



# Photo-electrochemical studies of the local dissolution of a hydrogen-charged X80 steel at crack-tip in a near-neutral pH solution

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## ABSTRACT

In this work, the photocurrent densities of a pre-cracked X80 pipeline steel under various applied forces in the absence and presence of hydrogen-charging were measured in a near-neutral pH solution through a photo-electrochemical system. Electrochemical impedance spectroscopy (EIS) was also measured on the steel electrode to obtain the local impedance under various test conditions. It was determined that, of the total photocurrent density measured at the crack-tip of the charged steel, the photo-induced current density, hydrogen-enhanced dissolution current density and photo-oxidative current density of hydrogen atoms contribute approximately 65.8%, 12.8% and 21.4%, respectively. An applied force increases the photocurrent density measured at crack-tip due to the enhancing stress concentration and the local electrochemical activity. In the presence of hydrogen-charging, the measured photocurrent density at individual applied force was higher than that measured on the uncharged electrode, which was associated with the hydrogen-induced dissolution current density and the photo-oxidation of hydrogen atoms. Under an identical applied force there was a higher photocurrent density at crack-tip than the region ahead of the crack for both charged and uncharged electrodes due to the high electrochemical activity at the crack-tip, resulting in more hydrogen atoms accumulating locally. The local electrochemical impedance measurements were consistent with the photo-electrochemical results to demonstrate the dependence of local dissolution rate of the steel at crack-tip on the applied force and hydrogen-charging.

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## 1. Introduction

Development of the high-strength steel pipeline technology has been paid much attention with the increasing oil/gas explorative and productive activities in the Arctic and sub-Arctic areas, where the high-strength steels, such as X80 steel, would enable the energy and pipeline industries to realize significant savings in the total cost of long-distance oil/gas transmission in view of the reduced wall thickness and increased operating pressure in pipelines [1,2]. However, stress corrosion cracking (SCC) has constituted a potential threat to the safe operation of the pipelines [3,4]. In particular, near-neutral pH SCC is a typical type of SCC occurring at an electrolyte, with a pH of about 6.5, trapped under the disbonded coating [3–6].

The near-neutral pH SCC of pipelines is attributed to a synergistic effect of hydrogen and stress on local dissolution of the steel at crack-tip [6–17]. A number of techniques, such as secondary ion mass spectroscopy [18] and hydrogen thermo-desorption spectroscopy [19,20], have been used to determine and image the

concentration and distribution of hydrogen in the steel. However, they are incapable of characterizing the contribution of hydrogen to the local anodic dissolution and corrosion of the steel [21].

Photo-electrochemical (PEC) technique provides a promising alternative to characterize *in situ* the effect of hydrogen on the corrosion and cracking behavior of the steel [22–25]. When a passivated steel is illuminated with the light of energy higher than the band gap of the passive film, a photocurrent is generated. Upon hydrogen-charging, the photocurrent is enhanced by the photo-oxidation of hydrogen to protons. Razzini et al. [22] used PEC to image the hydrogen distribution at the crack-tip in an unloaded X60 steel specimen after hydrogen-charging. It was demonstrated that a large amount of hydrogen was segregated at the high stress region with a plastic structure. In authors' previous work [26], it was found that the photo illumination would accelerate the anodic dissolution of the steel in a near-neutral pH solution. Moreover, hydrogen-charging increased the anodic dissolution current of the steel, compared to the uncharged steel electrode. Furthermore, Zeng et al. [27] reported that hydrogen might decrease the stability of the passive film on iron and stainless steels, and promotes localized corrosion. Apparently, the photocurrent response measured on a charged steel specimen upon photo illumination is quite complex, containing at least three components—the photo-oxidative

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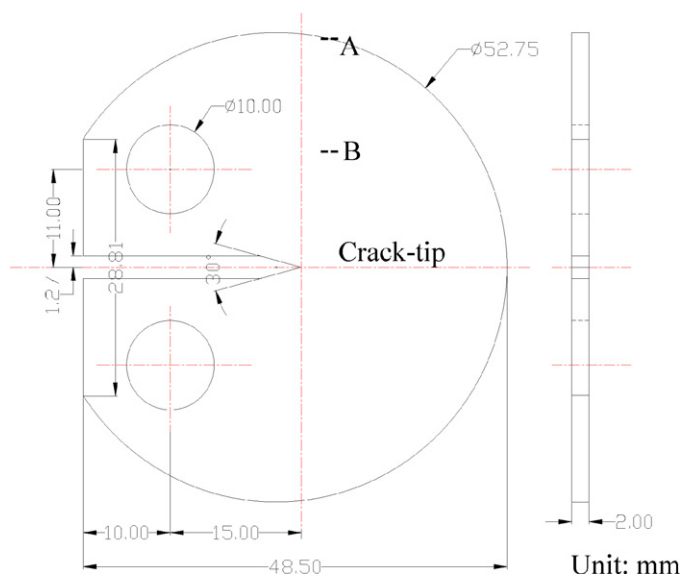


Fig. 1. Schematic diagram of the pre-cracked X80 steel specimen.

current of hydrogen atoms, the photo-enhanced anodic dissolution current of the steel, and the hydrogen-enhanced anodic dissolution current of the steel.

In this work, the distribution of hydrogen around the crack-tip in a pre-cracked X80 steel specimen and the resulting local corrosion behavior were characterized through an in-house PEC system. The contributions of photo illumination and hydrogen-charging to the measured photocurrent were determined quantitatively. The effect of the accumulated hydrogen on local dissolution of the steel was also investigated by electrochemical impedance spectroscopy (EIS).

