Case Studies of Harsh Application Environments, the Unforeseen Situations

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ABSTRACT
Rockwell Automation is a manufacturer of a full line of industrial automation controls, from large variable frequency drives to small sensors, with many varying products in between. As such, Rockwell products are exposed to many and varying application environments and endure many unadvised or unknown shipping and storage scenarios. With these varied environments comes many different types of degradation mechanisms that can affect electrical function. This project encompasses collected information from "typical" component level issues found in certain environments, to the strange, unusual, and unexpected cases that highlight analytical techniques and reinforce that making assumptions can be a hinderance when performing failure analysis. The paper will follow various case studies from inception to conclusion, highlighting the initial problem, the data collected, and the final resolution. These cases will encompass several environmental conditions from a range of industries, from pet food manufacturing to offshore oil platforms.

Key words: degradation, corrosion, contamination

INTRODUCTION
Degradation mechanisms in electronics and electromechanical devices have been experienced, studied, identified, and mitigated for many years. Issues with various electronic components due to specific contamination in application environments have been well documented. Industries such as mining, pulp and paper, marine, food and beverage, oil refining, water/wastewater, and others have the major contamination constituents reasonably well defined. The problem arises when dealing with contamination from application environments where the specific constituents, and their concentrations, are not well known. Relying on data from previous investigations for specific industries can be helpful in the realm of targeting probable degradation mechanisms during analysis, but making assumptions based on previous analysis may be a hindrance in determining actual root cause. Analysis can become even more challenging with lack of information of the application site, or when a product arrives at the customer with some kind of issue.

Shipping and storage can also present a myriad of additional environmental factors that can be difficult to define. Shipments can experience travel by air, boat, train, and/or truck through any number of varied environmental conditions, and be stored in environmentally uncontrolled facilities. It is very probable that a specific shipment will have to survive a combination of these situations.

This paper will present scenarios where environmental factors played a role in inducing various degradation mechanisms to Rockwell products. The paper will attempt to concentrate on the unusual scenarios, or where assumptions were made too early in the analytical process that likely hindered efficient determination of root cause. These scenarios are not the more common electronic product failure modes typically encountered, but are the more atypical in either cause, or degradation mode.

The data from analytical techniques used to identify mechanisms and contaminants will be reviewed. Combinations of analytical techniques are typically used for analysis, and all data collected from these techniques will not be included unless unique to the specific case. Analytical techniques may include, but not limited to: Scanning electron microscopy with energy dispersive spectrometry (SEM/EDS) ion chromatography (IC), Fourier-transform infrared spectrometry (FTIR), gas chromatography mass spectrometry (GCMS), X-ray diffraction (XRD), X-ray fluorescence (XRF), and optical microscopy.

CASE STUDY 1
Mysteriously cracking reed switch
When the need arises for switching power on or off to a device in a potentially explosive environment, like a grain mill or oil well environment, the need for shielding the arc generated by switching mechanical contacts is a benefit. A reed switch operates by an applied mechanical field. By placing the contact assembly in a glass envelope, which is backfilled with a controlled gas atmosphere and hermetically sealed, the arc is contained within the glass envelope preventing accidental ignition of the atmosphere. It is important for the glass envelope to remain intact (Figure 1).

An anomaly occurred to the reed switch glass aboard an offshore oil platform that was being moved from the Caribbean to the Mediterranean Sea. When the platform arrived at the prescribed destination, it was noted that many of the switch assemblies were not functional. Upon return receipt, analysis indicated the glass envelopes had cracked, which allowed moist air into the envelope, corroding the electrical contacts which raised contact resistance past acceptable levels.

The initial assumption was that the glass cracking was a mechanical issue, thus various tests were devised and implemented with little success inducing the exact cracking mode. After eliminating other various mechanical inputs as the cause, investigation centered on residual tensile stress in the tube itself. A small amount of residual compressive stress is needed to maintain the glass to metal seal of the switch assembly. Another factor that also indicated a possible stress issue, was that the furnace used to temper anneal the glass after hermetic sealing had been moved and refurbished. Thus, samples of pre and post furnace move were analyzed using light polarimetry to quantify the amount of stress remaining in the seal areas. While a slight compressive stress is needed to maintain the seal, excessive amounts can lead to a form of stress corrosion cracking of the glass in moist environments. It was found that the pre move samples contained significantly less stress at the seals than the post move samples. A design of experiments was performed to compare furnace temperature, belt speed, and actual temperature of the switch assemblies on the anneal fixture. A comparison was also done of the furnace temperature profile both pre and post move (Figure 2).

