

Conformal Coating Testing in Various Test Environments

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ABSTRACT

Conformal coatings have traditionally been tested by determining the mean time to failure of conformally coated hardware exposed to corrosive test environments. This test approach has serious shortcomings: The test temperatures are most often too high. At these high temperatures, the conformal coating properties may be quite different from those at the application temperatures. In addition, the times to failure are unacceptably long extending into many months. Overcoming these shortcomings is an iNEMI championed test that involves exposing conformally coated thin films of copper and silver to sulfur vapors at 40-50 °C in flowers of sulfur (FoS) chamber and using the corrosion rates of the coated metal thin films as a measure of the corrosion protection capabilities of the conformal coatings. The test temperatures are similar to the application temperatures, the test durations are no more than a week and can be conducted under various temperature and humidity conditions. The purpose of this paper was to determine if testing in the industry-standard mixed-flowing gas corrosion chamber would give similar results as those using the FoS chamber. Acrylic, fluorinated acrylate, and atomic layer deposition conformal coatings were tested in three environments: (a) flowers of sulfur (FoS), (b) mixed-flowing gas (MFG), and (c) iodine vapor. The performance of the coatings tested in the FoS and the MFG corrosion chambers were quantitatively similar. The iodine vapor test results were in qualitative agreement with the FoS and MFG test results.

INTRODUCTION

Conformal coatings protect coated hardware from the ill effects of moisture, dust, and corrosive gases. With ever-expanding markets for electronic goods, the world over, in environments ranging from benign to very harsh, the need for conformal coatings is ever increasing. Given a wide range of available conformal coatings and given a multitude of hardware on which they need to be applied, it behoves the industry to have standardized means of testing conformal coatings under the various application conditions.

Until recently, it was typical to test conformal coatings by applying them to actual hardware and determining the mean time to failure at high temperatures in the neighbourhood of 100°C in corrosive, generally flowers of sulfur, environments [1]. This approach suffers two shortcomings: One is that the conformal coating properties at high-test temperatures may be very different from those at the application temperatures; the other is that the test times are too long, extending into many months and sometimes longer. An iNEMI championed novel test approach overcomes these shortcomings by applying the coatings to metal thin films and characterizing the conformal coatings by the degree of corrosion protection they provide the underlying metal thin films [2]. The test temperatures are similar to the application temperatures and the test durations are as short as one week. In the first phase of the iNEMI project that studied this approach, testing was conducted in flowers of sulphur (FoS) chambers. Given that the industry-accepted environment for testing electronic hardware is the mixed-flowing gas (MFG) environment, iNEMI embarked on phase 2 of the project to compare conformal coating test results from testing in the FoS environment to those in the MFG environment. The iodine vapour test environment was included as the third environment in the project [3].

This paper compared these three environments in terms of how well they characterize conformal coatings. The conformal coatings chosen for testing provided a range of corrosion protection to the underlying metal thin films: acrylic coating provided moderate corrosion protection; fluorinated acrylate coating provided improved corrosion protection and the atomic layer deposited (ALD) coating provided excellent corrosion protection. The FoS and the MFG tests provided quantitatively similar results on the corrosion protection abilities of the conformal coatings. The iodine vapor test, taking no more than an hour to conduct, provided qualitative corrosion characterization of the coatings that agreed with the results of the FoS and the MFG tests.

TEST METHODS

Conformal coatings were quantitatively characterized by the degree of corrosion protection the coatings provided the underlying, 800-nm thick silver and copper serpentine thin films when exposed to corrosive FoS or MFG environments. Figure 1 illustrates a metal thin-film test coupon as a serpentine metal thin film deposited on silicon oxide on silicon mounted on a pegboard with 4-point resistance measurement connections to a receptacle connector. Conformal coatings were characterized by comparing the corrosion rates of the coated metal thin films to those of uncoated, bare metal thin films. The corrosion rates of the thin films in the FoS and the MFG environments were measured electrically via the rates of resistance increase of the films. Thin film electrical resistances were measured via the 4-point resistance method, in galvanostatic mode, in which known currents were pumped through the thin films and the potential drops across them measured. Tests were run in low (10%), medium (32-34%) and high (70-75%) relative humidity environments at 30°C in the MFG test chamber and at 40°C in the FoS test chamber.

