



Detection of under deposit corrosion in a CO₂ environment by using electrochemical noise and recurrence quantification analysis



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ABSTRACT

In this study, the corrosion of carbon steel immersed in CO₂ saturated aqueous solutions, in the presence and absence of sand deposits, were investigated by electrochemical noise measurement and recurrence quantification analysis. Uniform corrosion occurred at samples without sand deposit, while localized corrosion took place at the sand-covered steel samples. These two different corrosion types can be accurately predicted by random forest and principal component models based on recurrence quantification analysis of either electrochemical potential or current noise data, regardless of threshold values. The study provides a potential automated online corrosion monitoring scheme to ensure the integrity of pipelines.

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1. Introduction

For economic reasons, carbon steel pipelines are widely used for oil and gas transmission and distribution. However, when operating in CO₂ environments, these pipelines are vulnerable to corrosion. The situation is exacerbated in the presence of mineral deposits, such as silica sand, which can be transported along with fluids. Apart from causing erosion-corrosion of the pipelines, these solid particles can also induce severe localized corrosion (e.g. pitting and mesa attack), which is referred to as under deposit corrosion (UDC) when the solids settle at the bottoms of pipelines, owing to low fluid flow velocities or stagnant conditions during shutdowns [1–3].

The control of UDC is currently achieved by pigging and addition of corrosion inhibitors [4,5]. Effective application of these mitigation techniques should also be carefully considered and is closely related to monitoring pipeline corrosion. Conventionally, some corrosion monitoring techniques, such as linear polarisation resistance (LPR), electrochemical impedance spectroscopy (EIS) and electrical resistance (ER) are employed in laboratory testing or in the field for corrosion rate estimations. The results obtained from these approaches have some instructive value in the development

of corrosion control programs. Nevertheless, these techniques may not be particularly useful for monitoring of localized corrosion, which occurs in the case of UDC, since they can only provide information regarding the average corrosion rate [6,7]. One may find a pipeline with very low corrosion rates, suggested by these approaches, exhibiting severe pitting corrosion beneath a large volume of sediment [5]. Therefore, it is crucial that the occurrence of UDC can be detected and closely monitored.

Electrochemical noise (EN) measurement has been widely used for corrosion studies and field monitoring, owing to its ease of setting up, non-destructiveness, non-intrusiveness and particularly the ability to provide information on the initiation and propagation of localized corrosion events [8–12]. The challenge of this technique lies in the extraction of suitable feature variables from the noise data to distinguish between different types of corrosion. A number of parameters have been proposed for this purpose, including the roll-off slope of power spectral density plot [13], statistical parameters such as standard deviation, skewness and kurtosis [14], energy distribution plot from wavelet analysis of EN data [15] and characteristic charge [16], etc. Nevertheless, the optimal approach for the analysis of EN data remains uncertain, especially when the automated corrosion monitoring program is considered. Recently, studies have shown the variables extracted by recurrence quantification analysis of EN data, such as recurrence rate and determinism, could be used to characterize the EN signals associated with localized corrosion processes [8,17–24].

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A recurrence plot (RP) is a graphical tool first introduced by Eckmann et al. [25] to visualise the recurrence behaviours in dynamic systems based on phase space reconstruction [26]. It can be mathematically expressed as a matrix according to equation (1)

$$R_{i,j} = H(\varepsilon - \|\mathbf{x}_i - \mathbf{x}_j\|), \quad i, j = 1, 2, \dots, N \quad (1)$$

where \mathbf{x}_i and \mathbf{x}_j represent the states of the reconstructed phase space trajectory at time i and j , respectively; N is the total number of states in the trajectory; $\|\cdot\|$ calculates the distance between \mathbf{x}_i and \mathbf{x}_j ; ε is a user defined threshold and H is the Heaviside function which gives 0 and 1 depending on the sign of the content within the bracket. $R_{i,j}$ refers to the $(i, j)^{th}$ point in the recurrence matrix. The RPs can be quantified by a number of variables. The quantification of the RPs is called recurrence quantification analysis (RQA). EN signals could be characterised by the variables generated by RQA. Recently, the authors have proposed a corrosion type monitoring scheme [27] and an identification model [28] based on RQA of electrochemical current noise data. In these studies, the recurrence plots were generated without reconstructing the original time recordings to phase space trajectories. Instead, for a given signal, the Euclidean distance between each pair of the measured current values was computed and compared with a pre-defined threshold value. It was demonstrated that the variables extracted from the noise data by RQA were applicable in distinguishing between uniform, pitting and passivation (pseudo-passivation) corrosion processes [27,28].