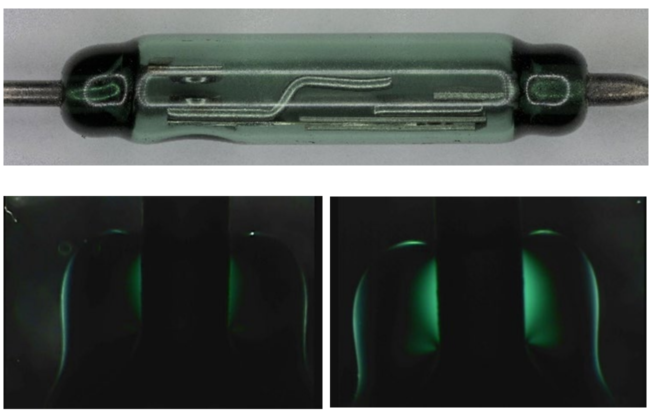


Figure 1. Image of the switch contact assembly (top). Polarimeter images of the stress field at a glass seal on a unit retained from production prior to furnace move (left) as compared to a unit produced using the post move furnace recipe (right).

A test method was devised using a commercial pressure cooker to induce the cracking mechanism. It was found that the glass envelope would crack relatively quickly with heat and moisture when the anneal was not sufficient to reduce the residual stress to an acceptable level, yet still maintain the seal during cyclic thermal testing. After furnace adjustments, this test method was used to verify adequate glass tempering.

While mechanical testing took some time to devise and implement, the testing did show that most mechanical inputs did not degrade the glass in the manner found from the field. Thus, the testing did provide some information to move focus to the manufacturing process, and the process improvements have improved yield.

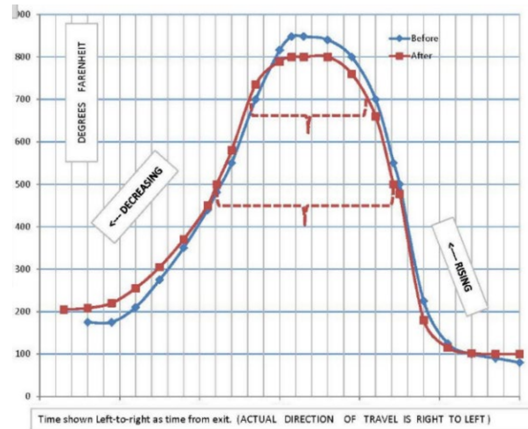


Figure 2. Furnace temperature profile comparing an earlier profile vs the later. The main issue was a ~50F peak temperature change.

CASE STUDY 2

Where is the contamination coming from?

Environmentally controlled rooms have been used to protect industrial process control electronics operating in less than clean environments for a long time. Typically, these rooms have air conditioning systems that both cool and filter the air. Degradation of electronics in control rooms is typically rare, if the room was correctly constructed. Thus, finding degradation within power electronics in a control room was a surprise. The control room was in a manufacturing facility that was producing pet food, thus contamination typical of a washdown environment was expected, along with the corrosion mechanisms typical of high pH anti-bacterial chemistries. These type of degradation mechanisms were not found. The power module was found to be suffering from dendritic sulfide corrosion growing across ceramic substrates bridging isolation gaps.

Figure 3. Image of the copper and silver coupons after exposure in the control room. The accelerated silver corrosion is typical of certain types of sulfur contamination in the environment.

Analysis of the power device removed from an affected drive indicted the presence of a sulfur bearing compound. The source of this compound was unknown. Cleaning agents did not appear to be the source. In an effort to determine the source, silver and copper corrosion coupons were exposed in various areas of the facility to generate an understanding of corrosion rates and corrosion types. Also, a filter sample was obtained from a rooftop air handler to analyzer the filter media. Debris samples were also collected from various area for analysis. Elemental compositions and crystalline structures were identified using SEM/EDS or XRF, and XRD techniques. It was noted that the coupons showed no visible signs of copper corrosion and slight corrosion/tarnishing of the silver (Figure 3). Elemental analysis found elevated carbon on both sets of copper coupons and elevated sulfur on both sets of silver coupons. These results suggest a presence of some type of organo-sulfide compound in the contamination. Elemental mapping of the power module indicates the corrosion was likely a sulfide of copper (Figure 4). Growth of these corrosion products are typically driven by contamination concentrations, temperature, moisture levels, and the potential difference across the ceramic.

