Evaluation of No-Clean Flux Residues Remaining After Secondary Process Operations

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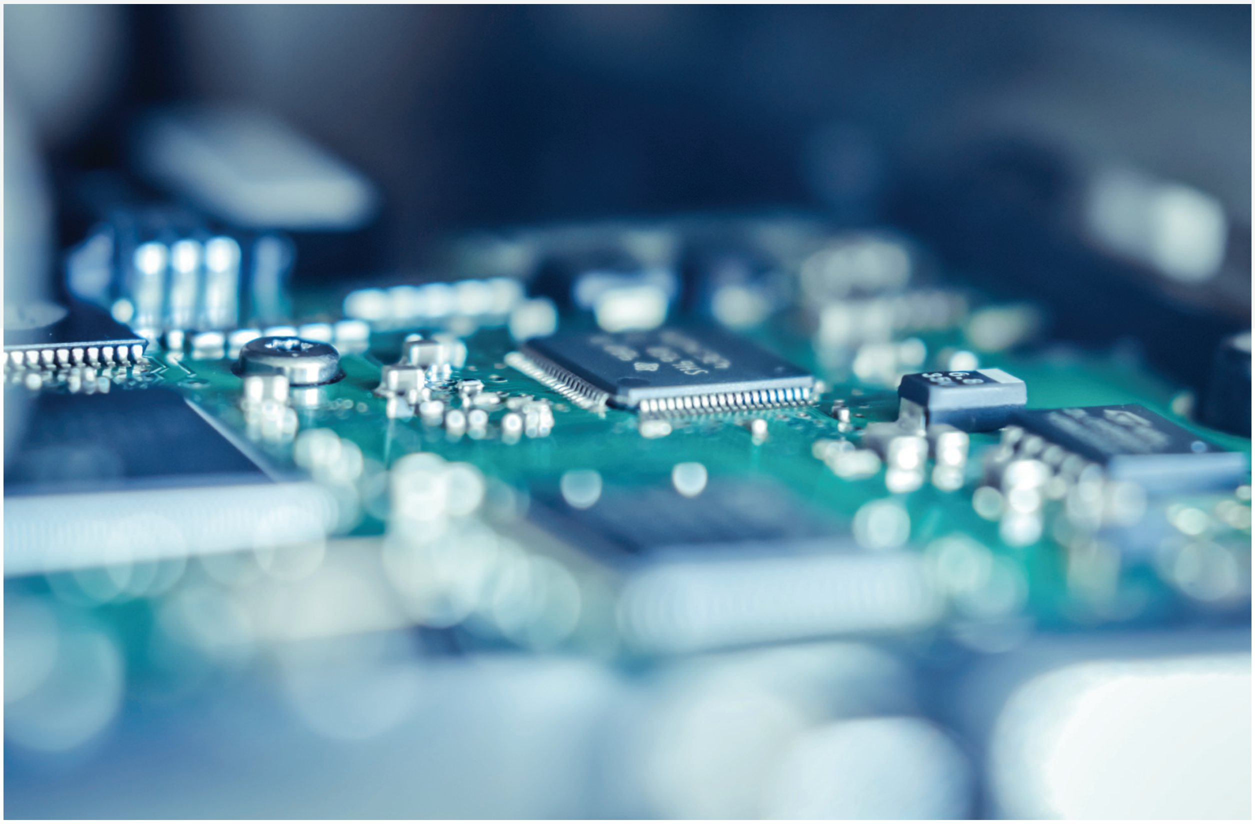

Abstract

In an ideal world, manufacturing devices would work all of the time, however, every company receives customer returns for a variety of reasons. If these returned parts contributed to a fail, most companies will perform failure analysis (FA) on the returned parts to determine the root cause of the failure. Failure can occur for a multitude of reasons, for example: wear out, fatigue, design issues, manufacturing flaw or defect. This information is then used to improve the overall quality of the product and prevent reoccurrence.

