

Influence of Salt Contamination and RH on Creep Corrosion of Immersion Silver (ImAg)

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ABSTRACT

Creep corrosion is the migration of corrosion products due to environmental corrosions of electronics in polluted environment with relative humidity and corrosive gas (NO_x , SO_x , Cl_2 and H_2S). Those gases react with copper and silver on the ImAg board, and the formation of corrosion products (Cu_xS , and Ag_xS) cause short circuit by creeping and bridging nearby components. Creep corrosion could be promoted by inherited surface features as well as external factors like contamination with salt residue. Although creep corrosion on ImAg has been studied extensively, the effect of commonly found salts on electronic products has been not well understood. In this work, various levels of salt contaminations have been applied on ImAg board, which were exposed to mixed flowing gas (MFG) environment (containing NO_x , SO_2 , Cl_2 and H_2S) at different relative humidity (RH) to investigate the combined effect of salt and RH on creep corrosion in the harsh environment.

Key words: Salt, RH, Creep Corrosion, ImAg, Contamination, Environmental Corrosion

INTRODUCTION

Due to the European Union's (EU) regulation of the RoHS (Restriction of Hazardous Substances), the PCB industry has been moving to alternative surface finish materials such as Immersion Silver (ImAg), Organic Solderability Preservatives (OSP), and Immersion Gold on Electroless Nickel (ENIG). Among these options, ImAg has become widely adopted due to its cost-effectiveness, good solderability, and electrical conductivity. However, ImAg surface is susceptible to corrosion induced by sulfur in the atmosphere. Moreover, electronics are gradually exposed to extreme environments more frequently due to severe air pollution and extreme natural phenomena which increases the risk of failure in general [1-2].

While corrosion prevention and mitigation techniques have made significant progress, electronic components in the field continue to experience failures, particularly due to corrosive atmospheres. One of the most common types of failure is creep corrosion, which arises in aggressive environments. As a result, creep corrosion phenomena have been studied in both industry and academia to establish creep corrosion mechanism by simulating the creep corrosion in lab condition [3-6]. With the contributions of multiple studies, creep corrosion mechanism has been accepted as occurring in two

separate steps: firstly, bare copper and silver metal corrosion occurs due to presence of pollutants and moisture. Secondly, corrosion products is spreading, mainly CuS_x , throughout the PCB surface which may eventually develop to the short circuit with nearby components [7-9].

In addition to the fundamental creep corrosion mechanism, many other different variables have been suggested that could possibly promote the creep corrosion of Cu and Ag. For example, Cu corrosion is highly dependent on the Relative Humidity (RH) and Sulfur containing gas concentration such as H_2S and SO_2 [10]. The mixture of NO_2 and H_2S gas has shown synergetic effect on Ag corrosion, and it is also known that Cl_2 could accelerate the Ag corrosion, although the influence of Cl_2 on Ag corrosion has not been fully understood yet [11].

Beyond corrosive gas, surface contamination—such as from organic acid flux—has been shown to promote creep corrosion on ImAg boards in corrosive environments. This suggests that creep corrosion is highly sensitive to both the chemical characteristics of the surface and environmental factors. Coastal regions, in particular, experience high humidity year-round and facilitate the deposition of various salts like chlorides and nitrates. Although the impact of the salt contamination was well-recognized, replicating the uniform salt distribution and getting consistent results has been challenging due to nature of PCB hydrophobic coating of solder mask.

This paper examines the probability of creep corrosion due to chloride and nitrate salts contamination in the moisture presence. We demonstrate that uniform salt distribution can be achieved by using surfactant, leading to consistent creep corrosion results throughout the ImAg board. Thus, it was successful to highlight the synergetic effects of salts concentration and RH.

EXPERIMENTAL

Specifically designed ImAg boards were evaluated under an optical microscope to any identify defects introduced by manufacturer prior to Mixed Flowing Gas (MFG) testing. Only defect-free ImAg boards were selected as test vehicles for further evaluation.

