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Coating degradation monitoring through combined acoustic emission and electrochemical impedance spectroscopy measurements

Romain HABİYAREMYE^{1*}, Yelitza DELGADO-GONZALEZ¹, Axel HOMBORG^{1,2},
Harleigh SEYFFERT¹, Arjan MOL¹, Lotfollah PAHLAVAN¹
¹ Delft University of technology, Delft, The Netherlands
² Netherlands Defense Academy, Den Helder, The Netherlands

Abstract. Corrosion is a leading damage mechanism in the degradation of marine assets. Organic barrier coatings are widely used as a corrosion mitigation measure, because their application suppresses the interaction between the metallic structure and the corrosive environment. Electrochemical impedance spectroscopy (EIS) is a well-established technique for the evaluation of coating performance. Acoustic emission (AE) monitoring has gained increasing interest as a technique for continuous monitoring of corrosion damage. In this experimental study, EIS measurements are combined with AE monitoring to investigate the degradation of different organic barrier coatings. Laboratory experiments were performed on 7 aluminum specimens covered with different types of coatings. Water uptake was achieved by immersing the samples in a salt solution for 24 hours prior to each test. Each sample was equipped with an electrochemical cell (Ag/AgCl reference electrode, platinum mesh counter electrode, acidic NaCl solution; pH=2) and an AE sensor (85-180kHz). The acidic salt solution was used to accelerate the degradation of the coatings for a period of 24 hours during which periodic EIS measurements were performed as well as continuous AE monitoring. AE signals detected during the test could be associated with a measured reduction in coating resistance and, in some cases, visible signs of surface degradation. The preliminary results from the study indicate that coating degradation generates AE signals in the same frequency range as that of typical AE from corrosion degradation. This is a promising perspective towards expanding the capabilities of AE based structural health monitoring systems.

Keywords: Acoustic emission; Electrochemical impedance spectroscopy; Coating degradation; Data fusion

Introduction

Shipborne aircraft are particularly prone to corrosion damage due to the nature of the environment where they are deployed. The combination of moisture and aggressive chlorine ions provides the ideal conditions for the development of corrosion. It is therefore not surprising that corrosion is one of the main driving degradation mechanisms in naval aircraft [1]. It is common to apply coatings to protect the structure from this corrosive environment. Among the wide range of types of coatings available on the market, organic polymer coatings are, by far, the most widely used in corrosion protection [2]. However, these coatings are also prone to degradation and ultimately, failure due to wear, aging, mechanical stress and environmental conditions. Maintenance directives require coatings to be visually inspected, based on qualitative metrics, during mandated surveys [3, 4]. These inspection campaigns are labor-intensive and lengthy, which keeps assets out of service for extended time periods. The combination of these factors makes inspections expensive endeavors. There is, for these reasons, a growing interest in structural health monitoring and predictive maintenance.

*Corresponding author Email: R.Habiyaremye@tudelft.nl



Electrochemical impedance spectroscopy (EIS) is a well-established non-destructive testing technique that can provide information about the properties of an electrochemical system in a steady state. EIS has a very wide range of applications, such as diagnostics and prognostics for batteries [5], bio-sensing [6] [7], and the non-destructive testing of coatings [8] [9] [10]. EIS works by applying a sinusoidal AC potential or current, over a range of frequencies, to an electrochemical system, and measuring the response of the system in terms of current or potential, respectively [10]. To obtain a more complete picture, typical EIS measurements test the impedance over a range of frequencies. The frequency-dependent response of the system can then be fitted to an equivalent electrical circuit consisting of resistances and constant phase elements (CPE) that best models the response of the electrochemical system. Using this model, the capacitance and resistance of the coating can be inferred and used as indicators of the state of the coating. EIS measurements can provide accurate and reliable data on the properties of a coating, however, in the context of structural health monitoring, they present two limitations. Firstly, measurements require a sweep through a range of frequencies, under the assumption that the system is in a steady state. This means that individual measurements are lengthy, which limits the technique to intermittent sampling instead of true real time monitoring. Secondly, the technique can only evaluate the coating at the location of the measurement; any coating defect outside of the electrochemical cell cannot be directly detected.

