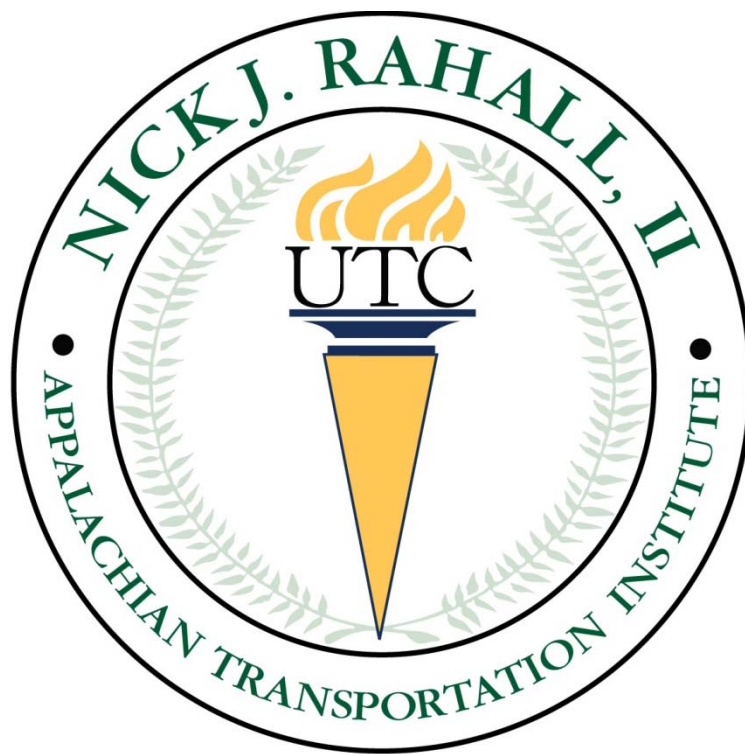


Use of Electroluminescence Technology for Highway Signage



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1.0 Introduction

1.1 Technology Background

Figure 1 illustrates the basic design of an electroceramescent lamp (ECL). The panel is comprised of a series of layers deposited upon a steel substrate. An AC potential applied across the electrodes (the steel substrate and the transparent conducting oxide) introduces a high field across the phosphor layer which causes electrons to accelerate to high energies and to activate luminescent centers in the phosphor. Light is produced when the centers relax to their ground state. The ECL panel can be driven by an AC power source over a wide range of voltages and frequency. Transformers and inverters are used to convert the primary power source (utility, battery, solar) to an appropriate voltage (typically 90 to 250 volts RMS) and frequency (typically 60 to 1000 Hz) for optimum SSCL performance. Typical illumination levels are in the 1 to 10 foot-lamberts range.

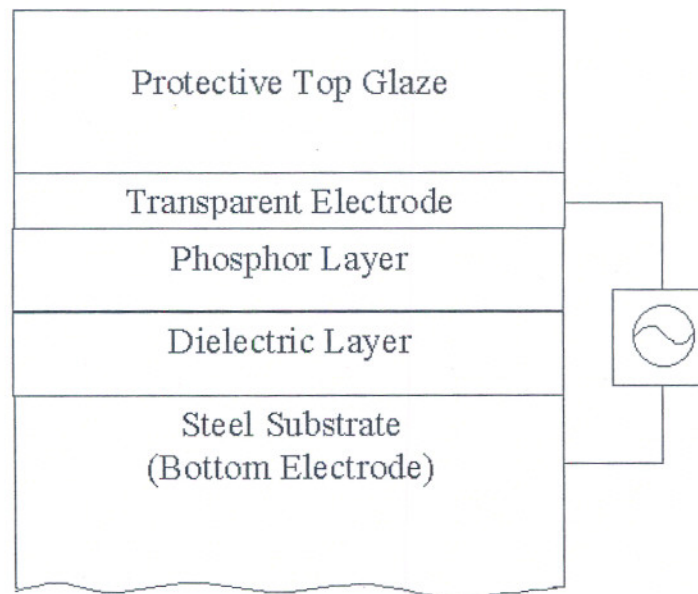


Figure 1. Schematic of ECL panel

1.2 Program History

In September of 1999, a two(2) year, R&D effort was initiated by MRE, Inc through a cooperative agreement with the U.S. Department of Energy (DE-FC26-99FT40631) with federal funding of \$1.6 million in a total effort exceeding \$2 million. The development team included the New York State College of Ceramics at Alfred University in Alfred, NY, Marshall University in Huntington, WV, and OSRAM Sylvania in Towanda, PA. The result is a new, high efficiency, light-emitting ceramic termed an "electroceramescent" system. The study addressed in this Progress Report was a part of this effort in collaboration with the Rahall Appalachian Transportation Institute (ATI TRP 99-05). The first commercial lamp products are planned for the summer of 2002.