If no defect is found, it is possible that in fact the product has no defect. On the other hand, the defect could be elusive and the FA techniques insufficient to detect said deficiency.

No-clean flux residues can cause intermittent or elusive, hard to find defects. In an attempt to understand the effects of no-clean flux residues from the secondary soldering and cleaning processes, a matrix of varying process and cleaning operation was investigated. Of special interest, traveling flux residues and entrapped residues were examined, as well as localized and batch cleaning processes. Various techniques were employed to test the remaining residues in order to assess their propensity to cause a latent failure. These techniques include Surface Insulation Resistance[1] (SIR) testing at 40^{0}C/90% RH, 5 VDC bias along with C3[2] testing and Ion Exchange Chromatography (IC). These techniques facilitate the assessment of the capillary effect the tight spacing these component structures have when flux residues are present. It is expected that dendritic shorting and measurable current leakage will occur, indicating a failing SIR test. However, since the residue resides under the discrete components, there will be no visual evidence of dendritic growth or metal migration.

Keywords: No-clean, Flux, Residue, Process, Fails, SIR, R0L0, R0L1, C3, and IC

Introduction

Some components are known to have a propensity for a higher level of failure during product operation. There are any number of reasons for this. One such reason is poor electrical design. Another reason is poor workmanship, or using components in applications for which they were never intended. As the industry continues its push towards miniaturization with higher circuit density and much tighter spacing, the opportunities for shorts or electrochemical migration (ECM) between adjacent conductors exponentially increases.[3]

Quad Flat No-Lead packages (QFN's) often seem to have a higher failure rate than other similar packages.[4] For this reason, they are of special interest in this study. They have no standoff, and depending on the ground, voltage, and signal configuration, they can entrap flux between signal pads and the ground pads. A mini-chamber is often created in this region between the leads, ground, and voltage pads.[5,6] This mini-chamber prevents the escape of volatiles from the flux during the reflow operation. In this environment, the flux remains viscous rather than going through a phase change to a benign glassy state (BGS).[5] In this viscous state, flux ions are more mobile, enabling ECM and dendrite growth. It is this condition that this report wishes to evaluate more closely.

Experiment

The test vehicle selected for this experiment was the Umpire 2 test vehicle, displayed in figure 1. Two sites were used for this evaluation, the LCC1 and QFN sites, outlined in figures 2 and 3.

As previously stated, the QFN sites were selected due to prior experience of them frequently being associated with printed circuit board assembly (PCBA) failures. The LCC1 sites, the SIR comb sites around the periphery, were selected as sites where solder paste is printed on alternating plus/minus mounting SIR pads. The comb pattern is also desired to simulate a poorly vented reflow condition,[7] which was the focus of this study. The experiment was run without components on the selected sites so that the flux, and any ECM or dendritic growth will be fully visible.

The variables targeted for this study are solder paste and QFN versus LCC1 sites. Two different solder pastes were selected, an R0L0 and a R0L1, the first having no detectable halides and R0L1 having less than 0.5% halides.[8] Both the R0L0 and R0L1 fluxes are from the same general flux family. Half of the test vehicles were assembled with R0L0 solder paste while the other half was assembled with R0L1 solder paste.