The salts used in this study included CuCl₂ (Copper (II) chloride dihydrate, 99%, Alfa Aesar), CaCl₂ (Calcium chloride, dried powder, 97%, Thermo Scientific), Cu(NO₃)₂ (Copper (II) nitrate trihydrate, 99%, Acros organics), and Ca(NO₃)₂ (Calcium nitrate tetrahydrate, 97+%, Thermo Scientific), and the salts solution concentration in cations contents per surface area (in²) were calculated based on the surface area of solution droplet as shown in the following example.

Example: Calculation 5 μg Cation/in² to CaCl₂ solution

1. 5 μg Ca
→ 5 μg × 1/40.078 mol/g (Calcium atomic mass)
= 0.1247 μmol Ca
2. 0.1247 μmol Ca
→ 0.1247 μmol × 110.98 g/mol (CaCl₂ molar mass)
= 13.84 μg CaCl₂
3. 13.84 μg CaCl₂ (per water drop, 5 μl=0.0744 in²)
→ 13.84 μg CaCl₂/in² × 0.0744 in²/5 μl
= 0.2059 g CaCl₂/l
∴ 5 μg Cation/in² = 0.2059 g CaCl₂/l

Total three different concentration levels, 5, 25, 50 μg of cation (Cu or Ca) / in² (based on water drop surface measurement), were established and the concentration calculated as Table 1.

Table 1. Salt Solution Concentration in g/l

Salts (g/l)	μg		
	5 Cation/in ²	25 Cation/in ²	50 Cation/in ²
CuCl ₂ ·2H ₂ O	0.1996	0.9980	1.996
Cu(NO ₃) ₂ ·3H ₂ O	0.2826	1.413	2.826
CaCl ₂	0.2059	1.029	2.059
Ca(NO ₃) ₂ ·4H ₂ O	0.4381	2.191	0.438

Total 45 solution droplets were applied on 12 different areas of the ImAg board to simulate consistent creep corrosion as shown in Figure 1.

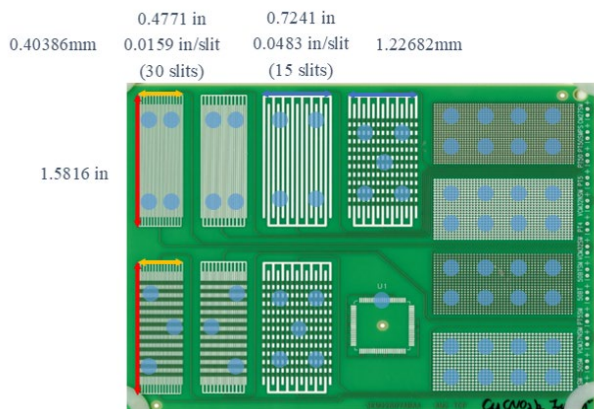


Figure 1. Solution Droplet Application on the ImAg Board Test Vehicle

In order to achieve uniform salt distribution, Isopropanol (2-Propanol, CMOS, J.T.Baker), Sodium Dodecyl Sulfate (Sodium dodecyl sulfate ≥98.5%, Sigma Aldrich), and Triton (X-100, Sigma Aldrich) have been added to the aqueous salt

solution. The samples' salt distribution were examined with Micro-Raman spectroscopy and SEM-EDS prior to MFG testing in order to ensure the uniform distribution. Figure 2 showed micro-Raman spectrum for the 50 μg/in² Cu(NO₃)₂ salt application with IPA and Triton as surfactants.

