patterns; process capability to avoid *solder bridges* and *missed joints* were evaluated using specific test patterns and
- Drilled via matrix was also used to evaluate process capability by creating acceptable *drilled via fill*. Soldering results on patterns were visually analysed, averaged and normalized to a range of 0-100%.

Non-coated/non-soldered and non-coated/soldered test boards provided a reference for some tests and test groups. A total of 38 test boards were included in the tests.

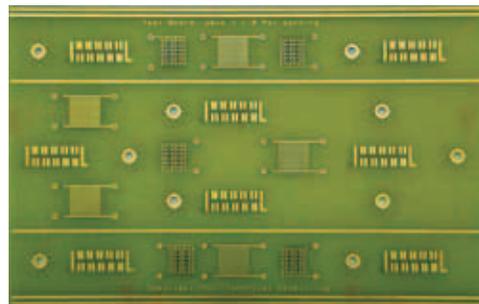


Figure 1. FMI test board for SIR and process capability measurements

Flux application

All fluxes were applied by an airless spray method. To record the applied solids content, all test boards were weighted before and after flux application. Solids content on the board is a key factor – both for process capability and reliability in environmental stresses. This was considered especially important as the fluxes selected for the evaluation represented very different solids contents (3.6-8.5% by weight).

Soldering process

The soldering process utilized an Erska ETS250 double wave soldering machine equipped with medium wave preheaters (Figure 2). Process parameters were set as follows:

- The contact conveyor speed was 0.7 m/min
- The preheat radiator source temperature was 550°C, the preheat time was 1 minute, the temperature gradient average on the top side of a 1.6 mm FR-4 laminate was 1.7C/s and the temperature on the top side at the end of the preheat was 130°C
- The contact length at the first wave was approximately 10 mm, at the second wave was approximately 40 mm. The second wave depth was 8 mm, the conveyor angle was 6°, the solder temperature was 258°C and the solder alloy was SnAg 3.8 Cu 0.7
- The PCB bottom surface height was 1 mm below the second wave surface (nominal)
- The same soldering carrier was used for all test boards to minimize mechanical inaccuracies between test groups



Figure 2. Ersas ETS250 double wave soldering machine

Protective coating application

The protective coating was applied by dipping. Boards were drained in a vertical position at a 45° angle until no drops fell. All test boards were given a 3 h/75°C drying treatment and settled at 21°C/24 h before entering into the 85°C/85% test. The contact measurement test was made for both air dried and cured coatings.

Effect of protective coating on electrical contact test

The purpose of this test was to determine the ease with which electrical contact can be made through the coating. This was evaluated by pressing a spring-loaded, Ag-coated tip of 0.75 mm radius against non-coated and coated immersion Au plated PCB surfaces (Figure 3). The contact force was measured using a Precisa BC1000J scale (10 mg sensitivity). The spring-loaded tip was attached to a benchtop device for linear movement and the PCB was located on the top of the scale. Contact force was slowly increased until electrical contact (<100 ohm) was detected by a Fluke 75 multimeter.

Five successive measurements were performed on the coated surface area. The tip of the contact tool was wiped with a microcloth after every measurement to prevent material accumulation at the contacting tool tip. The highest and the lowest force readings were eliminated and the result was calculated as an average of three measurements.

A non-coated immersion Au plated PCB surface was used as a reference.



Figure 3. The test setup for measuring force required to make electrical contact through coating

Drip water test (0.01% NaCl solution)

The purpose of the drip water test was to demonstrate the properties of a protective coating film against charge carriers (Cl⁻ ions) in an aqueous solution. A condensing/drip water soak test was performed by dipping one test board per each test group

into a 22°C, 0.01% NaCl solution for one minute. Close to the end of the 1 minute soak period (at t=50s), a 5.6V bias was connected into the test pattern for 10s via a 1 kohm bias resistor to initiate limited electrochemical reactions AND to measure the resistance of the test pattern when soaked (measurement taken at t = 60s).

Note: This test is not designed to demonstrate coating properties against gaseous chlorine.