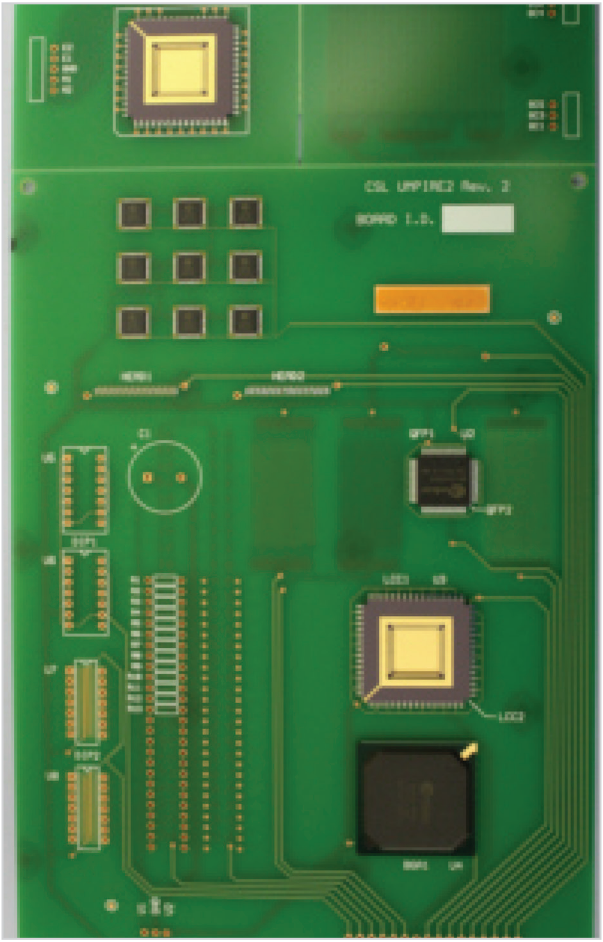

Figure 1: Umpire 2 Test Vehicle

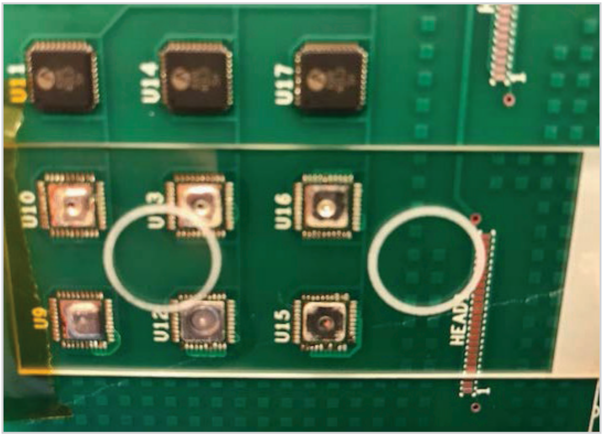

Figure 2: QFN Sites

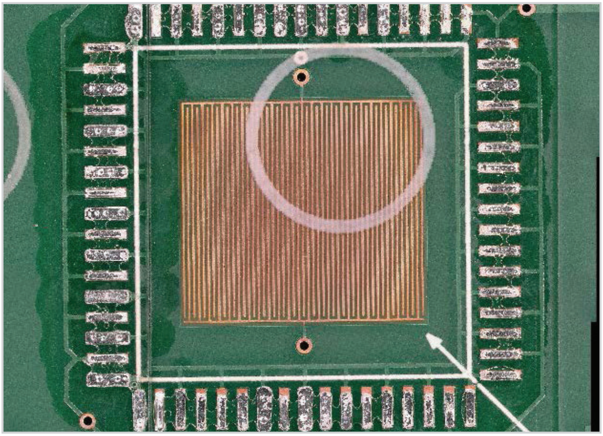

Figure 3: LCC1 Site

Process:

Solder paste was applied to the two locations of interest on the Umpire 2 test vehicle. One of the test vehicles was built as a control with no solder paste present. A glass slide was secured in place over the sites with polyimide tape prior to reflow. The glass slide was used in order to simulate the presence of a low stand-off component6 and to create a window to view the solder and flux, which would normally be enclosed by a component. Since there was no component for the solder to wet, in order to "pull" down the glass slide, a weight was placed on top of the glass slide to hold the glass slide in place and to counteract the surface tension of the solder. This was an attempt to compensate for a one-sided solder joint. A one-sided solder joint is more likely to dome and slightly lift the glass slide off of the site.

The test vehicles were then processed through a reflow oven with a standard reflow profile. Next, the test vehicles underwent surface insulation resistance testing.