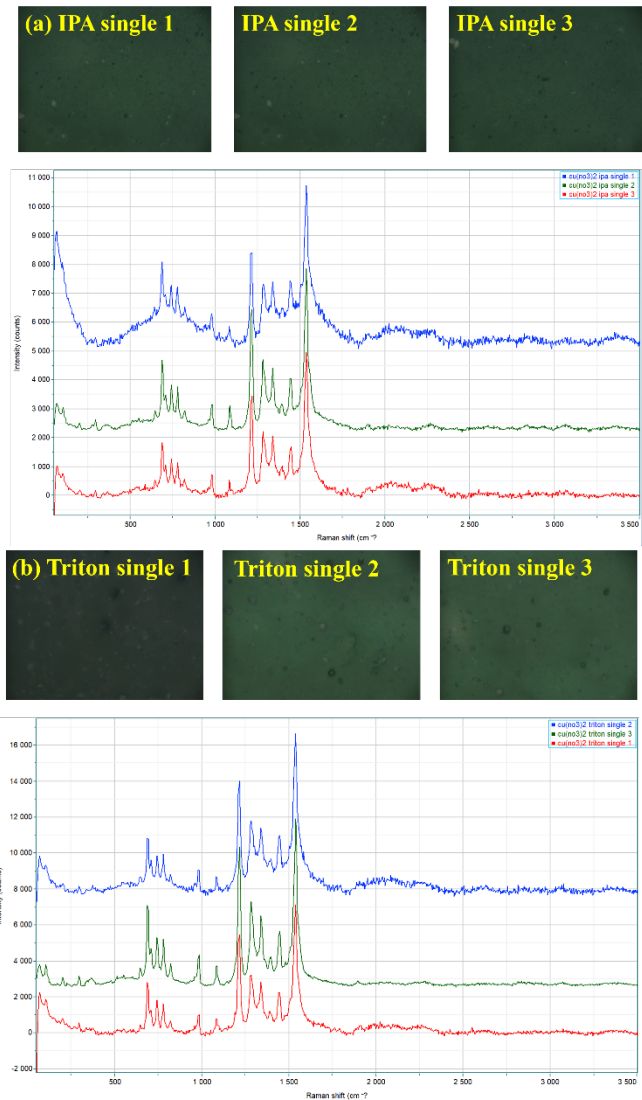


Figure 2. Micro-Raman Spectrum for Cu(NO₃)₂ Salt Application with (a) IPA and (b) 2% Triton

Micro-Raman spectroscopy is powerful analytical equipment to detect NO₃⁻ (1050 cm⁻¹) containing salt. However, due to extremely low concentration of nitrate salts, the observation of NO₃⁻ on the ImAg board was not available for both IPA and 2% Triton samples, but future measurements at higher concentration are planned to confirm the visibility of NO₃⁻ salt under Micro-Raman spectroscopy.

In addition to Micro-Raman spectroscopy, SEM-EDS was utilized for salt distribution observation. Figure 3 showed SEM images and EDS results of Cu(NO₃)₂ application with (a) IPA and (b) 2% Triton.

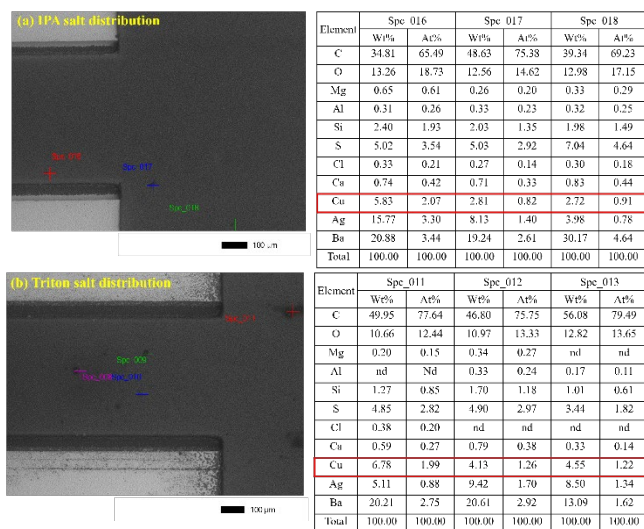


Figure 3. SEM-EDS for Cu(NO₃)₂ Salt Application with (a) IPA and (b) 2% Triton

Because of the nature of SEM-EDS, it is challenging to detect NO₃⁻, but it is possible to observe Cu under EDS. Cu element observation was possible on the ImAg board treated with Cu(NO₃)₂ and 2% Triton. Especially, Cu element detection on 2% triton treated board was possible throughout whole board including the middle of hydrophobic solder mask. It could be interpreted that 2% Triton assists uniform salt distribution on the board. On the other hand, Cu element observation on the ImAg with IPA application was only possible on the metal pad nearby solder mask. Due to poor salt distribution with IPA, IPA was not effective in generating uniform creep corrosion. Thus, 2% Triton was utilized as the most suitable surfactant for the testing since Triton does not generate micelle and disperse salts effectively.