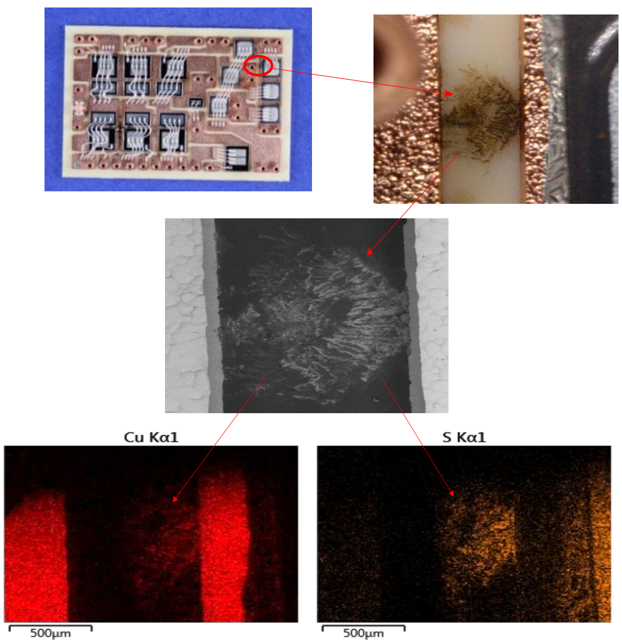


Figure 4. Optical images, SEM backscatter image and EDS elemental mapping showing the morphology and elemental composition of the dendritic growth found growing across the ceramic isolation layers in the power device. Note, not all elemental maps included.

A portion of both the collected debris and filters were analyzed for ionic contamination using IC. Analysis identified ionics in the form of chloride, bromide, nitrate and sulfate (Figure 5). The presence of ionics would accelerate corrosion mechanisms and contribute to electrical issues if accumulated on unprotected electronics/circuit boards in the presence of an electrical potential and elevated atmospheric moisture. The problem was that general corrosion of the PCBAs or other areas of the drive were not found. Portions of the coupons, debris and filters were solvent extracted and analyzed for volatile compounds using GCMS. The filters contained a variety of hydrocarbons, fatty acids, silicones, and aldehydes, all which were not expected to contribute to the sulfur corrosion. Several amine compounds were found which were suspected of inhibiting corrosion of the copper. However, no volatile sulfur containing compounds were identified in the samples. This would suggest that the source of sulfur would have been gas phase and may not have been captured in the debris/filter/coupon sampling methods.

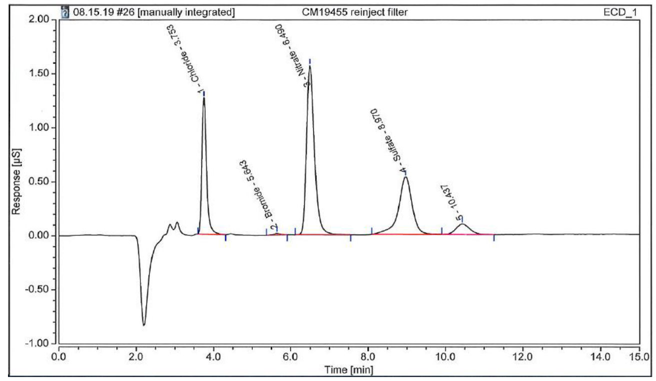


Figure 5. IC analysis results identifying the anions found in filter debris extraction.

The question remained as to why the control room contained concentrations of corrosion inducing sulfur compounds. It took additional investigation onsite to determine the probable cause. One aspect of the processing in this plant was long-term homogenization of various animal tissue with pressure and heat. To determine which processes may have been producing the contamination, and the possible path of ingress into the control room, active air sampling was performed near the processes, and in various air movement ducts.

Several National Institute for Occupational Safety and Health (NIOSH) defined air sampling methods were performed targeting specific gas phase compounds, along with non-standard for sulfur and organo-sulfur compounds, and other volatile corrosive compounds. These samples were evaluated both internally and externally using an accredited lab. Elevated levels of hydrogen sulfide, nitrogen dioxide, and elemental sulfur were found near the processing tanks, and in the control room. The levels of hydrogen sulfide were 10 times higher near the processing tanks as compared to within the control room. Hydrogen sulfide and elemental sulfur would both be sources for sulfur corrosion on copper and silver, while nitrogen dioxide acts to accelerate the corrosion on silver and steady the passive layer on copper. Both hydrogen sulfide and elemental sulfur are produced because of the breakdown of organic matter (meat and plants).

The likely source of the sulfur compounds within the control room was air handling. An investigation into plant exhaust airflow patterns found the external control room intake for "fresh" air was too close in proximity to the process fume exhaust. Process air was inadvertently being used to condition the control room, especially when the wind was blowing in certain directions. Filtration used on the HVAC system was meant for particulate, not gas phase contamination. Though the investigation was initially centered on cleaning chemicals, the investigation changed quickly once initial data was analyzed. Further investigation took some time, but resulted in positive changes for the customer.

