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Coating and interface degradation of coated steel, Part 1: Field exposure

S.M. Cambier[a,*], R. Posner[a,b], G.S. Frankel[a]

[a] Fontana Corrosion Center, The Ohio State University, 143 Fontana Labs 116 W. 19th Ave., Columbus, OH 43210, USA
[b] Present address: Henkel AG & Co. KGaA, Henkelstrasse 67, 40589 Düsseldorf, Germany

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ABSTRACT

The cathodic delamination (CD) rate of organic coatings on steel can be easily measured with a Scanning Kelvin Probe (SKP). In this paper, the measurement of CD by SKP (CD-SKP) was used for the first time to assess the interface stability after field exposure of polymer-coated steel. The uncoated region used as defect to initiate the CD in the humid SKP chamber was covered during field exposure. The CD rates of the field-exposed samples were compared to unexposed samples to evaluate the interface degradation. The CD-SKP assessment was related to the visual inspection of corrosion products through the transparent coating and electrochemical impedance spectroscopy (EIS) results. The CD rate measured after field exposure corresponded to the site severity that was estimated by the visual inspection. The EIS results were not always reproducible owing to the small exposed area, but overall the coating protection measurements were in accordance with the CD-SKP results. However, CD-SKP was more sensitive and faster than EIS. Furthermore, CD-SKP measurements exhibited clear evidence of improvement of interface stability during the initial stages of field exposure, which was followed by degradation of the interface. The CD-SKP technique can be used in practice to shorten the field exposure time of coated steel and provide early detection of the onset of interface degradation.

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1. Introduction

Coating lifetimes are best predicted by field exposure, but degradation of protective coatings can take several years even in aggressive field environments. Sensitive techniques have been used to measure the early stages of coating degradation [1] and interface failure [2] after short exposure time. Powerful tools such as Fourier transform infra-red (FT-IR) spectroscopy [1] and nuclear magnetic resonance [3] are available to assess the coating weathering degradation or the coating additive loss but cannot predict the mechanical integrity failure of coatings [4]. The onset of substrate corrosion is best predicted by the assessment of the coating/metal interface degradation. Electrochemical impedance spectroscopy (EIS) is commonly used to detect the coating water permeation or coating delamination [5], and has been applied after field exposure [2]. EIS can be used in bulk solutions or in humid environments [5]. Scanning acoustic microscopy is another technique to investigate blistering or other coating disbonding [6].

With the development of the Scanning Kelvin Probe (SKP), cathodic delamination (CD) of organic coatings has become easy to follow [7]. Several samples can be placed in the SKP chamber and their CD rates can be compared [8]. The measurement of CD by SKP (CD-SKP) was used in previous studies to compare coating performance or to study the mechanism of coating delamination from an artificial defect after salt fog exposure [9,10]. In this paper, the approach is developed to assess the coating/metal interface stability of coated steel after field exposure. CD-SKP is used to compare the delamination rates of coated steel samples pre-exposed in the field. CD is a degradation mode for coated samples that occurs as the result of a cathodic reaction taking place ahead of a delaminated zone [11]. CD can occur in the field, but that is not the object of this study. Instead, the rate of CD measured in the humid SKP chamber on pre-exposed samples is used as a measure of the degradation of the interface stability resulting from the field exposure.

The CD rate depends on the interface stability, oxygen reduction at the interface, and ion transport along the interface [11–13]. The ion transport along the interface and the oxygen reduction at this interface should be equivalent for samples with the same coating system. Therefore, for a given coating system, the CD rate measured by CD-SKP should only be related to the interface stability. In this

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* Corresponding author.
E-mail address: cambier.5@osu.edu (S.M. Cambier).

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case, the interface stability of pre-exposed and unexposed samples can be assessed by comparison of CD rates.

Steel substrates coated with poly(vinyl butyral) (PVB) were exposed in different climates where atmospheric and weathering data were collected. PVB was chosen as a coating system model for this study because it has good weathering stability and barrier properties. In addition, the relatively weak wet-adhesion of PVB facilitates its rapid assessment. The extension of this method to commercial coating will be discussed in future publications.

The first objective of this work was to show that the CD-SKP approach can assess the coating/metal interface degradation after environmental exposure. The CD-SKP results were compared to EIS measurements and visual inspection of the interface under the transparent coating. The field sites were ranked by each technique to understand and validate the CD-SKP results. The second objective was to apply the CD-SKP approach to characterize the environmental degradation of PVB-coated steel. A follow-on paper will discuss the environmental degradation of PVB-coated steel in the laboratory that was performed in an effort to develop a suitable accelerated test. Photo-oxidation of the coating and interface degradation in the field were assessed by FT-IR and CD-SKP, respectively. The degradation measured was correlated to the atmospheric and weathering data collected at each site. As a result, the important variables to input into a laboratory accelerated test could be determined.