Acoustic emission (AE) monitoring is a structural health monitoring technique that is capable of detecting a multitude of different damage mechanisms such as corrosion, and wear of coatings, [11, 12, 13, 14, 15, 16, 17]. The working principle of AE monitoring consists in the measurement of elastic stress waves caused by a rapid release of energy when irreversible changes occur in a material [18]. In thin-walled structures, these stress waves propagate as guided waves which allows them to be detected and localized at considerable distance by piezoelectric sensors. Additionally, AE monitoring allows for real time monitoring of damage. These aspects suggest that AE and EIS could be used to complement one another to obtain more accurate information on degradation.

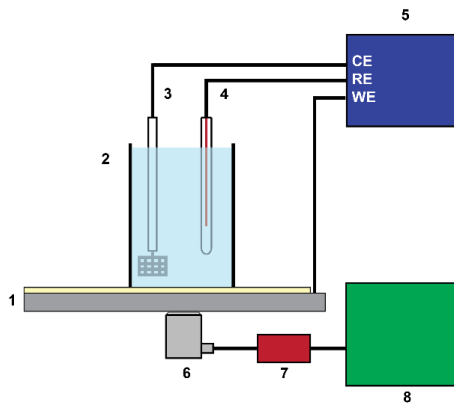
The aim of this study is to establish a laboratory technique that couples AE and EIS in order to obtain real time information on the degradation of barrier coatings. The combination of accurate intermittent readings of coating impedance and localizability and real-time monitoring of early onset of coating degradation and corrosion allow for a more holistic approach to coated metal degradation. The present work experimentally investigates the Acoustic emission behavior of the degradation of organic aerospace coatings. Accelerated degradation experiments were performed on 7 sample coatings. Degradation was accelerated by exposure to an acidic electrolyte solution. The degradation of the coatings was monitored through Acoustic emission measurements, periodic EIS measurements and open circuit potential measurement.

1. Methodology

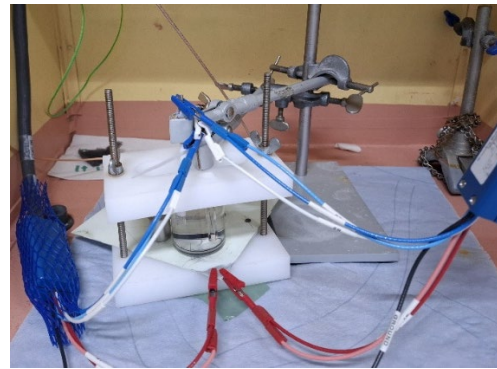
The degradation of 7 different coating types applied to aluminum panels was investigated. The specifications and thickness of the coatings are displayed in Table 1. Prior to each test, the sample panels were submerged in a neutral 3.5 wt.% solution of NaCl for 24 hours to allow the electrolyte to permeate and saturate the coating. A surface mounted corrosion cell was clamped to the coated surface of the panel. The experimental setup was instrumented for AE and electrochemical measurements and placed in a Faraday cage as shown in **Figure 1**.

Table 1: panel coatings and thicknesses

Panels	Thickness (μm)	Pre-treatment	Primer	Topcoat
P1	95.9 ± 2.9	Metaflex SP1050	Aerodur 2100 MgRP	Aerodur 5000
P2	82.8 ± 3.1	Metaflex SP1050	Aerodur HS2118	Aerodur 5000
P3	86.6 ± 4.9	7530 CF	7065 CF PU	CA8800
P4	73.4 ± 3.2	None	CA7521 CF EP	CA8311
P5	96.2 ± 5.4	Metaflex SP1050	PT17701 bare Al-rich	Aerodur 5000
P6	121.0 ± 10.9	Metaflex SP1050	PT23704 CF AL-rich	Aerodur 5000
P7	40.3 ± 4.5	Alodine 1200	Seevenax 113-24	Alexit 472-22



(a) schematic of experimental setup; 1: coated panel (Working electrode), 2: corrosion cell, 3: Platinum counter electrode, 4: silver chloride reference electrode, 5: Potentiostat, 6: AE sensor, 7: Preamplifier, 8: AE data acquisition system



(b) Laboratory set up

Figure 1: Illustration of the experimental setup used

The corrosion cell was then filled with an acidic 3.5 wt.% NaCl solution with a pH of 2, achieved by the addition of HCl. The circular surface of the coating exposed to the acidic solution amounted to 7.5 cm². The coated samples were exposed to the acidic solution for 24 hours before being rinsed with distilled water and examined again via optical microscopy.

