

Evaluation of Organic Coatings with Electrochemical Impedance Spectroscopy

Part 3: Protocols for Testing Coatings with EIS

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In Parts 1¹ and 2² of this Series, we discussed the technology of applying electrochemical impedance spectroscopy (EIS) to organic coatings on a metallic substrate such as aircraft, marine, or industrial maintenance coatings. This article describes several experimental protocols to evaluate these coatings with EIS. These experimental protocols differ primarily in the process used to stress the coating and accelerate the degradation of the coating.

There is no standard recipe for an EIS-based evaluation program that is guaranteed to work for every coating in every environment. This may come in time and, indeed, a standard for EIS evaluation of coatings is under development at ASTM and ISO.³ However, EIS can be employed in a variety of ways to evaluate virtually any coating.

It may be useful to think of EIS as a very sensitive detector that provides a snapshot of coating status. However, a single EIS measurement of an organic coating tells you nothing. To measure coating lifetime or performance, the coating must be stressed to bring about its failure. By making periodic EIS measurements during the stress process, a rate of coating failure can be estimated and a series of coatings may be ranked.

Even though some publications discuss the determination of the time-to-failure of a coating, this may be an unrealistic goal. There are too many variables that separate us from this "Holy Grail," most of which are not related to EIS. A more achievable objective is to use EIS in an experimental program that results in a performance ranking of a series of coatings for use in a specific environment.

The nature of the stress applied to the coating is, of course, very important in several aspects. The experimental design to prompt the failure of the coating must (1) simulate the service environment the coating will encounter and (2) it must not change the failure mechanism.⁴

To use EIS to evaluate a specific coating system, (1) place the coated sample in an environment designed to accelerate the degradation of the coating, (2) measure the EIS curves over time, and (3) identify an "index" that tracks coating quality. The index could be the Coatings Capacitance or the Pore Resistance, for example. The index can be very simple or more complex and we will look at several examples in this article. Unfortunately, all coatings do not fail in the same way, so there is no universal index for assessing coating quality with EIS.

This complex nature of coatings is no surprise to coatings scientists. A coating system may consist of the metal substrate, surface pretreatment, a primer, and one or more topcoats. Results can vary depending on types of coatings, thickness, number of layers, surface treatment, and the nature of the metal substrate.

EXPOSURE TESTS

For the purposes of this discussion, an "exposure test" implies a testing period equivalent to a typical standardized cabinet or atmospheric test. This may be as short as 15–30 days or as long as several years.

EIS and Atmospheric Exposure Tests

For the ultimate in coatings evaluation, atmospheric exposure is still the "gold standard." Every other test is an attempt to simulate the results of atmospheric tests. The problem with at-

mospheric tests, of course, is that they require a long, long time. Nothing can accelerate the deleterious effects of atmospheric exposure, but EIS can observe the deterioration of the coating long before visual defects appear.

Measure the EIS curve periodically during the exposure period. Place the sample in contact with the electrolyte in an electrochemical cell and measure the open-circuit potential (Eoc) as a function of time. The electrolyte can be chosen to simulate the particular atmospheric conditions of the exposure test. Run the EIS experiment when the sample has reached a steady state, signaled by a stable value of Eoc. Most computerized EIS instruments can measure the stability of Eoc. You can run the experiment when the stability is better than 0.1 mV/sec.

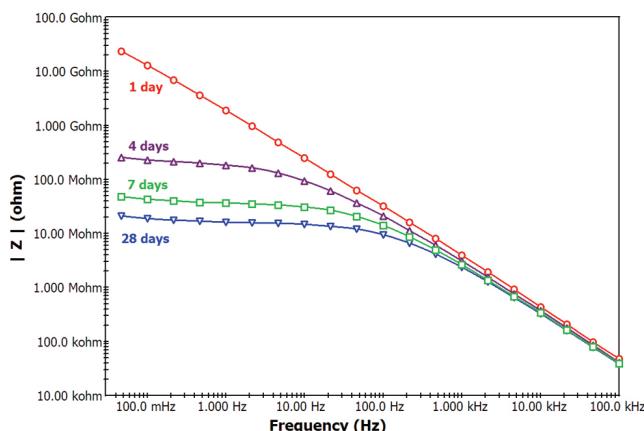
Immersion and Measurement of Impedance Magnitude at 0.1 Hz

