

Experimental Validation of a Micro-Sized Polarization Resistance Corrosion Sensor for Structural Health Management Applications

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ABSTRACT

This paper presents the theory and experimental validation of Analatom's Structural Health Management (SHM) system for monitoring corrosion. Corrosion measurements are acquired using a micro-sized Linear Polarization Resistance (μ LPR) sensor. The μ LPR sensor is based on conventional macro-sized Linear Polarization Resistance (LPR) sensors with the additional benefit of a reduced form factor making it a viable and economical candidate for remote corrosion monitoring of high value structures, such as buildings, bridges, or aircraft.

A series of experiments were conducted to validate the μ LPR sensor for AA 7075-T3. Test coupons were placed alongside Analatom's μ LPR sensors in a series of accelerated tests. LPR measurements were sampled at a rate of once per minute and converted to a corrosion rate using Analatom's SHM system. At the end of the experiment, pit-depth due to corrosion was computed for each sensor from the recorded LPR measurements and compared to the average pit-depth measured on the control coupons. The results demonstrate the effectiveness of the sensor as an efficient means to measure pit-depth for AA 7075-T3.

INTRODUCTION

Recent studies have exposed the generally poor state of our nation's critical infrastructure systems that has resulted from wear and tear under excessive operational loads and environmental conditions. SHM Systems aimed at reducing the cost of maintaining high value structures by moving from SBM (Scheduled Based

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Maintenance) to CBM (Condition Based Maintenance) schemes are being developed. These systems must be low-cost, simple to install with a user interface designed to be easy to operate. To reduce the cost and complexity of such a system a generic interface node that uses low-powered wireless communications has been developed by Analatom. This node can communicate with a myriad of common sensors used in SHM. In this manner a structure such as a bridge, aircraft or ship can be fitted with sensors in any desired location without the need for communications and power lines that are inherently expensive and complex to route. Data from these nodes is transmitted to a central communications Personal Computer (PC) for data analysis. An example of this is provided in Figure 1 showing Analatom's AN101 SHM system installed in the rear fuel-bay bulkhead of a commercial aircraft.

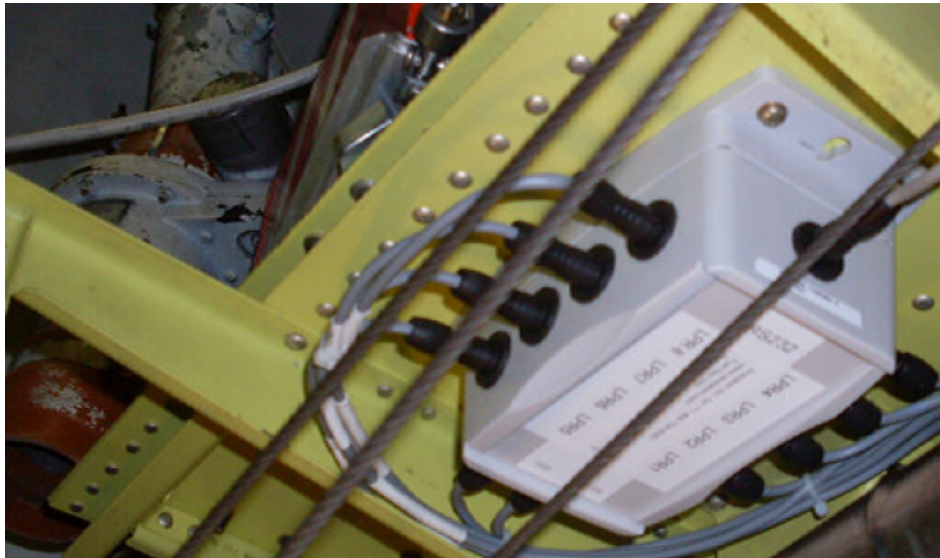


Figure 1. Analatom AN101 SHM system installed in the rear fuel-bay bulkhead of a commercial aircraft.