## 2. Experimental

### 2.1. Electrode and solution

Specimens used in this work were cut from a X80 steel pipe, with the chemical composition (wt%): C 0.026, Mn 1.6, Si 0.22, S 0.001, P 0.003, Ni 0.15, Cr 0.27, Cu 0.27, Nb 0.097, Ti 0.014, and Fe balance. The yielding strength of the steel was 640 MPa in longitudinal direction and 775 MPa in transverse direction, respectively, determined by tensile test in air [28]. Compact tension (CT) specimens were machined according to ASTM E647-05 [29], with the dimension shown in Fig. 1. The notch on the CT specimen was aligned with the roll direction of the steel. The specimen was ground sequentially down to 1200 grit SiC paper, polished with a 0.5  $\mu\text{m}$  emery paste, cleaned with acetone and distilled water, and dried in cold air. The working surface of the specimen was sealed with a 704 silicon rubber, leaving a working area of 0.2  $\text{cm}^2$  around point A, point B and the crack-tip, respectively, as indicated in Fig. 1.

The test solution was a near-neutral pH solution (NS4 solution), with the chemical composition: 0.483 g/L  $\text{NaHCO}_3$ , 0.122 g/L KCl, 0.181 g/L  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  and 0.131 g/L  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ . The solution was made from analytic grade reagents (Fisher Scientific) and ultra-pure water (18  $\text{M}\Omega \text{ cm}$  in resistivity). Prior to test, the solution was purged with 5%  $\text{CO}_2$  for 1 h to achieve an anaerobic and near-neutral pH condition (pH = 6.8). The gas flow was maintained through the test. All tests were carried out at room temperature ( $\sim 22^\circ\text{C}$ ).

### 2.2. Photo-electrochemical measurement

The photocurrent measurement was carried out using an in-house PEC system, with the schematic diagram shown in Fig. 2. The

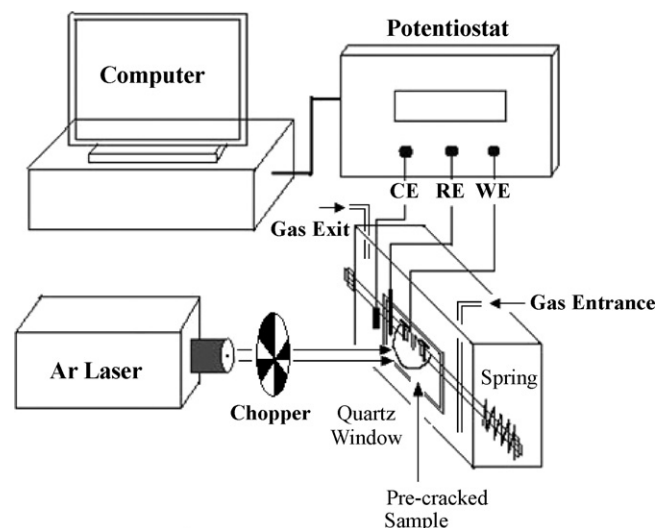


Fig. 2. Schematic diagram of the experimental set-up of the PEC system.

PEC system contained a 500 mW Argon ion laser source (Model 5490A ILT 6000 series, Ion Laser Technology Inc., USA). The light with a wavelength range of 457–514 nm travelled through a chopper and illuminated the steel specimen through a quartz window, and the beam diameter was 0.65 mm. The pre-cracked steel specimen was used as the working electrode (WE), a saturated calomel electrode (SCE) as reference electrode (RE), and a platinum plate as counter electrode (CE). Two tensile forces of 1600 N and 2700 N were applied on WE through a compressive spring installed at the side of the cell. The force applied was determined accurately by the change of the length of spring and its elastic coefficient. The stress distribution on the pre-cracked steel specimen under a 1600 N and a 2700 N of tensile force was calculated by finite element method [7], and shown in Figs. 3 and 4, respectively. The stress values at three positions marked in Fig. 1 under the force of 1600 N were  $\sigma_A = 69.2$  MPa,  $\sigma_B = 74.8$  MPa and  $\sigma_{\text{crack-tip}} = 637$  MPa. Under the 2700 N of applied force, the stress values were  $\sigma_A = 125$  MPa,  $\sigma_B = 142$  MPa and  $\sigma_{\text{crack-tip}} = 1070$  MPa. The resulting local stresses represented the amplitude of pressure fluctuation in natural gas transmission pipelines.

The photocurrent measurements were performed on the steel specimen at corrosion potential. The generated photocurrent was recorded by a Solartron 1280C electrochemical system. The PEC measurements were conducted at crack-tip and points A and B, as marked in Fig. 1.

The pre-cracked steel specimen was polarized galvanostatically with a cathodic current density of 10  $\text{mA}/\text{cm}^2$  for 2 h in NS4 solution for hydrogen-charging. The corrosion potential was measured immediately after hydrogen-charging for 1 h. The laser source was switched on to investigate the effect of photo illumination on corrosion potential.

### 2.3. EIS measurement

The EIS measurements were performed using a Solartron 1280C electrochemical system through a typical three-electrode cell, with X80 steel specimen as working electrode, a SCE as reference electrode and a platinum sheet as counter electrode. Prior to EIS measurement, the steel specimen was uncharged or pre-charged at a cathodic current density of 10  $\text{mA}/\text{cm}^2$  for 2 h. The EIS was measured at the crack-tip and points A and B, respectively, when the specimen was under various applied forces of 0 N, 1600 N and 2700 N. The EIS measurement frequency ranged from 0.01 Hz to 20,000 Hz, with an applied AC disturbance signal of 5 mV.

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