SECOND ROUND ROBIN EVALUATION OF INEMI CREEP CORROSION QUALIFICATION TEST

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ABSTRACT

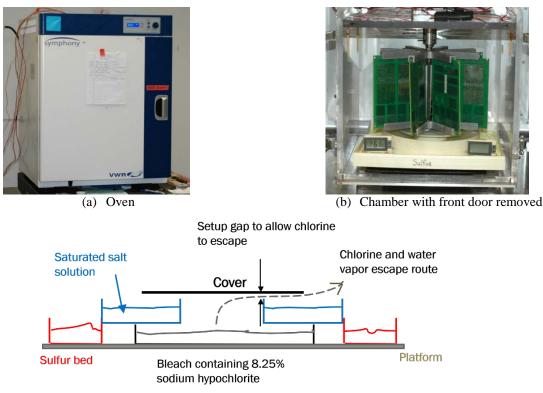
Creep corrosion on printed circuit boards (PCBs) is the corrosion of copper metallization and the creeping of the copper corrosion products across the PCB surfaces to the extent that they may electrically short circuit neighboring features on the PCBs. The paper will report the results of the second-round robin evaluation of the iNEMI creep corrosion qualification test and compare them to the first round robin test and to the recent mixed-flowing gas test results on the same iNEMI test PCBs. The modifications to and the differences in the 1^{st} and the 2^{nd} round robin test will be presented and discussed. The 2nd round robin test consisted of three 5-day test runs: the first 5 days at 31% relative humidity provided by MgCl₂ saturated salt solution; the 2nd 5 days at 59% relative humidity provided by NaNO₂ saturated salt solution; and the 3rd 5 days at 81% relative humidity provided by KCl saturated salt solution. The paper will show the round robin test results from three companies on immersion silver (ImAg), gold on electroless nickel (ENIG) and organic solderability preservative (OSP) finished PCBs soldered using organic acid (OR) and rosin (RO) fluxes. In the 1st round robin test, the ENIG finished PCBs suffered the most creep corrosion whereas in the 2nd round robin test, the ImAg finished PCBs suffered the most

creep corrosion. As expected, the OSP finished PCBs suffered very little creep corrosion. The paper will also list the copper and silver corrosion rates experienced during the test runs and discuss means of better control of these corrosion rates.

Key words: Creep corrosion, flowers of sulfur, test.

EXPERIMENT PROCEDURE AND RESULTS

The iNEMI flowers of sulfur (FoS) chamber, shown in Figures 1a and 1b, is a 300-mm cube acrylic box with a paddle wheel rotating at 20 revolutions per minute (RPM). Each paddle wheel can accommodate eight PCBs. The sulfur, 200 g in quantity, is contained in a 275-mm square tray, 20-mm deep, with a 195-mm circular opening in the center. The sulfur concentration is controlled by placing the chamber in an oven maintained at a constant 50°C. Clorox® household bleach, consisting of an aqueous solution of 8.25% sodium hypochlorite with NaOH added to control the pH at about 10.8, is contained in a 145-mm diameter petri dish that sits inside the sulfur tray on the same platform as the sulfur tray. The saturated salt solution is in a 190-mm diameter tray with a circular opening of 65-mm diameter in the center, which is covered by a 125-mm diameter circular



(c) Clorox setup is the source of chlorine gas that also controls the relative humidity in the chamber to the deliquesce relative humidity of the salt.

Figure 1: iNEMI FoS test chamber setup

plate with 1 to 3-mm gap to allow controlled escape of the chlorine gas from the household bleach, as shown in **Figure 1c**. The cover plate also throttles the escape of water vapor from the bleach such that the saturated salt solution can dominate the relative humidity in the chamber at its deliquescence relative humidity. The first step in the creep corrosion qualification test is to bake the test PCBs under flowing nitrogen gas at 100°C for 24 hours to drive off the volatile organic compounds (VOCs). The test PCBs are then subject to three 5-day test runs in an iNEMI FoS chamber, inspecting the PCBs after each test run for creep corrosion: In the 1st 5-day run, the relative humidity is maintained at

31% using MgCl₂ saturated salt solution; at 59% in the 2^{nd} 5-day run using NaNO₂ saturated salt solution; and at 81% in the 3^{rd} 5-day run using KCl saturated salt solution. In each of the three 5-day test conditions, the chamber reaches steady state in a few hours. The pass-fail criterion is the occurrence of creep corrosion bridging the gap between neighboring metallization features on the test PCB. The details of the test are described in **References 1-8**.