The EIS was performed using a BioLogic VSP-300 multichannel potentiostat with a three-electrode configuration. A small area of the coating was removed to ensure electrical contact with the panel serving as working electrode. The reference was provided by an Ag/AgCl electrode (3 M KCL), and a platinum mesh counter electrode was used. During the 24-hour test period, EIS measurements were performed every 4 hours. The EIS measurements were performed with a sinusoidal signal with a 100mV amplitude over a frequency range of 10⁻² Hz to 4×10⁵ Hz with 10 points per decade.

The acoustic emission behavior of the degradation of the specimen was measured with a single R15α 150 kHz resonant sensor manufactured by Physical Acoustics. This sensor was selected for its high sensitivity over the entire frequency range of interest. Front-end band pass filters were applied to input channels to provide frequency coverage between 85 kHz and 200 kHz. The 150kHz sensor was placed directly under the exposed area as shown in **Figure 1**. A silicon-based coupling gel was used to facilitate signal transmission to the sensors held in place by the cell clamp. The sensor output was amplified through a AEP5 preamplifier (Vallen Systeme) set to a gain of +40 dB. In this configuration, the 3 dB

bandwidth of the preamplifier extends from 2.5 kHz to 2.4 MHz. The measurements were recorded using an AMSY-6 digital multi-channel measurement system (Vallen Systeme).

2. Results

Electrochemical impedance spectroscopy

The periodic EIS measurements show clear variation over the exposure time for the majority of the panels. The sample coatings exhibited very different behaviors, as was expected because of the different elements and materials that were used for the preparation of the coatings.

The EIS data was used to fit Equivalent Electrical Circuits (EEC) in order to characterize the properties and behavior of the coatings. The EEC was fitted using Zview based on an assumed damaged coating model [19] as shown in **Figure 2**.

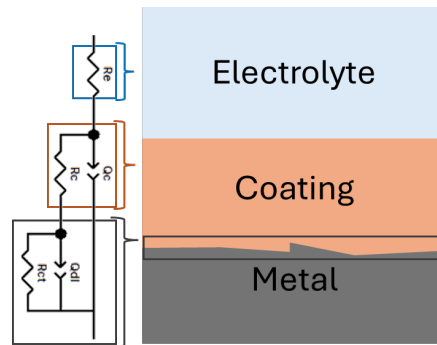


Figure 2: EEC used to model the electrochemical behavior of the degrading coating. R_e : Electrolyte resistance, Q_c : coating capacitance, R_c : Coating resistance, Q_{dl} : double-layer capacitance, R_{ct} : Charge transfer resistance

The degradation of the coating is estimated based on the reduction in the coating resistance (R_c), which is obtained by the fittings of EEC. Over time, the coating resistance is expected to decrease because the coating degrades and porosity increases. The electrolyte permeates in the pores and new defects and therefore more conductive channels are formed, reaching the metal substrate and eventually leading to the onset of corrosion.

Evolution of coating resistance degradation and effective coating capacitance are shown in Figure 3. Values of both graphs are normalized with respect to the value obtained at time zero for each panel. The initial values of the coating properties are shown in Table 1. resistor.