Dew point test

The purpose of the condensing/dew point test is to demonstrate protective coating properties in a condensing situation (low external ionics content, water film on coating). A continuous 15V bias was connected into the test pattern via a 150 kohm bias resistor. The dew point test was then performed by holding the energized test pattern in an atmosphere of water vapor (created by boiling deionized water) for 5 seconds. A resistance reading was taken at t=5s, when a water film had condensed on the cool sample.

Damp heat test

The purpose of this test is to demonstrate the protective coating surface insulation resistance (SIR) properties under elevated heat and humidity. This was performed at the VTT/Electronics laboratory in Oulu, Finland. A VTT automated measurement system was used to supply a 15V bias voltage to test patterns via a 100 kohm resistor. Forty-two measurement channels were used. Two SIR test patterns on each test board were selected for each test group. The two patterns were measured separately. Test vehicles were set up in an open subrack with a 15 mm distance between the boards (Figure 4). The voltage drop over the bias resistor was measured. Test results are presented as a test pattern surface resistance (SIR) value, calculated from voltage drop and bias resistor values.

The test conditions were 240 hours at 85°C/85% R.H. After 240 hours, the test condition was changed to 85°C/95% R.H. for 24 hours to demonstrate the samples' behavior under close to condensing conditions. The test was started by raising the chamber temperature to 85°C. Humidity was then raised after the temperature had stabilized. Test shut-off was performed by stopping humidification first, then slowly decreasing the temperature over 8 hours.

SIR values were re-measured 48 hours after the damp heat test at FMI (21°C/45% R.H.) by measuring all test patterns manually (15V bias fed through 1Mohm bias resistor and measuring voltage drop over bias resistor by Fluke 75 multimeter).

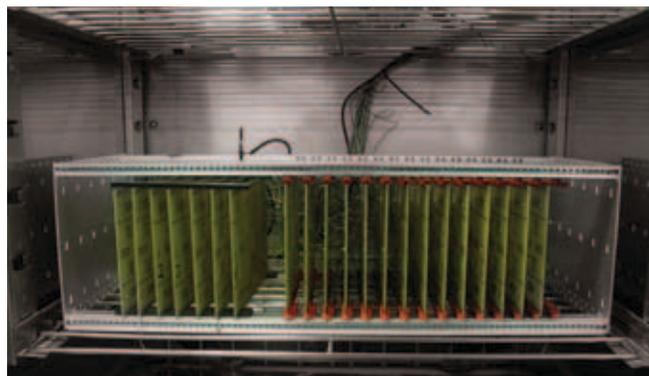


Figure 4. The damp heat test setup at VTT/Electronics laboratory, Oulu, Finland

Ionic contamination test

The ionic contamination of test boards after soldering (coated boards were not measured) was evaluated using an Alpha Metals Omega Meter model 600 SMD (Figure 5). The test solution was 50/50 H₂O/IPA at 40°C. The test time was set at 15 minutes. The test solution was sprayed continuously against sample surfaces. Results are expressed normalized at micrograms of NaCl equivalent per cm² (both sides of the test boards were taken into calculation). One board of each test group was measured.



Figure 5. Ionograph Alpha Metals Omega Meter 600 SMD

Process capability of fluxes test

Table 1. Matrix of test groups and tests conducted

Test Group	Electrical contact test	Drip water test	Dew point test	Damp heat test	Ionic contamination test	Process capability of fluxes
No flux, no coating	•	•	•	•	•	
No flux, Novec 1700	•	•	•	•		
Ref flux1, no coating		•	•	•	•	•
Ref flux1, Novec 1700		•	•	•		
Ref flux2, no coating		•	•	•	•	•
Ref flux2, Novec 1700		•	•	•		
Flux A, no coating		•	•	•	•	•
Flux A, Novec 1700		•	•	•		
Flux B, no coating		•	•	•	•	•
Flux B, Novec 1700		•	•	•		
Flux C, no coating		•	•	•	•	•
Flux C, Novec 1700		•	•	•		
Flux D, no coating		•	•	•	•	•
Flux D, Novec 1700		•	•	•		

Table 2. Protective coating data

Protective Coating	Carrier Base	Solids Content (w%)	Density (g/mL)	Viscosity (cP, @23°C)
3M Novec 1700	nonafluorobutyl methyl ether (3M Novec 7100DL)	2.0	1.50	0.6