The most straightforward use of EIS to characterize coatings is to immerse the sample in an electrolyte and periodically measure the impedance spectrum. This approach is exemplified by Gray and Appleman,⁵ who developed a method to determine the barrier protection properties of coatings. Samples were immersed in 5% NaCl solution, sealed, and placed in an oven at 65°C to accelerate attack. The panels were removed from the oven at 1, 4, 7, 14, and 28 days and the EIS curve was run. (See Figure 1.)

The limiting impedance at low frequency is equal to the sum of Pore Resistance (Rpore), the Polarization Resistance (Rp), and the Solution Resistance (Rs). Rp and Rpore are ini-

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Figure 1—EIS response of a pipeline coating in 5% NaCl at 65°C. (Figure 8 from reference 5.)



tially quite high and usually decrease with time as a result of the attack on the coating and initiation of under-film corrosion. R_s is usually very low and can be ignored. The log of the impedance modulus at 0.1 Hz was plotted as a function of immersion time. The data suggests that if the log of the impedance modulus is above 7 (impedance $> 10^7$ ohm-cm 2), then the coating was affording adequate corrosion protection to the surface. Below this impedance, the protection was poor. Above a value of 9 (impedance $> 10^9$ ohm-cm 2), the protection is good to excellent. These tests were conducted on both laboratory and field samples.

Long-Term Shelf-Life Tests

In an early publication,⁶ Tait detailed the results of extended shelf-life tests. A large population of internally coated metal containers was aged in "typical" proprietary electrolytes at normal storage temperatures (21 °C or 70°F) for longer than two years. Impedance measurements were made at periodic intervals. The coatings fell into three classes.

In the first class, the EIS response was essentially that of a capacitor (see Figure 3A of Part 2 of this series¹). After six months, the capacitance values were virtually the same as when the exposure test was started. After 24 months, no corrosion or coating delamination was observed upon physical examination of the samples. The constancy of the coating capacitance indicates little or no water uptake in the first six months.

In the second class of coatings, the EIS response closely followed the "coat-

ing model" (Figure 3 in Part 2 of this series). Tait found that the time-to-failure tracked the rate at which the polarization resistance value in the model dropped with time. If the R_p changed rapidly in the first days of exposure, the container failed after three months. Those containers with a slowly changing R_p lasted more than two years.

EIS and Cabinet Tests

The most common testing technique for coatings is exposure to a series of controlled aggressive conditions in a cabinet constructed for this purpose. The conditions include a variety of chemistries as well as exposure to UV radiation and cycles of wetness/dryness and heat/cold. These cabinets have been in common use for decades and attempt to simulate atmospheric or industrial conditions that can be used to degrade the coating in a realistic fashion. The goal is to correlate cabinet tests with actual exposure tests to predict time-to-failure. It is generally accepted that cabinet tests provide comparative results and not absolute results.

When used with cabinet tests, EIS acts as a quantitative detector of coatings quality. The EIS response of a sample

undergoing cabinet tests will follow the general trend described in Figure 3 of Part 2 in this series.² An EIS experiment is conducted on the specimens in the cabinet on a regular basis. If the samples are deteriorating rapidly, the EIS curves should be run daily. For more durable paints, a weekly EIS evaluation may be sufficient.

Some of the more popular cabinet tests are ASTM B 117 Salt Spray, ASTM D 5894 Cyclic Salt Fog/UV Exposure, and SAE J2234 Laboratory Cyclic Corrosion Test. The cabinet test standards specify the conditions for exposure in the cabinet, but do not provide testing methods or pass-fail criteria. This is addressed in ASTM D 1654, "Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments." All of these testing protocols are qualitative in nature.

Coupling EIS with these standard cabinet tests can provide a quantitative measure of coating deterioration.

