# 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 a round robin evaluation of the modified iNEMI creep corrosion qualification test. The earlier versions of the test involved testing in a specially designed flower of sulfur (FoS) chamber for 10 days at nominally 81% humidity level provided by KCl saturated salt solution. The modification to the iNEMI test was the change to three 5-day test runs, the first 5 days at 31% relative humidity provided by $MgCl_2$ saturated salt solution, the 2nd 5 days at 47% relative humidity provided by $NH_4NO_3$ saturated salt solution and the 3rd 5 days at 81% relative humidity provided by KCl saturated salt solution. The rest of the test procedure remained unchanged, with the iNEMI designed setup providing somewhat controlled and reproducible concentration of chlorine gas and a tray of sulfur for providing sulfur vapors. As usual the test was run at a constant 50ºC. The paper will compare the round robin test results from three companies on immersion silver (ImAg), electroless nickel immersion gold (ENIG) and organic solderability preservative (OSP) finished PCBs soldered using organic acid (OR) and rosin (RO) fluxes. As expected, the ENIG finished PCBs suffered the most creep corrosion while the OSP finished PCBs suffered the least 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.

## INTRODUCTION

Creep corrosion has been the dominant corrosion-related failure mechanism of electronic hardware since the implementation of ROHS in 2006. The restriction of lead metal in PCBs has forced the use of surface finishes, such as organic solderability preservation (OSP), immersion silver (ImAg), electroless nickel immersion gold (ENIG) and immersion tin (ImSn) to enhance PCB solderability. These finishes have made PCBs susceptible to creep corrosion failures in corrosive environments high in sulfur-containing compounds **[1-4].**Creep corrosion, a mass transport phenomenon in which solid corrosion products, typically sulfides, migrate over a surface without the influence of an electric field, was first reported by Egan and Mendizza for $Ag_2S$ on gold**[5].** They, and others subsequently, observed that creep corrosion is highly surface sensitive and only occurs on surfaces when two prerequisites are met: a site for generating corrosion product and a surface for supporting the creep of the generated corrosion product **[6-9]**.

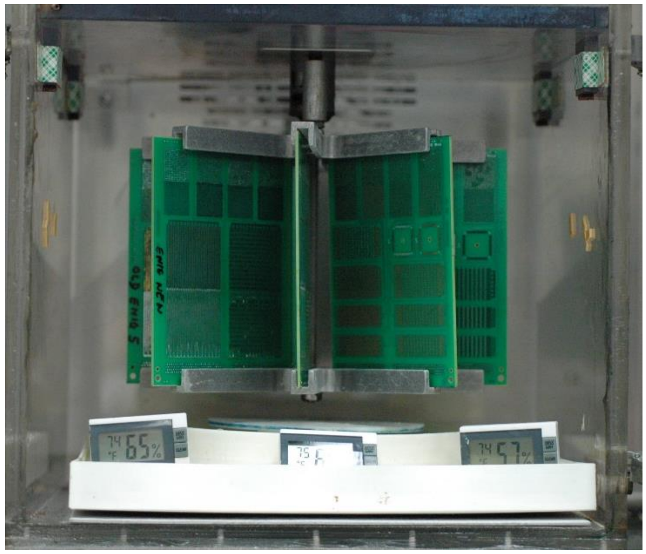


**Figure 1: FOS test setup loaded with test PCBs. The tray-like setup below the test PCBs provides the sulfur and chlorine gases and maintains the relative humidity in the chamber at the deliquescence relative humidity of the saturated salt solution in the setup**

The role of relative humidity on creep corrosion was studied recently by the iNEMI taskforce on creep corrosion **[10, 11]**. The taskforce conducted extensive testing at three companies on ImAg, ENIG and OSP finished PCBs soldered with OR and RO fluxes and concluded that creep corrosion has a strong and unexpected dependence on relative humidity. Contrary to the general belief that high relative humidity increases the propensity to creep corrosion, just as it increases corrosion of most metals, it was found that creep corrosion on some finishes can occur at relative humidity values as low as 11% but not in the higher humidity range; whereas, on other finishes creep corrosion may occur only under high humidity conditions.

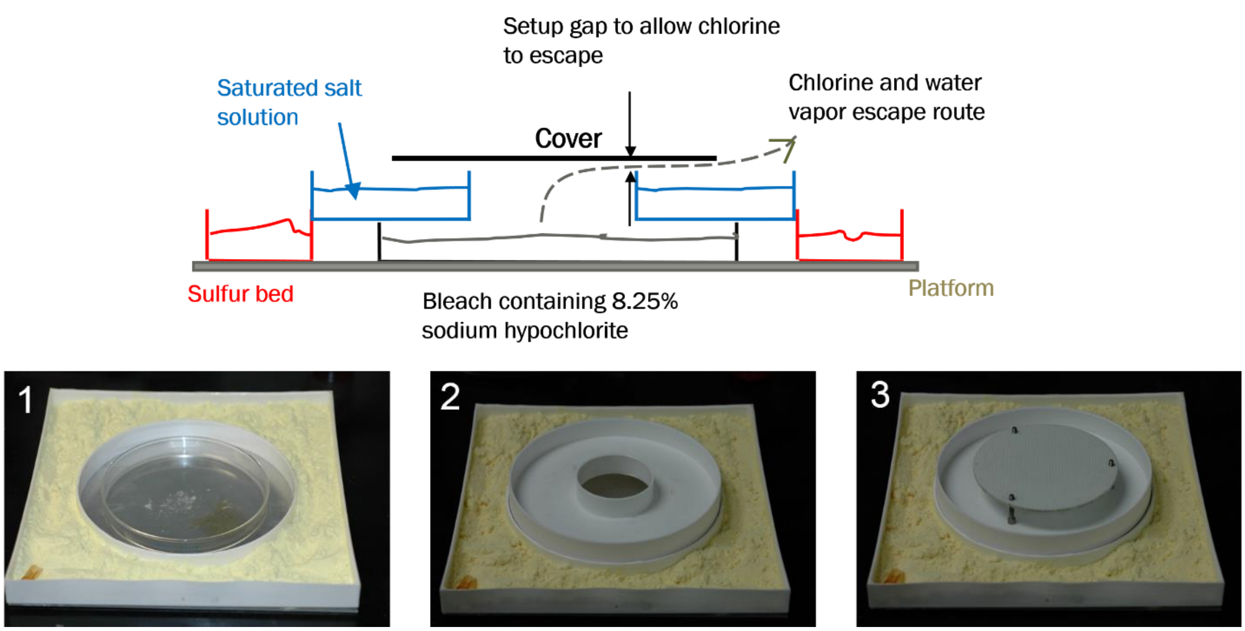



**Figure 2: Bleach setup in cross section and in pictorial format. The moisture coming off the bleach is forced to flow through a 1-mm gap and then over a saturated salt solution so that the air exiting the setup is in equilibrium with the saturated salt solution. The saturated salt solution, thus, controls the humidity of the air in the camber**

