

Corrosion Protection of Metal Surfaces from Sulfur by Using 3M™ Novec™ Electronic Grade Coatings

Introduction

A study was conducted to show the capability of 3M™ Novec™ 2701, 2704 and 2708 Electronic Grade Coatings to mitigate corrosion of exposed metal on printed circuit boards and electronic components under harsh environmental conditions.

Novec 2701, 2704 and 2708 electronic grade coatings are fluorinated polymers carried in segregated-hydrofluoroether fluids sold by 3M under the Novec trade name. These polymer coatings are designed for protection of printed circuit boards, components and a variety of surfaces from moisture and corrosion.

Novec 2701, 2704 and 2708 electronic grade coatings dry to a thin, transparent film with excellent hydrophobic and oleophobic properties. They do not require thermal curing and are easy to apply – whether by liquid dipping, spraying (controlled) or brush applying. The solution and polymer are both low in toxicity, non-ozone depleting and RoHS compliant. These coatings incorporate a yellow-orange dye into the polymer backbone that is designed to fluoresce under UV light to aid inspection and the quality control of the coating process.

Background

During the transition away from printed circuit board finishes that contain lead, many industries have reported corrosion when using circuitry plated with metals such as silver and tin.^{1,2,3} Industries that have cited these issues include petrochemical, water treatment, and rubber manufacturing.³ Circuitry subjected to the harsh environments associated with these and other industries is susceptible to corroding relatively quickly.^{1,2,3} In addition, geographic location can contribute heavily to this problem.³ The creep corrosion from exposure to these harsh environments often leads to electrical shorts and failures quickly because the characteristic dendritic growth can cause bridging.^{1,2,3} For industries that rely heavily on the use of electronics to function, creep corrosion needs to be mitigated, especially in cases where electronics must meet high minimum requirements.

Experiment

Test Boards

Standard IPC-B-25A test boards are commonly available and were used in the study. These printed circuit boards (PCBs) meet guidelines for the testing of solder masks (IPC-SM-804C) and conformal coatings (IPC-CC-830A).

Immersion silver (ImAg) finish is used in electronics as an alternative to lead-tin finishes.¹ Therefore, IPC-B-25A test

boards with ImAg and bare copper (Cu) finishes were both treated with Novec electronic grade coatings and tested. Additionally, some with ImSnPb finish, vias and solder mask were also tested. Boards of each surface finish that were not treated with Novec electronic grade coatings were used as control samples and tested under the same conditions as the boards which were treated.

It has been stated by some groups that flux residues, which result from the board construction process, may be necessary to simulate the dendritic growth involved in creep corrosion in the laboratory.¹ Because of this, in our study some boards were treated with flux and reflowed before being coated and tested. It was found that boards with no flux residues were just as susceptible to creeping corrosion as boards with flux residues. Therefore, the focus remained on the clean IPC-B-25A test vehicles, shown in Figure 1 below.

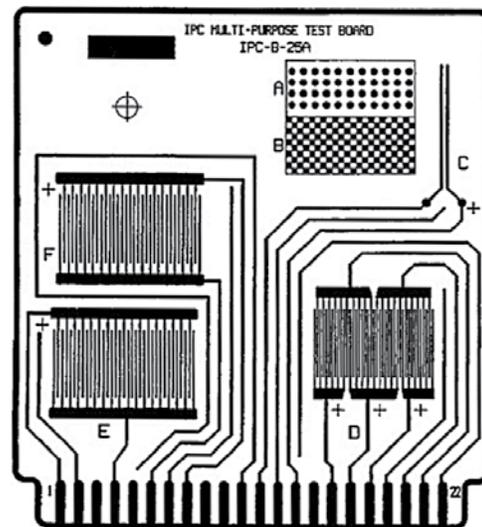


Figure 1. The IPC-B-25A test vehicle.

Coating Process

The IPC-B-25A test boards were cut in half vertically in order to accommodate the ASTM test conditions and the limited space in the test chamber. The cut boards were then cleaned with 3M™ Novec™ 72DA Engineered Fluid in a vapor degreaser. Novec 72DA fluid is effective at removing surface contaminants and particulate that, if left on the board, may have an impact on metal corrosion rates.