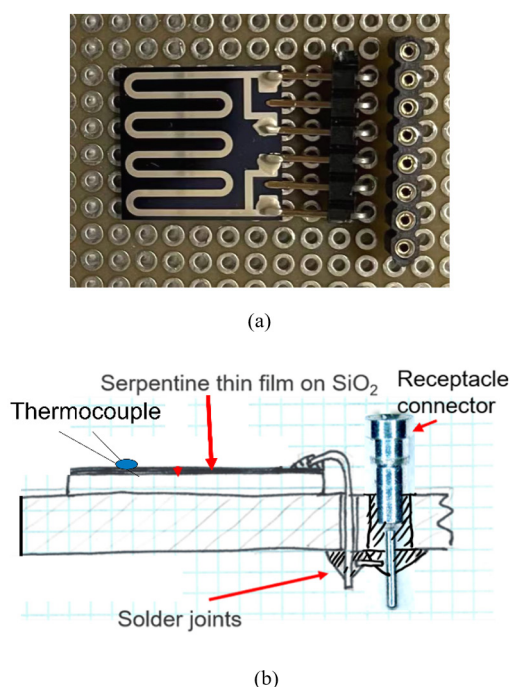


Figure 1: Thin-film test coupon showing (a) silver serpentine thin film on SiO₂/Si mounted on a circuit board and (b) schematic of cross-section of test coupon showing the way to connect the thin film to the receptacle connector.

The FoS chamber setup is described in Figure 2. The corrosive gas in the FoS chamber was sulfur gas with its concentration kept constant by keeping the chamber temperature constant. The relative humidity in the chamber was kept constant using a saturated salt solution with its deliquescent relative humidity matching the desired relative humidity. The thin films and the coatings housed in

a FoS chamber at 40°C were heated to and kept constant at various temperatures as high as 75°C, using joule heating. The currents used to measure the film resistances were also used to joule heat the thin films and the conformal coatings covering them. The temperatures of the coatings were measured using 50- μ m diameter T-type thermocouples glued to the coatings. The testing in an industry standard MFG chamber was like that in the FoS chamber with a couple of differences. The MFG chamber was held at 30°C and the gas composition was 200 ppb SO₂; 100 ppb H₂S; 200 ppb NO_x and 20 ppb Cl₂.

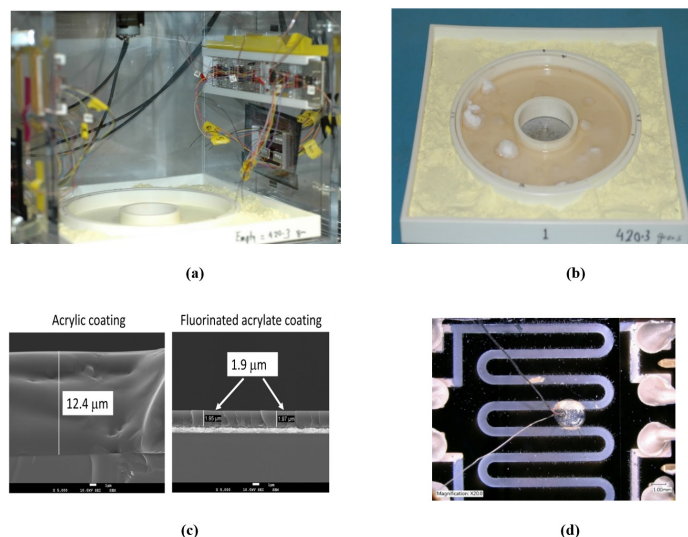


Figure 2: FoS chamber test setup showing (a) the inside of the chamber with thin films mounted on the chamber walls and sulfur and saturated salt trays on the bottom; (b) the flowers of sulfur tray surrounding the saturated salt solution tray; (c) cross sections of acrylic and fluorinated acrylate coatings, and (d) enlarged view of a silver serpentine thin film showing the thermocouple bonded to the surface adjacent to the thin film.

The corrosivity of the FoS and MFG environments were measured by measuring the corrosion rates of bare copper and bare silver thin films and foils. The thin metal film corrosion rates were measured using the rates of electrical resistance increase method. The metal foil corrosion rates were measured either by the mass gain method or by coulometric reduction [4]. Figure 3 summarized these corrosion rates as a function of relative humidity. The corrosion rates of thin metal films are in general higher than those of metal foils because thin films have finer grain size and have higher internal mechanical stresses.