1.2 Application Summary

Electroceramescent lighting (ECL) offers several potential advantages over traditional light sources, including:

- High visibility (direct, not reflected, area illumination)
- Low heat dissipation (no IR radiation)
- Low maintenance (no bulbs)
- Long life without catastrophic failure (up to 10 years)
- High visibility in adverse conditions (fog/smoke)
- High-quality light source (no glare, no flickering)

Numerical comparisons of the efficiency of EC lighting to traditional light sources are often misleading. For example, typical efficiency of incandescent and fluorescent lights exceed 15 and 75 lumens/watt (LPW), respectively, whereas the efficiency of an EC lamp may be as low as 4 LPW. Ironically, even a low efficiency EC lamp often uses dramatically less power than conventional light sources for a given task. For example, power requirements in commercial signage applications are approximately one-tenth the energy required for the same sign if conventional T-12 high output fluorescent lamps (550 W) had been used. An additional substantial energy saving (typically 50%) can be realized by forming the letters and symbols with laser cut ECL panels and literally lighting the individual characters. The flat, diffuse character of the ECL light produces virtually no glare or halo, even in adverse weather conditions. There is little wasted light. Reflected light configurations, widely used for internal and external signage, inherently waste much of the illumination energy lighting internal backlight cavities, the ground, or the sky. Typical ECL illumination levels are in the 1 to 10 foot-lambert range. Green, white, and orange phosphors are available.

Studies conducted by the United States Air Force in the 1980's demonstrated that EL lighting had several advantages over incandescent lighting in runway applications. Most notably, for equivalent power levels, EL lights were observed at greater distances than were incandescent lights. Moreover, the observation range of EL lights was not noticeably reduced during dust and haze conditions. These features present obvious advantages over conventional light sources for residential and commercial signs as well as for emergency lighting in industrial facilities. The Air Force study also recognized that EL is a high-quality light source without glare or flickering. Unlike incandescent lights, EL and ECL sources do not require energy-squandering diffusers to eliminate glare. In spite of dramatic differences in lumens/watt performance, the Air Force study demonstrated a human perception of equivalent brightness for an equivalent power level. One possible explanation is the unique spectrum and spatial sharpness of the EL or ECL light.

Spectral power distribution is another dimension of lighting economy. Our eyes are basically tetrachromatic: maximum sensitivity for red-sensitive cones is 570 nm, green-sensitive cones at 535 nm, rods at 505 nm, and blue-sensitive cones at 445 nm. The EL and ECL blue-green phosphors provide a narrow spectrum well matched to the region of

maximum human sensitivity. Hence, surface illumination at low power densities is highly visible.

Because of high visibility in inclement weather, low power consumption, durability, and long service life, ECL systems offer great promise for highway signage applications. For example because of the low power demand, solar power sources are practical for illuminated traffic signage remote to utility power availability.

1.3 Study Objectives

Highway applications involve extended exposure to extreme environments. The purpose of this study was to explore the degradation mechanisms for the glaze topcoat, edges, and rear substrate surface of the ECL lamp panel. A further objective was to evaluate possible coatings for improved environmental protection.

2.0 Experimental Set-up

Advanced imaging techniques were used to photodocument mechanisms of environmental failure of the glaze layer in prototype devices. The environmental test chamber used immersion in megapure water to accelerate corrosion testing. The environmental test chamber was constructed according to ASTM Standard D 870-97. This setup consisted of a lamp placed in a glass container partially filled with megapure water to cover approximately 1/2 of the lamp. The container was covered with a plexiglass sheet and sealed with high vacuum grease to prevent evaporation. Before each experiment, the initial water temperature was recorded, and monitored periodically. The solid state lamps were then characterized using scanning electron microscopy. Corrosion at interfaces was found to be the most important environmental failure mechanism. The substrate (steel) also corroded during sample testing. The efficacy of several protectants was evaluated as a means of deterring this corrosion process.