A variety of methods such as electrical resistance, gravimetric-based mass loss, quartz crystal micro-balance-based mass loss, electrochemical, and solution analysis methods enable the determination of corrosion rates of metals. The focus of this paper is on, Linear Polarization Resistance (LPR), a method based on electrochemical concepts to determine instantaneous interfacial reaction rates such as corrosion rates and exchange current densities from a single experiment. There are a variety of methods capable of experimentally determining instantaneous polarization resistances such as potential step or sweep, current step or sweep, impedance spectroscopy, as well as statistical and spectral noise methods [1]. This paper will focus on the former as Analatom's SHM system uses the potential step (or sweep) approach to measure LPR. This is followed with a description of an experimental setup and procedure used to validate the μ LPR sensor. The paper concludes with a summary of the findings.

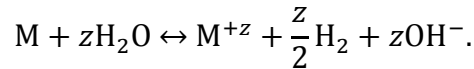
LINEAR POLIARIZATION RESISTANCE THEORY

Corrosion usually proceeds through a combination of electrochemical reactions; (1) anodic (oxidation) reactions involving dissolution of metals in the electrolyte and release of electrons, and (2) cathodic (reduction) reactions involving gain of electrons by the electrolyte species like atmospheric oxygen O_2 , moisture H_2O , or H^+ ions in an

acid. The flow of electrons from the anodic reaction sites to the cathodic reaction sites constitutes corrosion current and is used to estimate the corrosion rate. When the two reactions are in equilibrium at the equilibrium corrosion potential, E_{corr} , the net current on the metal surface is zero without an external source of current. The anodic reactions proceed more rapidly at positive potentials and the cathodic reactions proceed more rapidly at negative potentials. Since the corrosion current from the unstable anodic and cathodic sites is too small to measure, an external activation potential, E_a , is applied across the metal surface and the response current, I_a , is measured for electrochemical calculations.

Anodic and Cathodic Reactions

Corrosion reactions are electrochemical reactions occurring on the metal surface. Modern corrosion studies are based on the concept of mixed potential theory postulated by Wagner and Traud, which states that the net corrosion reaction is the result of two or more partial electrochemical reactions that proceed independently of each other [2]. For the case of metallic corrosion in presence of an aqueous medium, where z is the number of electrons lost per atom of the metal, the corrosion process can be written as,



It is assumed that the anodic and cathodic reactions occur at a number of sites on a metal surface and that these sites change in a dynamic statistical distribution with respect to location and time. Thus, during corrosion of a metal surface, metal ions are formed at anodic sites with the loss of electrons and these electrons are then consumed by water molecules to form hydrogen molecules. The interaction between the anodic and cathodic sites as described on the basis of mixed potential theory is represented by well-known relationships using current (reaction rate) and potential (driving force). For the above pair of electrochemical reactions, the relationship between the applied current I_a and potential E_a follows the Butler-Volmer equation,

$$I_a = I_{corr} \left[\exp\left(\frac{2.303(E_a - E_{corr})}{\beta_a}\right) - \exp\left(-\frac{2.303(E_a - E_{corr})}{\beta_c}\right) \right].$$

where β_a and β_c are the anodic and cathodic Tafel parameters given by the slopes of the polarization curves $\partial E_a / \partial \log_{10} I_a$ in the anodic and cathodic Tafel regimes, respectively [3].