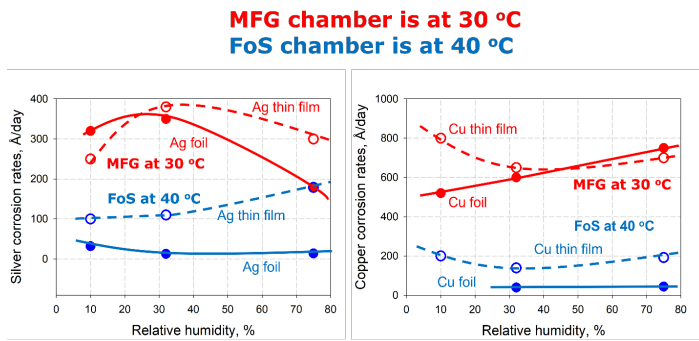


Figure 3: Corrosion rates of bare copper and bare silver thin films and metal foils in MFG and FoS chambers as a function of relative humidity were used to characterize the corrosivity of the chamber environments.

The iodine vapor test involved exposing coated serpentine metal thin films to iodine vapor at 40°C and 100% relative humidity for 30 and 60 minutes and optically estimating the extent of the metal film corrosion. The test setup is shown in Fig. 4a and 4b. The iodine vapors that come off an iodine saturated aqueous solution at 70°C in a sealed container are at 40°C, as is evident in the graph of Fig. 4c.

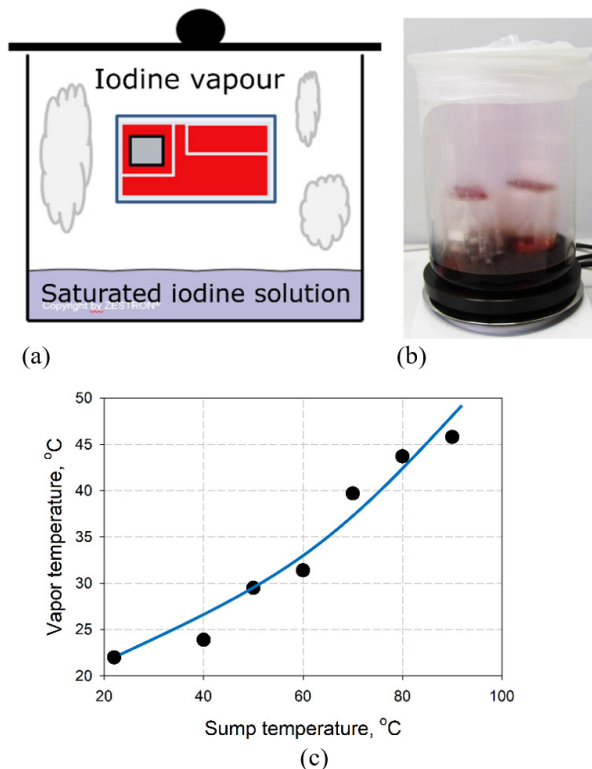


Figure 4: Description of the iodine vapor test: (a) Schematic of the setup; (b) Test chamber; (c) Plot showing that at 70°C sump temperature, the vapor temperature was 40°C. The relative humidity in the chamber was 100%.

In the FoS and the MFG chambers, the extents of the corrosion of bare and of conformally coated copper and silver thin films were measured as a function of time at various conformal coating temperatures. The corrosion rates at various coating temperatures were obtained from the slopes of these plots.

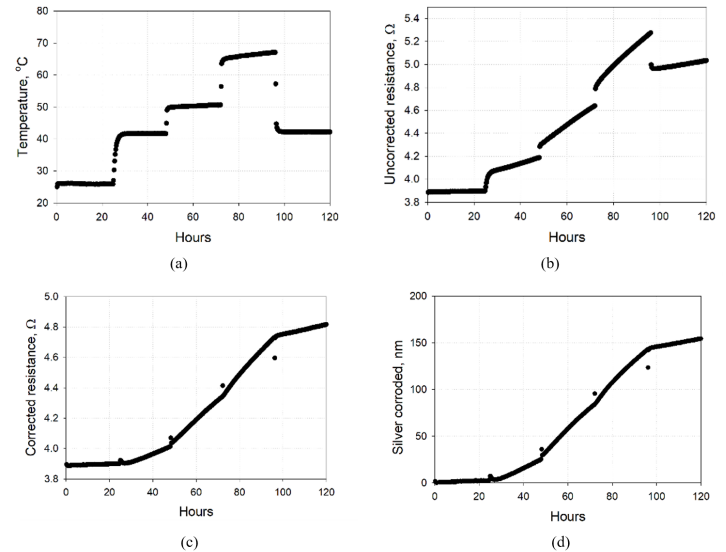


Figure 5: Steps are shown to obtain the thickness of silver corroded as a function of time. This example is for bare silver corroding in 40°C, 10% relative humidity environment. The thin-film temperature was stepped to various values by joule heating the film.