3.0 Control Experiment

It was determined whether light-emitting panels could function properly during immersion testing. As seen in Figure 2, there is no immediate change observable in the panels in the presence of water. However, in video recordings of an unprotected lamp, such as this one, corrosion became visible after only 20 hours of immersion. Observations indicated that surface defects grew to occupy approximately 75% of the entire panel following a week of the immersion process.

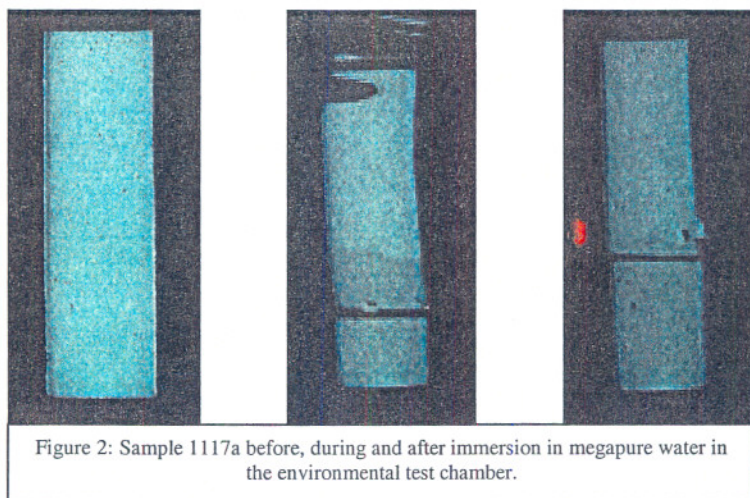


Figure 2: Sample 1117a before, during and after immersion in megapure water in the environmental test chamber.

Since pure water is unlikely to be present in a lamp's normal environment, the effects of adding an electrolyte were considered. However, the fact that damage occurs in the absence of an electrolyte indicates that a complex, corrosive environment is not necessary in order to study the environmental sensitivity of these devices.

4.0 Chemical Reactivity Reporter Probe

Phenolphthalein was chosen as a reporter system because of its properties as an acid/base indicator. A corrosion process or chemical reaction occurring at the sites of degradation yielding a base should produce a color change. A green phosphor, glazed panel was immersed in approximately 500ml megapure water. Three drops of phenolphthalein were added. A slight pinkish tint appeared after approximately 10 minutes, which grew in intensity over the next 24 hours and then diminished slowly throughout the duration of the study. Although some portions of the panel remained able to emit light for several days, the areas where damage did occur spread quickly. Images showing the progression of surface disintegration are presented in Figure 3. The presence of the weak electrolyte, phenolphthalein, did appear to accelerate the corrosion of the substrate, when compared to a sample immersed in megapure water.

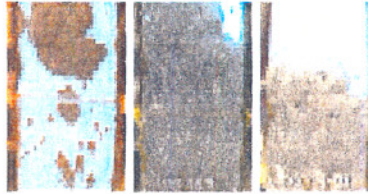


Figure 3: Degradation of panel immersed in phenolphthalein at days 8, 9, and 12, respectively.

5.0 Protection Systems

5.1 Edge Protectants

Apiezon Black Wax

It is anticipated that edge protection may be accomplished using materials with poor optical properties. To determine the ability of some materials to protect panel edges from corrosion, only these areas were initially coated. Apiezon black wax, usually employed as a vacuum sealing wax in air-sensitive applications, was the first of the edge protectants tested. The wax is packaged as small black strips. One strip was heated in the flame of a Bunsen burner to render it malleable. Once warmed, the liquid was spread along the edges of a panel, taking care to cover them completely, see Figure 4. When solidified, power was applied to the panel and it was left illuminated for 24 hours so that the wax could acclimate to the panel's temperature. At this stage the panel was immersed in megapure water in the environmental test chamber. Although signs of corrosion were observed with 24 hours, the resulting damage was insufficient to completely prevent function for 6 full days, as shown in Figure 4. This study leads to the conclusion that the interfaces present at the edges are not the only sites of corrosion. In fact, corrosion initiated at the front surface currently appears to be the chief failure mechanism. At this point efforts were redirected towards finding a protectant capable of preserving the front face of the panel itself.