Test:

The test vehicles were divided to receive two separate treatments, table 1 details the different SIR treatments. All of the parts were first subjected to a 40^{o}C and 90% relative humidity (RH) environment for 168 hours. After the first exposure, all parts went through a standard dry out to ambient conditions followed by an insulation resistance measurement. Next, the parts were subjected to two different treatment sets: 85^{o}C/85% RH and 85^{o}C/63% RH for 168 hours. After the second treatment, 85^{o}C/85% RH and 85^{o}C/63% RH, both sets went through a dry out to ambient conditions and another insulation resistance measurement. SIR testing was run with a 5 VDC bias.

Table 1: Surface Insulation Resistance Treatment Sample Size

	Temperature/Relative Humidity	
	40/90=>85/85	40/90=>85/63
R0L0	9	5
R0L1	10	5
Control	-	1

Results:

There were two response variables, measured surface insulation resistance and visible dendritic growth. If the surface insulation resistance value dropped below 1 x 108 ohms, this was considered an electrical fail. If there were visible dendrites found through visual inspection, this was also considered a fail. Tables 2 and 3 lists the insulation resistance fails from testing on a per card basis.

Table 2: Insulation Resistance Fails of the First Treatment

	40/90		85/85	
Flux	QFN	LCC	QFN	LCC
R0L0	1/9	0/9	9/9	6/9
R0L1	1/10	5/10	9/10	10/10
Control	-	-	-	-

Table 3: Insulation Resistance Fails of the Second Treatment

	40/90		85/63	
Flux	QFN	LCC	QFN	LCC
R0L0	0/5	0/5	0/5	0/5
R0L1	0/5	5/5	0/5	0/5
Control	0/1	0/1	0/1	0/1

The results of the two secondary treatments, 85°C/85% RH and 85°C/63% RH, are very different. Those assemblies in the 85°C/85% RH treatment had continuous insulation resistance fails and most of the remaining nets failed as well. In contrast to this, in the 85°C/63% RH, all of the failing nets transitioned back to passing nets and there were no additional failing nets. With the same temperature, and a RH difference between 63% and 85%, the contrast between the results was noteworthy. This would indicate that relative humidity is a more significant variable than temperature.

Figure 4 shows a plot of time versus resistance for a typical non-failing site that went through 40°C/90% RH. Figure 5 shows a plot of time versus resistance on a site that failed through 40°C/90% RH, as indicated by the drop in resistance.

Table 4 lists the visible dendrites found through inspection at the end of the 85°C/85% RH test.

Resistance v. Time

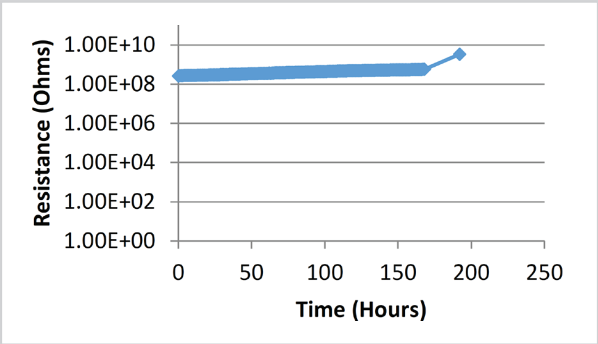

Figure 4: SIR Resistance, 40/90 R0L1 LCC Site Pass

Resistance v. Time

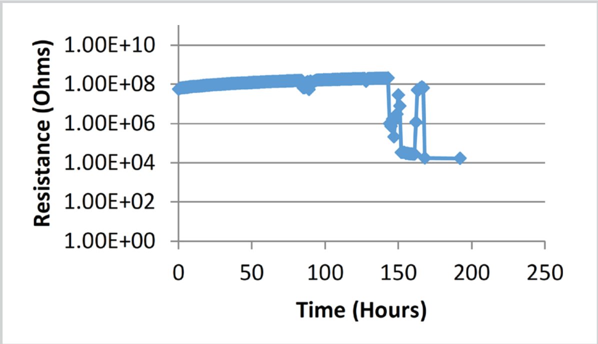

Figure 5: SIR Resistance, 40/90 R0L1 LCC Site Fail

Table 4: Visual Fails		
	First Treatment (40/90 => 85/85)	
Flux	**QFN**	**LCC**
R0L0	2/10	0/9
R0L1	2/10	7/10