Printed circuit boards with three different finishes, ImAg, ENIG and OSP, soldered with two different fluxes, OR and RO, were included in the 1st and the 2nd round robin tests.

Table 1: First round robin test: Relative humidity and corrosion rates in the three 5-day test runs at the three companies (A, B and C)

Site	Saturated salt	Bleach setup gap	Relative humidity %	Cu corrosion rate, Å/day, coul reduction/mass gain	Ag corrosion rate, Å/day, coul reduction/mass gain	AgCl formation rate, Å/day, coul reduction
А		1 mm	31-45	1667/1797	1244/1561	566
В	MgCl ₂	1 mm	38-42	1650/6591	1707/3455	598
С		1 mm	38-42	1544/	1017/	27
А		1 mm	46-57	604/904	558/719	40
В	NH ₄ NO ₃	1 mm	50-51	4105/2545	1236/3428	841
С		1 mm	51-53	1386/	1111/	16
А		1 mm	61-78	687/	376/	17
В	KCl	3 mm	73-75	1603/3178	882/2720	176
С		1 mm	72-73	1369/	704/	16

			L	ocatio	n on b	oard (Grey is	s for no	o corro	sion, y	ellow f	or cor	osion;	pink for light creep				
Site	Finish	Flux		corrosi	on and	dark r	ed for	heavy	creep c	corrosi	on. The	e resista	ance re	adings	are in	KΩ.)		
			1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	
Α			Open	Open	Open	Open	Open	Open	Open	Open	57	Open	Open	Open	Open	N/A	N/A	
В		OR	Open	Open	Open	300	Open	Open	Open	Open	Open	Open	Open	Open	520	N/A	N/A	
С	ImAg		Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A	
Α	IIIIAg		Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A	
B		RO	0.2	Open	0.13	Open	Open	Open	58	Open	Open	Open	Open	Open	720	N/A	N/A	
С			Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A	
Α			0	0.02	0.15	0.02	Open	0	Open	0.01	0.01	72.58	0.1	Open	Open	N/A	N/A	
В		OR	240	Open	Open	210	Open	Open	Open	Open	Open	Open	Open	Open	800	N/A	N/A	
С	ENIG		0.068	Open	Open	0.04	Open	Open	Open	0.045	Open	Open	Open	Open	Open	N/A	N/A	
Α	2.10		0	0	0.06	0.01	Open	0.01	Open	0.01	0.01	0.53	0.06	0.5	Open	N/A	N/A	
B		RO	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A	
С			Open	Open	Open	0.7	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A	
Α			Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A	
B		OR	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A	
С	OSP		Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A	
Α	0.51		Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A	
B		RO	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A	
С			Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A	

Table 2: First round robin test: Corrosion map after the 3rd 5 days with KCl saturated salt solution

First round robin test

The test PCBs were pre-baked at 100°C in flowing nitrogen gas for 24 hours to rid them of much of the VOCs. The iNEMI FoS creep corrosion qualification test method consists of three 5-day test runs each at a different relative humidity level. During the 1st round robin test, the first 5day test runs were at 31% relative humidity using MgCl₂ saturated salt solution; the second 5-day runs were at 47% relative humidity using NH₄NO₃ saturated salt solution and the third 5-day runs were at 81% relative humidity using KCl saturated salt solution. The 15-day iNEMI tests run thus tested for creep corrosion over a range of relative humidity levels expected in service.