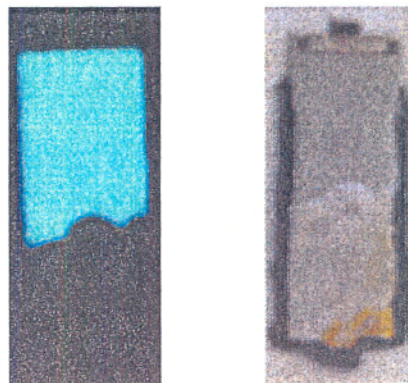


Figure 4: Left, sample 1215bw whose edges are reinforced with Apiezon black wax, before testing; center, 1215bw after 5-day immersion; right, sample 1215bw after 6-day immersion.

5.2 Front Face Protectants

Clear Coat

The first of the surface-protecting agents evaluated was Plasti-Kote Clear Coat, a spray-on acrylic lacquer employed in the automotive industry. A glazed panel was cleaned with 95% ethanol and left to air dry. The Clear Coat was applied according to manufacturer directions and left to dry overnight. At this point, the sample was immersed in the test chamber. After 4 days, water damage was observed on the panel above the water line. This was believed to be due to condensation within the chamber. After 8 days, extensive damage covered approximately 80% of the panel surface. This sample ceased to function after 17 days. This progression can be observed in Figure 5.

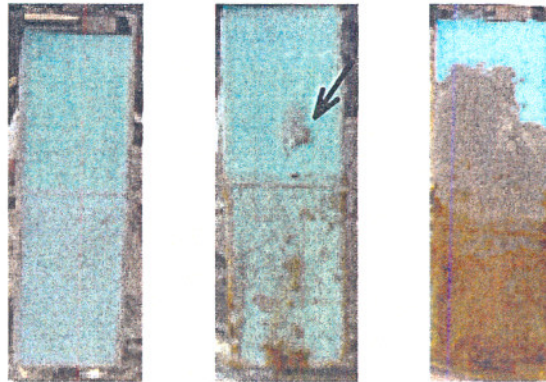


Figure 5: Sample 1229cc initial immersion in megepure water. Center, defect observed above water line at 5 days. At right, extensive damage reduces amount of area capable of emitting light.

Dow Corning Protective Coating

Dow Corning Metal Protective Coating, a spray-on organic wax, was the next protectant evaluated. A green glazed panel was cleaned as before, and the spray applied as directed. The sample was allowed to dry overnight before immersion. This proved to be one of the best protectants studied, in that, this coating proved to delay the effects of corrosion for a considerably longer time than any other application. After 3 days there was little observable damage, and after the corrosion process began, the panel was able to remain functioning for an extended period. This progression can be seen in Figure 6.

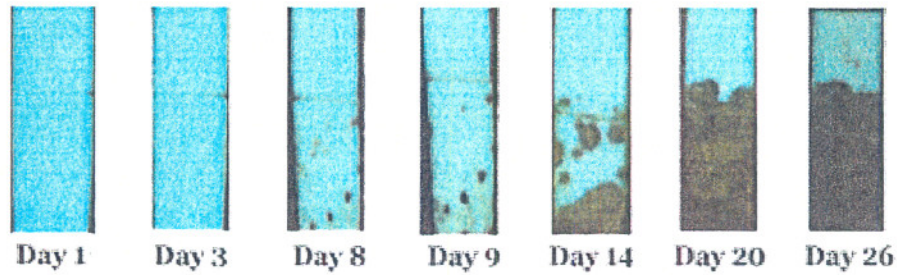


Figure 6: Corrosion progression of Sample 0102ow, a green glazed solid state ceramic lamp coated with Dow Corning Organic Wax.

Rustoleum

Another coating evaluated, Rustoleum, a commercially available clear enamel, proved to be a reasonably good protectant. Rustoleum was able to extend lamp performance to 9 days, with the first signs of corrosion only appearing after 3 days, as shown in Figure 7.

