No-Clean Flux:

The Use of Multiple Vendors Can Produce a Reliability Time Bomb

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Incompatibilities between similar solder flux products supplied by different vendors can create serious product reliability problems if not detected during initial product qualification. Due to the multitude of new fluxes being introduced to the industry and the proprietary and varied nature of these formulations, such incompatibilities are certain to arise occasionally.

At the Foxboro Co. (Foxboro, MA), during recent evaluations of no-clean fluxes supplied by multiple vendors, an incompatibility was found between one vendor's no-clean solder paste flux and a cored-wire no-clean flux that had been approved and in use at the facility for some time. This incompatibility resulted in excessive leakage current and could have had serious consequences for Foxboro's product line. The company's evaluation results underscore the need not only to evaluate and qualify new products, but to test for incompatibility between already approved or qualified materials and new ones.

No-Clean Flux/Paste Qualification

The Foxboro Co., a manufacturer of industrial process control systems and instrumentation, assembles both surface-mount and mixed-technology boards. The current phase-out of CFC solvent cleaning prompted the company to investigate alternative technologies. A no-clean soldering process was chosen, for which Foxboro had to find an appropriate no-clean solder paste, cored solder flux and wave-solder flux to replace the current RMA/RA chemistry. Through an evaluation process, two suppliers were selected (vendor X and vendor Y), and the following six-step final qualification procedure was used:

1. Samples of flux, solder paste and cored solder were requested, as well as product data sheets and material safety data sheets from vendors X and Y.
2. Leakage current/surface insulation resistance (SIR) testing was performed:
   a) Three IPC-B-25 boards per product tested were used; finger patterns were characterized for 1 X 10^{-10} amps max.
   b) Flux was brushed or liquid dipped on a test coupon and reflowed in an IR reflow oven using a standard profile.
   c) The initial leakage current was logged.
   d) The test chamber was set for 40°C, 95% RH and 30V bias.
   e) The chamber was run for eight weeks, with periodic checks during the test.
   f) Final measurements were taken after 1,344 hrs. at 1 X 10^{-8} amps max.
3. Ionic cleanliness testing was performed.
4. If found acceptable for solder paste, three test board assemblies were run through the process using samples of solder paste. Screening was checked. Solderability and flux residue were visually checked.
5. If still acceptable, regular production assemblies were tried. Screening was checked. Solderability and flux residues, ionic cleanliness and defect analysis were visually checked.
6. Results were formalized.

Initial Procedures and Results

Preparation for the qualification procedure involved a rigorous cleaning process to scrub off any dirt or debris that might cause shunting between the adjacent conductors. After this step-by-step wash, rinse and scrub process, the wires were attached. Electrical measurements were then taken, with only 1 X 10^{-11} amps of leakage current allowed. The comb patterns on the test coupons were tinned with copper.

During preparation of the test coupons, bus wires and test leads were hand soldered onto each coupon for measurement purposes. The no-clean cored-wire solder that was used was supplied by vendor Y and had been previously qualified and in use for rework and touch up at Foxboro for some time. Since the solder was a no-clean material, the residue was not removed.

Samples of flux supplied by vendor Y were brushed onto the comb patterns on the test coupons, reflowed and then subjected to the evaluation procedure. The current leakage test results indicated that vendor Y's no-clean material was usable but that its performance was marginal at best. Vendor X's material was then evaluated in the same manner. In both cases, the no-clean flux was brushed over existing no-clean residue from Y's cored wire solder on the test and bus wires.

First Signs of Trouble

Tables 1 and 2 summarize the test results using 6-mil and 12.5-mil line/space patterns on the test coupons. The tables indicate initial leakage current measurements and the range of measurements while samples were in the chamber, as well as the final measurement (degradation) after the samples were removed from the chamber and dried. The 6-mil spacing was intended to address SMT assembly applications, and the 12.5-mil spacing was meant to address wave-solder/through-hole applications. Initial results gave no sign of trouble, but the scenario changed dramatically as life and reliability tacking progressed over time.

Table 1*

Supplier	Material	Initial (Dry)	Leakage Current (Amps) During Exposure*	Final (Dry)
Control	None	8.5×10^{-11}	7.2×10^{-10} to 1×10^{-5}	4.35×10^{-8}
X	RMA Solder Flux	3.4×10^{-11}	5.95×10^{-8} to 1.25×10^{-2}	7.75×10^{-6}
X	RMA Solder Paste Flux	3.25×10^{-11}	8.4×10^{-8} to 1.75×10^{-3}	2.6×10^{-7}
Y	Solder Paste Flux	4.15×10^{-12}	7.25×10^{-8} to 3.3×10^{-7}	1.65×10^{-11}
Y	Solder Paste Flux	6.9×10^{-12}	1.7×10^{-7} to 4.8×10^{-7}	1.8×10^{-12}
X	Wave-Solder Flux	9.6×10^{-10}	3.9×10^{-8} to 1×10^{-4}	1.7×10^{-11}
Y	Wave-Solder Flux	1.4×10^{-11}	1.3×10^{-11} to 5.3×10^{6}	1.7×10^{-11}

Test results using 6 mil line/space patterns on test coupons.[1]

Although X's product yielded higher SIR values overall, it became clear that when the X and Y no-clean fluxes combined, they produced an unstable, corrosive material. The degradation was dramatic enough to be visible. The product produced by the blending of two harmless materials was actively destructive.

