Contents lists available at ScienceDirect

## Progress in Organic Coatings

journal homepage: www.elsevier.com/locate/porgcoat

## In situ measurement of pipeline coating integrity and corrosion resistance losses under simulated mechanical strains and cathodic protection

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#### ARTICLE INFO

Article history: Received 11 May 2016 Received in revised form 12 July 2016 Accepted 3 August 2016

Keywords: Pipeline coating Coating testing Coating degradation Mechanical strain Cathodic protection Electrochemical impedance spectroscopy (EIS)

#### ABSTRACT

A novel experimental assembly consisting of a specially designed tensile testing rig and a standard electrochemical flat cell has been designed for simulating buried high pressure pipeline environmental conditions in which a coating gets damaged and degrades under mechanical strain, and for studying the influence of mechanically induced damages such as the cracking of a coating on its anti-corrosion property. The experimental assembly is also capable of applying a cathodic protection (CP) potential simultaneously with the mechanical strain and environmental exposure. The influence of applied mechanical strain as well as extended exposure to the corrosive environment, coupled with the application of CP, has been investigated based on changes in electrochemical impedance spectroscopy (EIS). Preliminary results show that the amplitude of the coating impedance decreases with an increase in the applied strain level and the length of environmental exposure. The EIS characteristics and changes are found to correlate well with variations in coating cracking and degradation features observed on posttest samples using both optical microscopy and scanning electron microscopy. These results demonstrate that this new experimental method can be used to simulate and examine coating behaviour under the effects of complex high pressure pipeline mechanical, electrochemical and environmental conditions. © 2016 Elsevier B.V. All rights reserved.

1. Introduction

Coatings are widely used as a means of corrosion prevention for buried structures such as energy and water pipelines, with cathodic protection (CP) acting as a complementary corrosion protection technique [1]. Coatings on pipelines are subjected to mechanical stresses during pipeline installation processes such as hydrostatic testing and field bending and during the pipeline operation where the external soil loads and the internal pipeline pressure continuously impose mechanical stresses on coatings [1]. Coatings on the buried pipelines are also under the influence of the underground soil corrosion environment and cathodic protection (CP). This constitutes a highly complex system influenced by multiple factors in nature.

In order to probe the synergistic influence of three major factors on pipeline coating degradation, there is a requirement for an experimental method that is able to simulate such a complex con-

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http://dx.doi.org/10.1016/j.porgcoat.2016.08.007 0300-9440/© 2016 Elsevier B.V. All rights reserved. dition and measure its effects on coating degradation. In order to measure the effects of mechanical stress on the degradation of the pipeline coating, it has been reported in the literature [2–7] that a number of methods have been used to apply a mechanical stress on a coating in a controlled manner. These include uniaxial tension [2,4–9] and bending [3,10,11]. To impose a mechanical strain, the concept of uniaxial loading is seen to be the most common method that has been used by Bastos et al. [2], Zhang et al. [7], Zumelzu et al. [9], Elbasir et al. [8] and Klüppel et al. [5]. Kazimierz et al. [4] and Yuli et al. [6] employed a cyclic loading, albeit in the uniaxial tensile direction. The bending mode of mechanical stress application was employed by Nguyen Dang et al. [12], and Fredj et al. [10,11].

In order to measure the changes in the electrochemical properties associated with the degradation of the coating, various electrochemical cells have been employed to simulate buried pipeline conditions while electrochemical impedance spectroscopy (EIS) has been one of the preferred techniques used by many researchers to quantify changes in coating performance [13–18]. EIS has also been utilised to acquire information about the influence of coating degradation resulting from the application of mechanical strain [2–7,9–11,19]. A typical example of this is evident in







the work by Bastos et al. [2], where EIS measurements were carried out on polyester coated steel samples. They used thin coatings of a polyester primer with an inner coat of polyurethane on a steel substrate. The average thickness of the dual polymer coating, placed above a layer of electrodeposited zinc on the steel substrate, was 20  $\mu$ m. The coated samples were subjected to strains of 9, 11, 16, 19, and 23%, up to the point of fracture. All samples showed roughening of the surface, however, holes were observed only on coating samples which were strained till the point of rupture. These strained samples were then exposed to a solution of 5 wt% sodium chloride for a period of 50 days with EIS measurements conducted over the period of exposure. The impedance of the coating and the charge transfer resistance were seen to decrease with respect to an increase in the strain level, whereas the double layer capacitance increased with increase in the applied strain. A notable observation was that, in case of the coating under zero strain, the charge transfer resistance and the double layer capacitance evolved after a period of 30 days of exposure. On the other hand, in case of higher strains, these parameters were seen to evolve earlier. At the higher strain of 23%, no defects were found before exposure to the corrosive medium (5 wt.% NaCl), while the exposure appeared to result in the occurrence of defects and holes in the coating supposedly because of the dissolution of pigment particles. These observations imply a negative effect of the exposure environment in presence of an applied strain on coating's performance. Similar trends of the degradation and cracking of coatings was also observed using EIS by other researchers [7,9].