The prepared test vehicles exposed to MFG environment for specific amount of duration according to copper corrosion rate and the MFG condition shown in Table 2.

Table 2. MFG Test Condition in ppb

SO ₂	H ₂ S	NO _x	Cl ₂	RH	Temp
200	500	500	30-40	5, 40, 70%	30°C

The test vehicle and 2 cm × 5 cm Cu (Copper foil 0.1 mm, Alfa Aesar) and Ag (Silver foil 0.1 mm, Thermo Scientific) coupons were tested at the same time to monitor corrosion rate inside of the chamber after MFG testing by weight gain analysis using microbalance.

The corrosive gas concentration in the MFG chamber (ESPEC) was monitored with H₂S- SO₂ analyzer (iQ Series 450, Thermo Scientific), NO-NO₂-NO_x analyzer (iQ Series 42, Thermo Scientific), and Cl₂ analyzer (SPM Flex, Honeywell) in-situ. Beside of gas concentration, RH was also controlled and monitored with RH/Temp monitor (i.Server MicroServer, Omega Engineering) from 5%, 40%, to 70% for the synergetic effects with the salt application on the test vehicles.

RESULTS AND DISCUSSIONS

Results

Table 3 shows the change in corrosion products film thickness, calculated based on the weight difference before and after the MFG testing on the Cu and Ag coupon. MFG testing under 40%RH was conducted for 10 days, while testing at 5%RH was extended until 15 days to achieve similar total copper corrosion as 70%RH. Silver corrosion film thickness for 40%RH and 5%RH were similar but slightly thicker than 70%RH because silver corrosion is not highly dependent on the RH. In contrast, copper corrosion rate highly depends on the RH change [12].

Table 3. Thin Film Thickness of Corrosion Products (nm)

Relative Humidity	Coupon	Days	5 μg25 Cation/in ²	μg50 Cation/in ²	μg Cation/in ²	
5% RH	Cu	5	431.31			
		10	385.55			
	Ag	10	312.19			
		15	260.45			
	40% RH	Cu	5	536.59		
			10	503.88		
Ag		5	203.47			
		10	243.01			
70% RH	Cu	5	1351.44	1284.71		
	Ag	5	412.79	474.48		

Although 2% triton is suitable candidate for the uniform salt distribution with no micelle formation, it is crucial to understand the effect of surfactant on creep corrosion of ImAg surface finish. Figure 4 showed 2% triton treated board at different MFG exposure and throughout the whole exposure, 2% triton showed minor influence on the creep corrosion.

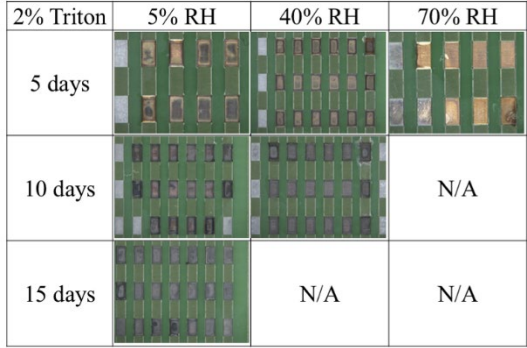


Figure 4. 2% Triton Effect on Creep Corrosion at Different Test Environments

Figure 5 shows creep corrosion induced by different kind of 50 μg/in² salt application on the ImAg board at 70%RH.