Linear Polarization Resistance (LPR)

The corrosion current, I_{corr} , cannot be measured directly. However, a-priori knowledge of β_a and β_c along with a small signal analysis technique, or LPR, can be used to compute I_{corr} . By applying the Butler-Volmer equation, the polarization resistance is defined as,

$$R_p \triangleq \left. \frac{\partial E_a}{\partial I_a} \right|_{|E_a - E_{corr}| \leq 10\text{mV}}.$$

By applying this definition to the Butler-Volmer equation and solving for I_{corr} produces the Stern-Geary equation,

$$I_{corr} = \frac{B}{R_p}, \quad \text{where } B = \frac{1}{2.303} \left(\frac{\beta_a \cdot \beta_c}{\beta_a + \beta_c} \right).$$

LPR Measurements

ASTM standards D2776 and G59 describe standard procedures for conducting polarization resistance measurements. Potentiodynamic, potential step, and current-step methods have all been described to determine the linear behavior of an electrode near E_{corr} [4,5]. LPR determined by potentiodynamic sweep is the most common method for acquiring R_p . For conventional macro-LPR measurements, a potentiodynamic sweep is conducted by applying E_a between $E_{corr} \pm 10$ [mV] at a slow scan rate, typically 0.125 [mV/s]. A linear fit of the resulting E_a vs. I_a curve is used to compute R_p . Performing this operation takes 160 seconds to complete.

Pit Depth

The rate of pit-depth growth due to corrosion is calculated by computing the pitting current density, i_{pit} ,

$$i_{pit}(t) = \frac{i_{corr} - i_{passive}}{N_{pit}}$$

where $i_{corr} = I_{corr}/A_{eff}$ is the corrosion current density, $i_{passive}$ is the passive current density N_{pit} is the pit density for the alloy (derived empirically) and A_{eff} is the effective surface area of the sensor. One critical assumption is the pH is in the range of 6-8. If this cannot be assumed, then a measurement of pH is required and $i_{passive}$ is needed over the range of pH values. Finally, by applying Faraday's Law to relate the charge flow to mass loss and assuming pits with a semi-circle geometry, then the computed pit depth is,

$$d(t) = 10 \left[\left(\frac{3EW}{4\pi\rho F} \right) \int_0^t i_{pit}(\tau) d\tau \right]^{1/3},$$

where $EW = 9.886$ [g/mol] is the equivalent weight for AA 7075-T3, $\rho = 2.810$ [g/cm³] is the density for AA 7075-T35, $F = 9.6485 \times 10^4$ [C/mol] is Faraday's Constant and d is the pit depth in [mm]. In practice R_p is not measured continuously, rather, periodic measurements are taken every T_s seconds. If it is assumed over this interval the R_p values change linearly then the mean value theorem for integrals can be applied to arrive at an alternative expression for d ,

$$d(t) = 10 \left[T_s \left(\frac{3EW}{4\pi\rho F} \right) \sum_{k=0}^{N-1} i_{pit}(kT_s) \right]^{1/3},$$

where N is the total number of samples.

μLPR CORROSION SENSOR

In this section, a μLPR is presented which uses the potential step-sweep method to compute polarization resistance. The μLPR works on the same principle as the macro LPR sensors and is designed to corrode at the same rate as the structure on which it is

placed. Although LPR theory is well established and accepted as a viable corrosion monitoring technique, conventional macro-sized LPR sensor systems are expensive and highly intrusive. The μ LPR is a micro-scaled LPR sensor inspired from the macro-sized version discussed in the previous section. Scaling the LPR sensor into a micro-sized package provides several advantages which include: a miniature form factor, two-pair electrode configuration and lower solution resistance.

Expertise in semiconductor manufacturing is used to micro-machine the μ LPR. Using photolithography it is possible to manufacture the μ LPR sensor from a variety of standard engineering construction materials varying from steels for buildings and bridges through to novel alloys for airframes. The micro sensor is made up of two micro machined electrodes that are interdigitated at 150 [μ m] spacing. The μ LPR sensor is made from shim stock of the source/sample material that is pressure and thermally bonded to Kapton tape. The shim is prepared using photolithographic techniques and Electro Chemical Etching (ECM). It is further machined on the Kapton to produce a highly ductile and mechanically robust micro sensor that is very sensitive to corrosion. Examples of the sensor and a fitted sensor underneath a coating are shown in *Figure 2*.