| 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 |
|---|---|---|---|---|---|---|
| A | | 1 mm | 31-45 | 1667/1797 | 1244/1561 | 566 |
| B | $MgCl_2$ | 1 mm | 38-42 | 1650/6591 | 1707/3455 | 598 |
| C | | 1 mm | 38-42 | 1544/-- | 1017/-- | 27 |
| A | | 1 mm | 46-57 | 604/904 | 558/719 | 40 |
| B | $NH_4NO_3$ | 1 mm | 50-51 | 4105/2545 | 1236/3428 | 841 |
| C | | 1 mm | 51-53 | 1386/-- | 1111/-- | 16 |
| A | | 1 mm | 61-78 | 687/-- | 376/-- | 17 |
| B | KCl | 3 mm | 73-75 | 1603/3178 | 882/2720 | 176 |
| C | | 1 mm | 72-73 | 1369/-- | 704/-- | 16 |

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

Given that creep corrosion can occur at both low and high humidity levels depending on the finish on the PCB, it was decided that the creep corrosion qualification test must include both low and high relative humidity levels. The iNEMI creep corrosion qualification test was accordingly modified to include three 5-day test runs at low, medium and high humidity. This paper describes the effort to qualify the modified iNEMI creep corrosion qualification test. The results of the modified test run at A, B and C companies on 6 different types of PCBs will be presented and discussed. The paper will also present improvements leading to better control of the atmosphere in the chamber.

## EXPERIMENTAL PROCEDURE AND RESULTS

The modified iNEMI flowers of sulfur (FoS) creep corrosion qualification test method consists of three 5-day test runs at three relative humidity levels in an iNEMI FoS test chamber. The first 5-day run is at ~31% relative humidity using $MgCl_2$ saturated salt solution; the second 5-day run at ~47% relative humidity using $NH_4NO_3$ saturated salt solution and the third 5-day run at 81% relative humidity using KCl saturated salt solution. The 15-day iNEMI test thus tests for creep corrosion over a range of relative humidity levels expected in service.

The iNEMI FoS test setup has been described in detail in **References 10 and 11** and repeated here for the convenience of the reader. The FoS chamber, shown in **Figure 1**, 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 setup to control the sulfur and chlorine gas concentrations and the relative humidity in the chamber is shown schematically and pictorially in **Figure 2**. The sulfur 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 50ºC. Household bleach, consisting of an aqueous solution of6-8.25% 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 circular plate with 1-mmgap to allow controlled escape of the chlorine gas from the household bleach. 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 relative humidity is maintained at ~31% in the 1$^{st}$ 5-days of the test using MgCl2 saturated salt solution; at ~47% in the 2$^{nd}$ 5-days of test using NH4NO3; and at ~81% in the 3$^{rd}$ 5-days of the test using KCl saturated salt solution. In each of the three 5-day test conditions, the chamber reaches steady state in a few hours.

**Table 2a: Corrosion map after the first 5 days in FoS chamber with $MgCl_2$ saturated salt solution. The relative humidity range across three test sites was 31-45%.**

| Test site | Finish | Flux | **Location on board** (C refers to sites with corrosion. The darker shaded boxes are where creep corrosion occurred. The values in the darker shaded boxes are the resistance values in Ohms. | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
| A | ImAg | OR | C | C | C | C | | C | C | C | C | C | C | | C | | C |
| B | | | 0.07 | -- | -- | 0.07 | C | -- | 53 | 384 | 25 | -- | -- | -- | 4.5 | | -- |
| C | | | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C |
| A | | RO | C | C | C | C | | | C | C | C | C | C | | C | | C |
| B | | | 24 | -- | -- | -- | C | -- | 0.0 | 580 | -- | 702 | C | C | 0.05 | | -- |
| C | | | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C |
| A | ENIG | | 0.02 | 0.23 | 21 | 0.04 | | 0.07 | -- | | -- | | | | 0.02 | | -- |
| B | | | 0.01 | 1 | -- | 0.03 | | -- | 0.0 5 | -- | -- | C | C | C | 0.02 | | -- |
| C | | | | C | C | C | | C | C | C | | C | | | C | | C |
| A | | RO | 0 | 0.44 | -- | 0 | | 0.07 | 0.1 | | 91 | | | | 0.01 | | -- |
| B | | | 0.05 | 0.74 | -- | 0.01 | C | 3.7 | 0.0 | C | -- | C | C | C | 0.01 | | -- |
| C | | | C | C | C | C | C | C | -- | C | | C | C | | | | C |
| A | OSP | OR | | | | | | | | | | | | | | | |
| B | | | | | | | | | | | | | | | | | |
| C | | | C | C | C | C | | C | C | C | | C | C | | C | | C |
| A | | RO | | | | | | | | | | | | | | | |
| B | | | | | | | | | | | | | -- | | |
| C | | | C | C | | | | C | C | C | | C | C | | | | C |

The iNEMI test boards used in the study were of the same design as described earlier by the iNEMI taskforce on creep corrosion**[10, 11].** Printed circuit boards with three different finishes (ImAg, ENIG and OSP) soldered with two different fluxes (OR and RO) were included in the round robin test. These 6 types of PCBs were subject to the modified creep corrosion test.

Three companies participated in the round robin creep corrosion test. 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. The copper and silver 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 1**.The table also lists the relative humidity conditions. The extent of creep corrosion was documented after each 5-day run. **Tables 2a-2c** list the creep corrosion results for the three participating companies under the 3 humidity levels after each 5-day test run for each of the 6 types of PCBs under test. **Table 3** shows the worst-case creep corrosion, after the three 5-day tests, on the six types of PCBs tested in company A.