CASE STUDY 3

The downfall of storing product before use

Multiple logic modules were returned from a customer several months after service with heavily deposited debris and intermittent electrical issues. The products were being installed into stainless steel enclosures and used in anchor pontoons within a bridge project as well as in ocean marine vessels. It was reported that the products were stored in plywood crates for an extended period near the service location before being put into service, at which time the customer had noticed debris accumulated on the product.

Initial testing of the returned units found them functional. During disassembly of the products, a large amount of white debris was noted across the entire exterior and interior of the magnesium chassis, as well as accumulated onto the internal PCBAs (Figure 6). Initially, moist sea air was identified as the problem. No signs of corrosion were found on the PCBA itself.

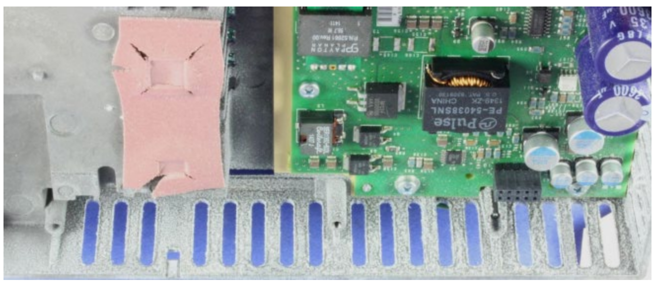


Figure 6. Image of the inside of one of the products as received, showing the metal chassis is covered by a white loose debris which appears to have accumulated onto the internal PCBA.

Elemental analysis of the contamination removed from the first chassis using SEM/EDS revealed high concentrations of oxygen, magnesium, carbon, sodium and aluminum; with lower levels of silicon, calcium, iron, chlorine, sulfur, potassium, zinc and chromium. IC analysis on the chassis and internal PCBA identified high levels on conductive ionic compounds with high levels of acetate and lower levels of chloride, nitrate and sulfate (Figure 7). Chloride would be expected in environments near the ocean and low levels of nitrate and sulfate can be found in general dust. Acetate is unusual to find, especially in the reported application environment. You might expect to find acetate in food manufacturing or washdown environments that would use acetic acid as part of the cleaning process. FTIR analysis identified the white debris as the salt of an organic acid confirming an acetate salt. XRD identified the crystalline species as a mixture of magnesium chloride, magnesium chloride acetate hydrate, magnesium acetate, and magnesium oxide.

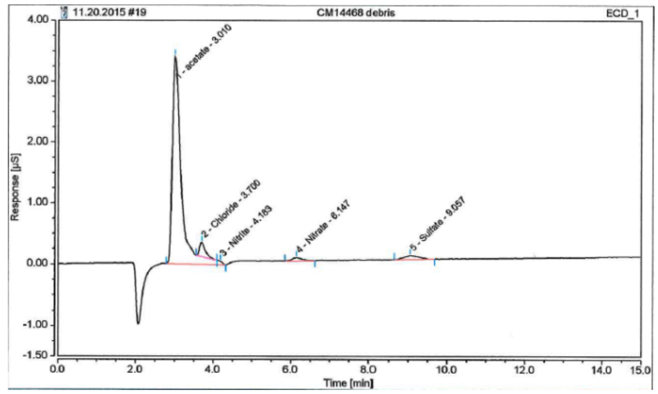


Figure 7. IC analysis results identifying high levels of acetate.

The intermittent issues were likely caused by ionics from corrosion products of the magnesium chassis accumulating onto the PCBAs combined with elevated moisture/humidity. Magnesium carbonate is a corrosion product of magnesium when exposed to high levels of moisture. The unusually high level of acetate was thought to be originating from the sealant used to seal the plywood crates the product was stored in. A byproduct of the curing of certain sealants (for example an acetoxy cure silicone) is acetic acid, which could form the acetate ion. This example highlights the unforeseen consequences of improper storage conditions.

CASE STUDY 4

When you can't see what's causing the product issues

Electrical issues were occurring at a specific customer in a paint shop on multiple drives. It was reported that the anomaly was occurring when the drives first powered up after some period of being down for maintenance. The event produced enough energy to open the enclosure doors. When units were returned, they appeared clean with no signs of particulate debris, corrosion or chemical attack.

Due to the nature of the application environment, the cause was thought to be due to an excess of organic solvents in the vicinity of the drives. Air sampling was performed at the customer site, which found levels of organic solvents well below the explosive range (0.0003% vs. 1.5%). If the application environment isn't causing the issue, then what is and why couldn't we see any evidence of it?

