



# Effect of surface finishing on early-stage corrosion of a carbon steel studied by electrochemical and atomic force microscope characterizations



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## ABSTRACT

In this work, the early-stage corrosion of a carbon steel with various surface roughness, which was created by different levels of surface finishing treatment, was characterized by an atomic force microscope and electrochemical measurements. It is found that the resulting surface roughness is at nano-meter scale. As the surface roughness increases, the corrosion activity of the steel is increased. The early-stage corrosion of the steel is featured with two stages of dissolution. While the first stage involves a rapid dissolution and increasing surface roughness of the steel, stage two is in an equilibrium state to have an approximately constant corrosion rate and surface roughness. Generally, the corrosion rate of the steel decreases when the surface finish of the specimen becomes finer. Local preferential corrosion occurs at surface irregularities, resulting in the deepening and widening of the features such as scratches with time.

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## 1. Introduction

Investigations of the early-stage features of metallic corrosion are essential to understand the mechanistic aspect of the initiation of corrosion [1–3]. By virtue of various surface analysis techniques, the corrosion process can be characterized *in-situ* by topographic imaging. In particular, the atomic force microscopy (AFM) is able to image the corroded metal electrode at a spatial resolution of nano-meter scale [4–8]. Moreover, the principle of AFM makes it possible to detect non-equilibrium features associated with corrosion initiation [6,9], which is beyond the capability of conventional electrochemical techniques that are usually suitable for equilibrium systems [10].

Considerable work has been performed to study pipeline corrosion [11], however, the majority of which investigated the steady-state corrosion behavior of pipe steels in corrosive environments. The authors' group used micro-electrochemical measurement techniques, including localized electrochemical impedance spectroscopy (LEIS) and scanning vibrating electrode technique (SVET), to characterize the local electrochemical activities of metallurgical features, such as non-metallic inclusions,

that are at a microscopic scale [12–14]. Frequently, corrosion of metals, including steels, starts from a preferential dissolution at finer metallurgical features and surface irregularities with a nano-meter dimension [15–17]. These irregularities can be induced by surface finishing, which is widely applied as final or intermediate procedure in steel milling [18].

To date, there has been few work to investigate the early-stage corrosion of steels induced by surface finishing. For those studying the steady-state corrosion of steels affected by their surface conditions, it was found that an increase in surface roughness of the specimen increases its corrosion rate and the susceptibility to pitting [19–23]. There were also observations [24] that there is no specific relationship between the surface roughness of steels and their corrosion rates. The controversial results are probably caused by the limited capability of the electrochemical methods, which cannot provide *in-situ*, but *ex-situ* characterizations. An *in-situ* investigation of corrosion of steels with varied surface finishes at nano-meter scale improves our understanding of the fundamentals of the early-stage corrosion.

In this work, an AFM, combined with electrochemical measurements, including corrosion potential, electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization curves, was used to study the early-stage corrosion of a X100 pipeline steel with various surface roughness at nano-meter scale. The effect of surface roughness on the steel corrosion was analyzed, and the correlation of the early-stage feature of corrosion with the surface

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roughness was established. Moreover, the corrosion current density was calculated based on changes of surface topographies of the steel derived by AFM images.

## 2. Experimental

### 2.1. Material and solution

Specimens used in this work were cut from a X100 pipeline steel plate, with a chemical composition (wt.%): C 0.07, Mn 1.76, Si 0.1, Ni 0.154, Cr 0.016, Mo 0.2, V 0.005, Cu 0.243, Al 0.027, S 0.005, P 0.018, and Fe balance. The microstructure of the steel consists of ferrite and bainite. The size of the polygonal ferrite ranges from 3 to 10  $\mu\text{m}$ . There is no obvious orientated structure induced by manufacturing processes [25].

The specimens used for *in-situ* AFM observation were machined into a rectangular shape with a dimension of 22 mm  $\times$  22 mm  $\times$  1 mm. To create various surface roughness, the specimens were ground using #400, #800 and #1200 emery papers, as well as polished by 1  $\mu\text{m}$  diamond paste, respectively. The specimens were then degreased in ethanol via an ultrasonic bath, rinsed with deionized water and dried by highly purified nitrogen. The specimens used for electrochemical measurements were sealed in an epoxy resin, leaving a working face with a dimension of 10 mm  $\times$  10 mm. The surface finishes of the specimen were identical to those for AFM observations.

The test solution, simulating a soil-extracted solution, contained 0.483 g/L  $\text{NaHCO}_3$ , 0.122 g/L KCl, 0.181 g/L  $\text{CaCl}_2 \cdot \text{H}_2\text{O}$  and 0.131 g/L  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ , with a pH of 7.8 [26]. All solutions were prepared using analytical grade chemicals and deionized water.