**Table 2b: Corrosion map after the second 5 days in FOS chamber with $NH_4NO_3$ saturated salt solution. The relative humidity range across the 3 test site was 46-57%.**

| **Test site** | **Finish** | **Flux** | **Location on board** (C refers to sites with corrosion. The darker shaded boxes are where creep corrosion occurred. The values in the darker shaded boxes are the resistance values in Ohms. | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
| A | ImAg | OR | C | C | -- | C | C | C | -- | C | C | C | C | C | -- | | C |
| B | | | -- | C | -- | 0.23 | C | -- | 0.8 | C | -- | C | C | C | | | C |
| C | | | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C |
| A | | RO | C | C | -- | C | C | C | -- | C | C | C | C | | -- | | C |
| B | | | -- | C | -- | -- | C | C | -- | C | -- | C | C | C | -- | -- | -- |
| C | | | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C |
| A | ENIG | | 0 | 0.0 | 0.1 | 0.01 | -- | 0.01 | 0.0 | -- | -- | -- | -- | -- | 0 | | -- |
| B | | | 160 | -- | -- | 320 | C | 980 | 1.6 | 72 | -- | -- | -- | -- | 270 | | -- |
| C | | | | -- | -- | -- | C | C | C | C | C | C | C | | -- | | C |
| A | | RO | 0 | 0 | 0.07 | 0 | -- | 0.01 | 0 | 0.5 | 0.1 | -- | -- | -- | 0.01 | | -- |
| B | | | 280 | -- | -- | 260 | C | 6.5 | 28 | 22 | 520- | 320 | -- | -- | 0.1 | | -- |
| C | | | -- | -- | -- | C | C | C | -- | C | C | C | C | | C | | C |
| A | OSP | OR | | | | | | | | | | | | | | | |
| B | | | | | | | | | | | | | | | | | |
| C | | | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C |
| A | | RO | | | | | | | | | | | | | | | |
| B | | | | | -- | C | | | | C | | | | | - | | |
| C | | | C | C | C | C | C | -- | C | C | C | C | C | | C | | C |

Following the round robin test runs, effort was focused on improving the reproducibility of the environment in the iNEMI FoS chamber. Tests were first run to determine the effect of the amount of sulfur in the chamber tray on the copper and silver corrosion rates. **Figure 3** shows the copper and silver corrosion rates as a function of sulfur content.

**Table 2c: Corrosion map after the third 5 days in FOS chamber with KCl saturated salt solution. The relative humidity range across the 3 test site was 61-78%.**

| Test site | Finish | Flux | **Location on board** (C refers to sites with corrosion. The darker shaded boxes are where creep corrosion occurred. The values in the darker shaded boxes are the resistance values in Ohms. | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
| A | ImAg | OR | C | C | -- | C | C | C | -- | C | 57 | C | C | C | -- | C | C |
| B | | | -- | -- | -- | -- | C | C | -- | C | -- | C | C | C | -- | | -- |
| C | | | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C |
| A | | RO | C | C | -- | C | C | C | -- | C | -- | C | C | C | -- | C | C |
| B | | | 0.2 | -- | 013 | -- | C | -- | 58 | C | -- | C | C | C | 720 | | -- |
| C | | | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C |
| A | ENIG | OR | 0 | 0.0 | 0.2 | 0.02 | | 0 | -- | 0.0 | 0.0 | 0.1 | -- | -- | -- | | 0.01 |
| B | | | 240<br>- | C | -- | 210 | C | -- | -- | -- | -- | -- | C | C | 800 | -- | -- |
| C | | | 0.1 | -- | -- | 0.04 | C | -- | 0.0<br>4 | -- | -- | C | C | | -- | | C |
| A | | RO | 0 | 0 | 0.1 | 0.07 | | 0.01 | -- | 0.0 | 0.0 | 0.5 | 0.06 | 0.5 | -- | | 0.01 |
| B | | | -- | -- | -- | -- | C | -- | -- | C | -- | C | C | C | -- | -- | -- |
| C | | | -- | -- | -- | 0.7 | C | -- | -- | C | C | C | C | | C | | C |
| A | OSP | OR | C | C | C | C | C | C | C | C | C | C | C | C | C | | C |
| B | | | -- | C | C | C | C | C | C | C | C | C | C | C | C | | C |
| C | | | C | C | -- | C | C | C | -- | C | -- | C | C | C | -- | C | -- |
| A | | RO | C | C | C | C | C | C | C | C | C | C | C | C | C | | C |
| B | | | -- | C | C | C | C | C | C | C | C | C | C | C | C | | C |
| C | | | C | C | -- | C | C | -- | -- | C | -- | C | C | C | -- | | C |

Next, tests were run to study the effect of household bleach dilution on copper and silver corrosion rates. The test results are listed in **Table 4.**

**DISCUSSION**

The objective of round robin test was to determine the reproducibility of the test conditions and the creep corrosion results. The important test conditions were the relative humidity level and the concentration of gases such as sulfur and chlorine in the iNEMI FoS chamber.

The relative humidity level was well controlled by design. The saturated salt solution had the dominant role in controlling the relative humidity. Some of the variability in the humidity levels listed in **Table 1** is probably more due to the accuracy of the relative humidity measurement than the actual variation in humidity.

| Saturated salt | $MgCl_2$ | $NH_4NO_3$ | KCl |
|---|---|---|---|
| Relative humidity range | 31-45 | 46-57 | 61-78 |
| Cu corrosion rate, coul redn/mass gain, Å/day | 1667/1797 | 604/904 | 687/-- |

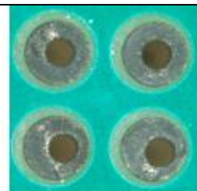
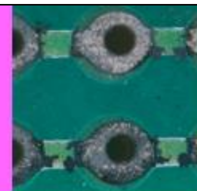
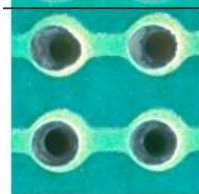
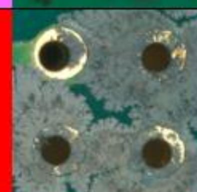
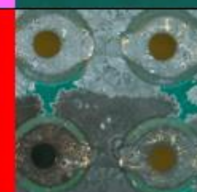
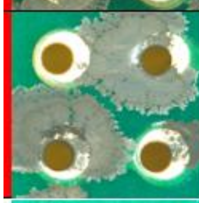
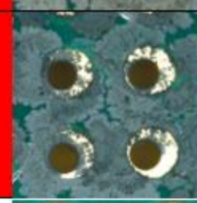
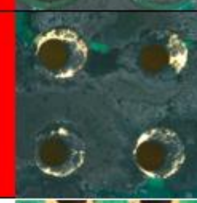
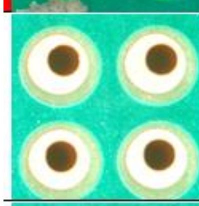
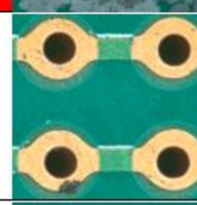
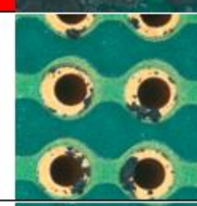
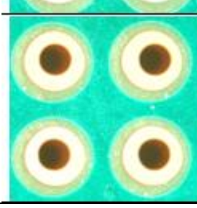
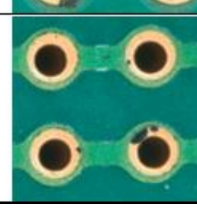
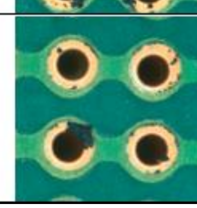
| | | | | |
|---|---|---|---|---|
| Ag corrosion rate, coul redn/mass gain, Å/day | | 1244/1561 | 558/719 | 378/-- |
| AgCl formation rate, Å/day | | 566 | 40 | 17 |
| Finish and flux | ImAg OR |  |  |  |
| | ImAg RO |  |  |  |
| | ENIG OR |  |  |  |
| | ENIG RO |  |  |  |
| | OSP OR |  |  |  |
| | OSP RO |  |  |  |