Upon further investigation, it was found that while the drives were cold, the airborne solvents could slowly absorb into the polyvinyl chloride (PVC) insulation wrapping on the electrolytic capacitors (Figure 8). When the drive suddenly warms up under a heavy load, the air-borne solvents evaporate on a much shorter time scale. Thermogravimetric analysis (TGA) demonstrated that transient solvent concentrations approaching the flammability limit were achievable within the drive enclosures as a result of the absorption-evaporation mechanism.

Figure 8. Representative image of drive capacitors

A second experiment demonstrated that when solvent molecules evaporate out of the PVC, an electrostatic charge separation also occurs. The buildup of separated charges in the drive enclosure therefore results in an eventual electrostatic discharge, which acts as an ignition source. The resolution was to convert the enclosures to a NEMA 12 type sealed enclosure to prevent air exchange with the application environment.

CASE STUDY 5

The crystals that just keep coming back

Multiple distributors were reporting what appeared to be crystal growth on the touchscreen display film of industrial computers when received across multiple date codes (Figure 9). The manufacturing plant that assemblies these units did not report any issues during assembly. The touchscreen supplier sent samples to an independent laboratory who identified the cosmetic anomaly as a mold, Aspergillus Restrictus. Many of these products go into food and beverage applications and hospitals, environments with strict safety regulations. Thus, the returned units were provided with a request to either confirm or dispute the previous findings.

A portion of the film was sent to an independent microbiology laboratory which found the film negative for mold growth. Upon disassembly, viewed under magnification, the cosmetic anomalies appeared to be crystalline in structure nucleating from several points across the film. The crystals were found to be highly water soluble and easily extracted.

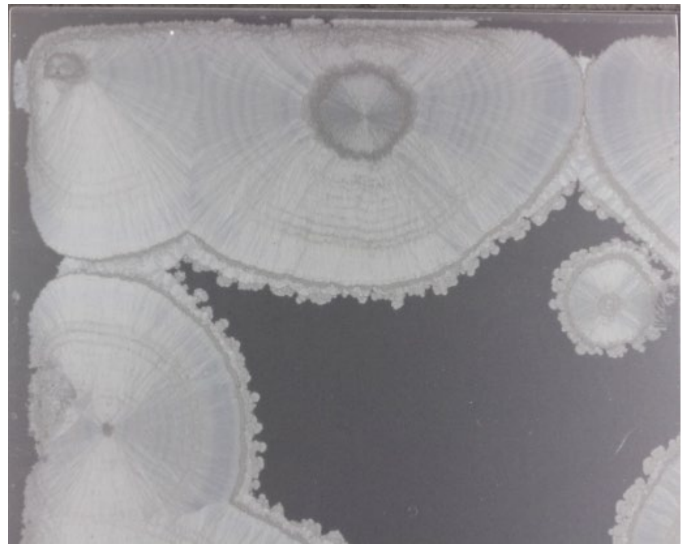


Figure 9. Image of cosmetic anomaly on diffuser film of returned computer.

FTIR analysis confirmed that they were the salt of an organic acid and IC analysis confirmed phthalic acid was present (Figure 10), however SEM/EDS analysis could find no counter ion, which was strange. Raman spectroscopy preformed at a local university found that the counter ion was likely ammonia, and the crystals were a good match to an ammonium salt of phthalic acid. The film was determined to be PET (polyethylene terephthalate), which is polymerized using terephthalate acid and butyl 1,4-butanediol. If the stoichiometry (ratio of starting materials) is off, excess terephthalate acid would be left on the film. But where was the ammonia coming from?

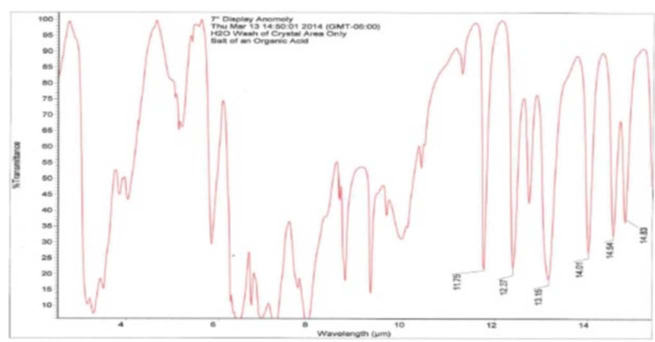


Figure 10. FTIR spectra of a water washing of the crystal area showing an ammonia salt of phthalic acid.