ASTM D 1654 discusses both scribed and unscribed panels. A scribed panel is generally used to simulate major damage to the coating that exposes the substrate-coating interface. The loss of adhesion is quantitated by measuring the length of "creepage" of the paint film from the scribe after air blow-off or scraping. Adhesion may also be measured with a tape pull-off test (ASTM D 3359). Tests on a scribed panel do not measure the barrier properties of the coating; they measure the ability of the coating-substrate to self-repair when the substrate is exposed.

The Knife Adhesion Test in ASTM D 6677 tests the adhesion of the coating on an unscribed sample, usually after a

Figure 2—Reversible behavior of a coating during thermal cycling. Open circles indicate heating, closed circles indicate cooling. (Figure 6 from reference 9.)

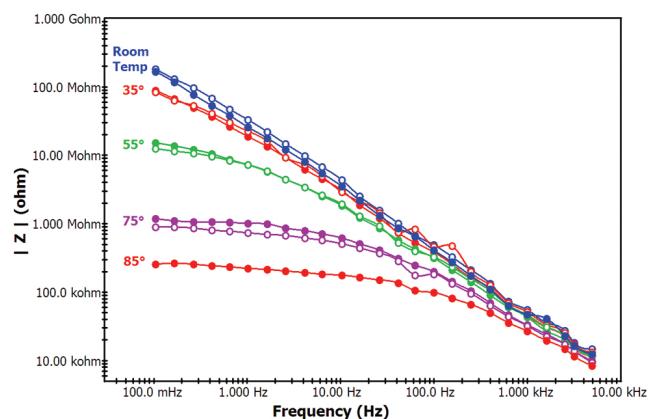
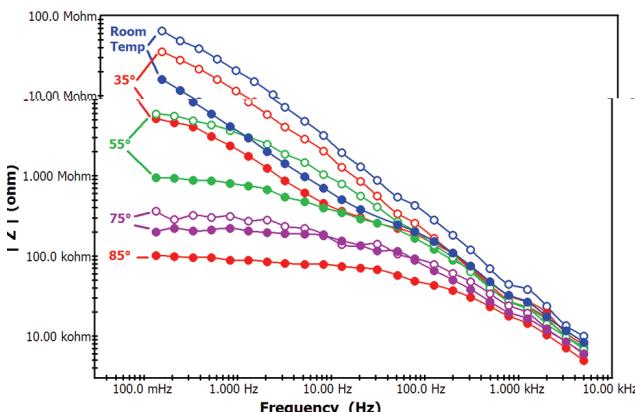


Figure 3—Irreversible behavior of a coating during thermal cycling. Open circles indicate heating, closed circles are cooling. (Figure 8 from reference 9.)



controlled exposure of some sort. There is a fundamental difference between pulling the coating off with tape and lifting the coating with a knife, and some workers prefer the latter.

An unscribed panel is used to test for rusting (ASTM D 610), blistering (ASTM D 714), or adhesion (ASTM D 3359) through the coating. Both D 610 and D 714 provide a semi-quantitative ranking technique involving the comparison of the tested panel to a series of photographs. The quantitative numerical results from EIS are seen as a major technical advancement in this area.

Since one of the key advantages of EIS is the ability to simultaneously measure the barrier properties and the corrosion properties, scribed panels are rarely used with EIS. The scribe inflicts physical damage to the paint film and underlying substrate and contributes to poor reproducibility.⁷

ASTM B 117 Salt Spray is the oldest standard cabinet test and, therefore, the test with the most history. B 117 is used to test bare metals and painted metals. With regard to painted metals, history has not been kind to ASTM B 117 and most users accept that B 117 does not correlate well with actual exposure. Nevertheless, it is still in common use, particularly for quality control applications or comparing different materials. ASTM B 117 uses a salt fog of 5% NaCl to accelerate the natural corrosion process.

Based on comments from within the industry, ASTM B 117 is not considered to be a useful technique for evaluating coatings and its use with EIS is not recommended.