In the present study, previously established approaches were applied to analyse the electrochemical noise data obtained from CO_2 corrosion of carbon steel samples with and without sand deposits. Detailed investigations of corrosion mechanisms associated with UDC have been carried out previously by range of electrochemical methods, including potentiodynamic polarisation, cyclic voltammetry and linear polarisation resistance [1,3,4,29]. However, these methods do not allow for in-situ monitoring of localized corrosion. It is expected that localized corrosion would occur at the carbon steel samples covered with sand while uniform corrosion would take place at steel without sand. The aim of the current study was to investigate whether the methodology for processing of electrochemical noise signals [27,28] could be used for UDC analysis. It was of particular interest to use EN to detect and monitor localized corrosion (pitting) that has been associated with the UDC. Moreover, comparative studies were conducted on the effect of different model parameters, e.g. the length of a data segment and the threshold value, with respect to the prediction accuracy of the model for identifying different types of corrosion.

2. Experimental work

2.1. Materials

The samples used in the present work were carbon steel (grade 1030) with chemical compositions of (wt%): C (0.37), Si (0.282), Mn (0.80), P (0.012), S (0.001), Cr (0.089), Ni (0.012), Mo (0.004), Sn (0.004), Al (0.01), and Fe (balance). The steel samples were soldered with conducting wires for electrical connection and then electro-coated using Powercron 6000CX to avoid potential crevice corrosion. Afterwards, the sample was mounted in epoxy resin (Epofix), leaving approximately 2 cm^2 as working surface. Prior to the test, the working surface was ground using silicon carbide paper up to 1200 grit, followed by rinsing with ultrapure water and ethanol and drying with nitrogen.

The test solution was prepared with analytical grade chemicals and ultrapure water (Milli-Q system, resistivity $18.2 \text{ M}\Omega \text{ cm}$), which consisted of 3 wt% sodium chloride (NaCl ; Merck, 99.7%) and

0.01 wt% sodium bicarbonate (NaHCO_3 ; Merck, 99.7%). Before performing a test, the solution was saturated with CO_2 (oxygen content $<10 \text{ ppb}$) at 30°C . The pH of the test solution was approximately 4.7. The silica sand used as deposit was purchased from Sigma Aldrich. The properties of sand and washing methods employed have been published previously [3].

2.2. Electrochemical noise measurement

Two nominally identical steel samples were used as working electrodes (WE1 and WE2). The two samples were positioned as shown in Fig. 1.

Two test cells were set up for the electrochemical noise measurement, one with sand-deposited steel (WE1 and WE2) and one with bare steel samples (no deposit). In both cells, the WEs were placed on a Teflon sample holder with working surfaces facing upwards. For the sand-covered WEs, a mounting cup (Struers) was used to hold the sand. The mass of the silica sand was 15 g, resulting in a thickness of approximately 8 mm above the steel samples. In a separate glass vessel, the test solution was heated up to $30^\circ\text{C} \pm 1^\circ\text{C}$ and purged with CO_2 for about 2 h. The two test cells, equipped with steel samples with (cell 1) or without sand (cell 2) deposit, were purged with N_2 for 30 min to create a deaerated environment. Afterwards, the CO_2 saturated test solution was pumped into the test cells. During all the tests, the temperature of the test solution was kept at $30^\circ\text{C} \pm 1^\circ\text{C}$ with the use of thermocouple controlled hotplates.

The electrochemical noise signals were measured using Gamry ESA410 software and Gamry Reference 600 potentiostat operating in the zero resistance ammeter (ZRA) mode. In addition to the working electrodes, a Metrohm single-junction Ag/AgCl (3 M) electrode was used as reference electrode and placed in Luggin capillary in close proximity to the working electrodes in order to minimize the IR drop between reference and working electrodes. The current flowing between the two WEs and the potential between the coupled WEs and the reference electrode were recorded simultaneously. In our previous studies with pitting corrosion systems [27,28], it was found that the sampling rate of 2 Hz allowed for detection of most pitting events. Therefore, the same sampling frequency was used in this study. During the EN measurements, the test solution was continuously purged with CO_2 . Low flow rates in the tests with and without sand deposit were controlled by a gas flowmeter to minimize an impact on the EN measurement.

The EN measurements were carried out for 30 and 17 days respectively for tests with and without sand deposits. Over the test

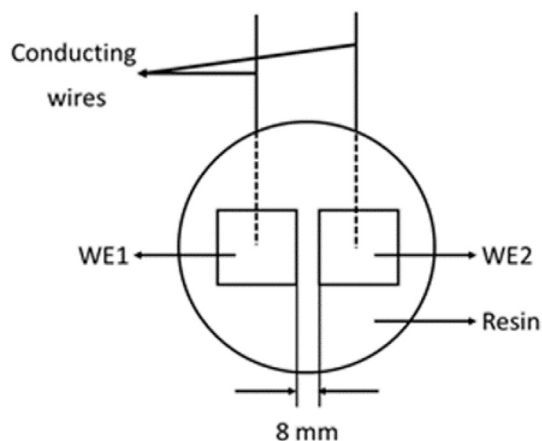


Fig. 1. Schematic diagram of the working electrodes for EN measurements (total working surface area 4.2 cm^2).

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