Table 1: Initial values of coating properties (0 hours) based on the fit EEC shown in Figure 2

sample	P1	P2	P3	P4	P5	P6	P7
Resistance ($\Omega \text{ cm}^2$)	1,20E+09	7,21E+08	1,08E+08	4,81E+08	1,10E+09	1,34E+09	1,30E+10
Capacitance (F cm^{-2})	3,01E-11	2,41E-12	1,23E-10	1,11E-11	1,10E-11	5,35E-12	3,85E-12

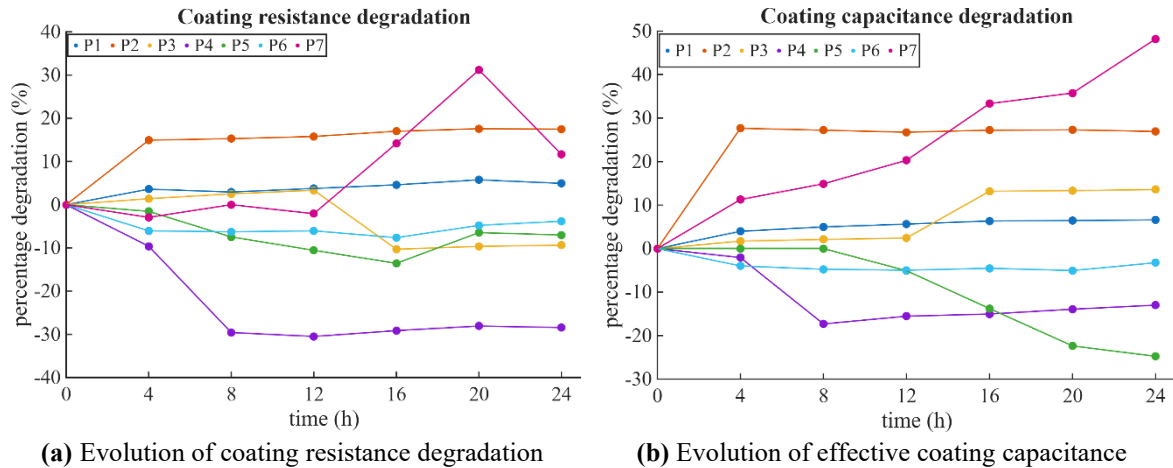


Figure 3: Evolution of coating properties over time

The coating resistance and capacitance for the 7 panels exhibit an initial large change in the first 4-8 hours before stabilizing and remaining relatively stable over the remaining time of testing. Nevertheless, some samples (e.g. panel 4), showed a decline in degradation, meaning that the resistance increased rather than decreasing as expected. This behavior could reflect the formation of subproducts, which blocked the pores and cracks that may have formed. Meanwhile, an increment in the coating resistance degradation is observed for panel 1. These differences in behavior exhibit how relevant the composition of the coating is.

Nevertheless, it is shown that almost all panels have undergone significant change with respect to the initial state during the first 4 hours, which could be attributed to the fast electrolyte uptake, however all the panels were submerged previously to saturate the coatings for 24 hours. The solution used for this initial saturation did not contain the hydrochloric acid used for the accelerated degradation process. Longer experiments to test this acid addition and its interaction with the coatings for resistance evaluation are recommended for further research.

Furthermore, the increase of coating resistance observed in some panels could denote the constriction of conductive pathways inside the coating [21, 22]. This behavior was not expected; a decrease should appear because of the saturation of the coating. Additionally, some discontinuities can be observed for some samples, caused by range switching in the potentiostat. A clear example of this behavior was observed for panel 3. Consequently, these correspond to an equipment measurement error, which was often observed for these long-term tests.

Overall, no corrosion was observed in any panel that showed a high coating resistance. Corrosion was observed if the coating resistance was less than $1 \cdot 10^4$, if the value is $1 \cdot 10^4 < R_c < 1 \cdot 10^6$ then the coating was moderate, if $1 \cdot 10^6 < R_c < 1 \cdot 10^8$ the coating was good and if $R_c > 1 \cdot 10^8$ the coating had excellent properties. Therefore, based on these considerations, it can be said that all panels have excellent coating property at the start of the test. Exceptionally, panel 7 showed relatively high coating resistance, with a value of $1.3 \cdot 10^{10} \Omega \text{ cm}^2$ at time zero and $1.1 \cdot 10^{10} \Omega \text{ cm}^2$ after 24 hours of test. Where panel 7 showed only limited decrease in coating resistance, the effective capacitance exhibited much more significant and sustained increase.