Table 3. Liquid flux data

Flux	Descrip.	Carrier Base	Solids Content (w%)	Density (g/mL)	Acid number (mg/OH)
Multicore MF200	Ref flux1	IPA	6.4	0.830	48
Interflux 2005C	Ref flux 2	IPA	3.4	0.814	28
Cobar 396-TNP	Flux A	NVOC (water)	3.0	1.007	28
Multicore MF300S	Flux B	NVOC (water)	4.6	1.012	37
Interlux 2009MLF	Flux C	NVOC (water)	3.6	1.00	25
Qualitek 358	Flux D	NVOC (water)	8.5	1.030	52

3. Results

Effect of protective coating on electrical contacting properties

Table 4. Contacting properties results (force required for the test pin to break coating and create a contact)

Sample (immersion Au plated PCB surface)	Contact force to break through coating (contact tip radius 0.75 mm)
Reference surface (Immersion Au PCB plating, no coating)	<0.1mN
Novec 1700 air dried	0.3N (average of 3 measurements)
Novec 1700 oven cured 3h/75°C	0.1N (average of 3 measurements)

A large variance in individual measurements was detected. Oven cured film shows a lower contacting force than air dried, which may be a result of the oven dried film being harder. Contact force to make an electrical contact is acceptable for most connector types.

Applied flux quantity and ionic contamination results after dual wave soldering

Table 5. Comparison of solids applied and resulting ionic contamination level

Flux	Sample ID	Applied flux (g)	Applied flux (g/m ²)	Solids content (w%)*	Solids applied (mg)*	Meas. ionics (µg/cm ²)	Solids applied/meas. ionics
Control	X	0	0	NA	0	0.2	NA
Ref Flux1	41	0.72	45.0	6.4	46	2.4	19.2
Ref Flux2	32	0.52	32.5	3.4	18	0.9	20.0
Flux A	9	0.67	41.9	3.0	20	1.0	20.0
Flux B	13	0.78	48.8	4.6	36	1.8	20.0
Flux C	23	0.63	39.4	3.6	23	1.4	16.4
Flux D	25	0.69	43.1	8.5	59	2.9	20.3

* Solids content and calculated results refer to manufacturers specification for solids content

The solids content of the fluxes varied in the range of 3.0-8.5w%. Flux application on the board should be adjusted to achieve a balance between the soldering results and the resulting ionics on the board. In this evaluation, flux amount was not adjusted according to flux solids content. This needs to be taken into account when interpreting the results. A high solids amount increases ionics on the board after soldering, but a low solids amount may affect soldering results.

A high solids content of flux may be beneficial, as less carrier (water) is applied to the board. A spray fluxer has to be accurate enough for application of small flux quantities.

The solids applied on the board seem to correlate with the amount of ionics measured on a board; each 10 mg of solids content creates an amount of 0.5 µg of NaCl equivalent ionics content on a board for both IPA and water based fluxes. The result for Flux C makes a small difference in this respect.

Process capability results

In the interpretation of results below, it has to be taken into account, that:

- Set process parameters were general and not specifically adjusted for any of the fluxes
- Fluxes selected for testing had different solids content and flux applied on the board was not adjusted according to solids content

Table 6. Results of flux process capability evaluation

Flux	Sample ID	Applied Flux (g)	Solids Applied (mg)	Solder Bridging (%)	Missed Joints (%)	Via Fillet (%)	Average (%)
Ref Flux1	42	0.75	48	96.3	85.0	100	93.8
Ref Flux2	35	0.53	18	70.0	56.3	55.6	60.6
Flux A	8	0.56	17	92.5	78.8	99.4	90.2
Flux B	12	0.60	28	93.8	76.3	100	90.0
Flux C	18	0.67	24	95.7	87.5	97.5	93.6
Flux D	30	0.76	65	87.5	91.3	63.0	80.6

A missed joints evaluation was performed as an average of the four test patterns in the middle of the board and a solder bridging evaluation was performed as an average of all 8 test patterns. A via fillet evaluation was performed on all five 6 × 6 via matrixes (criteria: solder wetting to hole knee min. 50% of circumference). 100% reading refers to no anomalies found.