Figures 6 and 7 shows one of the several dendrites that were found through inspection at the end of 85^{o}C/85% RH testing. Notice that not all of the dendrite is in focus, due to the depth of field; this image was taken looking down through the glass slide. In this image, the flux reaches from the surface of the PCB to the bottom of the glass slide.

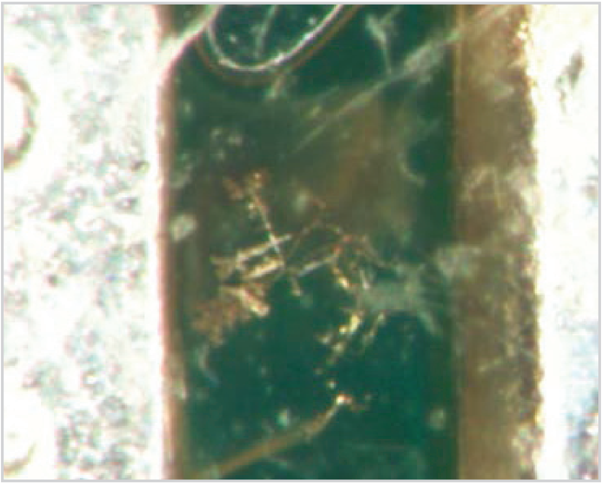

Figure 6: Example of a Dendrite Found through Visual Inspection

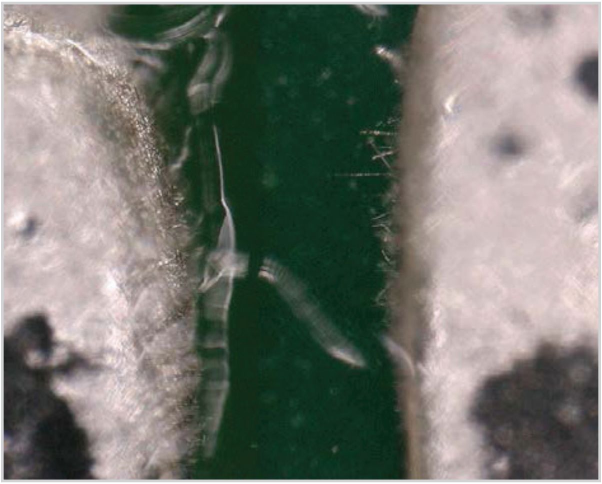

Figure 7: Example of a Dendrite Found though Visual Inspection

All five of the healed assemblies that failed in 40^{o}C/90% RH and healed in 85^{o}C/63% RH were again placed in 40^{o}C/90% RH with a 5 VDC bias for 24 hours. All five returned to their failing condition. On one of these assemblies, an inspection for dendrites was performed. An observed dendrite can be seen in figure 8.

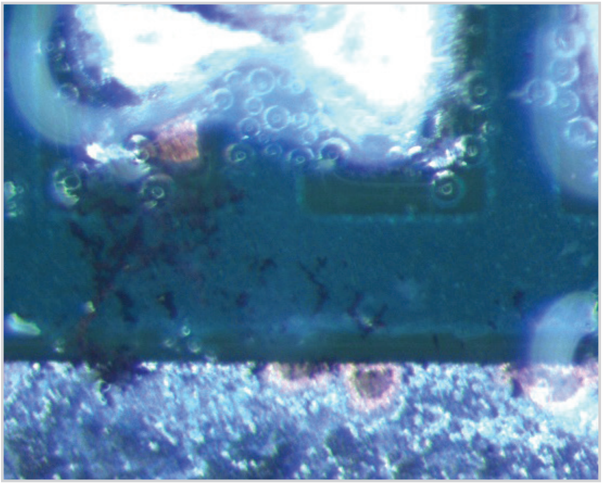

Figure 8: Dendrites observed post after 40/90 followed by 85/63 followed by 40/90 testing.