In talking to the manufacturing location, it was determined that the operators were wiping down the displays with glass cleaner to remove any fingerprints on the display immediately before packaging. The ammonia in the glass cleaner sealed up with the left-over terephthalate acid on the film would form the ammonium salt of phthalic acid that was found on the surface of the film when the distributors received the product. The crystal growth was replicated by spraying a portion of new film with an ammonia-based cleaner and sealing it in a glass desiccator at room temperature for several days. The solution to the problem was to have the operators clean with a non-ammonia based cleaner.

A few years later, more field returns were received with a similar cosmetic anomaly, but this time the displays had been in service for some time (Figure 11, 12). All the returns were from the food and beverage industry, specifically from beverage bottling lines. The initial assumptions were that it was the same issue and that the operators had resumed cleaning with an ammonia-based cleaner.

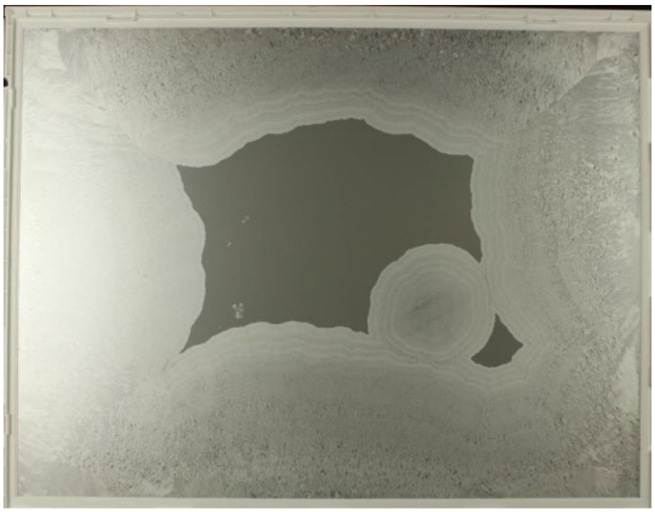


Figure 11. Image of anomaly on diffuser film of returned computer.

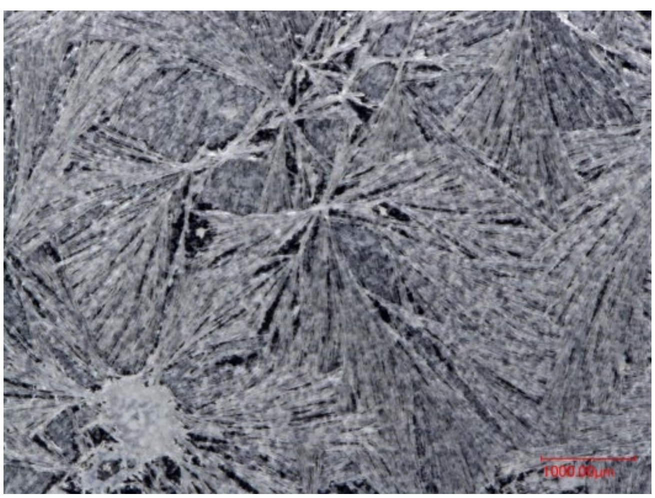


Figure 12. Image of anomaly on diffuser film under magnification.

This time, FTIR analysis of the crystals found bisphenol A and confirmed that the film material inside the display was a polycarbonate (PC). It was discovered that the film supplier had recently changed, and the film material had changed from a PET to PC. This change shouldn't have caused any issues, a plastic is a plastic after all. Bisphenol A is a monomer/starting material used in the polymerization of polycarbonate. PC is also susceptible to hydrolytic degradation with prolonged exposure to warm/humid environments, which results in a breakdown of the polycarbonate to the bisphenol A monomer. The crystal growth was replicated in the lab by placing a sample of the film in a humidity chamber.

All the end use environments were found to use steam/high humidity as part of the sterilization process, which appeared to be accelerating the crystal growth on film that had excess Bisphenol A starting material. This anomaly appeared to be isolated to one lot of material and the issue never returned.

A few years later, more field returns with a similar cosmetic anomaly; this time the crystals, apparent on both the film and plastic bracketing, were noticed after the distributor received them but before being commissioned at customer sites (Figure 13). It was thought to be a storage issue, possibly high humidity based on the previous analysis. During conversations about the storage conditions, it was discovered that the distributor was placing VCI (vapor corrosion inhibitor) pucks in with the product during storage.

Figure 13. Image of anomaly on diffuser film of returned computer that had been stored with a VCI emitter.