The lower process capability found for Ref Flux 2 may be a result of low solids content and insufficient application of flux. Flux D shows lower process capability results than fluxes A, B and C, although applied solids are at a high level. Flux A shows relatively good performance despite the low quantity of solids applied. Fluxes B and C performed the best. Flux B was found to have an effective wetting agent (surfactant), thus improving fillet soldering. Flux normally does not effect missed joints but, based on results, it seems to have had an effect in this case. Reference Flux 1 shows process capability equal to NVOC fluxes B and C, but the applied solids amount is rather high.

The results in the front of the board were found to be significantly lower than the center and back-end. The comparison in table 6 shows the difference. Results are taken from the two test patterns located in the front of the board and the four in the middle. It is believed that the higher density of water (compared to IPA) will

initiate liquid flow along a tilted conveyor. The front of the test boards (20 mm) for all water based fluxes had solder particles, showing low flux quantity/activity on the board surface. The IPA-based fluxes showed no solder particles, but test patterns showed somewhat lower process capability for them.

Table 7. Process capability difference at the front of board vs. board center

Flux	Sample ID	Solder bridging in the center of test board (%)	Solder bridging in the front of test board (%)
Ref. Flux1	42	100	97
Ref. Flux1	35	75	63
Flux A	8	100	90
Flux B	12	100	90
Flux C	18	100	63
Flux D	30	95	73

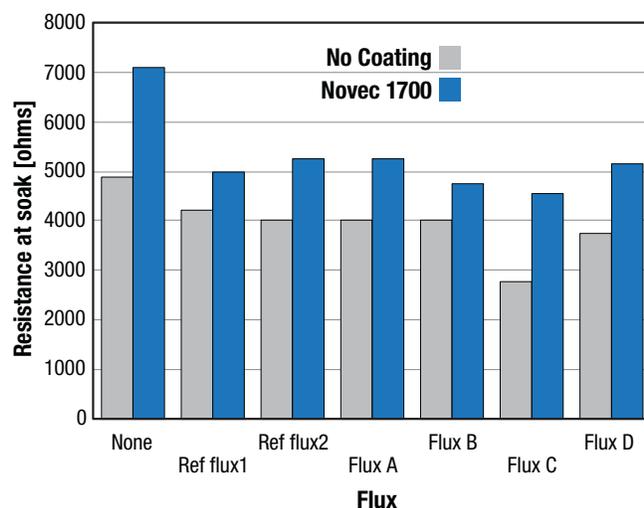
Drip water test in 0.01% NaCl solution

The 6 × 6 via matrix has a natural resistance of 4.9 kohms (no flux, no coating) in the 0.01w% NaCl solution. The application of flux and soldering will lower test pattern resistivity to around 4K ohms by adding ionic impurities (fluxed samples, no coating). Flux C is an exception (2.8K ohms, no coating), but the result may be due to the limited number of samples.

Thin protective coatings appear to provide some benefit in preventing charge carriers (Cl-ion) from traveling through when soaked in a NaCl solution. The use of Novac 1700 has demonstrated an approximately 20% improvement in resistance when compared to non-coated samples.

Note: This test is only meant to demonstrate coating behavior against chloride (Cl-) ions and is not designed to demonstrate coating properties against gaseous chlorine.

Figure 6. Results from the drip water test in 0.01%-w NaCl solution



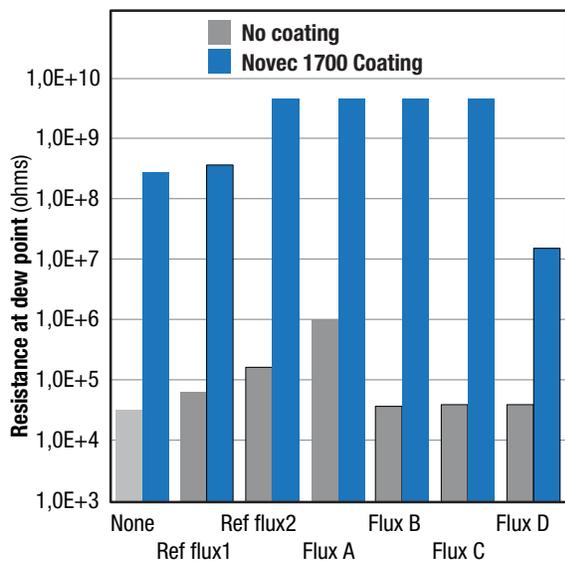
Dew point test

The 6 × 6 via matrix test pattern was used for the dew point test. This test demonstrates the effects of sudden ambient temperature and relative humidity changes. Examples of when this could occur include when taking cold appliances into a hot humid environment or when powering up long-term stored equipment in a humid environment.

