

Evaluation of Organic Coatings with Electrochemical Impedance Spectroscopy

Part 3: Protocols for Testing Coatings with EIS

David Loveday, Pete Peterson, and Bob Rodgers—Gamry Instruments*

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-

* 734, Louis Dr., Warminster, PA 18974; Voice: 215-682-9330; Fax: 215-682-9331; Email: brodgers@gamry.com.

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