FTIR analysis confirmed the crystals and film material were the same composition as the pervious returns, bisphenol A and PC. This time the end use environments and storage conditions did not have high humidity. In evaluating the VCI emitter chemicals, it was discovered that one of the chemicals, 2-diethylaminoethanol, is incompatible with PC. The crystal formation was replicated by placing a sample of the film and bracketing in a sealed glass desiccator with the specific VCI emitter used. This analysis confirmed that this time the crystal formation, although the same chemical structure, was a result of chemical incompatibility. The VCI emitters were added routinely when product was stored to prevent corrosion of any exposed metal. While typically helpful, the chemicals used can have unintended negative reactions with some materials. The solution was to use a different VCI emitter with chemicals that were compatible with the plastics used in the product.

These examples in this case study, although not related to surface mount technology, highlight the importance of not making assumptions and the need to understand the product history including storage and application environments before determining the cause of the anomaly. This also highlights the unintended consequences of material changes. If the film had not changed from PET to PC, it would not have been susceptible to degradation from humidity or the VCI emitter.

CASE STUDY 6
The flying contamination source

A set of cooling fans from a motor drive supplying power to an air conditioning unit were returned with the request to determine why the fan control boards for the fans were severely corroded. The drive was located on a rooftop HVAC unit located in southern California, and other than normal outside environmental conditions, the corrosion was unusual as the drive was reasonably well protected. The drive itself contained some dust, but nothing that would be considered unusual for a rooftop application. Other areas of the enclosure and some of the electronics were showing signs of degradation also. Sea air was suspected.

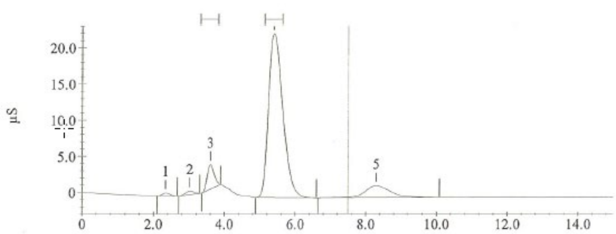
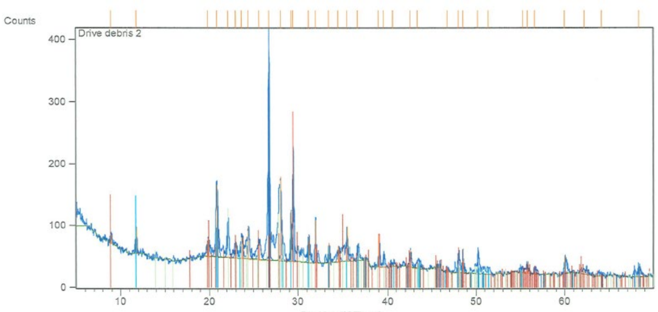


Figure 14. IC data indicating a significant amount of nitrate (peak 4).



No.	Visible	Ref. Code	Compound Name	Chemical Formula
1	☑	01-086-1560	Quartz	Si O2
2	☑	01-085-0850	Sodium nitrate(V)	Na N O3
3	☑	00-006-0046	Gypsum	Ca S O4 !2 H2 O
4	☑	01-089-5401	Muscovite 2\ITM\RG#1	K Al2.20 (Si3 Al)0.975 O10 ((O H)1.72 O0.28)
5	☑	00-020-0554	Albite, ordered	Na Al Si3 O8

Figure 15. XRD data indicating the presence of sodium nitrate and gypsum, both which may be ingredients in a fertilizer.

Elemental analysis with EDS of the debris found a majority of carbon, oxygen, aluminum, silicon, calcium and iron, with smaller amounts of sulfur, chlorine, potassium, phosphorus, magnesium, sodium, titanium, manganese, and zinc. IC analysis in conjunction with XRD indicted the presence of several minerals, which is not unusual in a dust analysis, plus a significant amount of nitrate, likely sodium nitrate. Finding large amounts of nitrate is unusual in a dust analysis, especially on a rooftop of a multistory building.

Further investigation was required. The building was in an area that was surrounded by agricultural crops. Crop dusting was routinely performed. It was suspected that wind driven fertilizer was being drawn into the drive and the combination of the fertilizer and environmental moisture accelerated the corrosion mechanisms.

This case study points out the fact that the application environment is not always easy to determine. All aspects of the local environment need to be evaluated.

CASE STUDY 7
When electronics stop conversing

Communications between PCBAs inside many electronic products is done over ethernet protocols using optical transceivers, commonly called small form-factor pluggable (SFP) network interface modules. One such module in application had an issue and communications were erratic. The module as replaced and submitted for analysis. The module's issue was recreated in a test stand. The application was in the Tire & Rubber industry, and the assumption was made that the sulfur corrosion susceptible resistors on the uncoated PCBA internal to the device were to blame.