Non-coated samples demonstrate problematic behavior. Initial resistance (at 23°C/45% R.H.) of the test pattern drops from 0.45G ohm (maximum measurable) to approximately 30k ohms. Test variant Flux A, having the lowest solids content and lowest measured ionic contamination (of the NVOC fluxes), shows 1M ohm resistance at dew point.

3M™ Novec™ 1700 Electronic Grade Coating samples show at least 5 decades higher resistance values than uncoated samples. The resistance change could not be measured from the Novec 1700 coated samples Ref flux 2, Flux A, B, or C, because results exceeded maximum measurable resistance (0.45G ohms). The Novec 1700 coated sample Flux D shows lower resistance, as expected, due to high solids ionic contamination (**Note. Flux D would have required a lower volume applied on the test board, due to its higher solids content. The result should be interpreted more as an excess flux application rather than a problem in flux performance.**)

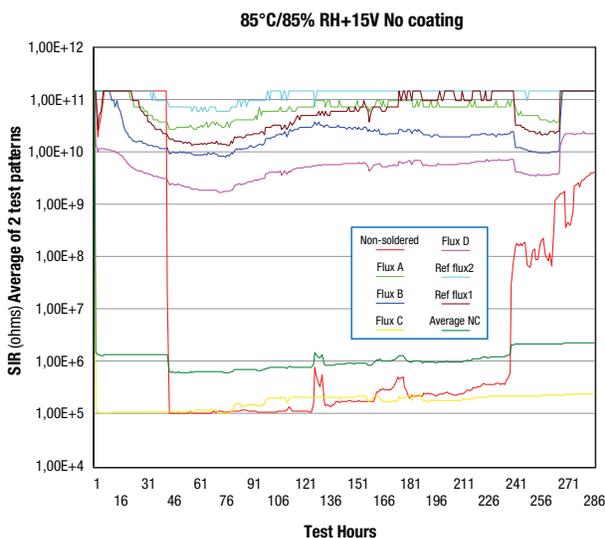
Figure 7. Results from the dew point test



Damp heat test

(85°C/85%R.H.+15V bias 240 hours, followed by 85°C/95%R.H.+15V bias 24 hours)

Figure 8. Results from 85°C/85% R.H.+15Vbias test for non-coated samples (NC states “no coating”)



Non-coated samples showed two pattern failures (the other of two patterns averaged in the results) for test variants “non-soldered” and “Flux C”. The other pattern on the same board for both variants had performed well. The results show the average.

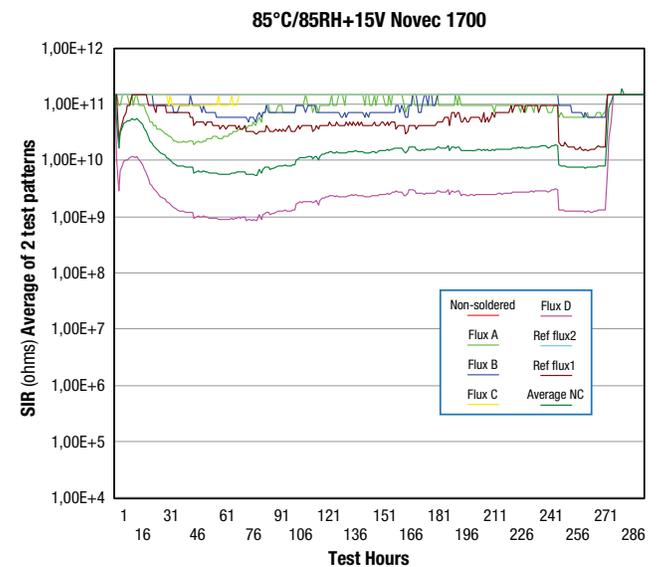
Failure analysis:

- Sample ID 1, non-coated, non-soldered sample, test pattern 1 of 2: Microscopic analysis reveals a contaminated (by unknown liquid material) area of approx. 0.2 × 0.02 mm in a 45° angle between fingers of test pattern (gap 0.2 mm). Visual examination could not reveal the nature of the contamination. No migration was found.
- Sample ID 19, non-coated, Flux C, test pattern 2 of 2: With 40X magnification, no failure location or failure mechanism could be identified. The test pattern shows some local darkening of conductor surfaces but nothing could be detected on the insulating gaps.