Three companies participated in the round robin creep corrosion test. The differences in the test runs at the three companies, that may explain the differences in the test results, were as follows:

- Clorox setup: All tests, but one, were run with 1-mm setup gap to throttle the outflow of moisture from the household bleach and to ensure that enough chlorine gas entered the FoS chamber. The one exception was the 3-mm gap by company B for the KCl test run.
- Test boards: Company B used new test boards for each 5-day test run. Company A and C used the same boards for all the three 5-day test runs.
- Amount of sulfur in chamber: Company A used ~300g sulfur, Company B used ~800g, Company C used ~180g.
- Company C used a different Clorox bleach product from company A and B. The Clorox type used by company C released lesser amount of chlorine gas, especially at lower relative humidity, as evidenced by the rate of AgCl corrosion product formation.

The copper and silver corrosion rates and the rates of formation of AgCl measured during each 5-day period using the coulometric reduction and the mass gain methods are listed in **Table 1**. The table also lists the relative humidity conditions. The relative humidity level was well controlled by design. The saturated salt solution had the dominant role in controlling the relative humidity. The variability in the humidity levels listed in **Table 1** is probably more due to the accuracy of the relative humidity sensors than the actual variation in humidity.

The concentrations of gases in the chamber were indirectly measured by measuring the copper and silver corrosion rates. It was assumed that the AgCl formation rate is directly related to the chlorine concentration and that the corrosion rates of copper and silver are related to a combination of sulfur and chlorine concentrations and the humidity and the temperature of the air. In earlier iNEMI research, it was discovered that in the relative humidity range above about 50%, corrosion rates decrease with humidity, probably because of the absorption of chlorine gas by moisture condensed on surfaces in the chamber. This trend of decreasing corrosion rates with increasing humidity is evident in Table 1 especially for the AgCl growth rate. The AgCl growth rate decreases dramatically as the relative humidity is increased. The exception to this behavior is the high AgCl growth rate observed in company B at 50-51% relative humidity. The AgCl growth rate in company C was lower than that of company A because the household bleach company C had used may have been in storage too long, well beyond its shelf life. Table 1 shows that in general there was reasonable agreement between the copper and silver corrosion rates of company A and company C; whereas, the copper and silver corrosion rates of company B were higher.

Table 3: First round robin test results from company A: Worst case creep corrosion and location on test PCB. The red cell background indicates heavy creep corrosion and the pink cell background indicates light creep corrosion.

Saturated salt		MgCl ₂	NH ₄ NO ₃	KCl		
Relative humidity range		31-45	46-57	61-78		
Cu corrosion rate, coul redn/mass gain, Å/day		1667/1797	604/904	687/		
Ag corrosion rate, coul redn/mass gain, Å/day AgCl formation rate, Å/day		1244/1561	558/719	378/		
AgCl form	ation rate, Å/day	566	40	17		
	ImAg OR)= () = ())= () = ()			
	ImAg RO			5 0°		
Finish	ENIG OR	0 °				
and flux	ENIG RO			00		
	OSP OR		• • • • •			
	OSP RO					

The creep corrosion results are listed in **Tables 2.** The tables shows that overall the ENIG finished boards suffered the most creep corrosion and the OSP finished boards suffered the least creep corrosion. A major discrepancy in the results is that the ImAg finished boards suffered heavy creep corrosion in the company B chamber even in the low

humidity range of 38-42%; while in company A and company C, ImAg finished boards suffered little to no creep corrosion. The explanation for the high creep corrosion on ImAg finished boards in company B may lie in the high levels of chlorine gas in the company B chamber as evidenced by the

Site	Saturated salt	Bleach setup gap	Relative humidity	Cu corrosion rate, Å/day, coul reduction/mass gain	Ag corrosion rate, Å/day, coul reduction/mass gain	AgCl formation rate, Å/day, coul reduction
Α		1 mm	28-30	991/1086	462/771	60
В	MgCl ₂	1 mm	42-44	940/1020	624/536	160
С		1 mm	42-43	1322/1205	1343/885	91
А		1 mm	55-57	846/694	439/480	92
В	NaNO ₂	1 mm	55-62	470/257	895/648	48
С		1 mm	55	702/862	941/801	64
А		3 mm	81-83	1406/751	427/488	44
В	KC1	3 mm	68-75	874/557	780/968	80
С		3 mm	73-75	1118 / 960	861/1037	75

Table 4: Second round robin test: Relative humidity and corrosion rates in the three 5-day test runs at the three companies (A, B and C)

high rate of AgCl formation. Overall the creep corrosion test results from the three companies were in general agreement. **Table 3** shows the worst-case creep corrosion, after the three 5-day tests, on PCBs tested at company A.