In the above examples, however, the applied strains were in the plastic region of the steel substrate, so that the deformation in the sample was retained even after the strain was no longer applied. As a result, the effect of strain on the polymer coatings was studied ex situ, and the effects of strain in the elastic region of the steel cannot be studied using these experimental methods, since such a study of the effect of strain in the elastic region requires in-situ testing methods. In an attempt to demonstrate an in situ testing method under uniaxial tension, Klüppel et al. [5] constructed a setup, which facilitated the extension of the coated steel in a horizontal plane, rather than a vertical plane. In this case the samples used were steel plates coated with a coating of a prepolymer on top of a conversion coating. The coated sample was subjected to increasing strains in two electrolytes, namely a phosphate buffer and a 0.05 M sodium chloride solution, and EIS measurements were carried out at each stage. High strain levels between 5 and 25% were used, with the observed cracks (detected using SEM) increasing in size from less than a micron to a few microns with increasing strain from 5 to 25%. This correlated to the drop in the impedance noted for the higher strain values.

Similar in-situ methods were reported by Elbasir et al. [8] and Yuli et al. [6], who tried to look at the additional effect of CP, but from the point of view of delamination of the coating only. In case of Elbasir et al. [8], a setup was constructed which made it possible to apply a constant uniaxial tensile load to a coated sample installed in a horizontal plane whilst a CP potential was applied. However, the coating used in this case was not an intact coating, but had an artificial defect. The motive of the study was to measure the increase in the area under the delaminated coating, which was taken to be a measurement of the influence of CP on the coating. Yuli et al. [6] also considered the measurement of the delaminated area as the main parameter to explain the effect of the applied potential. In this particular instance, the mode of strain application was cyclic loading. In both of these cases, mechanical cracking of the coating was not induced nor was the EIS technique utilised.

In other studies, Nguyen Dang et al. [12] and Fredj et al. [10,11] employed low bending stresses (low in magnitude) to impose strain on testing samples in the elastic region of the polymer. The motive was to compare the influence of tensile and compressive stresses on the coating properties. The two coatings used by Fredj et al. [10,11] were epoxy based coatings, with one coating (an epoxypolyamide/amine) having a thickness of 150 µm and the other coating (a solvent-free polyamine epoxy) having a thickness of 320 µm. Their tests focused only on the elastic and viscoelastic stresses of the polymer, which were 3 MPa and 4 MPa respectively. The coated samples were bent to the curvature corresponding to these stresses, and then the coating were exposed to 3 wt% sodium chloride solution. The period of exposure was 4 months in each case. After this long period of exposure, it was seen that the tensile stress had a more severe effect on the degradation of the electrochemical properties of the coating as compared to the zero stress and the compressive stress. This was suggested to be a result of an interplay between the enthalpic and the entropic parts of the diffusion process, as well as to the rigidity of the polymer chains resulting from the applied visco-elastic stress [10,11]. Formation of defects was reflected by a drop in the coating resistance and a rise in the coating capacitance. However, these defects may not have been cracks, as the applied stress was restricted to the elastic region of the polymer. In case of cyclic loading, Kazimierz et al. [4] used an in situ mode of operation, wherein the sample was continuously undergoing a cyclic loading in a uniaxial manner. However, here the stresses were restricted to the elastic region of the underlying steel. The EIS measurements were carried out on a coating of thickness of  $182 \,\mu\text{m}$  in an electrolyte of 3% sodium chloride. The coating resistance was seen to increase initially and then decrease, a phenomenon which was attributed to the release of energy in the polymer, rather than to changes occurring in the coating surface as a result of the applied cyclic loading.

In addition to these examples described above, a more detailed literature review [20] shows that, although there has been work done on the influence of mechanical stress, environment, and CP, on organic coatings, these are typically limited to very thin coatings, and the combination of only two of the three key factors. Therein lies a need to study the cracking of pipeline coatings under the combined effects of three major factors, mechanical stress, corrosive environment and CP.

In this work a new experimental method has been developed in order to facilitate the control of the applied strain on coated samples, either in the elastic region or the plastic region. It also aims to enable the insitu study of the combined effect of mechanical strain on coatings exposed to corrosive media under CP using the technique of electrochemical impedance spectroscopy.

#### 2. Experimental

#### 2.1. Methodology development

The design of a new testing assembly has taken two major aspects - mechanical and electrochemical - into consideration. The mechanical aspect requires that it should be able to apply a mechanical strain with a provision to monitor the strain applied. Although a workshop based tensile testing machine can be used to apply such strain on a coated sample, a full scale machine is not practical if one has to do long term and many tests at the same time. In the literature, there have been two types of smaller scale testing setups constructed. One setup is for the horizontal tensile straining [5,8], while the other setup is for the application of a bending stress [3,10,11]. However the real time monitoring of the strain has not been performed, and for this reason in one prior setup the applied strain was only estimated based on the curvature of the sample corresponding to the bending stress of interest [3,10,11]. Hence, in the present work, the mechanical part of the assembly has been designed such as to obtain controlled mechanical strain as well as to monitor it.

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