ASTM D 5894 Cyclic Salt Fog/UV Exposure of Painted Metal differs from B 117 in three key factors: UV exposure, more dilute salt solution, and wet/dry cycles. The intense UV radiation at 340 nm photochemically degrades the coating and is a key factor in simulating exposure in exterior conditions. The wet and dry cycles provide realistic conditions for corrosion. The test involves:

- A one-week (168 hr) exposure cycle of 4-hr UV at 60°C and 4-hr condensation at 50°C followed by:
- A one-week (168 hr) fog/dry cycle of 1-hr fog (0.05% NaCl and 0.35% NH_4SO_4) at ambient temperature and 1-hr dry-off at 35°C.
- The cycles may be repeated if agreed by the parties involved.

ASTM D 5894, unlike B 117, was developed specifically for coatings and is generally agreed to give more realistic results. These results, however, are comparative and not absolute. ASTM D 5894 is also referred to as a Prohesion Test, from "Protection by Adhesion."

ASTM D 5894 has enjoyed wide acceptance by the coatings community. Bierwagen⁴ recommends the use of D 5894 with weekly EIS analysis to track the status of the coating.

To employ EIS as a quantitative sensor of coating degradation during D 5894, remove the panel from the fog cycle, immerse the panel in an electrolyte of 0.05% NaCl and 0.35% NH_4SO_4 , allow the sample to equilibrate for 30 min, then run the EIS curve. To obtain the most consistent results, the samples should be removed from the cabinet at the same point in the cycle. If possible, coordinate multiple panels so they are outside of the cabinet for the same amount of time.

SAE J2334 Laboratory Cyclic Corrosion Test⁸ was developed by the automotive and steel industry specifically for automotive coatings and is widely used by both

the automobile manufacturers and their vendors. The development involved comparing several different test conditions on standard panels that had been exposed to an urban industrial environment for five years. The test conditions that gave the best correlation to the exposure tests were selected. The conditions of J2334 were selected primarily to simulate the effects of road salts. It is interesting that J2334 does not employ UV exposure.

One 24-hr cycle of SAE J2334 consists of three stages:

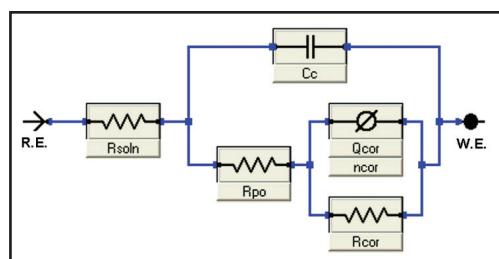
- Humid Stage—50°C and 100% relative humidity for 6 hr.
- Salt Application Stage—Dip, fog, or spray a salt solution (0.5% NaCl, 0.1% CaCl_2 , 0.075% NaHCO_3) for 15 min.
- Dry Stage—60°C and 50% relative humidity for 17 hr, 45 min.

The typical SAE J2334 test is conducted for 60 cycles for coated samples. The test allows for either manual or automatic operation. Because SAE J2334 was developed for a relatively specific sample, there is a correlation to actual exposure time: 80 cycles of SAE J2334 corresponds to about five years of exposure.

ACCELERATED TESTS

Even though a cabinet test is faster than real-world exposure, it still takes a long time. One cycle of ASTM D 5894 requires two weeks. One cycle of SAE J2334 takes 24 hours and the normal test requires 60 cycles, or two months! Since nothing is ever fast enough, several attempts have been made to develop quicker tests for paints. These short-term tests introduce more aggressive stress conditions to induce failure in a shorter time. The user of the accelerated tests must be concerned that (1)

Figure 4—Equivalent circuit used in the REAP procedure. The Constant Phase Element is indicated by \emptyset .



the stress method does not change the mechanism of failure and (2) the stress method is sufficiently analogous to the service conditions to be relevant. The ultimate goal is a short test that produces a predictive result.

Thermal Cycling

Bierwagen⁹ has investigated the acceleration of coatings failure by high temperature. An increase in temperature will increase the rate of diffusion of the electrolyte into the coating, reducing the barrier properties of the coating and possibly enhancing the chemical and physical "aging effects" from attack by the electrolyte.