**Table 4: Copper and silver corrosion rates as a function of bleach dilution. 100% means no dilution. 50% means 1 part of water added to 1 part of bleach. All test runs except for the C9 run were for 5 days. The c9 run was for one day.**

| Run # | %RH | Bleach concentration | Bleach pH | Corrosion rates by coulometric reduction, Å/day | | | Corrosion rates by mass gain, Å/day | |
|---|---|---|---|---|---|---|---|---|
| | | | | Cu total | Ag total | AgCl | Cu total | Ag total |
| C5 | 60-63 | 100% | 10.8 | 769 | 668 | 59 | 928 | 739 |
| C7 | 58-61 | 50% | 10.8 | 1350 | 1058 | 53 | 1170 | 925 |
| C8 | 58-59 | 100% | 10.8 | 930 | 1296 | 21 | 1197 | 1058 |
| C9 | 58-59 | 50% | 10.8 | 1482 | 1349 | 30 | 1697 | 2590 |

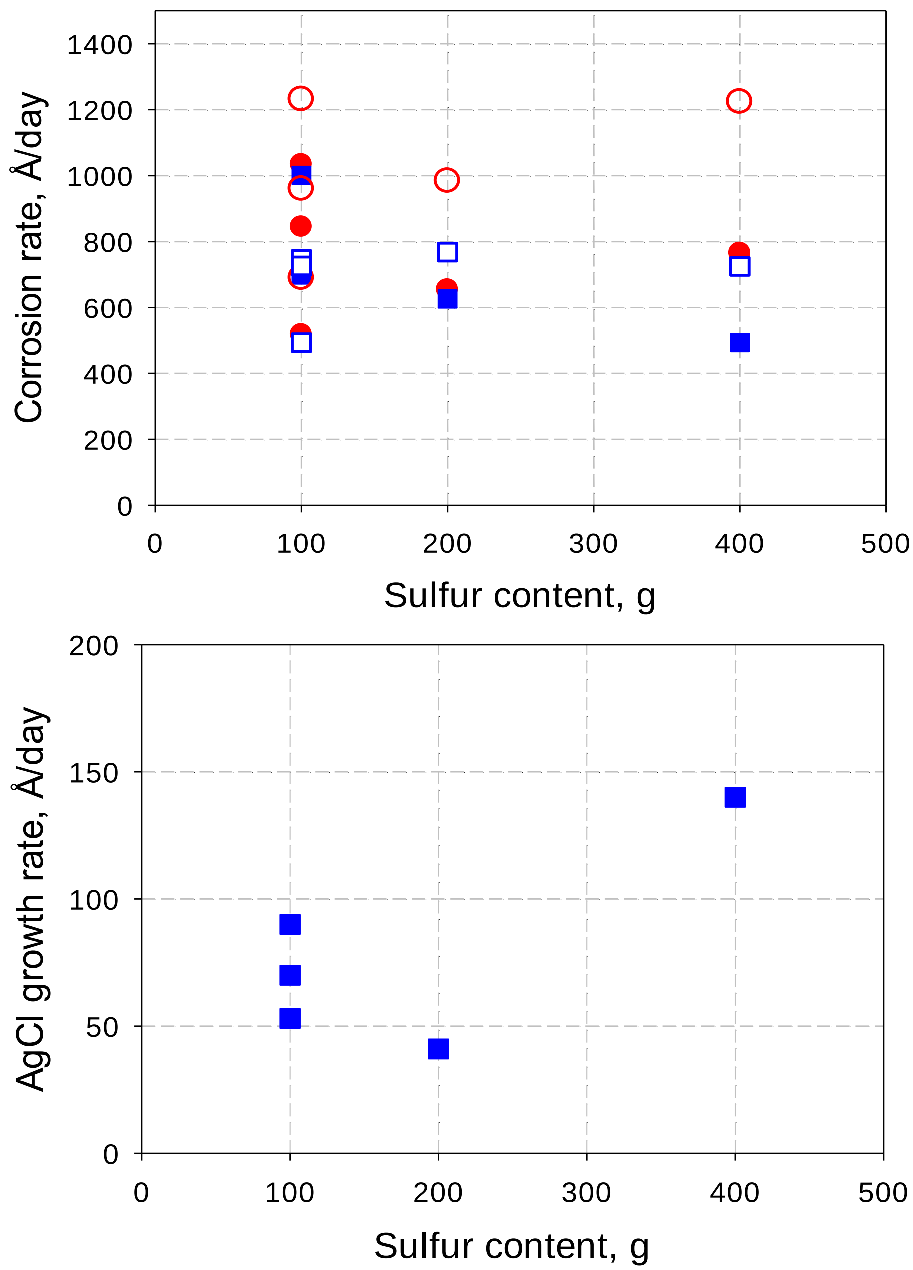



**Figure 3: Effect of sulfur content on the copper and silver corrosion rates. The red circular dots are for copper corrosion rates and the blue square dots are for silver corrosion rates. The open dots are for mass gain method and the solid dots for coulometric reduction method. The relative humidity of 46-57% was obtained using ammonium nitrate saturated salt solution.**

The concentrations of gases in the chamber were indirectly measured by measuring the copper and silver corrosion rates using the coulometric reduction and the mass gain methods. 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 clearly 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. **Table 1** shows that in general there is 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.

The creep corrosion results from the three participating companies on 6 different PCBs under three different humidity conditions are listed in **Tables 2a-2c.**A quick glance at 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 high rate of AgCl formation. Overall the creep corrosion test results from the three companies were in general agreement.

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 as shown in **Figures 3a and 3b**. 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 household bleach chemistry. Another source of variability may be variations in the synergistic effects of chlorine, sulfur and moisture in the air on the AgCl growth rate. We decided to study the role of household bleach dilution on the chlorine concentration as measured by the AgCl growth rate during 5-day test runs. The results are summarized in **Table 4**. One surprising result is that dilution of the household bleach by 50% did not decrease the rate of copper and silver corrosion. Work continues to improve the means of controlling the chlorine concentration in the chamber.