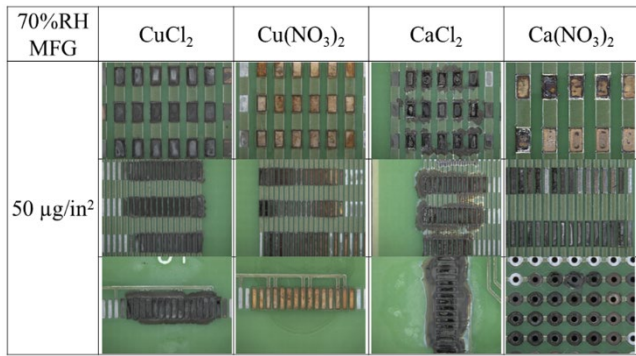


Figure 5. 50 μg/in² Salts Effects on Creep Corrosion Under 70%RH

50 μg/in² salt concentration causes creep corrosion on ImAg board and the variation of creep corrosion severity due to different type of salts is visible from the chloride and nitrate application. Both 50 μg/in² CuCl₂ and CaCl₂ showed significant creep corrosion on the ImAg board whereas 50 μg/in² Ca(NO₃)₂ and Cu(NO₃)₂ showed less creep corrosion on the ImAg board. There may be differences between area on the board, but the 50 μg/in² chloride salts treated test vehicles formed wide range of short circuit where the equipment could possibly fail rapidly. On the other hand, 50 μg/in² nitrate salt treated test vehicles did not generate short circuit regardless of the pattern of the board. However, longer exposure of nitrate salt applied board will fail eventually due to short circuit formation from creep corrosion as well.

Figure 6 shows the results of 25 μg/in² salts concentration applied samples after MFG testing.

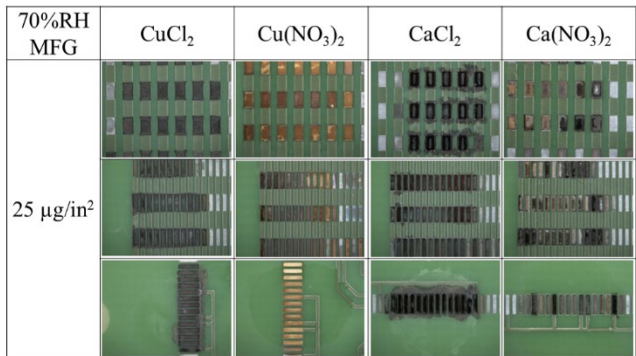


Figure 6. 25 μg/in² Salts Effects on Creep Corrosion Under 70%RH

Unlike results from 50 μg/in² chloride salts treated boards, 25 μg/in² CuCl₂ and CaCl₂ showed moderate creep corrosion with a few short circuit formations. Comparably, 25 μg/in² Cu(NO₃)₂ and Ca(NO₃)₂ showed minor creep corrosion without short circuit formation.

Lastly, Figure 7 shows the MFG result with 5 μg/in² salt applications.

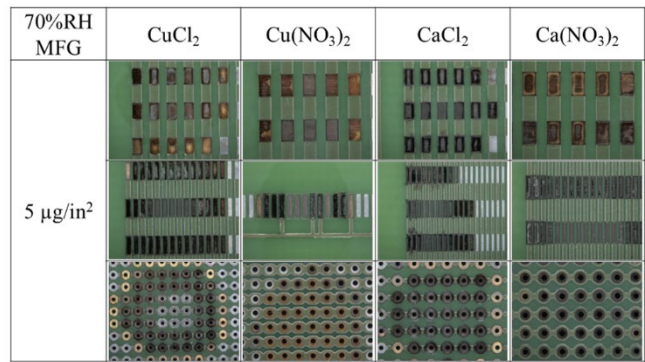


Figure 7. 5 μg/in² Salts Effects on Creep Corrosion Under 70%RH

All 5 μg/in² treated ImAg board showed minor creep corrosion.

Since 25 μg/in² and 5 μg/in² salts treated ImAg board showed minor or less creep corrosion under 70 %RH, there is very low possibility that the lower salts concentration treated ImAg boards shows sufficient creep corrosion at low RH. Therefore, further 40%RH and 5%RH testing was conducted with 50 μg/in² salts concentration for all 4 salts.

Figure 8 shows 40%RH MFG test results for 10 days to match the copper corrosion rate with 70%RH for 5 days. As mentioned earlier, since copper corrosion rate highly depends on the RH, test conditions which is lower RH than 70%RH are extended to match the copper corrosion rate of 70%RH of copper coupon.