The sample is immersed in the electrolyte (0.05% NaCl and 0.35% NH_4SO_4) from ASTM D 5894. An EIS curve is obtained at room temperature, 35°, 55°, 75°, and 85°C, then in the same sequence back to room temperature. The EIS data is obtained at each temperature after equilibrating for 20 min. A complete test procedure consists of three temperature cycles, followed by a three-day immersion, and a final EIS scan. A complete test period requires about one week. To obtain similar results with a Prohesion Test may require 4–12 weeks.

The behavior of the EIS curves during the thermal excursions provides an indication of coating quality and corrosion resistance. As the temperature is increased, the total impedance at low frequencies is reduced. When the sample is cooled, the low frequency impedance may (Figure 2), or may not (Figure 3), return to its original value. The return of the impedance at low frequency to its initial value is an indication of good corrosion resistance of the coated sample.

If the temperature range of the thermal cycling test includes the glass transition temperature (T_g), it might be wise to run two tests: one that remains below T_g and another that incorporates the normal temperature limits.

Rapid Electrochemical Assessment of Paint (REAP)

In 1996, Kendig and coworkers published an electrochemical approach to a 24-hour determination of the time-to-failure of an automotive coating on mild steel.¹⁰ The Rapid Electrochemical Assessment of Paint (REAP) protocol incorporates an impedance measurement

to determine the barrier properties of the coating and a cathodic disbonding procedure to determine the damage caused by corrosion at the metal-paint interface. To our knowledge, the REAP test is the only published procedure to combine an EIS measurement with a physical test for paint adhesion.

The EIS measurements were performed in 0.5 M NaCl. The impedance was measured immediately after immersion and again after 24 hr. Although a frequency sweep from 10^4 to 0.1 Hz is sufficient to characterize the sample initially, a lower frequency of 0.01 Hz is needed for the later scan. The lower frequency is necessary to define the EIS curve after the development of a Pore Resistance and a Polarization Resistance.

The equivalent circuit shown in Figure 4 was used to model the system. Notice that the authors chose to use a Constant Phase Element (CPE) instead of a capacitor to model the coating-metal interface. A CPE has been described as an "imperfect capacitor" and is mathematically expressed as:

$$Z_{\text{CPE}} = (1/Y_0)/(\text{j}\omega)^\alpha$$

Y_0 is a constant, $\text{j} = (-1)^{1/2}$, $\omega = 2\pi f$, and α is a constant between 0 and 1. If $\alpha = 1$, Y_0 is the capacitance. The use of the CPE as an element in the equivalent circuit is left to the discretion of the user. Use of a CPE can sometimes give a better fit with a model. Even though the CPE does not have a simple explanation, it is relatively popular in the electrochemical literature.

The cathodic disbonding experiment is performed on a second identical sample. The sample is scribed through the

Figure 5a—Impedance response of an epoxy coating on steel to the AC-DC-AC procedure. (Figure 3 from reference 12.)

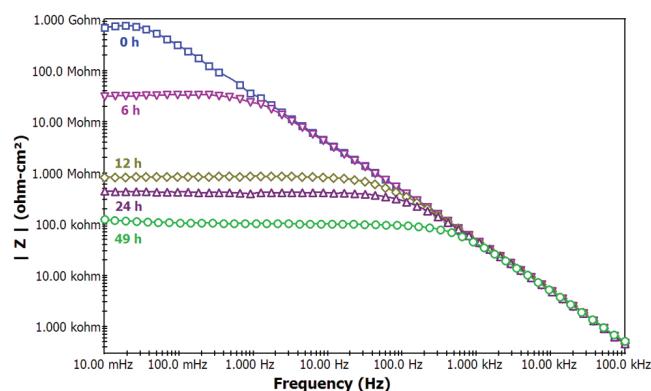
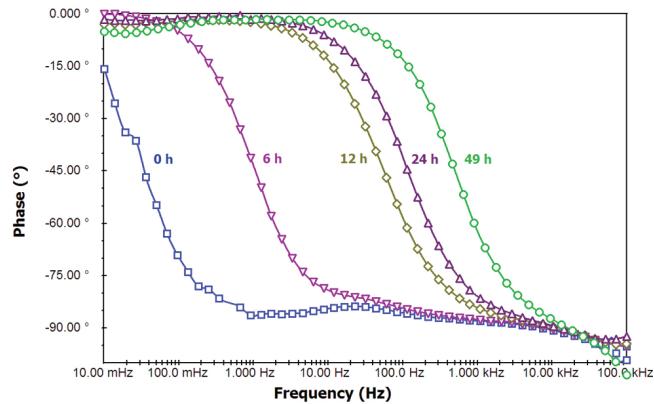