2. Experimental

2.1. Materials

PVB, which is composed of 80 wt% vinyl butyral, 20 wt% vinyl alcohol and less than 1 wt% vinyl acetate, was purchased from Sigma-Aldrich. PVB granules were dissolved at 15 wt% in methyl alcohol to enable application on the steel surface.

1018 steel sheets, cold rolled to 1/8” thickness, were purchased from McMaster-Carr. The substrates were cut into rectangular shapes of 6.35 cm by 1.9 cm. The surface was ground to 600 grit and cleaned with a detergent for all experiments. Coatings were applied by a draw-down bar on freshly ground and cleaned steel surfaces. The sample long edges were covered with electrical tape prior to coating application. PVB coatings were air dried for 24 h after coating application. Each set of samples prepared for weathering exposures included control samples. All samples were shipped for outdoor exposure in individual airtight containers. The control samples were sent with the other samples, but were kept indoors in their airtight containers. As a result, the control samples were handled exactly the same except for not being exposed to the outdoor environment.

2.2. Field exposures

The samples were exposed at different field sites. The selection of sites was intended to cover a wide variety of weathering conditions. At most sites, the samples were exposed on a rack at about 1 m from the ground at a 45° angle. Thompson Farm, NH, which was operated by the University of New Hampshire (UNH), is a rural site located near Durham, NH. Daytona Beach, FL site is facing the ocean and is located on Battelle Memorial Institute property. Sites at Lyon Arboretum, HI, Waipahu, HI, and Mauna Loa, HI are maintained by the University of Hawaii (UH). Some samples were also exposed at the Ohio State University campus (first floor balcony of the Newman Wolfram building, facing west). Weathering and atmospheric data were collected by UNH [14], UH, and the National Climatic Data Center [15–18]. Details of the conditions at each site are presented in tables in the Appendix. Table A.1 shows the general characteristics of each site and the temperature range during the first month of exposure. Table A.2 displays the time of wetness (TOW), and concentrations of chloride and sulfur. The deposition of airborne chloride and sulfate at the Hawaiian field sites was collected in wet candles. Each month the concentration was measured and reported in total daily mg/m^2. The details of the total radiation are shown in Table A.3. The wavelength range depended on the sensitivity of the UV sensor.

Two different time of wetness (TOW) values are given in Table A.2: TOW$_{ISO}$ and TOW$_{SEN}$. ISO 9223 defines TOW to be the number of hours when the conditions RH $\geq$ 80% and T > 0 °C are met during one year of exposure. The TOW$_{ISO}$ values in this report represent the number of hours these conditions were met during the first month of exposure and were calculated from RH and temperature data that were collected every hour. The TOW$_{ISO}$ values were left as hours per month and not normalized to hour per year. In the Hawaiian field sites, the voltage output of painted latex leaf wetness sensors was recorded every minute. The dry condition corresponded to a resistance of 1MΩ and a voltage output of 2.3 mV. TOW values, designated as TOW$_{SEN}$, were calculated from the sensor data to be the number of hours when the voltage output was above or equal to 2.3 mV.

2.3. Instruments

A Spectrum 100s FT-IR instrument from Perkin Elmer was used to analyze coated metals. The spectrometer was used with a two-mirror infrared reflection-absorption spectroscopy (IRAS) setup and with a triglycine sulfate (TGS) pyro electric detector.

A height-regulating SKP from K&M Soft Control was used to follow the progress of CD. The instrument was calibrated before each use with the constant potential of Cu in saturated CuSO4 (0.316 V/SHE). Potential line scans were recorded with a step size of 100 μm over a 20000 μm distance.

The sample preparation method for CD-SKP was borrowed from Leng et al. [19]. A piece of insulating tape covered part of the top of the sample before coating application. The coating was then applied on top of the metal and the taped area. The sample was exposed to various outdoor environments with the tape in place. The tape was removed before CD-SKP measurement when the sample was dried. Removing the tape left a clean coating edge, which represented an artificial coating defect. This defect was covered with 0.5 M NaCl electrolyte to initiate cathodic delamination. The SKP chamber was maintained near 100% relative humidity (RH) during CD measurements. Several samples were tested in parallel in the SKP chamber to ensure identical test conditions and to facilitate sample comparison [8]. The measurements were started immediately after covering the defect with electrolyte.

EIS measurements were performed after the CD-SKP test on the non-delaminated coated side of the sample. A plastic tube was sealed vertically with silicone on the sample. The exposed area, was 1.06 cm^2 and contained about 3 mL of 0.1 M NaCl, a saturated calomel reference electrode and a platinum wire as counter electrode. EIS measurements were taken over a 12 day period with a Gamry 600 potentiostat. A 10 mV signal about the open circuit potential was applied over a range of frequency from 100 kHz to 100 mHz.

3. Results

3.1. Environmental characteristics of the exposure sites

The information in the tables in the Appendix provides a basis for comparison of the different exposure sites. The temperatures at Thompson Farm and Mauna Loa were the lowest. The TOW$_{ISO}$

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