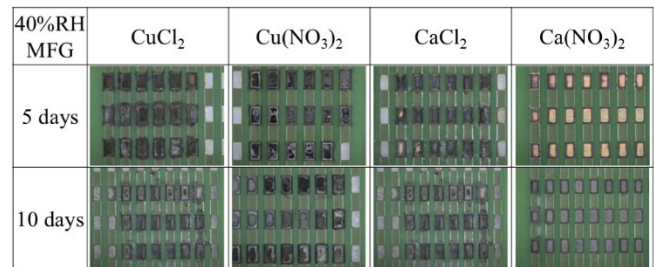


Figure 8. 50 μg/in² Salts Effects on Creep Corrosion After 5 days and 10 days of MFG Exposure Under 40%RH

The difference in the degree of creep corrosion between salts under 40%RH is more noticeable than 70%RH MFG exposure after 5 days. CuCl₂ and CaCl₂ after 5 days of MFG exposure under 40%RH showed severe creep corrosion with extensive short circuit formation as much as the 70%RH tested ImAg board. Compared to severe creep corrosion of chloride salts, Cu(NO₃)₂ and Ca(NO₃)₂ treated test vehicles showed minor creep corrosion with no short circuit formation after 5 days. Moreover, nitrate salts showed moderate corrosion with short circuits similar as the results of 70%RH exposure after 10 days of MFG exposure.

Figure 9 showed 5%RH exposure salts applied ImAg board until 15 days.

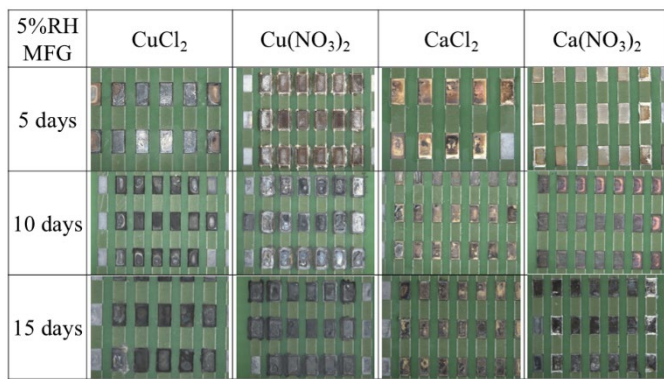


Figure 9. 50 µg/in² Salts Effects on Creep Corrosion until 15 days of MFG Exposure Under 5% RH

Most of salts treated ImAg board showed very minor creep or no creep corrosion at 5%RH except Cu(NO₃)₂ treatment. Test vehicles with CuCl₂, CaCl₂, and Ca(NO₃)₂ showed minor or no creep corrosion without short circuits formation. Surprisingly, Cu(NO₃)₂ treated ImAg board showed different behavior with other three salts. Until 10 days of MFG exposure, Cu(NO₃)₂ treated sample showed moderate creep corrosion with localized short circuit formation. Although nitrate showed less creep corrosion than chloride in most cases, Cu(NO₃)₂ treated board showed more creep corrosion at 5%RH than 70%RH sample after 15 days of MFG exposure. Due to the morphology difference of the corrosion products, some of corrosion products formed on Cu(NO₃)₂ sample at 5%RH MFG exposure showed brighter corrosion products while the samples tested at 70%RH MFG condition showed dark corrosion products.

Discussions

It is evident that creep corrosion strongly depends on the type of salt present. In general, chloride salts (CuCl₂, CaCl₂) significantly accelerated creep corrosion compared to nitrate salts (Cu(NO₃)₂, Ca(NO₃)₂). Creep corrosion has showed strong dependency on chloride in higher RH environment. Especially, MFG testing under 70%RH and 40%RH with 50 µg/in² salt application, CuCl₂ and CaCl₂ showed severe creep corrosion, whereas Cu(NO₃)₂ and Ca(NO₃)₂ created very minor creep corrosion as shown in Figure 5. Chloride provides creepable surface that supports corrosion products, mostly copper sulfide, formation and spreading. In contrast, nitrate salts are less effective than chloride in providing creep supportive surface. Therefore, the field failure with creep corrosion due to chloride salt is more likely than nitrate salt.