Failures, such as those shown here, are normally somewhat random in nature. A significant drop in SIR can be generated by minor surface effects if the PCB surface is not protected.

Test variant Flux D shows the lowest SIR due to the high solids content left on the board (this is due merely to excess application rather than the property of the flux).

Figure 9. Results from 85°C/85%R.H.+15Vbias test for Novec 1700 coated samples



Novec 1700 coated samples exhibited no failures. The coating on Flux D samples did not improve SIR when compared to the non-coated samples. Fluxes B and C performed the best: The SIR is at the measurement limit throughout the test, even during the 95% R.H./24 hour period (test hours 240-264). Non-soldered, non-fluxed and Ref Flux 2 Novec 1700-coated samples showed the maximum detectable SIR throughout the test. **Note: The red line of the non-soldered test pattern is hidden by the light blue line of test variant Ref Flux 2.**

At the end of the test, all test patterns recovered back to the maximum measurable SIR.

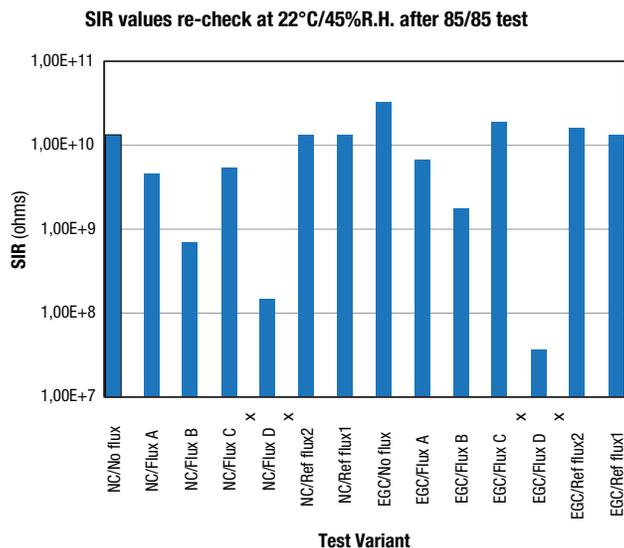
Note for all test variants:

FMI Technical Consulting had performed similar evaluations. The SIR values in those tests have been significantly lower (1-2 decades) than those measured here.

One difference with earlier measurements is that all samples in this test have been dried for 3h/75°C, settled at 21°C/24 hours, and then damp heat tested. Earlier tests have not included this pre-dry procedure. Pre-drying will remove/reduce polyalcohols and other hygroscopic and ionizing materials on sample surfaces which most probably will explain the differences.

All possible re-checks on samples, sample numbering, wiring and test system setup at VTT have been performed.

Figure 10. Results of SIR measurements 48 hours after damp heat test



SIR values were re-checked at FMI in 22°C/45% R.H. conditions. A test voltage of 15 V was fed into the test pattern while measuring the voltage drop over a 10 Mohm bias resistor. Calculations from the voltage division and bias resistor values resulted in the SIR. Results are expressed as an average of two test patterns per test variant.

Resistance levels of all test variants were lower than expected, compared to results measured at VTT. Both VTT and FMI measurement systems were thoroughly checked, and results should be considered to be valid. It is assumed that the moisture still contained within the PCB laminate after the long-term damp heat conditions could have affected the results.

Test variant Flux D shows a permanently lowered SIR, even at room temperature. Because the SIR is significantly lower than measured immediately after completion of the 85°C/85% RH test, it can be assumed that the remaining moisture within the PCB laminate and the high contamination level of variant Flux D enabled chemical reactions in the test pattern area. Visual evaluation under 40X magnification shows darkened conductor metal surfaces, especially on the positively biased electrode, but these changes are minor. Conductor gaps show signs of flux residues but no metals migration paths.