The steps followed to determine the thickness of metal film corroded away as a function of time during five periods, each period at a constant temperature lasting one day in the FoS test, are shown in Fig. 5. The periods were shorter in the MFG test (4 to 16 hours) because of the higher corrosion rates in the MFG environment. The example shown in Fig. 5 is for testing in the FoS environment. During the first period which was the first day in the FoS test, the test chamber was at room temperature. For the remaining four periods, each lasting a day in the FoS test, the chamber temperature was raised to and held constant at 40°C. During day 2, the joule heating resulting from 100-mA current raised the film temperature slightly above the ambient to about 42°C; during day 3, the 200-mA current raised the film temperature to about 52°C; during day 4, the 300-mA current raised the film temperature to about 68°C; and during day 5, the 100-mA current lowered the film temperature to about 43°C. The jogs in the as-measured resistance plot, shown in Fig 5b, we term “uncorrected” resistance, at the instant of change of temperature of the film, were the direct result of the temperature coefficient of electrical resistivity of the metal films. We can compensate for this effect by calculating what would be the film resistance if the film was to be cooled to 40°C. This corrected film resistance, with the jogs smoothed out, is plotted in Fig. 5c. Knowing that the film thickness is inversely proportional to its electrical resistance, the thickness of metal film corroded away can be calculated. The above-described procedure was repeated in

low, medium, and high relative humidity ambiances at 40°C in FoS and at 30°C in MFG environments, on copper and silver thin films coated with the three coatings under test along with the uncoated thin films.

RESULTS AND DISCUSSION

An example of coated and uncoated silver and copper thin films corrosion in the FoS chamber is shown in Fig. 6 for 32% relative humidity and 40°C test run. The plots of the thickness of silver and copper corroded versus time have almost no jogs associated with temperature changes, indicating well-chosen values of the silver and copper coefficients of electrical resistivities. Anyway, it was discovered that the selected coefficients of resistivity values had little effect on the calculated corrosion rates. From the slopes of a metal corroded plot (Fig 7a) at various temperatures, the corrosion rates can be obtained and plotted in an Arrhenius fashion as shown in Fig. 7b.

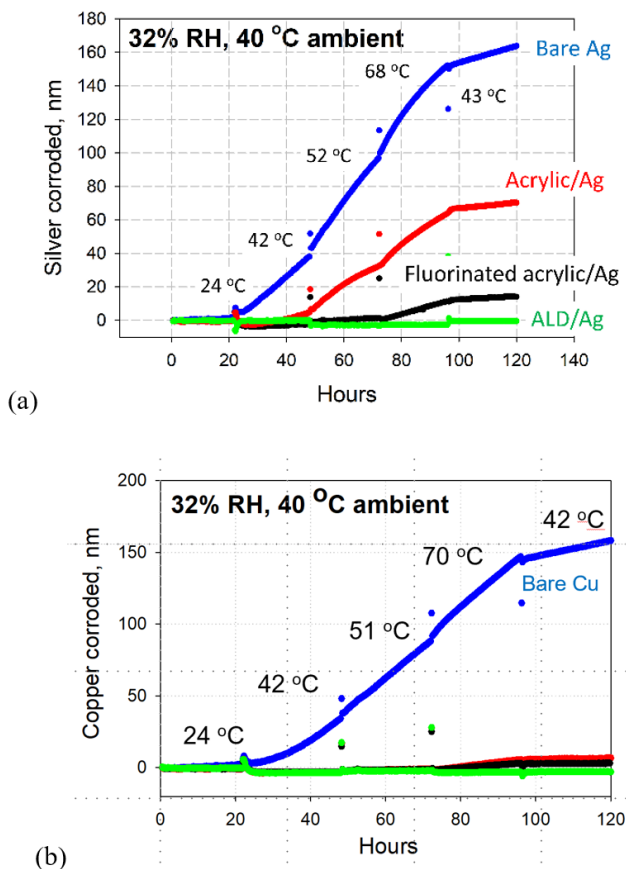


Figure 6: Example of corrosion in FoS chamber at 32% relative humidity and 40°C. (a) Thickness of Ag corroded; (b) Thickness of Cu corroded. Note the low corrosion rates of coated Cu compared to coated Ag, even though the coated Cu and Ag film corrosion rates were similar.