**Table 4: Copper and silver corrosion rates as a function of bleach dilution. 100% means no dilution. 50% means 1 part of water added to 1 part of bleach. All test runs except for the C9 run were for 5 days. The C9 run was for one day.**

| **Run #** | **%RH** | **Bleach concentration** | **Bleach pH** | **Corrosion rates by coulometric reduction, Å/day** | | | **Corrosion rates by mass gain, Å/day** | |
|---|---|---|---|---|---|---|---|---|
| | | | | Cu total | Ag total | AgCl | Cu total | Ag total |
| C5 | 60-63 | 100% | 10.8 | 769 | 668 | 59 | 928 | 739 |
| C7 | 58-61 | 50% | 10.8 | 1350 | 1058 | 53 | 1170 | 925 |
| C8 | 58-59 | 100% | 10.8 | 930 | 1296 | 21 | 1197 | 1058 |
| C9 | 58-59 | 50% | 10.8 | 1482 | 1349 | 30 | 1697 | 2590 |

## CONCLUSIONS

The round robin creep corrosion tests conducted on 6 different test PCBs at three companies gave quite similar results with some differences, the major one being that the ImAg PCBs suffered creep corrosion in company B with little to no creep corrosion occurring in company A and company 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.

If it is determined that the level of chlorine concentration control achievable today is not acceptable, more work will be need to devise means and determine precautions to tighten the standard deviation of the AgCl growth rate.

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# Round Robin Evaluation of iNEMI Creep Corrosion Qualification Test


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Simon Lee and Geoffrey Tong, The Dow Chemical Company, Tao Yuan Hsien, Taiwan

Chen Xu, Nokia, Murray Hill, NJ, USA

# Agenda

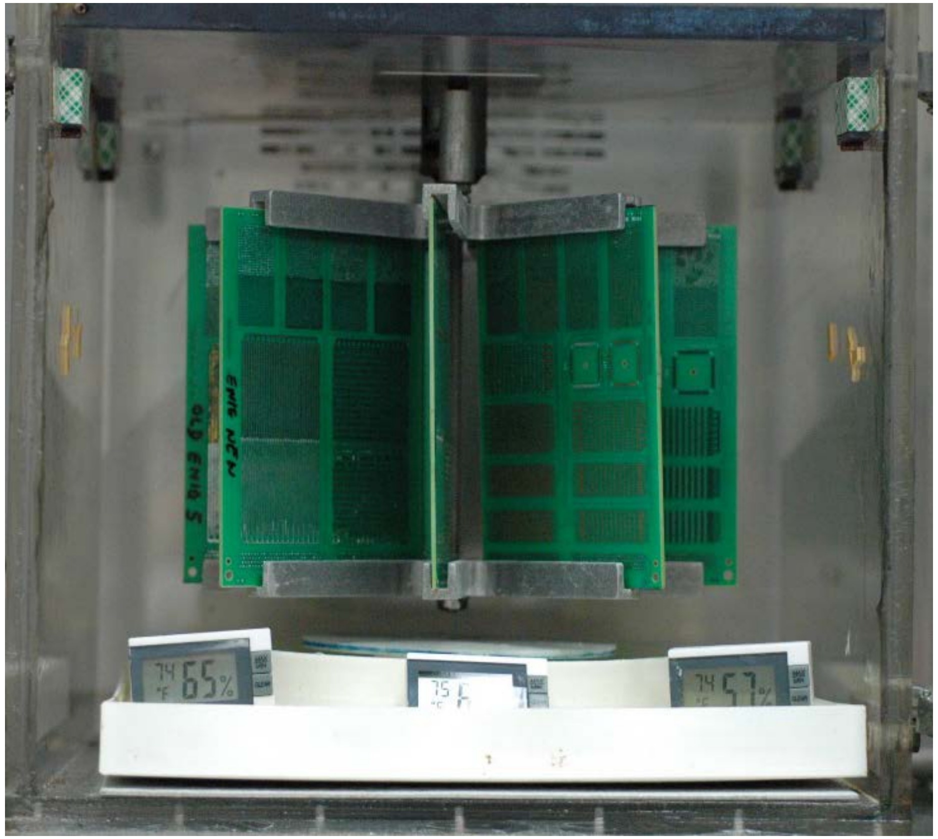
- Objective
- iNEMI flowers of sulfur chamber design
- Relative humidity control and corrosion rates in the chamber
- Creep corrosion round robin test results
- Chamber control
  - *Effect of sulfur*
  - *Effect of household bleach dilution*
- Conclusions