Each board was coated by a dip coating process. The process began with a chamber filled with one of the 3M™ Novec™ 2701, 2704 or 2708 Electronic Grade Coatings. The chamber sat on a table which moved up and down at a controlled rate. The rate at which the boards were removed controlled the thickness of the coating. In general, the faster the board is removed, the thicker the coating. The boards were dipped, held in solution for 30 seconds and removed from the coating solution at a rate of 12 inches per minute. The boards were allowed to dry and then placed into a flowers-of-sulfur test chamber as described below.

To simulate the type of conditions that might occur in the field, some boards were treated with flux prior to being tested. To do this, the coating process was modified slightly for boards that would be treated with flux. These test vehicles were first cleaned as stated above, the chosen flux was applied and the boards were then reflowed. The boards were allowed to cool to room temperature and then coated by the dip coating process as described above.

High Humidity - High Sulfur Test

“Flowers-of-Sulfur” (FoS)

A variety of methods can be used to test the porosity of coatings and protective finishes. The ASTM B809 method provides a standard method by which to induce the corrosion of various metal finishes.⁴ The test is designed to recreate the problematic high hydrogen sulfide gas and high humidity environment found in many industries.

The testing setup is shown in Figure 2. A 10 L glass desiccator was used as the test vessel. Grease was never used to seal the lid to the chamber and there was a vented stopper which allowed for equilibration of the system without pressure buildup. The test vessel contained a potassium nitrate solution in which there was a Petri dish containing elemental sulfur floats. The samples were suspended at least 75 mm above the sulfur powder. The samples were held in place above the sulfur source by an apparatus and the clips were not affected by the sulfur.

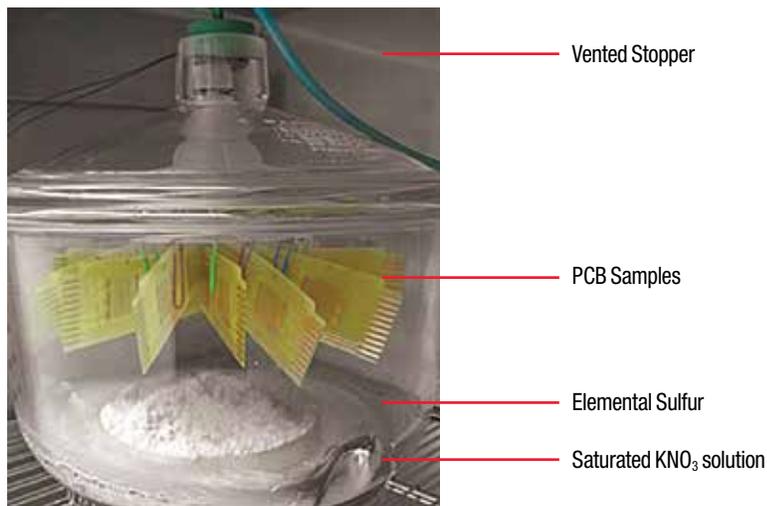


Figure 2. Samples in the flowers of sulfur chamber setup as described in the ASTM B809 test method.

Data

“Flowers-of-Sulfur” (FoS)

The FoS test method was used to study how the finish of a circuit board behaves in a corrosive, sulfur-containing environment. The method was designed to show whether attempts to mitigate corrosion, specifically creep corrosion, with a protective coating were successful.

Treated and untreated Cu finish test vehicles were exposed to the corrosive high sulfur environment in this study. After 10 days of exposure to the FoS test, untreated Cu finish boards were found to have succumbed to severe tarnish and creeping corrosion.