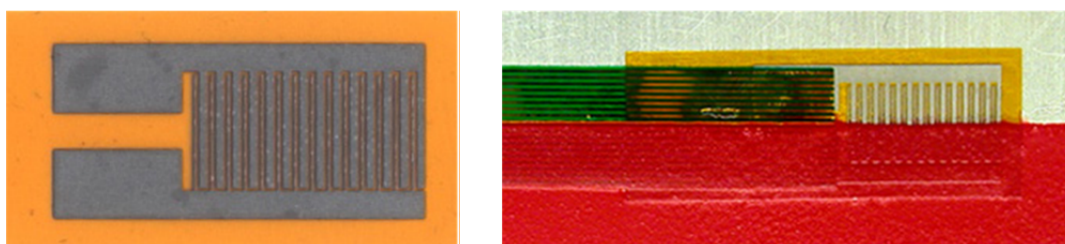


Figure 2. Thin μ LPR exposed (left) and underneath a coating (right).

EXPERIMENT

Equipment / Setup

The experiment consisted of twenty-four (24) μ LPR sensors and three (3) control coupons. The coupons and μ LPR sensors were made from AA 7075-T3. Each coupon was placed next to a set of μ LPR sensors. Each sensor was held in place using a non-reactive polycarbonate clamp with a nylon fitting. All the sensors and coupons were mounted on an acrylic plexiglass base with the embedded hardware placed on the opposite side of the frame. An electronic precision balance (Tree HRB-203) with a calibrated range of 0 – 200 [g] (± 0.001 [g]) was used to weigh the coupons before and after the experiment. Finally, a weathering chamber (Q-Lab QUV/spray) promoted corrosion on the coupons and μ LPR sensors by applying a controlled stream of tap water for 10 seconds every five minutes.

Procedure

First, the surface of each coupon was cleaned using sandblasting. Then, each coupon was weighed using the analytical balance. The entire panel of coupons and μ LPR sensors were placed in the weathering chamber for accelerated testing. The experiment ran for approximately 17 days. During the experiment, Analatom's

embedded hardware was logging the polarization resistance from each μ LPR sensor. The sample rate was set at one sample per minute. Once accelerated testing was finished, the coupons were removed and the μ LPR data was downloaded and archived for analysis. The corrosion byproducts were removed from each coupon by applying micro-bead blasting to the coupon surface. Finally, the cleaned coupons were weighted using the analytical scale to compute the relative corrosion depth during the experiment.

RESULTS

Coupon Corrosion

The corrosion byproducts were carefully removed using micro-bead blasting. The pitting depth, d , of each coupon was calculated for $N_{pit} = 15$,

$$d = 10 \left(\frac{3\Delta m}{4\pi\rho N_{pit}SA} \right)^{\frac{1}{3}}$$

where values for the mass loss Δm , exposed surface area SA and resulting pit depth, d , of each coupon is provided in Table 1.

Table 1. Experimental measurements of coupon corrosion.

Coupon	Area [cm ²]	Init. Mass [g]	Final Mass [g]	Δm [g]	d [mm]
1	58.05	77.253	77.215	0.038	0.1548
2	57.99	76.927	76.896	0.031	0.1446
3	58.05	76.897	76.869	0.028	0.1398

μ LPR Corrosion

The linear polarization resistance measurements were used to compute corrosion pit depth for each μ LPR sensor. The average pit depth computed for each sensor over time was compared to the measured pit-depth of the coupons as shown in *Figure 3*.