The control of the FoS chamber environment is important. Instead of directly measuring the chemical composition of the

gases in the chamber, it is more convenient to measure the rates of copper and silver corrosion using coulometric reduction technique. Not only does one get the formation rates of cuprous and cupric oxides, copper sulfide, silver sulfide and silver chloride, these rates are cumulative in nature, being averaged over the duration of the test. It was determined that sulfur content in the tray did not have any significant effect on copper and silver corrosion rates. This result should come as no surprise because according to thermodynamics, the equilibrium concentration of sulfur in the air in a sealed chamber is only a function of temperature. The sulfur tray can conveniently hold 200 g sulfur. It was therefore decided that from henceforth we will standardize the test to 200 g sulfur. Chlorine concentration control is a challenge. We measure chlorine concentration in the air indirectly by measuring the AgCl growth rate on silver foils. There is much variability in the rate of formation of AgCl during the 5-day test runs most probably due to variability of the chemistry of the household bleach used by the three companies. Another source of variability may be variations in the synergistic effects of chlorine, sulfur and moisture in the air on the AgCl growth rate.

Table 5: Second round robin test. Corrosion map after the 3^{rd} 5 days with KCl saturated salt solution. The relative humidity range across three test sites was 68-83%

			Location on board (Grey is for no corrosion, yellow for corrosion; pink for light creep co								corre	osion					
Site							y creep	p corrosion. The resistance readings are in $K\Omega$.)									
			1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Α			29.4	Open	Open	6.67	Open	Open	7.21	0.886	3	Open	7.7	Open	122.3	N/A	N/A
В	True A co	OR	1.42	8.4	0.3	1.27	230	2.6	0.05	86	66	630	6.3	Open	0.38	N/A	N/A
B *	ImAg	UK	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A
С			Open	Open	Open	0.695	Open	Open	0.142	1.917	4320	Open	Open	Open	0.298	N/A	N/A
Α			1.262	Open	Open	5.6	Open	55.9	21.6	180.3	1.042	45.0	Open	Open	36.5	N/A	N/A
В	T A	во	2.34	1.41	7.15	6.65	16.4	7.3	1.52	47.1	50	1150	42	Open	5.6	N/A	N/A
B *	ImAg RO	ĸO	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A
С			Open	Open	Open	0.615	Open	Open	0.113	0.186	0.973	21.37	Open	Open	0.146	N/A	N/A
Α			0.650	Open	Open	44.59	Open	Open	7.22	4.988	Open	Open	Open	Open	Open	N/A	N/A
В	ENIG	OR	0.53	Open	Open	Open	Open	Open	1900	Open	Open	900	Open	Open	Open	N/A	N/A
С			0.02	Open	Open	0.083	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A
Α			96.7	Open	Open	326.9	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A
В	ENIG	RO	0.24	Open	Open	5.9	Open	Open	0.1	Open	Open	Open	Open	Open	Open	N/A	N/A
С			0.04	Open	Open	0.147	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A
Α			Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A
В	OSP OR	OR	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A
С			Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A
Α			Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A
В	OSP	RO	15.4	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A
С			Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	Open	N/A	N/A

			k cell background indicates			
Saturat	ed salt	MgCl ₂	NaNO ₂	KCl		
pH Value (b	efore/after)	10.8/9.47	11.0/9.47	10.99/9.17		
Relative humidi	ity (5 readings)	28-30	55-57	81-83		
Cu corrosion rate gain, Å/day	, coul redn/mass	991/1086	846/694	1406/751		
Ag corrosion rate gain, Å/day	, coul redn/mass	462/771	439/480	427/488		
AgCl formation ra	ate, Å/day	60	92	44		
	ImAg OR					
	ImAg RO			0.0		
Finish and flux	ENIG OR					
r misii anu mux	ENIG RO					
	OSP OR					
	OSP RO	• • • •				

Table 6: Second round robin test results at company A. Worst case creep corrosion and location on test PCB. The red cell background indicates heavy creep corrosion and the pink cell background indicates light creep corrosion.