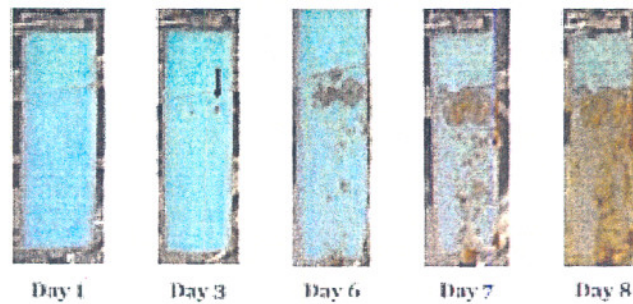


Figure 7: Corrosion progression of Sample 0116R, a panel coated with Rustoleum. The arrow in the Day 3 image indicates an area analyzed by SEM, to be discussed below.

Protective Overlay Film, POF 1160

A piece of 3M's protective overlay film, POF 1160, was cut large enough to wrap around the front and side edges of a panel. A second piece was applied to the back of the panel to ensure an airtight seal. This film, developed to protect traffic signs, was considered to be a promising protectant; but in actuality was only able to protect this panel for 19 days. This protectant was revisited in an attempt to improve its usefulness. One method of application is to cut the pieces to cover the front and back of the panel to be the same size, and to apply them so that they cover the panel like an envelope. Signs of corrosion began to appear after only 1 day, as seen in Figure 8. Delamination of this film should be further investigated. Unusual arcing effects have also been observed when using this film, maybe due to the organic solvents outgassed from the film or its adhesive layer.

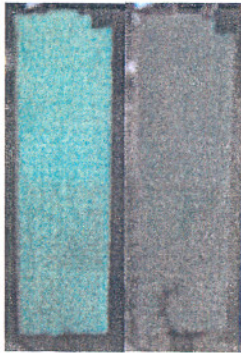


Figure 8: Sample covered with POF 1160. On the left, an image of the sample on Day 1 of immersion. On Day 2, as seen on the right, the beginning stages of corrosion are apparent in the bottom left of the image.

Sol Gel

A substantial number of corrosion resistance studies were performed using acid- and base-catalyzed sol gel, not only as a protective coating, but also as a replacement for the glass encapsulation itself. Our first experience with sol gel, called for a base-catalyzed preparation. This involved adding 19.0ml megapure water with approximately 10 drops of concentrated NH_4OH (to $\text{pH}=10$) to 15.5ml 95% EtOH and 15.0ml tetraethyl o-silicate. This solution was sonicated for 20 minutes. Resulting sol gel mixtures were evaluated for their ability to adhere both to the phosphor and a metal substrate, as discussed below.

The amount of phosphor to be suspended in sol gel was determined by first measuring the density of a solidified piece of sol gel and using this value to calculate the amount of phosphor needed to achieve a 4:1 ratio of sol gel to phosphor. This was an approximation of the amount of phosphor and glass present in an actual panel. Roughly 0.4g phosphor were used in each experiment of this segment of the study. Attempts to obtain large, transparent fragments of this phosphor-impregnated sol gel curing in air at room temperature and at 60, 80, and 100°C for varying amounts of time were unsuccessful. After 20 to 30 experiments carried out in this manner, it became clear that these mixtures were highly sensitivity to experimental conditions. This suggests that further experiments may produce favorable results. Similar attempts to bind sol gel to an alternative metal substrate, Nilo K, were also unsuccessful. For this reason, our attention was turned to acid-catalyzed sol gel.

Acid-catalyzed sol gel can be prepared in the same manner as previously described, with the exception that 3 drops of concentrated HCl (to $\text{pH}=3$) are substituted for the NH_4OH . With acid catalysis, the resulting solid appeared to be clearer and less brittle than base-catalyzed sol gel. Applying sol gel to a glazed panel was realized in several ways. Dipping, spin-coating, and applying drop wise were assessed. Of these, dipping provided the most encouraging results. Panels receiving spin-coating and drop wise applications were able to function for 12 days, while those receiving a dipping application could function for 21 days. The effects of preparation temperature were also investigated. Samples coated with sol gel and “cured” in an oven at various temperatures were not as resilient as those whose coatings were allowed to cure more slowly at room temperature.