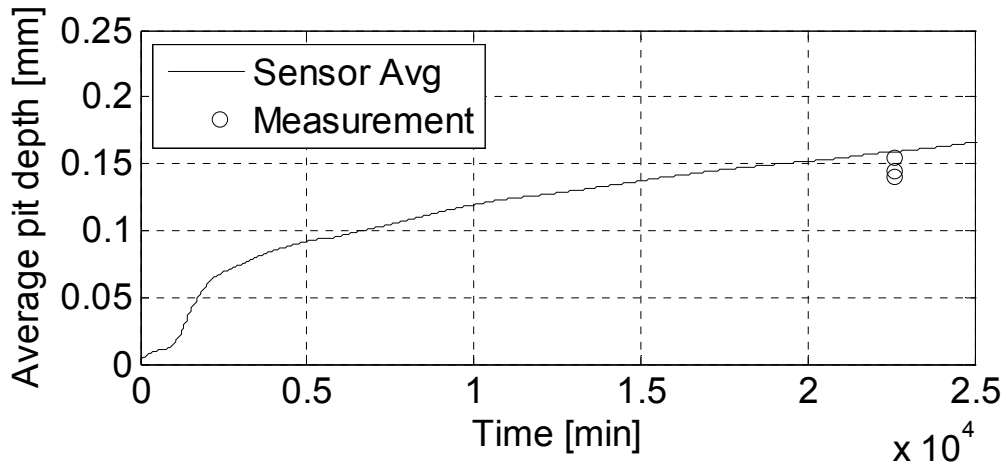


Figure 3. Comparison of the average pit depth from the LPR sensors and the measured pit-depth from the coupons.

A histogram of the computed pit depth for the LPR sensors 22,638 minutes after the start of the test (total coupon exposure time) is provided in *Figure 4*. The mean and standard deviation computed from the distribution are $\bar{X} = 0.1590$ [mm] and $s = 0.0221$ [mm], respectively. Assuming a normal distribution, the 95% confidence interval for the computed pit depth from the LPR sensors is (0.1157, 0.2023) [mm]. This agrees with the measured coupon pit-depth to within the statistical uncertainty.

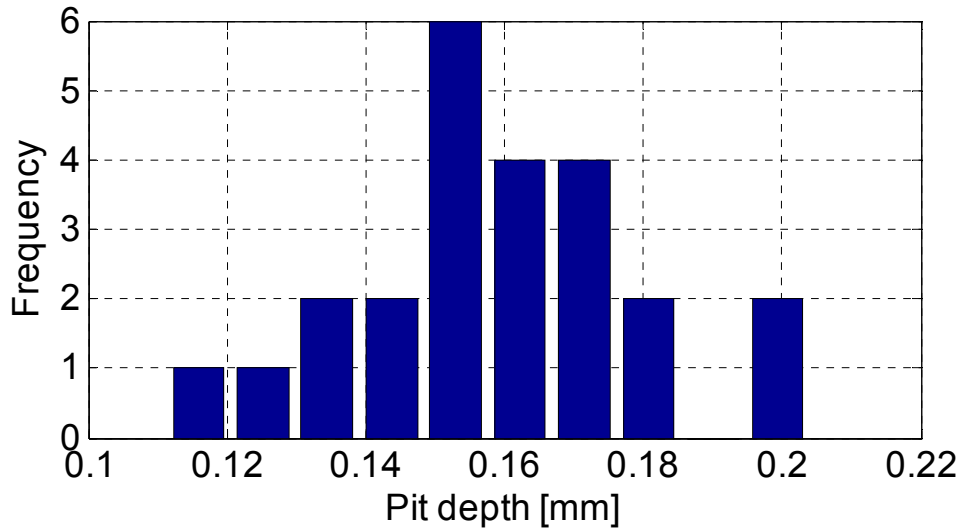


Figure 4. Histogram of the pit-depth computed from μ LPR sensor measurements.

SUMMARY

A micro-sized LPR (μ LPR) sensor was presented for corrosion monitoring in Structural Health Management (SHM) applications. An experimental test was performed to compare corrosion measurements from twenty-four μ LPR sensors with three coupons. Both the coupons and sensors were constructed from the same material, AA 7075-T3. According to the results, the pit-depth measured on the coupons fell within the 95% confidence interval computed from the pit-depth measured on the μ LPR sensors. The results indicate multiple μ LPR can be used to provide an accurate measurement of corrosion.

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