Test variant No flux (not soldered, low contamination level, all coating variants) shows the highest SIR, as expected.

IPA based reference fluxes and Flux C show higher SIR than others.

4. Conclusions, discussion

Protective coating evaluation

Protective coatings can be evaluated in many ways. The test designer should be well aware of what coating property each test is demonstrating.

The ionics contamination level under a fluorinated coating seem to affect dielectric performance. The ionic contamination level of PCB assemblies should be kept below a 2 µg/cm² NaCl-equivalent level for acceptable results. The flux wet weight (g/m³) in wave soldering systems should be controlled carefully. When the level of ionic contamination is decreased below 1.5 µg/cm², according to test results, final performance is comparable to cleaned assemblies. Cleaning PCB assemblies prior to coating is, however, recommended for the highest reliability requirements.

The 3M™ Novac™ 1700 coating provided a significant improvement in all test pattern SIR and general reliability in all tests against non-coated samples. This demonstrates that the Novac 1700 coating was found to be compatible with all tested fluxes.

The electrical contacting properties through the coating layer are found to be acceptable. The coating will allow reliable electrical contacting. For extremely low contact pressure connectors, separate testing will be necessary.

Application of thermal energy (curing) over air drying did not significantly change the layer properties in electrical contacting. Achieving electrical contact through cured coating film was, in fact, slightly easier.

NVOC fluxes evaluation

General

The SIR performance was found to be mainly dependent on the amount of ionic contamination left on the board after soldering. All NVOC fluxes tested exhibit different solids content, which has to be taken in account when adjusting flux application. Recommended flux application quantities based on the presented SIR and soldering performance tests are given here:

Table 8. Recommended flux application

Flux	Flux Wet Weight (g/m ³)	Flux Solids Weight (g/m ³)	Evaluated ionics left on board after soldering µg/cm ² NaCl equivalent
A	40-50	1.2-1.5	0.96-1.20
B	28-33	1.29-1.52	1.00-1.22
C	35-42	1.26-1.51	1.00-1.21
D	17-22	1.45-1.87	1.16-1.50

Test results suggest that the amount of solids applied to the board will result in approximately 1/1250 µg/cm² ionic contamination (0.08%).

It may prove difficult to apply a small enough quantity of Flux D. Flux D requires an accurate and stable spray fluxer for constant results.

Results of the NVOC fluxes, when compared to the IPA-based reference fluxes, were generally found to be equal for both soldering results and SIR/leakage current.

Note 1: When implementing water-based flux systems to wave soldering equipment, the compatibility of machine structures and ventilation with water-based materials has to be evaluated carefully. Most commercial wave soldering equipment will exhibit some problems that need to be overcome.

Note 2: Water-based fluxes showed a tendency to flow along tilted PCB bottom surfaces toward the back-end of the board. It may be a good idea to use a stand-alone fluxer with a horizontal conveyor. Because most of the water has been evaporated, the PCB may then be transferred on a tilted conveyor. The 3D form of a real PCB assembly surface may reduce the flowing tendency.

Flux A

Flux A has the lowest solids content (3.0%-w) of the tested fluxes. It was found difficult to apply enough flux to achieve sufficient activity in the soldering process. Along with flux, plenty of water has to be applied, which may be difficult to remove prior to preheating/soldering. Despite its low solids content and low acid number, Flux A had acceptable soldering properties but may perform best in single wave systems. Despite low ionics detected on the board, Flux A only had an average SIR performance.

Flux B and C

Fluxes B and C represent an average solids content (4.6 and 3.6 respectively). Soldering performance was found to be very good. Performance in the damp heat and other tests was found to be acceptable (mostly dependent on the ionics contamination level).

Flux D

Flux D represents exceptionally high solids content and will require very careful spray application. High solids content may prove useful in process, as much less water will be sprayed on boards, thus making it easier to dry and preheat assemblies.

Flux D suffered from excess application in both the process capability and SIR tests. Some spray systems may not be able to spray low flux quantities accurately enough.

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