After X-ray analysis by SEM showed chloride deposits on the surface of sol gel-coated panel, alternative acids for catalysis were evaluated. In addition to re-evaluating the performance of a solution prepared with concentrated hydrochloric acid, solutions catalyzed with 85% phosphoric acid and glacial acetic acid were applied to green unglazed panels. The sample coated with acetic acid-catalyzed sol gel proved to be the most durable, outlasting by an average of 4 days. Sol gel as an encapsulant showed great potential in that degradation appeared after 3 days, rather within the first 24 hours as with the traditional glass encapsulation. In the progression below, days 1, 3, 7, 9, 11, and 13 are shown, respectively.

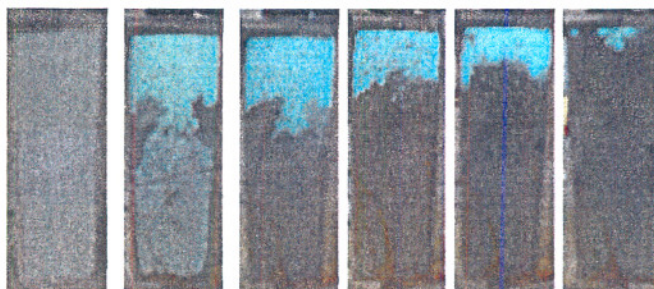
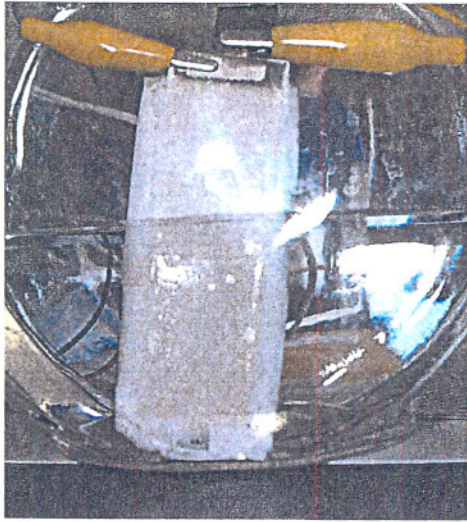


Figure 9: Progression of corrosion on an acetic acid-catalyzed sol gel coated unglazed panel. Notice that the panel was able to function in areas where damage had not extended. This panel ceased to function when phosphor damage reached the silver bus, located at the top of the images.

Torr Seal and POF 1160 Combination

Torr Seal is an ultra high vacuum epoxy that has been used in several previous investigations with only marginal success: it alone will only prolong the life of a panel by 8 days. 3M's protective overlay film, POF 1160, also showed marginal results when used alone in previous investigations, in large part due to the airtight seal that was supposedly attained likely to not have been present during previous work. This investigation attempts to use these two products in combination to examine their effects upon preventing corrosion.

A white phosphor glazed panel was cleaned with ethanol and wiped clean with a Kemwipe. A piece of POF 1160 was cut and applied to the illuminating surface of the test panel. Torr Seal was mixed according to the manufacturer's directions and a thick coat of the epoxy was applied to all corners of the panel. A second piece of POF 1160 was then applied to the back of the sample immediately following the application of the Torr Seal, meaning that the epoxy did not have adequate time to dry. This, in effect, sandwiched the epoxy between the layers of overlay film. The sample was allowed to dry to hardness (for 6 hours) in hopes of an airtight and waterproof seal being attained. The panel was then immersed in distilled water and illuminated.



Day 1



Day 18

This combination has proven to be quite successful. Not only has the panel maintained all of its function, but it has also shown absolutely no signs of corrosion over the 16 days of the ongoing trial. The only problem that has surfaced during this investigation has been the air bubbles that have formed within the POF 1160 film. After discussing the problems with a road sign manufacturer, careful application of the overlay film should eliminate this problem. Further investigations are planned for future work

5.3 Back Face Protectants

Hot Paint

With rust being prominent in all images showing signs of corrosion, it was agreed that the metal substrate must also be protected. A commercial product manufactured by Plasti-Kote, Hot Paint, used in the automotive industry as an engine protectant, was applied to the back surface of a green phosphor, glazed panel and allowed to air dry for 1 hour. At this point the panel was placed in a 650°C oven for 1 hour to cure. After cooling, the sample was immersed, as was an untreated panel to serve as a control. Unfortunately, there was no noticeable degree of difference in the amount of rust present after a 10-day test period.