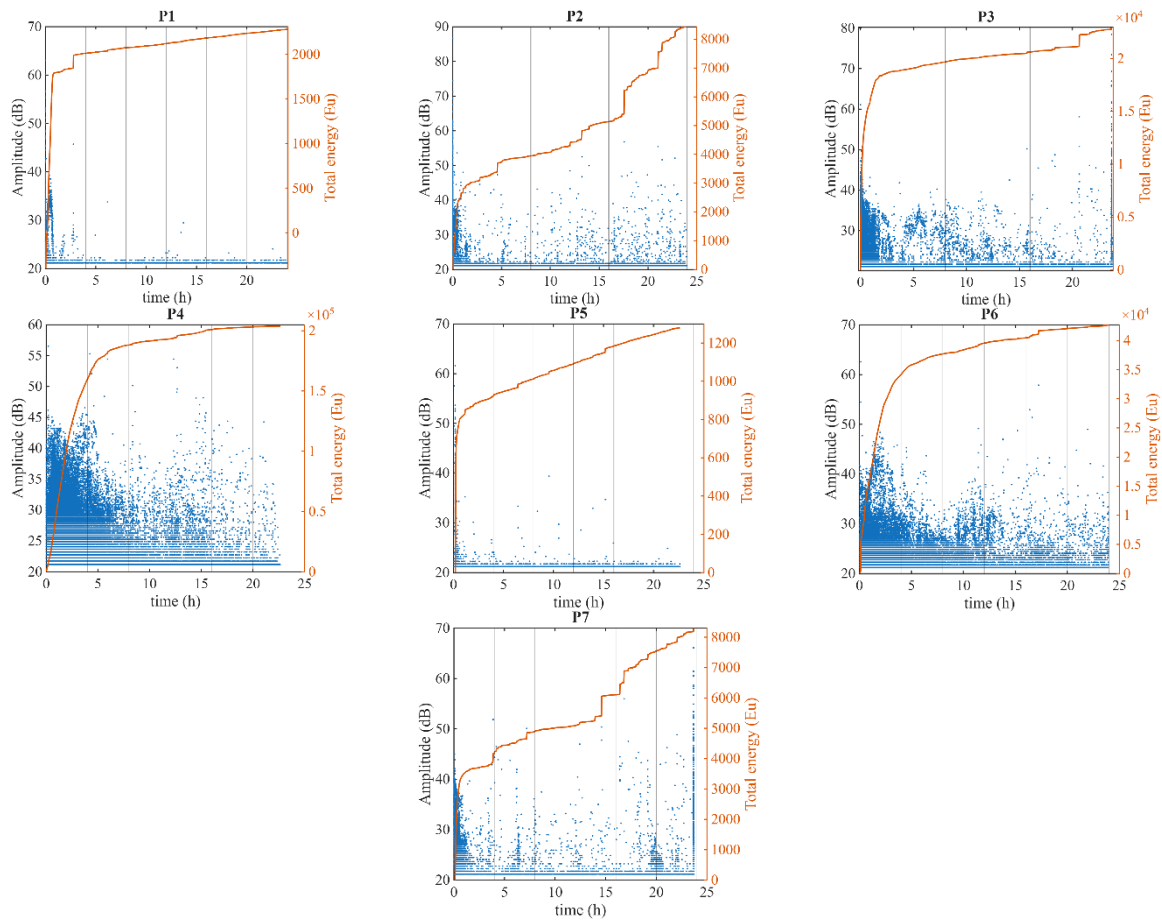


Figure 4: AE signal amplitude distribution over time along with the cumulative AE energy