Figure 5b—Phase response of an epoxy coating on steel to the AC-DC-AC procedure. (Figure 3 from reference 12.)



paint to expose the underlying metal. The scribed sample is immersed in 0.5 M NaCl and a potential of -1050 mV is applied for 24 hr. The primary reaction is the reduction of oxygen.

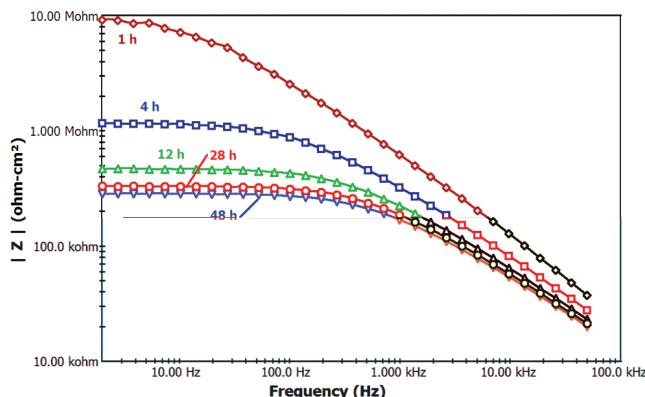


The alkaline environment produced by the cathodic reaction is particularly detrimental to the adhesion of the coating to the metal. The coating is further stressed by the oxidation of the metal (usually iron) to the oxides, which have a higher volume than the base metal.

After completion of the cathodic polarization, delamination of the coating is measured by placing tape across the scribe and pulling to remove the portion of the coating that has disbanded.

The goal of the REAP technique is ambitious, since the authors not only had to define the parameters to predict time-to-failure, they also had to define time-to-failure itself, a not insignificant

Figure 6—EIS response of a free PVC plastisol film during immersion in 3% NaCl. (Figure 2 from reference 13.)



task that, regardless of the definition, is certain to attract critics. ASTM B 117 salt fog was chosen as the accelerated test, not so much for its successful prediction of lifetimes, but because of its widespread use in the coatings industry.

The best correlation was obtained by using R_{cor} , % water uptake, and pull-back (dx/dt) to estimate time-to-failure. Note that R_{cor} and R_p are identical.

$$TTF = -830.1 + 118 \log R_{cor} - 169.2 \log \frac{dx}{dt} - 48.03 (\% \text{water})$$

The water uptake was calculated by measuring the initial EIS spectrum and again after 24 hr. Coating capacitances (C) were evaluated from the EIS measurements.

$$\text{Volume fraction of water} = \frac{\log (Ct/Co) \log 80}{\log 80}$$

The concept of combining EIS to measure the barrier properties and more conventional physical techniques to evaluate adhesion is attractive to many researchers. The consensus seems to be that barrier properties and adhesion are both important, but very different. Since adhesion is a function of chemical, electrochemical, and physical properties, EIS may not always serve to evaluate adhesion. In addition to the tape pull-back test, adhesion can be tested with ASTM D 6677 (Knife Adhesion Test). It can also be useful to combine cathodic disbonding with tests such as D 6677. In the case of D 6677, cut the coating with the knife, then apply a potential of about -1 volt vs. SCE to encourage cathodic disbond-

ing. Assess the disbonding as described in D 6677.

cally polarized to generate an alkaline environment and stimulate delamination; and (3) an EIS curve is run to assess the condition of the coating after delamination. Steps 2 and 3 may be repeated to apply additional stress to the sample if desired (Figure 5).

The cathodic potential (a negative potential is termed "cathodic" because it prompts a reduction reaction) generates hydrogen and hydroxide ions at the surface of the metal beneath the coating.