Alternative Substrate

In another effort to lessen the presence of what appeared to be common rust, alternate metal substrates were investigated. The most promising of these was Nilo K, commonly called Kovar. Special Metals, Inc manufactures Nilo K, which is an alloy containing 29% nickel, 53% iron, and 17% cobalt. Since this alloy has a coefficient of expansion very similar to that of borosilicate glasses, it is commonly used in glass-to-metal seals. Since glass encapsulation is being employed, such a seal is necessary. The higher percentage of nickel present would understandably reduce the amount of rust produced in the presence of moisture. These traits would make this alloy ideal for our purposes. A sample 4 in. wide and 0.6mm thick was obtained for study. As mentioned earlier, some

pieces were used to test adhesion to base-catalyzed sol gel. Other portions of this sample were submitted to MRE so that a prototypical panel could be made using this as a substrate. These prototypes have not yet been made available for testing.

6.0 Potentiometric Indicator Investigations

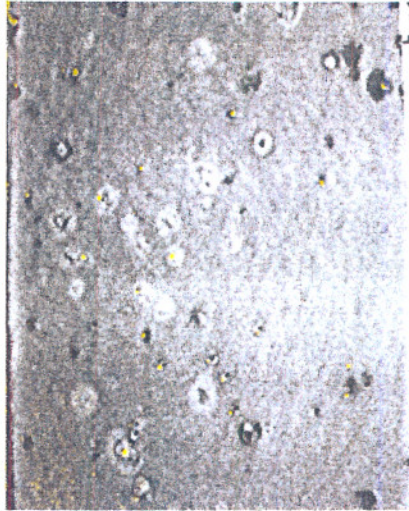
Previous work done upon mapping the locations of the corrosion sites using fluorescent dyes did not produce good results. These queries examined the efficacy of methylene blue, a potentiometric dye, and its possible use as an indicator of the future sites of corrosion.

Potentiometric indicators such as methylene blue are an important class of compounds known as redox indicators. These are substances that do not participate in the redox titration, but whose oxidized and reduced forms differ in color. When added to a solution containing the analyte (in our case being the illuminated panel), the indicator changes color in response to the electrochemical potential and not to the presence of a specific species. Methylene blue has an oxidized color of deep blue and is colorless when reduced. Only the indicator that is in contact with the metal substrate should be capable of being reduced. Our studies examined the effects of this dye.

Potentiometric Indicator Timing Study I

Our first experimental setup involved a white phosphor with glaze panel that was cleaned with ethanol and wiped with a Kemwipe. A 110 Volt AC electric source was connected with the positive electrode going to a piece of copper wire, and the negative electrode connecting to the testing panel. A third wire also connected the panel to the copper wire. Both the copper wire and panel were immersed into a solution containing methylene blue and distilled water. The electrode was then plugged in and the panel was illuminated for five minutes. The panel was then disconnected from the electrodes and cleaned, again using ethanol and a Kemwipe. One could visually see the many locations where there appeared to be locations where the potentiometric dye had become trapped within the panel. Using the L-W Scientific camera, the sites where the potentiometric dye had attached were mapped out.

The panel was then attached to a power supply, dipped in distilled water at a temperature of 23°C, and allowed to corrode over a seventeen-hour period. The sample was then taken back to the L-W Scientific camera and pictures documenting the corrosion followed.



Time Lapse Photo Showing the Sites of Methylene Blue Entry in Yellow

The initial photographs were overlaid directly on top of the image of the corroded sample. The picture above shows one portion of the panel under investigation where the methylene blue was changed from its blue color to a bright yellow color to aid in seeing it contrast against its background. A surprisingly high 91.8% of those (102 actual imperfections) sites that took on the methylene blue led to corrosion on the following day. While over 90% of those sites where the methylene blue there was still 60 corrosion sites, 37% of the total number of corrosion sites, which cannot be accounted for in this experimental setup. More investigations into these findings will follow, chiefly changing the amount of time that the panels are allowed to be exposed to the methylene blue and the corrosion time to see how much of an effect that it could have upon the results.