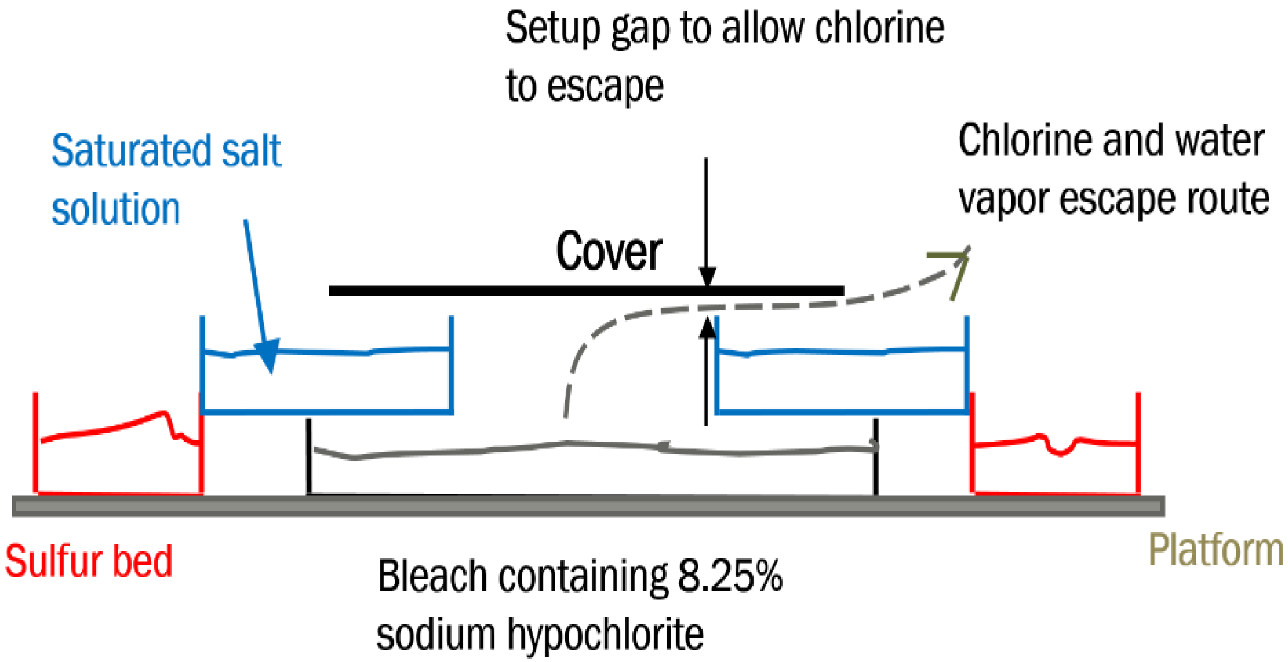
# Objective

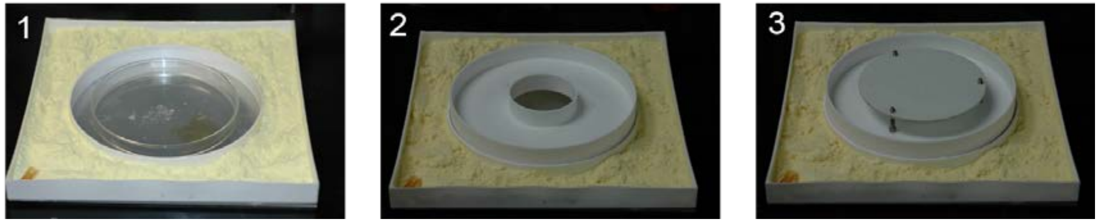
- The objective was to compare the round robin test results
  - *from three companies on*
  - *immersion silver (ImAg), electroless nickel immersion gold (ENIG) and organic solderability preservative (OSP) finished PCBs*
  - *soldered using organic acid (OR) and rosin (RO) fluxes.*
- The objective was also to evaluate and improve the reproducibility of the relative humidity control and the copper and silver corrosion rates in the iNEMI FOS chamber.

# iNEMI flowers of sulfur chamber

- The chamber is at 50ºC.
- Paddle wheel rotated at 20 rpm.
- 8 paddles per wheel. One paddle reserved for copper and silver corrosion foils.





# Relative humidity and corrosion rates in the three 5-day teat runs at company A, B and C

| Site | Saturated salt | Household bleach container 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 |
|---|---|---|---|---|---|---|
| A | $MgCl_2$ | 1 mm | 31-45 | 1667/1797 | 1244/1561 | 566 |
| B | | 1 mm | 38-42 | 1650/6591 | 1707/3455 | 598 |
| C | | 1 mm | 38-42 | 1544/– | 1017/– | 27 |
| A | $NH_4NO_3$ | 1 mm | 46-57 | 604/904 | 558/719 | 40 |
| B | | 1 mm | 50-51 | 4105/2545 | 1236/3428 | 841 |
| C | | 1 mm | 51-53 | 1386/– | 1111/– | 16 |
| A | KCl | 1 mm | 61-78 | 687/– | 376/– | 17 |
| B | | 3 mm | 73-75 | 1603/3178 | 882/2720 | 176 |
| C | | 1 mm | 72-73 | 1369/– | 704/– | 16 |

# First 5-day run at 31-45% RH (Resistance is in kΩ)

| Site | Board | 1L | 2L | 3L | 4L | 5L | 6L | 7L | 8L | 9L | 10L | 11L | 12L | 13L | 14L | 15L |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A | ImAg/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| B | ImAg/OR flux | 0.07 | -- | -- | 0.07 | -- | -- | 53 | 384 | 25 | -- | -- | -- | 4.5 | -- | -- |
| C | ImAg/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | ImAg/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| B | ImAg/RO flux | 24 | -- | -- | -- | -- | -- | 0.03 | 580 | -- | 702 | -- | -- | 0.05 | -- | -- |
| C | ImAg/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | ENIG/OR flux | 0.02 | 0.23 | 21 | 0.04 | -- | 0.07 | -- | -- | -- | -- | -- | -- | 0.02 | | |
| B | ENIG/OR flux | 0.01 | 1 | | 0.03 | -- | --- | 0.05 | -- | -- | -- | -- | -- | 0.02 | | |
| C | ENIG/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | ENIG/RO flux | 0 | 0.44 | -- | 0 | -- | 0.07 | 0.01 | -- | 91 | -- | -- | -- | 0.01 | | |
| B | ENIG/RO flux | 0.05 | 0.74 | -- | 0.01 | -- | 3.7 | 0.02 | -- | -- | -- | -- | -- | 0.01 | | |
| C | ENIG/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | OSP/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| B | OSP/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| C | OSP/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | OSP/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| B | OSP/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| C | OSP/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |

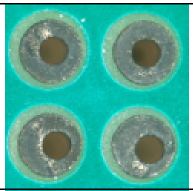
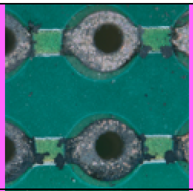
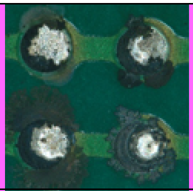
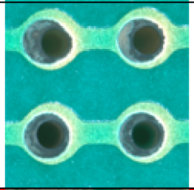
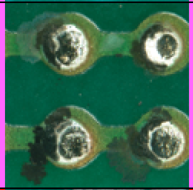
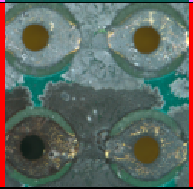
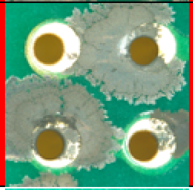
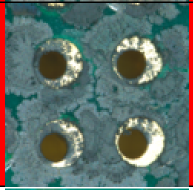
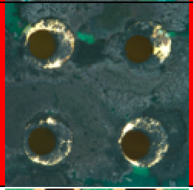
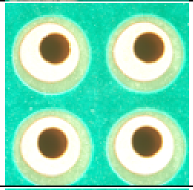
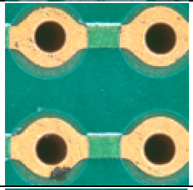
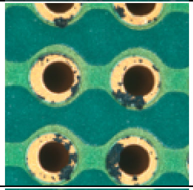
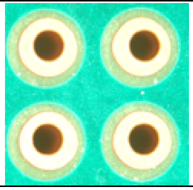
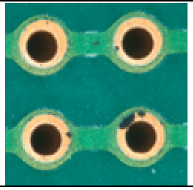
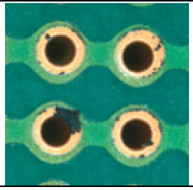
# Second 5-day run at 46-57% RH (Resistance is in kΩ)