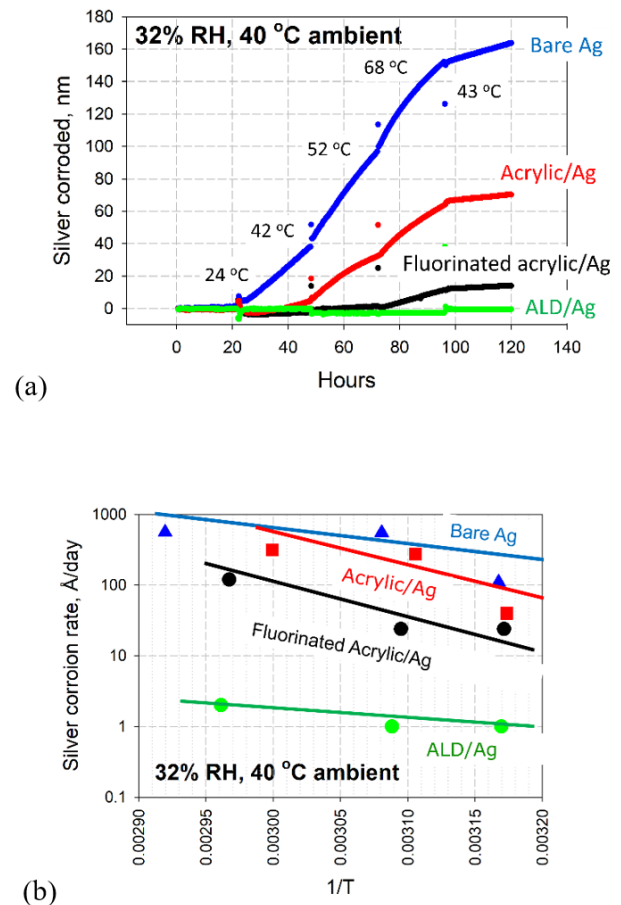


Figure 7: Example of converting thickness of silver corroded vs time at various temperatures into an Arrhenius plot.

Before summarizing/displaying the data on Arrhenius plots, it is worth delving into the advantage of Arrhenius plots: They allow a wide range of corrosion rates stretching over many orders of magnitude to be plotted on one plot and equally importantly the slopes of the plots shed light on the rates of corrosive gas transport through the conformal coatings and the reaction rates between the gases and the metal. Data lying on a linear plot with no break in slope indicates one controlling mechanism, whereas a change in slope indicates changing mechanisms over the temperature range of measurement. Fig. 8 and 9 capture the corrosion rates data of coated and uncoated silver and copper thin films over three orders of magnitude of corrosion rates. However, the standard deviations of the corrosion rates are too high to be of use in discussions on the mass transport mechanisms and reaction rates at the metal thin film surfaces. The purpose and discussion of the Arrhenius plots in the following paragraphs are therefore generally limited to the concise presentation of corrosion rate data rather than the mass transport and chemical reaction rates and their mechanisms and the associated activation energies.