Acoustic emission

The amount of acoustic emission signals captured during the test period varied widely between different coatings. Based on the initial observations, the EIS measurements seem to have no influence on the AE measurements. No significant change in AE behavior is observed during or after EIS measurements as shown in **Figure 4**. All samples exhibit an increased AE activity at the very beginning of the experiments. This behavior is also reflected in the cumulative energy curves. The number of measured signals decreases most significantly during the first four hours of the exposure time. This behavior is similar to the behavior observed based on the EIS measurements where the highest activity occurred in the first hours of the test. If we assume that the energy released in the form of AE signals comes from the physical and chemical processes that govern the degradation of the coating, one could reasonably assume there would be a correlation between the AE behavior and the degradation of a given material. The acoustic emission hit rate (signals detected per minute) along with the evolution of the coating resistance (R_c) are shown in **Figure 5**. The periods of high AE activity correlate well with the periods where the coating resistance changes are significant for panels 1, 2, 4, and 6. The transient behavior of the coating properties, as they are reflected by the rapid change in resistance at the beginning of the test aligns with the high AE hit rate at the start of the measurement. For all aforementioned panels, more than 50% of the total AE energy is emitted within the first 4 hours of the experiment.

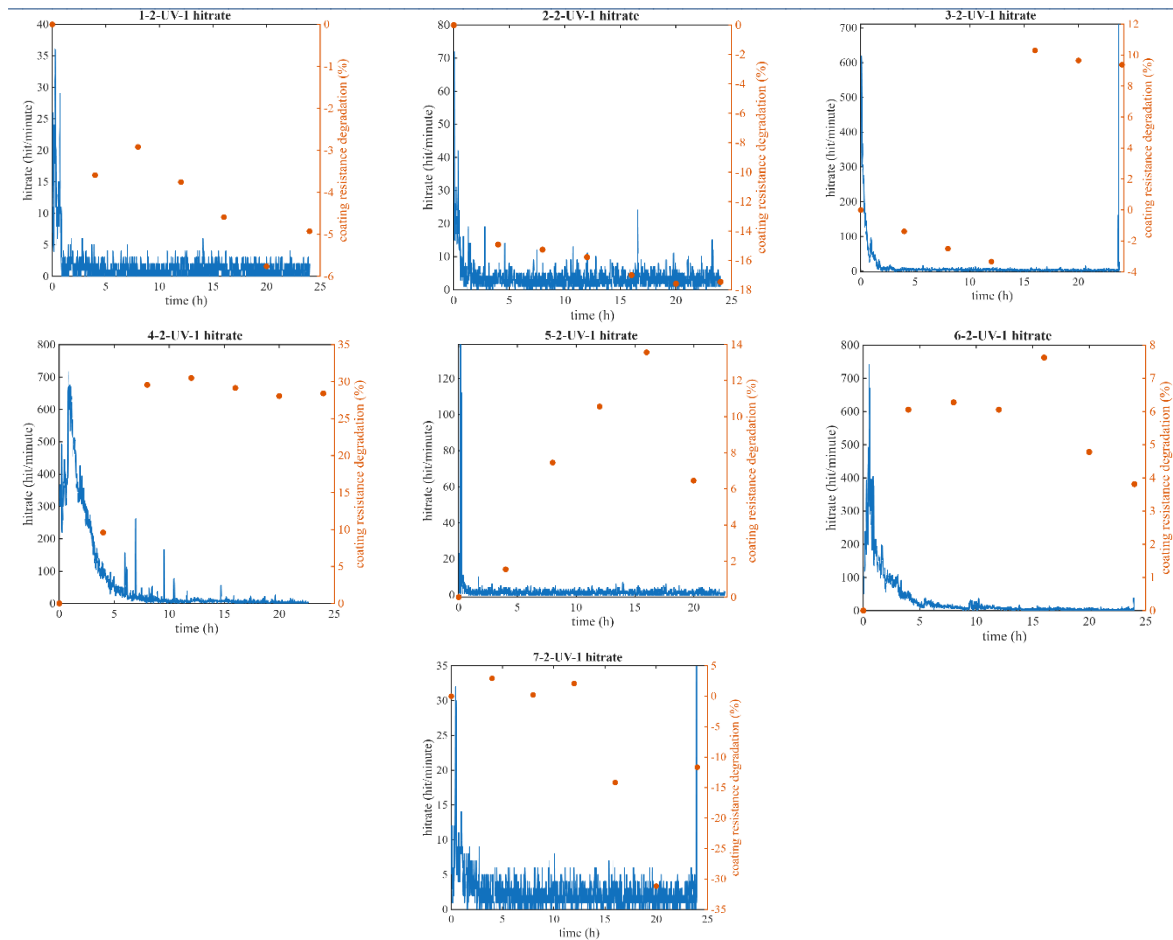


Figure 5: evolution of coating resistance over time along with the AE hit rate

The remaining samples exhibit similar decaying AE activity trends; however, this activity could not be directly correlated with the evolution of the coating properties. The parallel between the degradation of the coating and the AE activity suggests that there is a possibility to estimate the rate of degradation of the coating which could serve to interpolate the behavior of the coating between periodic EIS measurements

3. Discussion

Regarding the unexpected electrochemical behavior of some coatings, two main explanations are suspected. The increase in coating resistance (negative degradation) was attributed to the reaction between the coating and the acidic electrolyte, the products of this reaction, subproducts, may have obstructed the pores in the coating, thereby restricting contact between the electrolyte and the substate and hence increasing resistance, due to this, the corrosion was inhibited due to this phenomenon. The non-monotonic behavior of coating properties over time is believed to be caused by measurement and EEC fitting errors.