| Site | Board | 1L | 2L | 3L | 4L | 5L | 6L | 7L | 8L | 9L | 10L | 11L | 12L | 13L | 14L | 15L |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A | ImAg/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| B | ImAg/OR flux | | | | 0.23 | | | 0.75 | | | | | | | | |
| C | ImAg/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | ImAg/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| B | ImAg/RO flux | | | | | | | | | | | | | | | |
| C | ImAg/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | ENIG/OR flux | --0 | 0.02 | 0.09 | 0.01 | -- | 0.01 | 0.01 | -- | 0.11 | -- | -- | -- | 0 | -- | -- |
| B | ENIG/OR flux | 160 | | | 320 | | 980 | 1.63 | 72 | | | | | 270 | | |
| C | ENIG/OR flux | 0.872 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | ENIG/RO flux | 0 | 0 | 0.07 | 0 | -- | 0.01 | 0- | 0.46 | 0.06 | -- | | | --0.01 | -- | -- |
| B | ENIG/RO flux | 280 | | | 260 | | 6.5 | 28 | 22 | 520 | 320 | | | 0.1 | | |
| C | ENIG/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | OSP/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| B | OSP/OR flux | | | | | | | | | | | | | | | |
| C | OSP/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | OSP/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| B | OSP/RO flux | | | | | | | | | | | | | | | |
| C | OSP/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |

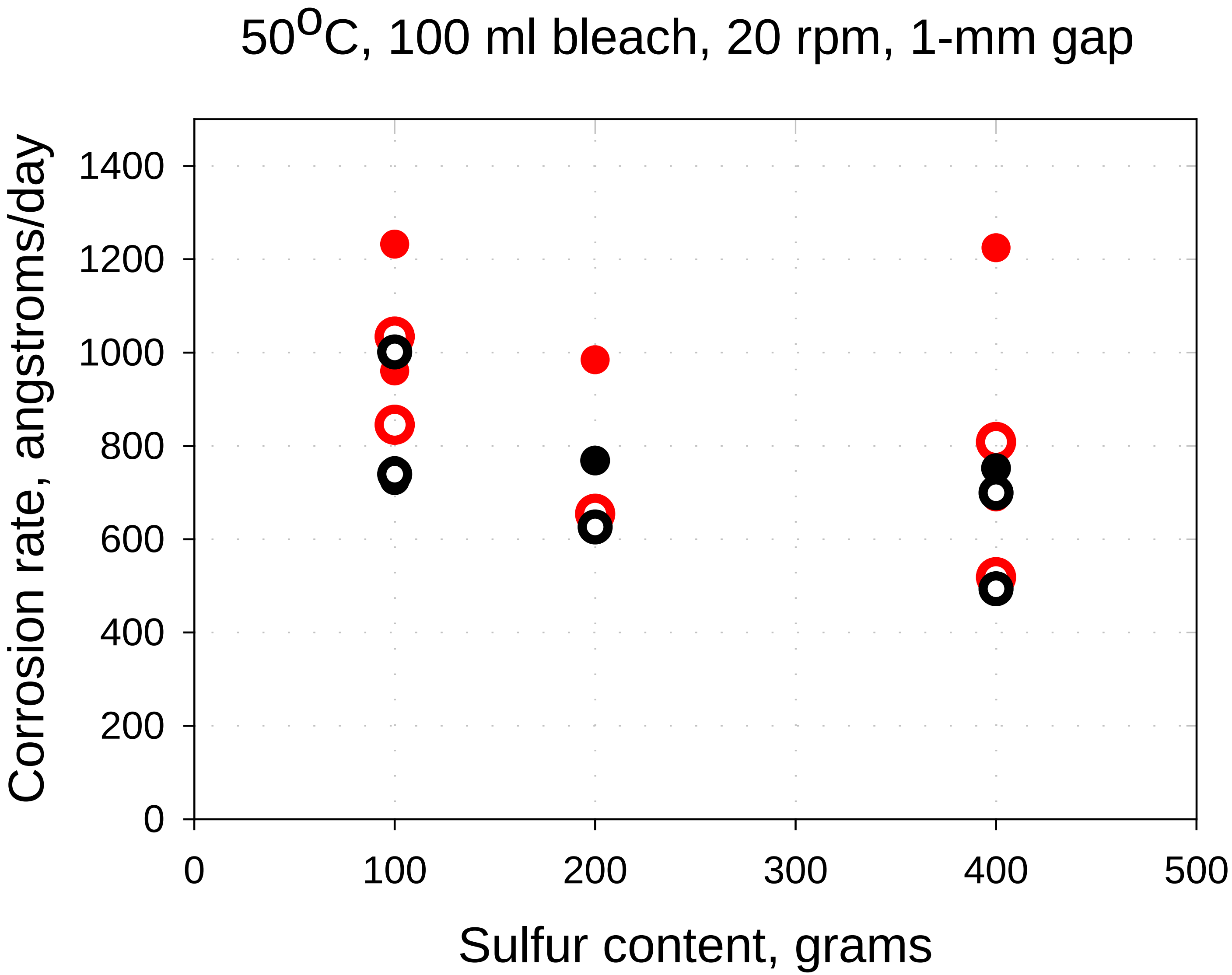
# Third 5-day run at 61-78% RH (Resistance is in kΩ)

| Site | Board | 1L | 2L | 3L | 4L | 5L | 6L | 7L | 8L | 9L | 10L | 11L | 12L | 13L | 14L | 15L |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A | ImAg/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | 57 | -- | -- | -- | -- | -- | -- |
| B | ImAg/OR flux | 2.8 | | | 300 | | | | | | | | | 520 | | |
| C | ImAg/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | ImAg/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| B | ImAg/RO flux | 0.3 | | 0.13 | | | | 58 | | | | | | 720 | | |
| C | ImAg/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | ENIG/OR flux | 0 | 0.02 | 0.15 | 0.02 | -- | 0 | -- | 0.01 | 0.01 | 72.58 | 0.1 | -- | -- | -- | 0.01 |
| B | ENIG/OR flux | 240 | | | 210 | | | | | | | | | 800 | | |
| C | ENIG/OR flux | 0.068 | -- | -- | 0.0457 | -- | -- | -- | 0.045 | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | ENIG/RO flux | 0 | 0 | 0.06 | 0.01 | -- | 0.01 | -- | 0.01 | 0.01 | 0.53 | 0.06 | 0.5 | -- | -- | 0.01 |
| B | ENIG/RO flux | | | | | | | | | | | | | | | |
| C | ENIG/RO flux | -- | -- | -- | 0.706 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | OSP/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| B | OSP/OR flux | | | | | | | | | | | | | | | |
| C | OSP/OR flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| | | | | | | | | | | | | | | | | |
| A | OSP/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| B | OSP/RO flux | | | | | | | | | | | | | | | |
| C | OSP/RO flux | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |