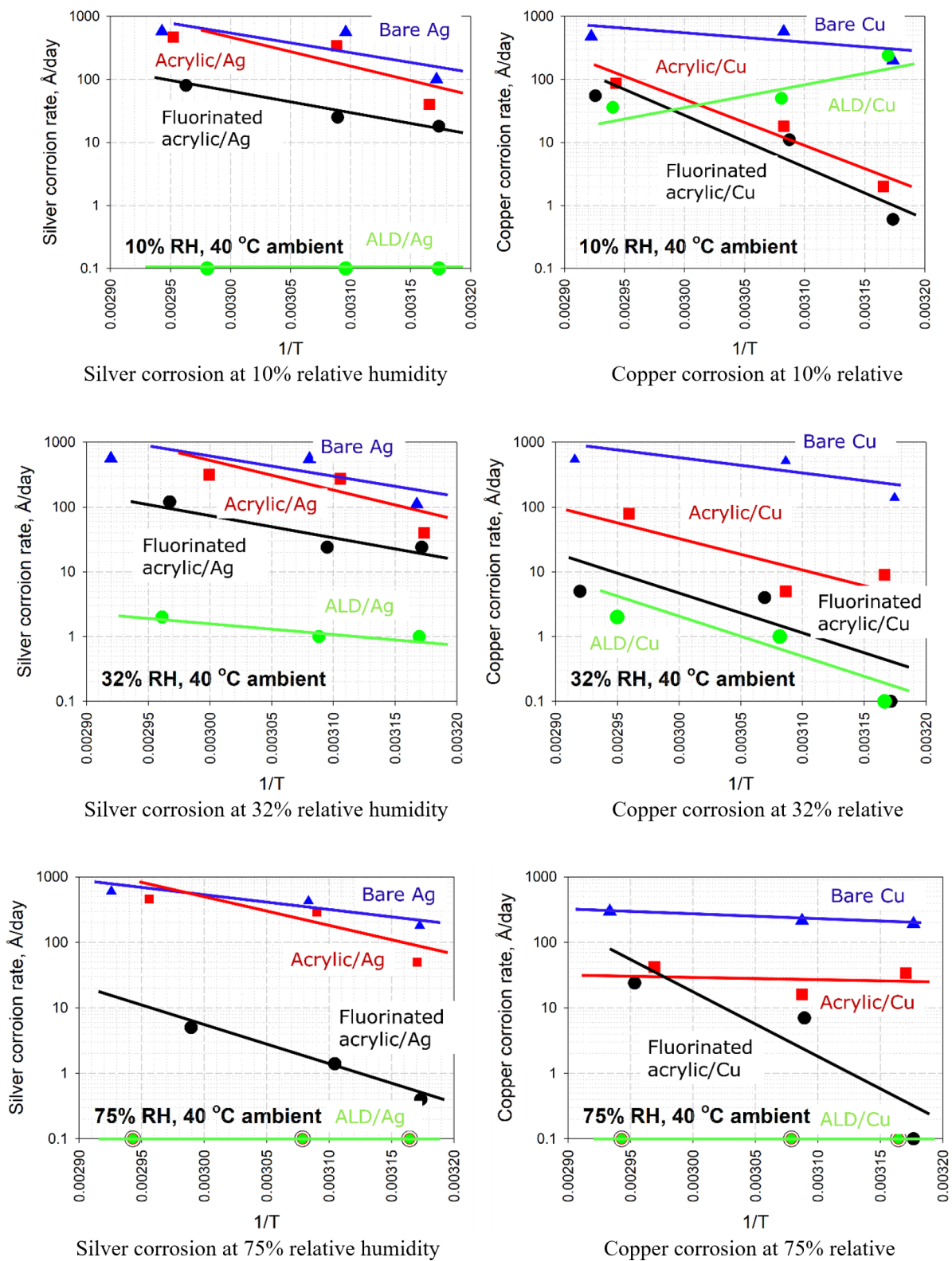
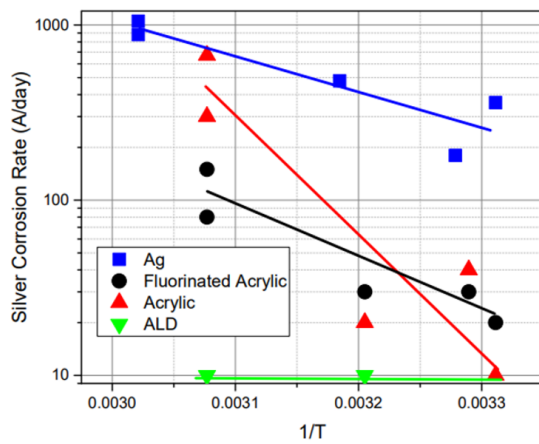
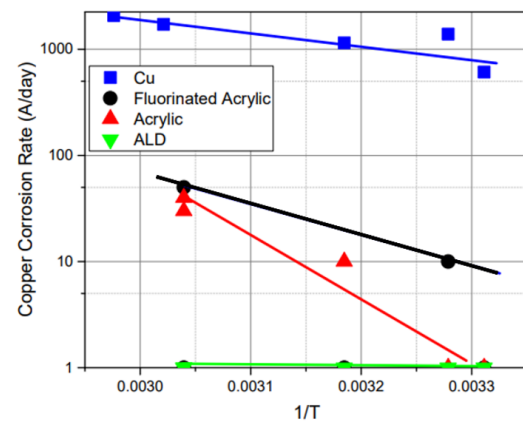


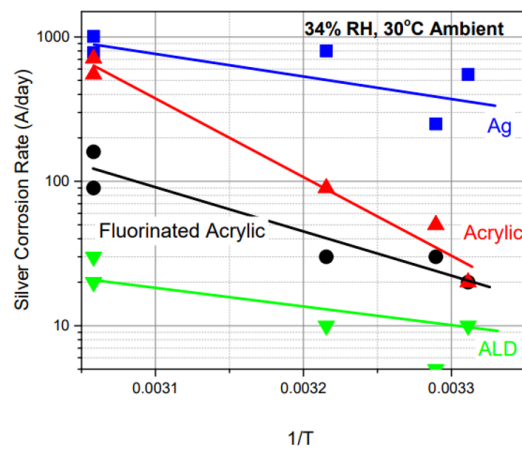
Figure 8: Arrhenius plots of silver and copper corrosion rates in FoS chamber at various relative humidity levels at 40°C chamber temperature.