Second round robin test

Based on the Clorox setup gap analysis from the 1st round robin creep corrosion test, as well as the study of corrosion rates as a function of sulfur content, and as a function of Clorox dilution, the FoS test setup was finalized to include 200g sulfur and use of 100 ml of concentrated Clorox with 8.25% sodium hypochlorite. The same three companies participated in the 2nd round robin creep test. Printed circuit boards with three different finishes (ImAg, ENIG and OSP) soldered with two different fluxes (OR and RO) were included in the 2nd round robin test. Each of the 6 types of PCBs occupies one slot of the paddles. The other 2 slots were loaded with silver and copper foils on the dummy boards to monitor the corrosion rates. All the 3 companies used the PCBs from the same manufacturing lot. The test PCBs were pre-baked at 100°C in flowing nitrogen gas for 24 hours to rid them of much of the VOCs. The test PCBs were then exposed to three 5-day runs in the above described FoS chamber at 50°C at three relative humidity levels: (a) relative humidity of 33% using MgCl₂ saturated salt solution during the 1st 5 days; relative humidity of 59% using NaNO₂ saturated salt solution during the 2nd 5 days; and relative humidity of 81% using KCl saturated salt solution during the 3rd 5 days. The copper and silver

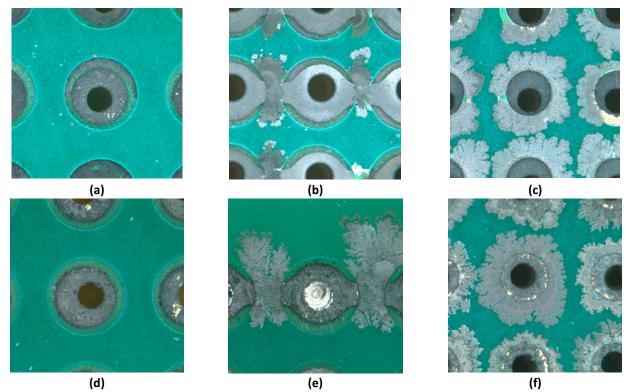


Figure 2: Role of solder mask on creep corrosion from plated-through-hole pads with exposed copper: The top three micrographs are of ImAg PCB soldered with NR330 flux from FOS run #11 with 13-19% relative humidity. The bottom three micrographs are of ENIG PCB soldered with EF8000 flux from FOS run #15 with 74-80% relative humidity. Figures (a) and (d) show no creep corrosion when the solder mask does not overlap the copper metallization. Figures (b) and (e) show creep corrosion emanating only from where the solder mask overlaps the copper metallization. Figures (c) and (f) show creep corrosion occurring all around the PTH where the solder mask overlaps the copper metallization.

corrosion rates and the rate of formation of AgCl measured during each 5-day period using the coulometric reduction and the mass gain methods are listed in **Table 4**. The table also lists the relative humidity conditions. The extent of creep corrosion was documented. **Tables 5** shows the creep corrosion results after the 3rd 5-day run. At site B, the two ImAg boards had heavy creep corrosion after 10 days test, so in the 3^{rd} 5-day test run, these 2 boards were replaced by two pre-baked fresh ImAg boards (site marked B* in **Table 5**) to verify the creep corrosion sensitivity to high relative humidity. **Table 6** shows the worst-case creep corrosion, after the three 5-day tests, on the six types of PCBs tested at company A.