Potentiometric Indicator Timing Study II

The previous potentiometric indicator study showed a high correlation to those sites that trapped the potentiometric dye at a specific location leading to corrosion within a seventeen-hour period. A problem with the previous experimental setup was that one could not adequately tell if the dye was being entrapped within a pore of the glass or if the dye was actually passing all of the way through the glass layer and onto the metal surface. The potentiometric dye was an excellent indicator to use within this setup because if it were reduced, it would turn colorless only at those sites directly in contact with the metal. Using Adobe Photoshop, detailed photos were taken of a panel that was stained with the methylene blue, then pictures in its reduced state, and overlay both of these on top of a picture of the panel once it was corroded. Those colorless sites should be the sites where corrosion has undoubtedly taken place.

The first portion of this experiment was exactly like the previous potentiometric investigation. A green with glaze panel was cleaned with ethanol and wiped with a Kemwipe. A 110 Volt AC electric source was connected with the positive electrode going to a piece of copper wire, and the negative electrode connecting to the testing panel. A third wire also connected the panel to the copper wire. Both the copper wire

and panel were immersed into a solution containing methylene blue and distilled water. The electrode was then plugged in and the panel was illuminated for five minutes.

The panel was then disconnected from the electrodes and cleaned, again using ethanol and a Kemwipe. One could visually see the many locations where there appeared to be locations where the potentiometric dye had become trapped within the panel. Using the Pixera camera, the sites where the potentiometric dye had attached were mapped out. The panel was again cleaned with ethanol a Kemwipe. A 9 Volt DC electric source was connected with the positive terminal connected to a piece of copper wire, and the negative terminal connecting to the testing panel. A third wire also connected the panel to the copper wire. Both the copper wire and panel were immersed in 0.13 M sodium chloride. The electrode was attached in this configuration for five minutes. Photographs using the Pixera camera were again taken.

The panel was then attached to a power supply, dipped in distilled water at a temperature of 23°C, and allowed to corrode over an eighteen-hour period. The sample was then taken back to the Pixera camera and pictures documenting the corrosion followed. No photographs were available for this report.

The results of this investigation were quite promising. Out of 86 "hits", that is, where the potentiometric indicator once was but was then reduced to colorless 93, 62% matched up to corrosion sites. As in the previous study, more investigations need to follow to examine the effects of changing the amount of time that the panels are allowed to be exposed to the methylene blue and the corrosion time to see how much of an effect that it could have upon the results.

7.0 SEM Analysis Results

It is believed that corrosion of all test panels occurred through essentially the same mechanism. A mechanism involving water penetrating the lamp at some point was inferred from SEM analysis. Once an entrance pathway for water has been established, it can diffuse through the respective layers until it reaches the dielectric/metal interface. Once reaching this area, water is trapped and accumulates. The increasing volume of water separates the dielectric layer from those above it and causes delamination. Using SEM imaging and Quantitative Analysis corrosion defects such as the one indicated in Figure 7 were further interrogated. Figure 10 shows one corrosion site that possibly caused the failure mechanism. The secondary image (on the left) supports the delamination theory because the area around the defect is distended. The mapgroups show the location of the observed elements and their relative intensity, implying that all layers above the dielectric/metal interface were penetrated.

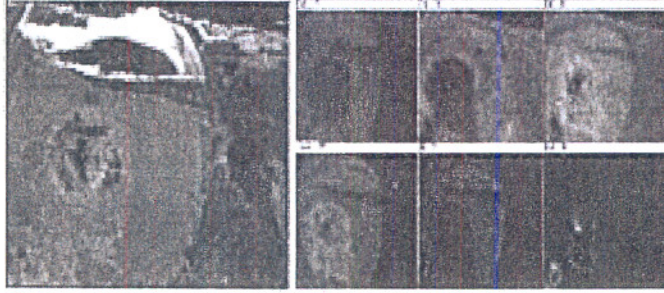


Figure 10: The left image is a secondary image of a defect such as the one indicated by an arrow in Figure 7. It was taken at 50x and 15kV by a JEOL 5310-LV Scanning Electron Microscope. The images at right are a mapgroup of the same area determined by X-ray analysis.

8.0 Conclusions

None of the tested coatings served as a perfect protectant, although some were able to slow the corrosion process. An untreated panel can undergo sufficient corrosion to prevent function in as little as 12 hours. Treated panels can maintain function for several days, even though beginning signs of corrosion can appear as quickly as 24 hours.

Current and future work will include probing the effectiveness of new protectant coatings and combinations thereof. Surface defects will be further studied. These defects will be characterized using scanning electron microscopy and elemental analysis.

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