The adhesion of the coating is compromised by the alkaline environment and delamination is further encouraged by the pressure of the hydrogen beneath the coating.

For fresh, intact coatings, it is necessary to apply a pronounced negative potential (from -2 to -3 volts) to induce a stress. Based on the interpretation of the structure of a paint film on a metallic substrate, the cathodic polarization step must attack the coating by opening pores, allowing access to the metal surface. From the EIS response to this applied stress, that is exactly what is happening as noted by the reduction in the limiting impedance at low frequency.

The AC-DC-AC test can be conducted on a paint panel in a typical three-electrode electrochemical cell. It has also been successfully employed on routine samples or on samples outside of the laboratory by using the EIS instrument in "two-electrode mode" and

contacting the sample using a copper disk and filter paper moistened with the appropriate electrolyte.

STUDIES OF FREE PAINT FILMS

The effect of the paint film can be separated from effects of the metal substrate or the metal-paint interface by studying the free paint films. The free films can be produced by applying to glass, plastic, or smooth metal and carefully removing. The free films are mounted in an electrochemical cell that allows an electrolyte to be placed on either side of the film. The EIS curve is generated using a "four-terminal" or "four-electrode" measurement, in which a reference electrode and an inert electrode (usually platinum) are placed on either side of the membrane.

Permeation of the coating with ions or water can be precisely studied with free films.^{13,14} The changes in impedance of a free film after immersion are similar to the changes observed in coatings applied to substrates, but they occur faster. The EIS response typically displays a decrease in impedance and an increase in capacitance as water penetrates the film (see Figure 6).

PRACTICAL ISSUES

EIS and Coating Thickness

A newcomer to EIS may have concerns regarding the maximum coating thickness that can be measured. The thickness of the coating is not the issue; the impedance of the coating is the figure of merit. Thickness is immaterial. For example, a two-inch filled polymeric coating on the high-strength steel hull of an ocean-going vessel can be evaluated using EIS. The impedance is about 10^{13} ohms-cm², which is very high. The EIS measurement was assisted by using a 12 in.² sample.

Precision of EIS Measurements

To have confidence in a scientific measurement, it is important to understand the accuracy and precision of the measurement. The accuracy and precision of modern EIS instrumentation is

typically $\pm 1\%$ for impedances between 1 and 10 megaohms at frequencies between 10 μ Hz and 100 kHz. For organic coatings with impedances higher than 10 Mohms (10^7 ohms), the user should confirm that the EIS instrument is capable of proper operation in this impedance region by running an Open Lead Curve. (See Part 1 of this Series.)

The greatest source of error is the variation in coating thickness. This variation can occur on the surface of an individual sample and from sample-to-sample and can be remarkably high. These issues are discussed for coated aerosol containers by Tait.¹⁵ When dealing with sample variability, it is best to increase the number of sample replications. Tait recommends eight replicates for coated samples.

Cable Length

In the field, the sample can be a long distance from the EIS instrument. This is particularly true for towers, aircraft, or watercraft. This requires the use of long cables that, because of the additional capacitance they add to the system, can cause serious problems with noise pickup. Cable length should be kept as short as possible. In the event that extended cables are required, seek the advice of the manufacturer and, if possible, purchase the cables from the manufacturer. In spite of this potential problem, EIS measurements have been successfully performed on an aircraft in an operating hangar with 80-ft (25 m) cables on aircraft coatings with impedances as high as 10^{11} ohms.

FUTURE DIRECTIONS

EIS is a unique measurement tool in that it provides a quantitative test on the complete coating system (metal substrate and coating). We expect that the steady proliferation of EIS into the coat-

ings community will lead to a greater understanding of the breadth of applications to various coatings. Within general classes of coatings, it is likely that failure mechanisms will be classified and routinized.

The need for more rapid results will drive the continued development of accelerated tests. To incorporate delamination into testing procedures, the combination of EIS and adhesion testing will probably be exploited. As EIS gains acceptability by coatings researchers, there will be a need for multichannel instruments for greater sample throughput at lower cost. If cabinet tests remain popular, there will be a demand for automated EIS measurements during the exposure period.

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