Post visual inspection, eight of the sample sites went through SIR testing at 40^{0}C/90% RH, 85^{0}C/85% RH, and again 40^{0}C/90% RH were selected for local ionics testing. Four of the sample sites were made with a R0L0 flux and the other four of the sample sites were made with a R0L1 flux. All of the R0L0 samples passed local ionics testing and all of the R0L1 samples failed local ionics testing. The aliquots of samples removed from the sites were submitted for Ion Exchange Chromatography (IC) testing. Again the sample sites made with R0L0 flux all passed and the sample sites made with the R0L1 flux all failed. The failing species was the halide, specifically the bromide ions. The results of IC testing are shown in table 5.

Table 5: Local Ionics and IC Test Results

all values in ug/in^2		Ion Chromatography (Dionex ICS 3000 at Foresite) n/a = not applicable																	C3 Tester	
	Sample Description	F^-	$C_2H_3O_2$	HCO_2^-	Cl^-	NO_2^-	**Br^-**	NO_3^-	PO_4^{3-}	SO_4^{2-}	WOA	MSA	Li^+	Na^+	NH_4^+	K^+	Mg^{2+}	Ca^{2+}	Results	Time(sec)
Foresite recommended limits (NC Paste SMT)		<3	<3	<3	<3	<3	**<10**	<3	<7	<3	<25	N/A	N/A	N/A	N/A	N/A	N/A	N/A	Clean	>120
ID	**C3 Extractions of each sample site**																			
1	ROL1 #1 QFN	0	2.98	0	0.26	0	**12.65**	0.05	0	2.14	0	0	0	3.65	3.05	0	0	0	Dirty	74
2	ROL1 #1 LCC	0	3.66	0	0.99	0	**11.54**	0.01	0	1.25	0	0	0	3.01	2.95	0	0	0	Dirty	104
3	ROL1 #2 QFN	0	2.14	0	0.69	0	**15.64**	0.04	0	1.98	0	0	0	3.62	3.11	0	0	0	Dirty	81
4	ROL1 #2 LCC	0	2.06	0	0.35	0	**12.34**	0.08	0	1.43	0	0	0	2.78	2.77	0	0	0	Dirty	98
5	ROLO #1 QFN (#U9)	0	0.65	0	1.34	0	**0.05**	0	0	0.25	12.35	0	0	1.36	0.69	0	0	0	Clean	180
6	ROLO #1 LCC	0	0.28	0	0.70	0	**0.01**	0	0	0.36	10.21	0	0	1.09	0.58	0	0	0	Clean	180
7	ROLO #2 QFN (#U9)	0	0.85	0	1.34	0	**0.05**	0	0	0.27	13.06	0	0	1.33	0.99	0	0	0	Clean	180
8	ROLO #2 LCC	0	0.27	0	0.70	0	**0.01**	0	0	0.36	11.21	0	0	0.89	0.55	0	0	0	Clean	180
9	ROL1 Paste only 0.1g Raw	0	7.84	0	0.15	0	**30.71**	0.08	0	1.88	0	0	0	15.64	5.53	0	0	0	Dirty	53
10	ROLO Paste only 0.1g Raw	0	2.87	0	0.03	0	**0.01**	0	0	0.51	107.84	0	0	2.71	1.04	0	0	0	Clean	178

Analysis:

There are several significant findings from the SIR results and visual inspection. The expected result was that the QFN sites would fail at much greater frequency than the LCC sites, however, the LCC site had many more fails, both electrical and visual, than the QFN sites. The QFN sites were expected to have a higher failure rate due to their propensity to create mini-chambers, but mini-chambers were observed more frequently on the LCC sites. Fourteen locations with dendrites were found on the assemblies. All fourteen were processed with the R0L1 solder paste flux. None were found on the assemblies with the R0L0 solder paste flux.[9] It was in the LCC mini-chambers that all of the dendrites grew. On the QFN sites where dendrites were observed, it was determined that only one mini-chamber was formed due to the surface tension of the solder causing doming of the solder such that the glass slide was lifted off of the flux. At the same time, the geometry of the LCC mounting pads did not cause significant doming of the solder so a mini-chamber of flux was created between the LCC mounting pads with much greater frequency.

The base non-failing flux insulation resistance in 85^{0}C/85% RH was between one and two orders of magnitude less than it was in 40^{0}C/90% RH, as seen in table 6. These measurements were made where there were no electrical fails. This decrease in insulation resistance would indicate a much greater degree of ion mobility in the 85^{0}C/85% RH SIR environment.[10]

Table 6: Average Insulation Resistance as a Function of Flux Type and SIR Environment

SIR Environment	**40/90**		**85/85**	
Flux Type	**R0L0**	**R0L1**	**R0L0**	**R0L1**
Base Flux Resistance	2.0×10^9	2.0×10^8	2.0×10^7	5.0×10^6

The order of magnitude difference in insulation resistance between the R0L0 and R0L1 flux subjected to the same conditions, 40^{0}C/90% RH, was not expected. So, in addition to the 85^{0}C/85% RH having an increase in ion mobility over 40^{0}C/90% RH, the presence of halides, even in small quantities, also increased the ion mobility within the flux.

When exposed to ambient conditions, the passing samples from the 85^{0}C/85% RH environment base non-failing flux insulation resistance increased to between 1×10^{11} and 1×10^{12} within three to four hours. To the limits of detection, the circuits were open.

For the assemblies which were run at 85^{0}C/63% RH, the base non-failing flux insulation resistance was significantly higher than either the 40^{0}C/90% RH or the 85^{0}C/85% RH cells. See table 7.

Table 7: Base Non-Failing Flux Insulation Resistance at 85^{0}C/63% RH

SIR Environment	85/63	85/85
Flux Type	**R0L0**	**R0L1**
Base Flux Resistance	1.3×10^{11}	1.0×10^{11}

Referring back to table 2 where the five fails from the 40^{0}C/90% RH R0L1 cell were observed, all five fails healed in the 85^{0}C/63% RH environment. There were no electrical fails observed after the 85^{0}C/63% RH condition. The high level of the non-failing insulation resistance may explain why the samples healed. The level of electrical current is very low. On the other hand, it is also known that dendrites can form and disappear in a matter of minutes. High current flow between test measurement intervals can destroy dendrites that bridge a gap between conductors, which would have otherwise caused a short.[11] Thus, failures caused by the presence of dendrites are intermittent in nature.[12] This may also be a reason why an assembly electrically fails in application but passes by the time it gets back to be tested. The medium for the electrical fault may have dried in transit and no longer conducts electricity at a noticeable level. As can be seen in table 7, the reduction in moisture increases the insulation resistance and also reduces the difference in base non-failing insulation resistance between the R0L0 and the R0L1 fluxes.

It is clear that by the difference in the number of electrical fails and presence of dendrites, which even the presence of a low amount of halides in some environmental conditions has a deleterious effect on the insulation resistance reliability. In corrosion chemistry, it is well known that halides accelerate the metal corrosion reaction. If the halide was not consumed in the reaction, it would be considered a catalyst.