Besides of salts type dependency of creep corrosion, the concentration of the salt strongly affects the creep corrosion. For instance, under the identical MFG environment, 50 µg/in² and 5 µg/in² salt application showed significantly different creep corrosion results as shown in Figure 5 and Figure 7. Higher salt concentration, which could be interpreted as highly contaminated area, is more prone to field failure due to creep corrosion from salt contamination. In the absence of salt contamination, RH alone is not a critical parameter for the creep corrosion unless condensation occurs. However, once an ImAg board is contaminated with salt

residue or precipitates, it becomes much more sensitive to RH. The presence of salt residues facilitates the formation of a surface that promotes creep corrosion. With salt residue on the board, higher RH environment tends to produce more creep corrosion as shown in Figure 5, Figure 8, and Figure 9. The reason higher RH promotes more creep corrosion is due to the dissolution of salt residues. Moisture in a high RH environment dissolves the salts, transforming the board's surface into one that encourages creep corrosion. This process, driven by the presence of salt residues, is illustrated in Figure 10.

In terms of microenvironment, due to the nature of metal wettability, metal pad surface is desirable for moisture to accumulate from the atmosphere. The present salt contaminant nearby metal pad could be dissolved in the moisture. Additionally, distributed salt contaminant on the hydrophobic solder mask nearby metal pad could retain moisture. Eventually, hydrophobic surface turns into slightly hydrophilic surface due to ion present in accumulated water.

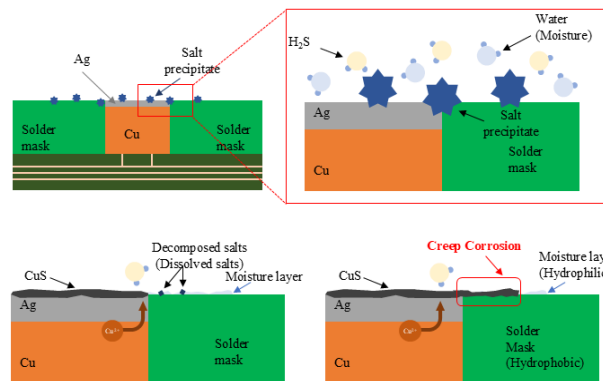


Figure 10. Illustration of Salt Assisted Creep Corrosion in Microenvironment

In addition to RH in the atmosphere, the concept of Critical Relative Humidity (CRH) can be considered. CRH is the threshold of RH at which a specific salt begins absorbing moisture from the atmosphere [13-14]. Since the creep corrosion is affected by the salt contaminants nearby the metal pad, and the salt initiate to absorb moisture from atmosphere at certain RH, CRH could be possibly related to creep corrosion. It could be assumed that if the influence of CRH was significant, creep corrosion should not occur under the RH lower than CRH. However, the correlation between CRH and creep corrosion is not strong, as demonstrated by the results of 40%RH and 5%RH MFG exposure.

Although CRH of CaCl₂ (29.5%RH) and Cu(NO₃)₂ (35%RH) lower than 40%RH, both salts still caused creep corrosion under 40%RH MFG exposure. Furthermore, ImAg boards with Cu(NO₃)₂ contamination exhibited creep corrosion even at 5% RH. Additionally, although all 4 salts CRH (CuCl₂:67%RH, Ca(NO₃)₂:46.7%RH) is lower than 70%RH, the nitrate salts showed moderate or minor creep corrosion. These findings demonstrated that there is no strong correlation between CRH of the salts and the susceptibility of creep corrosion.

Although it was demonstrated that chloride promotes more creep corrosion than nitrate, and salt contaminated ImAg board showed more creep corrosion under higher RH, $\text{Cu}(\text{NO}_3)_2$ stimulates more creep corrosion than any other salts under low RH environment according to Figure 7. While the MFG chamber and the salt solution application were carefully controlled, there is still a possibility of irregular salt distribution or variation in gas concentration. To address this, an additional three sets of the samples were exposed to same MFG condition with continuous monitoring of gas concentration. Figure 11 showed additional results for $\text{Cu}(\text{NO}_3)_2$ and $\text{Ca}(\text{NO}_3)_2$ contaminated ImAg board exposed to 5%RH MFG condition for up to 15 days.