Cu finish boards that were treated with Novec 2708 coating, however, had minimal tarnish and no creep corrosion after 10 days of exposure. There was also substantially less tarnish and corrosion on Cu finish boards which were treated with Novec 2704 and 2701

“Flowers-of-Sulfur” (FoS) Chamber Test Results (60°C, >90% RH)

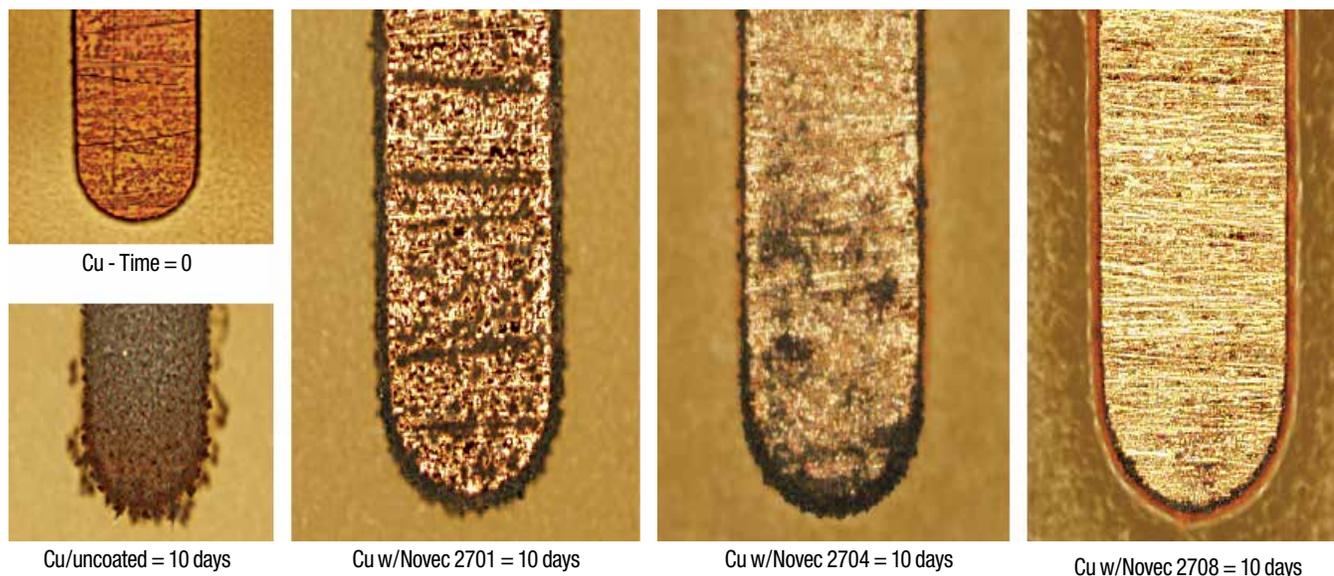


Figure 3. The pictures on the top and bottom left show uncoated Cu finish B-25A test vehicles before and after 10 days exposure in the FoS chamber. The three larger pictures on the right show Cu finish B-25A test vehicles which were coated with 3M™ Novec™ 2701, 2704 and 2708 Electronic Grade Coatings after 10 days exposure in the FoS chamber.

coatings after 10 days than on untreated boards. The testing showed that the characteristic dendritic growth of creep corrosion was drastically reduced by the presence of 3M™ Novec™ 2701, 2704 and 2708 Electronic Grade Coatings.

The conclusion was that treatment of circuitry with Novec coatings mitigated damages caused by exposure to the corrosive environment inside the FoS chamber. This difference in corrosion growth is shown in Figures 3 and 4.