Discussion/Conclusions:

There are several conclusions that can be made from this study:

1. In previous reports it has been stated that it is required for flux to achieve full reflow temperatures to become safe.[13] In this report it shows that when flux is effectively encapsulated, entrapping volatiles and activators, achieving full reflow may not be enough to render the flux safe.
2. The retention of volatiles in the flux, such that the flux remains viscous and does not phase change to a benign glassy state, allows for greater ion mobility and the potential to create ECM and dendrites.
3. In this viscous state, flux activators such as halides can have a much greater deleterious effect.
4. When a PCBA has components which entrap flux, such as QFN's, in a crevice, pocket or mini-chamber, thus preventing a full dry out or cure of the flux, those assemblies may be prone to grow dendrites.
5. Local ionics test and IC test results both support the presence and mobility of ions; in this case Br-.
6. Ion mobility in flux is inversely related to flux insulation resistance.
7. There is a relationship between ion mobility, insulation resistance, and the probability of creating ECM or dendrites.
8. A twenty two percent increase in relative humidity had a much greater effect on ion mobility, the probability of creating ECM or dendrites, than a 45^{o}C increase in temperature.
9. Product that has failed in an application but passes system test may have dried out in-transit, thus reducing ion mobility and disrupting the failing site, causing no defect to be found.
10. Rehydrating the electronic assembly prior to system test with a non-condensing moisture, 40^{o}C/90% RH, can improve the probability of finding the defect, which might otherwise be considered "no defect found."

All three of these factors work to increase the mobility of ions in flux. It was also noted that the non-failing insulation resistance is related to the ion mobility; a lower insulation resistance also correlates to an increased ion mobility. When these variables work in conjunction with each other, they can easily lead to ECM and dendritic growth.

Acknowledgements:

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References:

[1] IPC 9201A, “Surface Insulation Resistance Handbook.” IPC Press, 2007, Bannockburn, IL.

[2] C3 is a registered trademark of the Foresite Company. Kokomo, Indiana.

[3] Bixenman, M., McMeen, M., & Tynes, J. “Reliable Micro-Electronic Assembly Process Design Test Methods-A Non-Standard Approach.” 2017 SMTA Toronto Expo and Technology Forum Proceedings.

[4] Bixenman, M., Chan, J. & Loy, T. C., “Advanced Packaging and Electronic Assembly Cleaning Fluid Innovation.” 38th International Electronic Manufacturing Technology Conference 2016 proceedings.

[5] Isaacs, P. & Munson T., “What Makes No-Clean Flux Residue Benign.” SMTA Pan Pacific Conference, 2016 Proceedings.

[6] Tellefsen, K., “SIR and ECM Testing of Soldering Materials vs. Soldering Processes.” 2015 SMTA International proceedings.

[7] Lee, N-C., & Chen F., “A Novel Solution for No-Clean Flux not fully dried under Component Terminations.” Presentation at the Boston Chapter of the SMTA

[8] IPC J-STD 004, “Requirements for Soldering Fluxes.” IPC Press, 2004, Bannockburn, IL.

[9] Guene, E. & Ducoulombier, A., “Reliability Study of No Clean Chemistries for Lead Free Solder Paste in Vapour Phase Reflow.” 2016 18th Electronics Packaging Technology Conference Proceedings.

[10] Bixenman, M., Lober, D., McMeen, M. & Tynes, J. “Characterizing Materials at the Component Interface can Improve Reliability.” 2016 SMTA Houston Expo and Tech Forum Proceedings.

[11] Michalkiewicz, R., Green J. and Opperhauser, S., “Surface Insulation Resistance Testing of Solder Pastes and Fluxes.” 2001 SMTA Pan Pacific Conference proceedings.

[12] Tegehall, P. E., “Impact of Humidity and Contamination on the Surface Insulation Resistance and Electrochemical Migration.” Chapter 10, The ELFNET Book on Failure Mechanisms, Testing Methods, and Quality Issues of Lead-Free Solder Interconnectors, DOI: 10.1007/978-0-85729-236-0 10, Springer-Verlag London Ltd., 2011.

[13] Isaacs, P., Kobeda, E. & Zhang, J., “Pb-free Flux: A Chemical View of Reliability.” SMTA Pan Pacific Conference, 2014 Proceedings.