All tests were performed at ambient temperature ( $\sim 20^\circ\text{C}$ ) and open to air.

### 2.2. Electrochemical measurements

Electrochemical measurements were performed through a Solatron 1280C electrochemical system on a conventional three-electrode cell, where the steel specimen was used as working electrode, a carbon rod as the counter electrode (CE), and a saturated calomel electrode (SCE) as the reference electrode (RE). Upon immersion in the solution, the open circuit potential (OCP) of the working electrode was measured for 1 h until a steady-state value was reached. The EIS was measured in the frequency range from  $2 \times 10^4$  Hz to  $10^{-2}$  Hz, with an applied disturb signal of 10 mV. The EIS measurements were carried out at OCP. Potentiodynamic polarization curves were measured at a potential sweep rate of 0.167 mV/s.

All tests were conducted at least three times to ensure the reproducibility of the testing results.

### 2.3. In-situ AFM characterization

An AFM (Keysight 5500 scanning probe microscope system) was used for topographic characterization of the steel specimen. A scanner carrying a long rectangular cantilever with a spring constant of 0.2 N/m (apex radius  $< 10$  nm) was placed above the specimen. The scanning mode was configured as contact, with a scanning rate of 1 Hz and a resolution of  $512 \times 512$  pixel. The specimen was installed in a perforated plastic cell, which served as the solution container. The size of the cell was sufficiently large for easy installation of the specimen, and also avoid evaporation of the solution.

Prior to addition of the solution in the plastic cell, the topography of the steel specimen was imaged within an area of  $50 \mu\text{m} \times 50 \mu\text{m}$ . A region with the size of  $10 \mu\text{m} \times 10 \mu\text{m}$  was selected for further AFM characterization of the specimen as corrosion proceeds. The obtained images were processed to remove background signals,

and to extract results such as surface roughness and topographic profiles. The surface roughness ( $R_t$ ) is defined as the height difference between the peak and the valley in the imaging region [27].

## 3. Results

### 3.1. AFM characterization of surface topography of steel specimens prior to corrosion

Fig. 1 shows the surface topography of steel specimens prepared with various surface finishing methods, i.e., #400, #800 and #1200 emery papers grounding, and 1  $\mu\text{m}$  diamond paste polishing, respectively. It is seen that there are obvious parallel scratches induced by surface finishing. With the finishing condition becoming finer, the steel surface is smoother and the surface scratches are less obvious.

In order to quantify the surface roughness of the specimen under various finishing conditions in Fig. 1, a total of 13 points is selected on each specimen, as shown in Fig. 2a, to measure the roughness by AFM, where the sampling scale is 50  $\mu\text{m}$ . The statistical results and standard deviations are shown in Fig. 2b. It is seen that both the surface roughness of the specimen and the standard deviation of the measured results decrease when the surface finishing condition becomes finer. For example, the specimen grounding with #400 paper has a surface roughness of 223 nm, and the surface roughness of the specimen polished by 1  $\mu\text{m}$  diamond paste is about 4 nm.

It is noted that a measurement of the average roughness of each sample is more accurate than scattered measurements at individual points. However, the AFM is not capable of measuring the average surface roughness of the test specimen due to its limited scanning range. In order to improve the measuring accuracy, it is tried to measure the surface roughness at many points.

### 3.2. Electrochemical measurements

Fig. 3a shows the time dependence of the OCP of the steel electrode with various surface roughness in the test solution. It is seen that, upon immersion in the solution, the OCP drops rapidly until a knee point is reached. Then the OCP gradually becomes constant. Moreover, a longer time is required to achieve the approximately stable OCP for the specimen with a finer surface finish. The steady-state OCP is more negative when the surface polishing of the specimen is coarser.

Fig. 3b shows the potentiodynamic polarization curves measured on steel specimens with different surface roughness in the solution. It is seen that all the measured curves exhibit a similar characteristic, i.e., an active dissolution featured anodic branch and a mass transfer controlled cathodic branch. Moreover, the polarization curves almost copy each other.

The corrosion potentials obtained from Fig. 3a and b are summarized in Fig. 3c. It is seen that the corrosion potential is shifted less negatively with the decrease of the surface roughness. Generally, the corrosion potential of a metal electrode indicates the corrosion tendency of the metal in corrosive environments, where a more negative potential is associated with the increased corrosion activity of the metal in the environment [28]. It is thus seen that, as the surface roughness decreases, the corrosion activity of the steel reduces. Moreover, the corrosion potentials derived from the polarization curves in Fig. 3b are more negative than those obtained from Fig. 3a. This is attributed to the cathodic pre-polarization applied on the steel electrode during potentiodynamic polarization measurements.

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