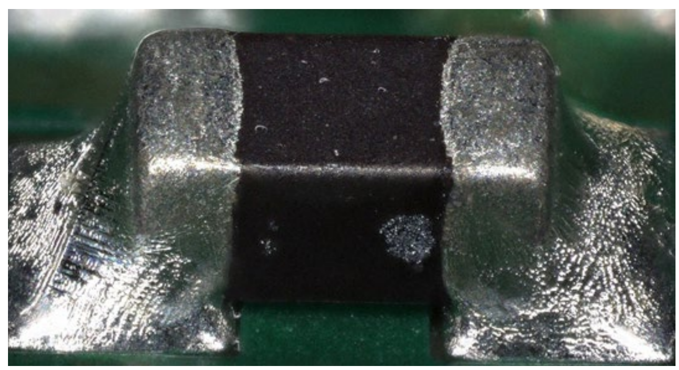


Figure 16. Optical image of a surface mount inductor showing corrosion mid -body through the epoxy coating.

What was found, after inspecting many resistors very carefully, both optically and in the SEM, was not an issue with the surface mount resistors, but rather with some surface mount inductors. Silver corrosion product was found on the surface of the epoxy molding. SEM/EDS analysis indicated the debris contained silver and sulfur, likely silver sulfide (Figure 17). The internal construction of the inductor uses silver, which was too close to the surface of the epoxy. The contaminant diffused into the outer coating, and corrosion occurred internally. The change in volume of the corrosion product cracked the case, which allowed direct access to the external environment, thus accelerating the degradation mechanism. The board was not conformally coated.

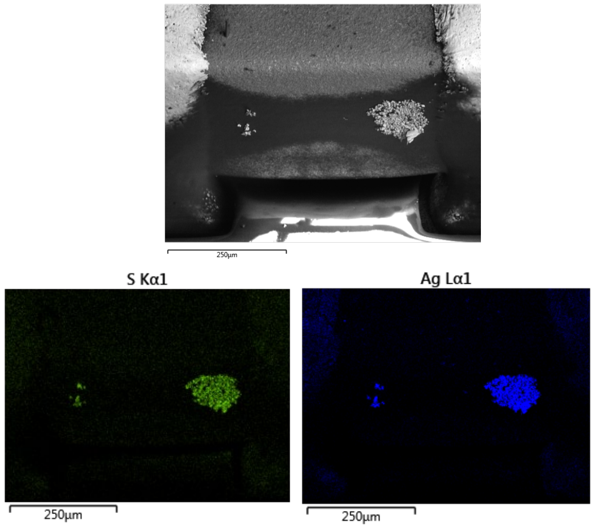


Figure 17. SEM backscatter image and EDS elemental maps of the inductor in question. Note, not all elemental maps included.

SUMMARY

The preceding studies highlight several investigations that were unusual in nature, either due to the application environment, or the degradation mechanism, or both. Some of these cases highlight situations where certain initial assumptions led the investigation down an unproductive path that sacrificed valuable time and resources. Sometimes miscues are part of the investigative process, especially when application information is sketchy, but can lead to useful information. Implementation of a general analysis plan to collect data for evaluation of a possible root cause is typically a good initial path to take.

ACKNOWLEDGEMENT

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BIOGRAPHIES

Chris Genthe is a Senior Principal Engineer with Rockwell Automation's Chemistry and Materials Engineering Group. He has over 35 years of experience in materials, including metallurgy, corrosion identification and control, material selection, failure analysis, processing, and accelerated testing. Chris has coauthored several publications, has three patents pertaining to corrosion control of electronic assemblies, received seven Rockwell innovation awards, and was a member of the 2018 Rockwell Team of the Year. Chris was awarded the Rockwell Engineer of the Year in 2020 for his work on the development of gas phase accelerated corrosion test methods that target specific corrosion mechanisms in electronics. He earned a BS and MS in Materials Engineering from the University of Wisconsin – Milwaukee, and is an adjunct professor teaching Environmental Degradation of Materials at the same institution.

Kelly Flanagan is a Principal Chemist in the Chemistry and Materials Engineering group at Rockwell Automation in Milwaukee, Wisconsin. She has been with Rockwell since 2007. Kelly has undergraduate degrees in Chemistry and Biology from UW-Stevens Point. Kelly's area of expertise involves the use of chromatographic and spectrometric techniques used in root cause failure analysis evaluations; in particular contamination identification related to printed circuit board failures and coating evaluations. Kelly has received six Rockwell Automation Innovation Awards and was a member of the 2018 Team of the Year Award.