Flowers-of-Sulfur (FoS) Chamber Test Results (60°C, >90% RH) 10 Days

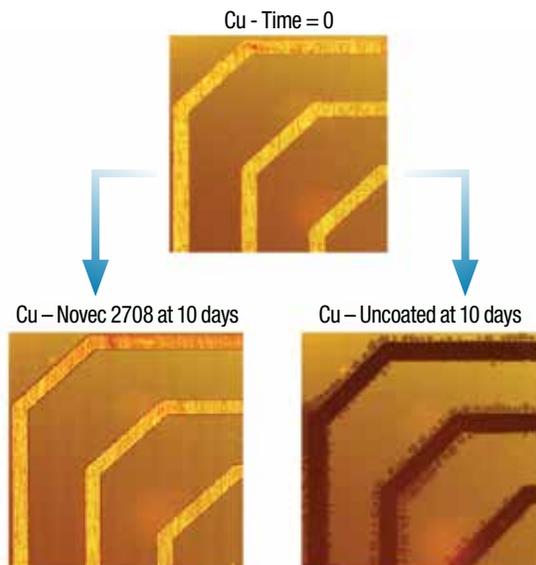


Figure 4. The pictures on the top and bottom right show uncoated Cu finish IPC-B-25A test vehicles before and after 10 days exposure in the FoS chamber, respectively. The bottom left picture shows a Cu finish IPC-B-25A test vehicle which was coated with 3M™ Novec™ 2708 Electronic Grade Coating after 10 days exposure in the FoS chamber.

Since ImAg and other finishes are often used to protect Cu circuitry, alternate finishes were also included in the study. Figure 5 shows results of coated and uncoated boards with these alternate finishes.

The IPC-B-25A design was used for the ImAg finish boards and a 3M-designed test board was used for the ImSnPb finished boards. The latter was done in addition to the IPC-B-25A boards to determine whether the creep corrosion phenomena could be mitigated on a typical solder mask, which is present on circuit boards in most cases. The coatings did mitigate corrosion on both alternate finishes tested.

Flowers-of-Sulfur (FoS) Chamber Test Results (60°C, >90% RH) Alternate Finishes After 34 days

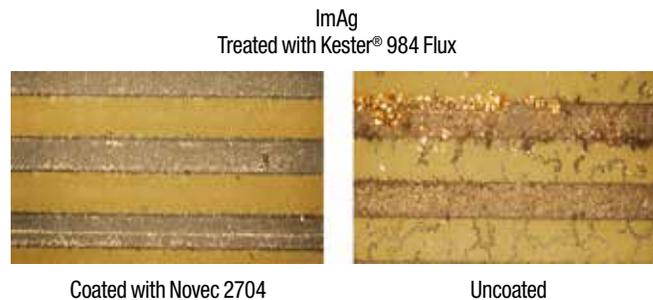
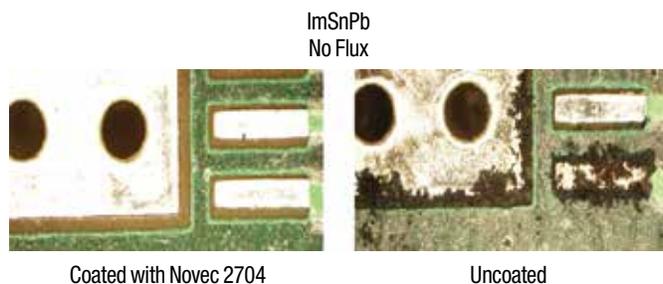


Figure 5. The pictures on the top left and right pictures show coated and uncoated ImSnPb finish test vehicles after 34 days exposure in the FoS chamber, respectively. The bottom left and right pictures show ImAg finish B-25A test vehicles coated and uncoated after 10 days exposure in the FoS chamber, respectively.

Summary and Conclusions

Creep corrosion can be driven by many factors and is an increasing concern for many industries, end customers and the circuit board industry. Tests were completed to demonstrate whether coating printed circuit board circuitry would help to reduce the progress of creeping corrosion caused by sulfur in the environment.

In this study, 3M™ Novec™ 2701, 2704 and 2708 Electronic Grade Coatings were applied over a variety of metal and metal finished electronic circuitry surfaces. Uncoated versions were used as a control and compared over time to the coated versions.

A FoS chamber was chosen as the test method for inducing creep corrosion. It simulated the high hydrogen sulfide gas and high humidity environments in which circuitry is increasingly being used.

This testing procedure resulted in a dramatic difference with the coated versions showing significantly less corrosion development. The conclusion was that Novec 2701, 2704 and 2708 electronic grade coatings help mitigate the formation of creeping corrosion of exposed metal on circuit boards caused by exposure to high levels of sulfur and humidity.