Although some of the results obtained by EEC fittings were not expected, it demonstrates the high relevance of the composition of the coatings. It would be recommended to perform multiple repetition of the test to average the results and reduce the bias associated with electrode placement, measurements errors and local variability of coating properties. This

would help to highlight the trends of coating property evolution and verify the origin of unexpected results.

The combination of AE and EIS as it is presented in this study could be integrated into an SHM strategy involving real time monitoring and periodic inspections as illustrated in **Figure 6**. Acoustic emission can be used to detect and localize the initiation of coating degradation after which targeted EIS measurements can be performed to evaluate the integrity of the coating locally. At risk and/or damaged areas may then be monitored to track unusual AE activity related to further degradation.

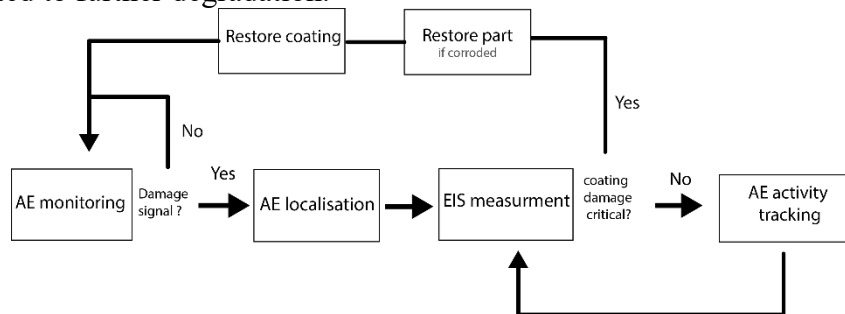


Figure 6: SHM strategy combining periodic targeted EIS measurements with continuous AE monitoring

4. Conclusions and future work

The acoustic behavior of the degradation of several organic coatings was experimentally investigated. A laboratory technique that couples AE and EIS in order to obtain real time information on the degradation of barrier coatings was presented. Coated aluminum samples were tested under accelerated degradation conditions by exposure to an acidic electrolyte solution. Correlations could be found between the Acoustic emission activity in the form of AE hit rate and the evolution of the properties of the coating resistance data obtained by the fitting of EEC.

In future work, it would be recommended to investigate coated samples at different degradation rates by varying the acidity of the electrolyte. Additionally, applying these techniques over prolonged exposure times would make it possible to capture the different stages of degradation of coating metal structures, from coating degradation to onset of corrosion of the substrate. Furthermore, the comparison of the results obtained using a two-electrode system and gel electrolyte for EIS is advised taking into consideration on-site measurement since classical EIS is not easily adaptable to self-contained sensor technology for SHM applications.

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