DISCUSSION

The 1st round robin creep corrosion tests gave quite similar results with some differences, the major one being that the ImAg PCBs suffered heavy creep corrosion in company B while the ImAg PCBs suffered little to no creep corrosion in companies A and C. This discrepancy may be explained on the high corrosion rates suffered by copper and silver in the company B chamber, especially the high rate of formation of AgCl. Overall the round robin test results are in general agreement with the industry that amongst the PCB finishes tested, the OSP finished PCBs suffered little to no creep corrosion and the ENIG finished PCBs suffered the most creep corrosion. The 2nd round robin test setup and PCBs were similar for all the three companies: (a) The Clorox setup gap was 1 mm for the first two 5-day runs and 3 mm for the third 5-day run that used the KCl saturates salt solution; (b) The sulfur content was 200g; (c) the Clorox contained 8.25% sodium hypochlorite; and (d) the test PCBs were from the same manufacturing lot. The 2nd round robin test results differed from the first in that the ImAg finished PCBs suffered much more creep corrosion than the ENIG finished PCBs. This difference may be a natural outcome of the aging of the PCBs as noted below where it was observed that PCBs from lots that had suffered creep corrosion in the field stopped suffering creep corrosion after 2-years of storage in an office environment. In other words, PCBs age over time and their creep corrosion behavior changes. In both the round robin tests, the OSP finished test PCBs suffered little to no creep corrosion.

Effect of solder mask edge: Solder mask edge plays a dominant role in promoting creep corrosion as documented in Figure 2. When the copper metallization is not covered by solder mask, as is the case in Figures 2a and 2d, the tendency to creep corrosion is minimal. In cases of the copper metallization only partially covered with solder

mask, as in the case in **Figures 2b and 2e**, the creep corrosion mostly emanated from the edge between the mask and the metallization. In cases of the copper metallization surrounded by solder mask, as is the case in **Figures 2c and 2f**, the creep corrosion radiates out from the metallization-mask edge circle. The reason for the creep corrosion occurring at the metallization-mask edge may be due to the solder flux trapped in the edge. Mask edge designs with less of a trapping tendency may be the solution to eliminating creep corrosion.

Effect of storage time: We have evidence that the duration the printed circuit board assemblies (PCBAs) are stored may reduce their tendency to creep corrode. PCBAs from lots from two different companies that had suffered creep corrosion in the field were tested in the iNEMI FoS test. Fresh PCBAs, from both companies, with very short storage times, suffered creep corrosion in the iNEMI FoS test as shown in **Figure 3**. When stored in normal office environment for 2 years and then tested, PCBAs from neither company suffered creep corrosion in the iNEMI FoS test (**Figure 3**). This observation of storage time reducing the tendency to creep corrosion may be explained if we consider that creep corrosion happens mostly at the solder mask edge. There may be processing chemical trapped at the solder mask edge which may be the root cause of creep

corrosion. Over extended storage time, the trapped chemicals may be consumed in the reaction with the surrounding copper, while at the same time forming a protective corrosion product layer. The consumption of the trapped chemicals and the passivating corrosion product layer may be the root cause hindering creep corrosion on PCBAs with extended storage time.

CONCLUSIONS

The iNEMI taskforce on creep corrosion has developed an innovative, general-purpose FoS corrosion chamber that is inexpensive and easy to maintain. A creep corrosion qualification test has been developed that has been successful in two round robin tests at three companies on test PCBs from two different lots, with three different finishes (ImAg, ENIG and OSP) soldered with two different fluxes (rosin flux and organic acid flux). In the first-round robin, the ENIG finished boards suffered the most creep corrosion and the OSP finished boards suffered the least creep corrosion. In the second-round robin test, the ImAg finished boards suffered more creep corrosion than the ENIG finished boards and the OSP finished boards suffered the least creep corrosion. The change in the order of creep corrosion propensity between ENIG and ImAg finished boards may be explained based on the processing chemicals trapped at the solder mask edge reacting and passivating the neighboring copper and thus changing the creep corrosion behavior of the PCBs.

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