¹Xu, C., Smetana J. Franey, G. Guerra, D. Flemming, W. Reents, Dennis Willie, Alfredo Garcia, Guadalupe Encinas, and Jiang Xiaodong. "Creep Corrosion of PWB Final Finishes: Its Cause and Prevention." *IPC APEX EXPO Proceedings* (n.d.): n. pag. Web.

²Kenny, Jim, Karl Wengenroth, Ted Antonellis, ShenLian Sun, Cai Wang, PhD, Edward Kudrak, and Joseph Abys, PhD. "PWB Creeping Corrosion Mechanism and Mitigation Strategy." Enthone Inc., Crookstone Electronics, n.d. Web.

³Schuessler, Randy, PhD. "Creep Corrosion on Lead-Free Printed Circuit Boards in High." *Dfrsolutions.com. SMTA International Proceedings*, Oct. 2007. Web. 19 Aug. 2013.

⁴"ASTM B809 - 95(2008)." Standard Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor ("Flowers of Sulfur"). N.p., n.d. Web. 15 Aug. 2013.

The 3M™ Novec™ Brand Family

The Novec brand is the hallmark for a variety of patented 3M compounds. Although each has its own unique formula and performance properties, all Novec products are designed in common to address the need for safe, effective, sustainable solutions in industry-specific applications. These include precision and electronics cleaning, heat transfer, protective coatings and surface modifiers, fire protection, lubricant deposition and several specialty chemical applications.

3M™ Novec™ Engineered Fluids • 3M™ Novec™ Aerosol Cleaners • 3M™ Novec™ 1230 Fire Protection Fluid • 3M™ Novec™ Electronic Coatings • 3M™ Novec™ Electronic Surfactants

United States	China	Europe	Japan	Korea	Singapore	Taiwan
3M Electronics Markets Materials Division 800 810 8513	3M China Ltd. 86 21 6275 3535	3M Belgium N.V. 32 3 250 7521	Sumitomo 3M Limited 813 3709 8250	3M Korea Limited 82 2 3771 4114	3M Singapore Pte. Ltd. 65 64508888	3M Taiwan Limited 886 2 2704 9011

Technical Information: The technical information, recommendations and other statements contained in this document are based upon tests or experience that 3M believes are reliable, but the accuracy or completeness of such information is not guaranteed.

Product Use: Many factors beyond 3M's control and uniquely within user's knowledge and control can affect the use and performance of a 3M product in a particular application. Given the variety of factors that can affect the use and performance of a 3M product, user is solely responsible for evaluating the 3M product and determining whether it is fit for a particular purpose and suitable for user's method of application.

Warranty, Limited Remedy, and Disclaimer: Unless an additional warranty is specifically stated on the applicable 3M product packaging or product literature, 3M warrants that each 3M product meets the applicable 3M product specification at the time 3M ships the product. 3M MAKES NO OTHER WARRANTIES OR CONDITIONS, EXPRESS OR IMPLIED, INCLUDING, BUT NOT LIMITED TO, ANY IMPLIED WARRANTY OR CONDITION OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE OR ANY IMPLIED WARRANTY OR CONDITION ARISING OUT OF A COURSE OF DEALING, CUSTOM OR USAGE OF TRADE. If the 3M product does not conform to this warranty, then the sole and exclusive remedy is, at 3M's option, replacement of the 3M product or refund of the purchase price.

Limitation of Liability: Except where prohibited by law, 3M will not be liable for any loss or damage arising from the 3M product, whether direct, indirect, special, incidental or consequential, regardless of the legal theory asserted, including warranty, contract, negligence or strict liability.



Electronics Markets Materials Division

3M Center, Building 224-3N-11
St. Paul, MN 55144-1000
www.3M.com/novec
1-800-810-8513

Please recycle. Printed in USA.
© 3M 2014. All rights reserved.
Issued: 4/14 9728HB
60-5002-0724-0

Kester is a registered trademark of ITW Kester.
3M and Novec are trademarks of 3M Company.
Used under license by 3M subsidiaries and affiliates.