The significant increase in leakage current in a short period of time indicated that whatever residue was left on the surface tended to be hygroscopic (taking in moisture) and caused some shunting. This was only true of the test coupons that mixed the no-clean residues of vendor X's no-clean flux and vendor Y's cored-wire flux.

Table 2*

Supplier	Material	Initial (Dry)	Leakage Current (Amps) During Exposure*	Final (Dry)
Control	None	1.65×10^{-11}	6.75×10^{-10} to 1.3×10^{6}	2.15×10^{-11}
X	RMA Solder Flux	1.45×10^{-11}	1.5×10^{-8} to 1.65×10^{-4}	3.9×10^{-7}
X	RMA Solder Paste Flux	2.35×10^{-11}	3.6×10^{-8} to 4.15×10^{-3}	8.4×10^{-7}
Y	Solder Paste Flux	2.5×10^{-12}	3.95×10^{-8} to 1.65×10^{-7}	1.7×10^{-11}
Y	Solder Paste Flux	3.1×10^{-12}	4.8×10^{-8} to 8.25×10^{8}	1.75×10^{-11}
X	Wave-Solder Flux	4.5×10^{-12}	1.55×10^{-8} to 2.4×10^{-5}	1.6×10^{-11}
Y	Wave-Solder Flux	4.7×10^{-12}	8×10^{-11} to 2.6×10^{-7}	1.8×10^{-12}

Test results using 12.5 mil line/space patterns on test coupons.[2]

During visual inspection, green corrosion was noted on the hand-soldered bus wire. In all likelihood, this was corrosion of the copper, but no additional analysis has been performed. Furthermore, it is not known whether the corrosion caused the shunting or the incompatible reaction of the fluxes, but the consensus was that the corrosion was more likely to be a consequence of the reaction, rather than a cause. The options available at this point included SEM examination and/or energy-dispersive X-ray spectroscopy (EDS) to determine what the corrosion products were and thereby validate the assumption of chemical incompatibility.

Multiple Products, Multiple Vendors

Before this evaluation, it was assumed that one could pick and choose the best products from several vendors if necessary to fill requirements. This approach must now be reconsidered in light of the potential incompatibilities that may be encountered with multiple vendor sources.

When multiple vendors are involved, it may be necessary to test all products for compatibility with one another. Some vendors may have to be eliminated, even though their products are excellent; previously qualified products may

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*Exposure condition: 40°C, 95% RH, eight week duration (MIL-STD-202, Method 103, Test Condition D)
Test was terminated after five weeks based on failure to test criteria.
Leakage current test criteria: 1×10^{-8} amps max. for SMT PWAs-high impedance circuitry; 1×10^{-7} amps max. for through-hole-standard circuitry; three orders of magnitude change during exposure.

have to be re-qualified; or alternate process steps may have to be implemented (such as cleaning), when multiple materials are used that are known to be incompatible. Whenever possible, however, all needs should be met by a single vendor.

Discussion

When changing to a no-clean process, flux compatibility is critical. Some manufacturers use the same designation for all no-clean products, which can have detrimental effects on the final assembly. Typically, wave-soldering flux, cored-wire solder flux and solder paste must be cross-checked.

Surprisingly, residues from an in-line process are less of a problem than those from a batch or hand assembly process. During an in-line process, all flux sees proper activation heat, rendering it neutral. In a hand-soldering operation with rosin-based fluxes, a liquid flux is often used in conjunction with a cored wire. But the liquid flux never sees the necessary passivating temperature. The most important fact to remember about no-clean soldering is that no-clean fluxes are only safe after they, in their entirety, have seen passivation temperature.

Regarding the reaction itself, Foxboro is looking for possible side reactions from the combination of one or more dicarboxylic acids, namely malic, adipic and oxalic, and an amine that will yield a green or grayish product at high temperatures (abietic, ascorbic or citric).

Two reactions typical of carboxylic acids are of interest:

- Conversion into amides (usually requires a chloride to form an acid chloride before reacting with the amine).
- The Hell-Volhard-Zelinsky reaction (a nucleophilic displacement and elimination of the alpha-halogenated carboxylic acid).

With dicarboxylic acids, there are two acid groups available to react with the amine. As a result, several products (rather than a single one), may be the cause of the problem. The typical reaction of the amines is conversion into substituted amides. Reactants are a primary or secondary amine and an acid chloride of a carboxylic acid or dicarboxylic acid.

Because Foxboro is reacting dicarboxylic acids, which may or may not react both carboxyl groups with an amine, reaction with a large number of different products is possible.

The end user should watch for potential incompatibilities between old products still in use and new products.

Conclusion

The potential for intermittent and ultimately catastrophic failure of a circuit over time due to flux incompatibility is cause for serious concern. By relying on a single source for all flux products, the end user may be safe, but if multiple vendors are sourced, all products should be evaluated for compatibility. In addition, the end user should watch for potential incompatibilities between old products still in use and new products, even if they are from the same supplier. No matter what the result, the impact will be significant.

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