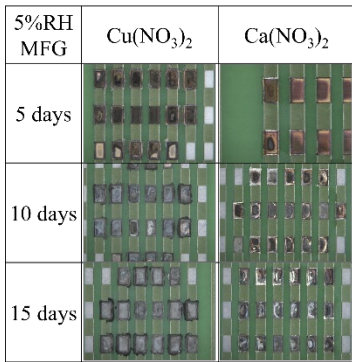


Figure 11. 50 $\mu\text{g}/\text{in}^2$ Salts Effect on Creep Corrosion After 15 days of MFG Exposure Under 5% RH

It is possible to obtain repeatable and reliable results that the creep corrosion under low RH with $\text{Cu}(\text{NO}_3)_2$ contamination was present. The creep corrosion on the $\text{Cu}(\text{NO}_3)_2$ contaminated ImAg board under 5%RH is extent, although the exact mechanism is not fully understandable and similar result has not been previously reported. Therefore, further investigation is required to explore the factors driving $\text{Cu}(\text{NO}_3)_2$ assisted creep corrosion under low RH and to clarify the environmental factors that contribute to this phenomenon.

To better understand the corrosion products from $\text{Cu}(\text{NO}_3)_2$ at 5%RH are identical, the corrosion products were analyzed under SEM-EDS, and the results shown in Figure 12.

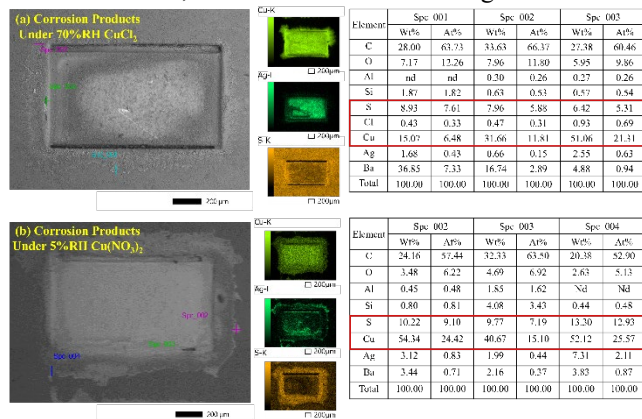


Figure 12. Creep Corrosion Products on ImAg Board under in 70%RH CuCl_2 contamination (a), and under 5%RH in $\text{Cu}(\text{NO}_3)_2$ contamination

For comparison, corrosion products generated from CuCl_2 at 70%RH was utilized as a reference group. Qualitatively, corrosion products from $\text{Cu}(\text{NO}_3)_2$ at 5%RH is similar to the corrosion products from CuCl_2 at 70%RH. In both cases, the dominating creeping species is copper sulfide. The only difference between those two corrosion products was the presence of chloride in the CuCl_2 -treated ImAg board, which was absent in the $\text{Cu}(\text{NO}_3)_2$ samples due to the use of CuCl_2 .

CONCLUSIONS

Conclusion

This study demonstrates the following key findings.

1. Triton-X (a non-ionic surfactant) does not significantly cause creep corrosion but promotes uniform salt distribution.
2. Salt contamination on the ImAg board leads to various degrees of creep corrosion and short circuit formation.
3. Chloride salt contamination promotes more creep corrosion than nitrate salt contamination.
4. Higher concentration of salt contamination generates more creep corrosion.
5. In the present of salt contamination, higher RH environment caused more creep corrosion than lower RH.
6. The Critical Relative Humidity (CRH) of salts does not have a direct correlation with creep corrosion.
7. $\text{Cu}(\text{NO}_3)_2$ generates more creep corrosion under low RH than high RH.

However, the study is limited by $\text{Cu}(\text{NO}_3)_2$ MFG testing under low RH. Future investigation is needed to explore the clear relation between nitrate salts, especially $\text{Cu}(\text{NO}_3)_2$ and creep corrosion. Overall, this study provides important insights into the impact of both minor and major contamination on creep corrosion, offering new perspectives on reliability in the field. We hope it will stimulate further research and practical applications in the electronics industry.

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