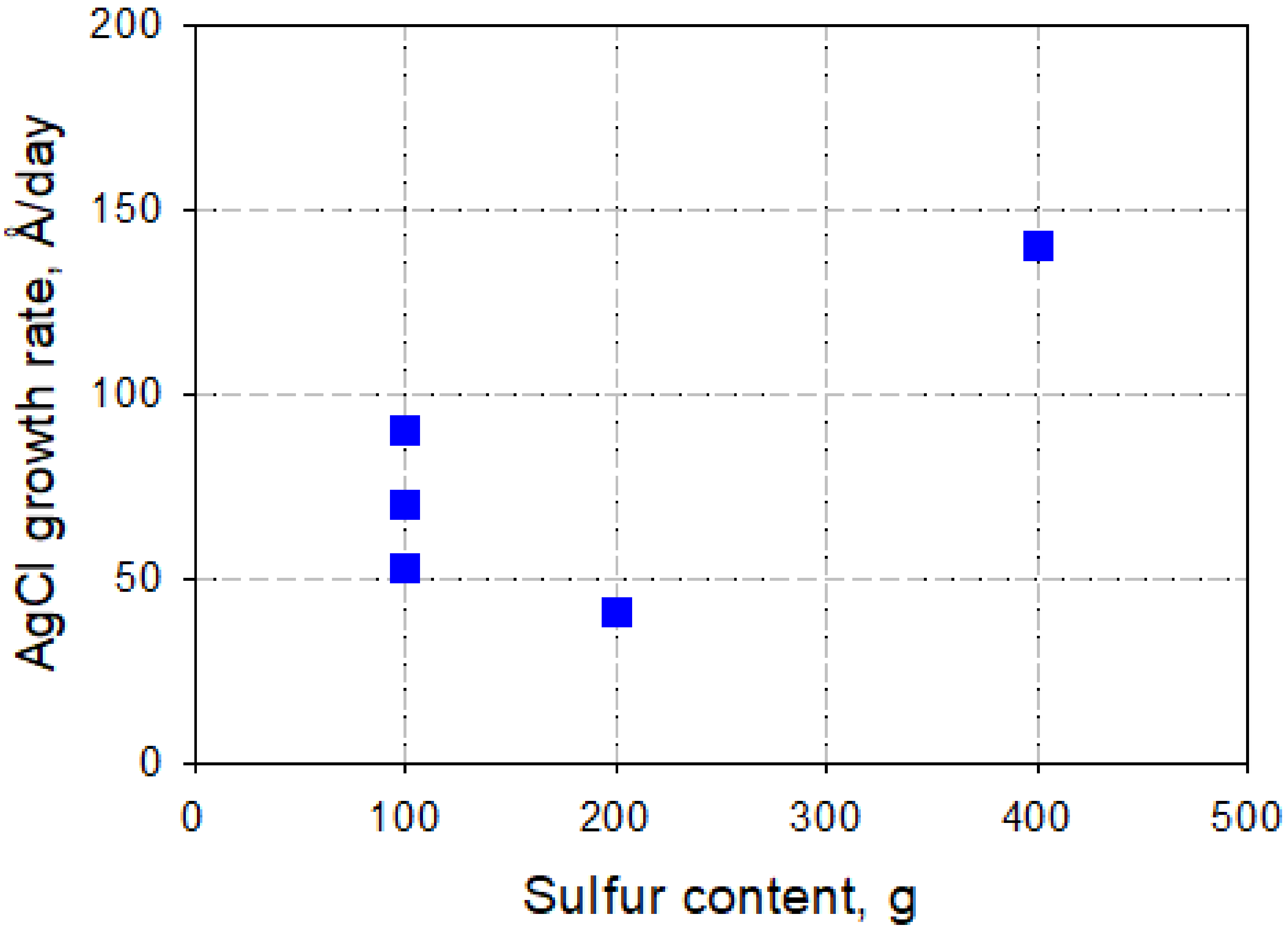
# Worst-case creep corrosion during the three 5-day test runs in company A

| Saturated salt | | $MgCl_2$ | $NH_4NO_3$ | KCl |
|---|---|---|---|---|
| Relative humidity range | | 31-45 | 46-57 | 61-78 |
| Finish | ImAg, OR |  |  |  |
| | ImAg, RO |  |  |  |
| | ENIG ,OR |  |  |  |
| | ENIG, RO |  |  |  |
| | OSP, OR |  |  |  |
| | OSP, RO |  |  |  |

# Effect of sulfur conent on copper and silver corrosion rates



# Effect of sulfur content on AgCl growth rate



# Effect of bleach dilution on corrosion rates

FOS with 200 g sulfur, $NaNO_2$ saturated salt solution, 100 ml bleach, with 8.25% sodium hypochlorite, 50ºC, 20 rpm, 5 day runs.
100% dilution means no dilution.
50% dilution means 50 g bleach (with 8.25% sodium hypochlorite) and 50 g DI water.

| ID | %RH | Bleach dilution | Bleach pH before and after | Corrosion rates by coul reduction, Å/day | | | Corrosion rates by mass gain, Å/day | |
|---|---|---|---|---|---|---|---|---|
| | | | | Cu | Ag | AgCl | Cu | Ag |
| C5 | 60-63 | 100% | 10.8/9.4 | 769 | 668 | 59 | 928 | 739 |
| C6* | 60-61 | 50% | 10.8/-- | 1910 | 1046 | 173 | 2095 | 994 |
| C7 | 58-61 | 50% | 10.8/-- | 1350 | 1058 | 53 | 1170 | 924 |
| C8 | 58-59 | 100% | 10.8/-- | 930 | 1296 | 21 | 1197 | 1058 |
| C9** | 58-59 | 50% | 10.8/9.3 | 1482 | 1349 | 30 | 1697 | 2590 |

* Bleach spilled on aluminum plated steel; may have released gas. **One day run.

# Conclusions

- The results of the round robin test are in general agreement within the limits of the reproducibility expected of corrosion phenomenon.
- The test results in general agree with the industry position that
  - *ENIG finished PCBs are very prone to creep corrosion and that*
  - *OSP finished PCBs are very resistant to creep corrosion.*
- The amount of sulfur has no effect on the Cu and Ag corrosion rates.
- The effect of the dilution of the household bleach and the control of chlorine gas in the chamber needs further study.