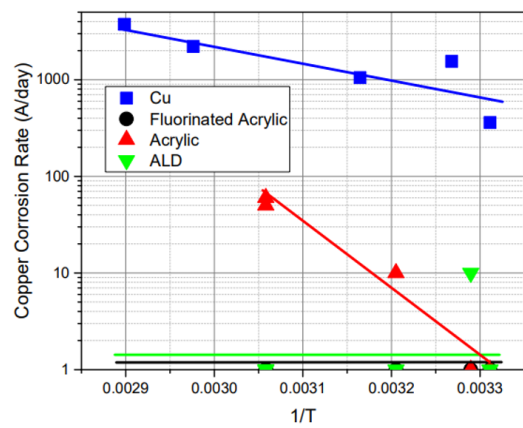
Silver corrosion at 10% relative humidity



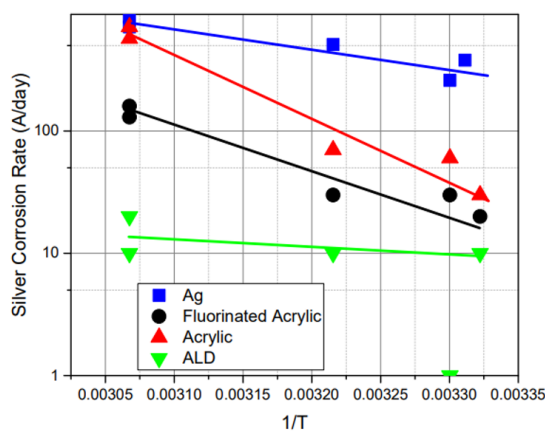
Copper corrosion at 10% relative humidity



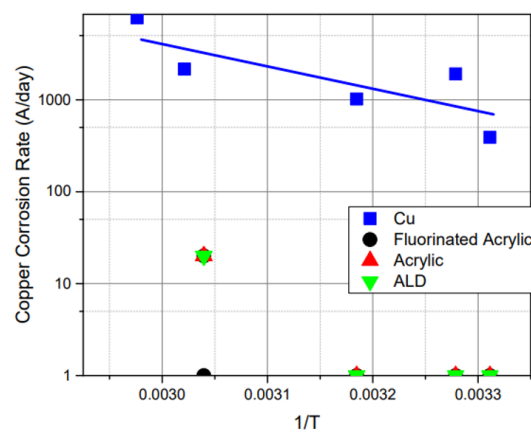
Silver corrosion at 34% relative humidity



Copper corrosion at 34% relative humidity



Silver corrosion at 70% relative humidity



Copper corrosion at 70% relative humidity

Figure 9: Arrhenius plots of silver and copper corrosion rates in MFG chamber at various relative humidity levels at 30°C chamber temperature.

Table 1: Iodine vapor test results

Coating	Thin film	After 30 minutes in iodine vapors	After 60 minutes in iodine vapor
Acrylic	Ag	Corroded except at the edge where the coating was thicker	Fully corroded
	Cu	No corrosion	Corrosion occurred except in areas of thicker coating
Fluorinated acrylate	Ag	Corroded	Corroded
	Cu	Corrosion started	Corrosion progressed
ALD	Ag	No corrosion	Corrosion started on the edges
	Cu	Slight corrosion	Corrosion proceeded

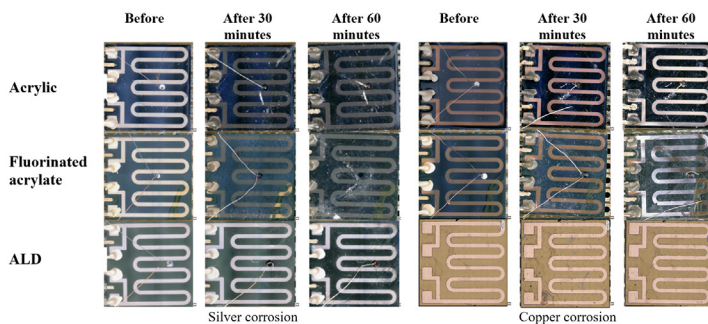


Figure 10: Visual inspection of the coated thin films subjected to iodine vapors for 30 and 60 minutes under a low magnification microscope indicates that the thin films under the acrylic coatings corroded the most, the fluorinated acrylate coated thin films corroded less and the ALD coated thin films corroded the least. The ALD-coated thin copper films look different because the thin films were precleaned before the deposition of the ALD coating.

The corrosion rates of uncoated and coated copper and silver thin films in 10, 32, and 75% relative humidity FoS environments are summarized in the Arrhenius plots of Figure 8. Notice that in general the corrosion rates of coated copper thin films are less than those of coated silver thin films indicating that copper is better protected from corrosion by the conformal coatings.

The corrosion rates of uncoated and coated copper and silver thin films in 10, 34, and 70% relative humidity MFG environments are summarized in the Arrhenius plots of Figure 9. Notice that, in this case too, in general, the corrosion rates of coated copper thin films are less than those of coated silver thin films indicating that copper is better protected from corrosion by the conformal coatings.

As expected by the more corrosive environment in the MFG chamber, the corrosion rates of uncoated and coated thin films were in general higher in the MFG chamber compared to in the FoS chamber; though the results from the coatings tested in the two environments trend in a similar manner: The ALD coating

provided the best corrosion protection to the coated metal film; the fluorinated acrylate the next best protection and the acrylic coating provided lesser protection.

The relatively linear nature of the Arrhenius plots of Figs. 8 and 9 indicate that the transport mechanism of corrosive chemical species through the conformal coatings remains the same over the 40 to 75 °C range of temperature employed in this study. In both the FoS and the MFG tests, the temperature had a strong degrading effect on the acrylic and the fluorinated acrylate coatings. The corrosion rates of the ALD coatings were too low to observe any temperature effects.

The iodine vapor test results are shown in Fig. 10 for silver and copper thin films and are tabulated in Table 1. Silver and copper thin films under the ALD coatings corroded the least; the films corroded a little more under the fluorinated acrylate coatings and the most under the acrylic coatings, in agreement with the test results in the FoS and the MFG chambers.

CONCLUSIONS

The flowers of sulfur (FoS) and the mixed-flowing gas (MFG) test results were in quantitative agreement in their characterization of the corrosion protection provided by the acrylic, the fluorinated acrylate, and the atomic layer deposited coatings. The coatings tested provided much better corrosion protection to the underlying copper than to silver, probably because of the better adhesion of the coatings to copper.

The FoS chamber is of simple construction, and easy to operate [5]. It is a couple of orders of magnitude less expensive to own and operate compared to the MFG chamber. This study demonstrated that the FoS chamber is as effective an environment as the industry standard MFG chamber in testing conformal coatings. The iodine vapor test can be done in an hour. Its characterization of conformal coatings is in qualitative agreement with the results from the FoS and the MFG tests.

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BIOGRAPHIES



Prabjit Singh is a Senior Technical Staff Member in IBM Poughkeepsie, with 45 years of experience in the metallurgical engineering aspects of mainframe computer power, packaging, cooling and reliability. For many years he taught a graduate course on Power Electronics at The State University of New York at New Paltz. He authored the ASHRAE book on data center gaseous and particulate contamination. He has 88 issued patents and is an IBM Master Inventor. He is a Distinguished Alumnus of the Indian Institute of Technology and an iNEMI Fellow. He received B.Tech. degree in Metallurgical Engineering from the Indian Institute of Technology in 1969 followed by a M.S. and a Ph.D. in Metallurgy from the Stevens Institute of Technology, Hoboken, New Jersey, in 1973 and 1977. Later he received a M.S. in Micro-Electronic Manufacturing from the Rensselaer Polytechnic Institute, Troy, New York, and a M.S. in Electrical Engineering from Walden University, Minneapolis.



Dr. Chen Xu is a Distinguished Member of Technical Staff with Nokia Bell Labs CTO. He has extensive experience in the fields of semiconductor and electronic packaging, electronic manufacturing, surface finishes and coatings, failure mode analysis and reliability assessment. In the last 15 years, he has been focusing on the impact of environmental conditions (such as corrosive, dusty and salty environments) on the reliability and performance of electronic equipment. Chen Xu received his BS Degree in Chemistry from Tong-ji University in Shanghai, China and his Ph.D. in Physical Chemistry from Ruhr-University Bochum, Germany.



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Khaw Mei Ming holds a bachelor's degree in Electronics and Computer Engineering and boasts 25 years of PCA manufacturing expertise in the EMS and OEM sector. She is certified by BSI as a Lean Sigma Black Belt, overseeing Keysight's Corporate PCB/PCA design guidelines and quality acceptance standards.



Marko Pudas received his M.Sc. degree in Chemistry and Doctorate degree in Electronic manufacturing at University of Oulu. He has worked 10y with ALD, on applications such as corrosion and tin whiskers mitigation. He has contributed to >50 publications and >50 patents families.



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