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# In situ corrosion characterization of simulated weld heat affected zone on API X80 pipeline steel

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ABSTRACT

Subject to Gleeble processing, microstructures of weld heat affected zone were simulated in X80 steel. Corrosion behavior of the simulated specimen with a microstructure gradient was studied by polarization, local electrochemical impedance spectroscopy and scanning vibrating electrode technique. Microstructure of granular bainite mixed with ferrite (region B) showed the highest charge transfer resistance and the most positive current density value. Acicular ferrite base metal displayed the lowest charge transfer resistance and the most negative current density. Both the positive and negative peak current densities increased at the first few hours of immersion followed by a decrease.

## 1. Introduction

Localized corrosion in welds is the most common failure mode for the high strength pipeline steels buried underground [1]. Zhang and Cheng [2] have found that weld heat affected zone (HAZ) of X70 pipeline steel has the largest anodic dissolution current upon hydrogen-charging in near-neutral pH solution. Chaves and Melchers [3] have found that the maximum pit depth in weld HAZ of X56 pipeline steels is much greater than that in the adjacent base metal (BM) when exposed to natural Pacific Ocean seawater. It has been proposed [2–4] that corrosion susceptibility of HAZ is enhanced due to phase changes during welding process as a result of the extreme temperature gradient. Minor differences in composition and microstructure between HAZ and BM can result in an electrochemical galvanic effect, affecting the corrosion reaction of the steel. Earlier works have shown that different phases and microstructures in steel provide sites for the anodic and cathodic reactions [5]. Great efforts [6,7] have been taken to clarify the electrochemical differences between different phases. It is generally accepted [8–11] that corrosion susceptibility of pearlite and austenite is higher than ferrite and lower than lath martensite. Clover et al. [8] found that steels with a banded ferrite/pearlite structure performed poorly in terms of localized corrosion and this was attributed to a segregated distribution of cementite. Cui and Lundin [12] have found that initial corrosion attack for 316 stainless steel weld deposits in $FeCl_3$ is from austenite instead of ferrite when the ferrite is distributed in a discontinuous cellular dendritic microstructure. The research [13,14] on 2205 duplex stainless steel show that the selective dissolution in acidic chloride solution is related to the potential applied to the steel. Sedriks et al. [15] found that small amounts of δ ferrite in the austenite matrix was detrimental to pitting resistance by providing favorable sites for pitting initiation.

Investigation of microstructure effect on electrochemical and corrosion properties is also applied to high strength low alloy pipeline steels [16–19]. Luo et al. [16] have found that pipeline steels with a microstructure of fine grained bainite–ferrite have a better combination of strength and SCC resistance than those with ferrite-pearlite structures. Ren et al. [20] have found that elemental segregation and coarse grains lead to the high corrosion sensitivity of HAZ. Because HAZ in a welded joint is too narrow to get available test specimen, it is very difficult to analyze the effect of a characteristic microstructure on corrosion using the true weldment. The research method usually used is to simulate various microstructures by heat treating. Nowadays, with the development and application of thermodynamic simulation technique in welding field, research on the relation between microstructures and properties for the welded joint becomes easy [21–23]. Mohammadi et al. [4] used conventional electrochemical techniques such as open circuit potential (OCP), potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) to study the effect of different post welding microstructures achieved by thermodynamic simulation on corrosion behavior of X80 steel.

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In this work, we used micro-electrochemical measurements including scanning vibrating electrode technique (SVET) and localized electrochemical impedance spectroscope (LEIS) to study corrosion behavior of the simulated weld HAZ with a microstructure gradient for API X80 steel. These two local methods have been used in the past to investigate different corrosion behavior of various microstructures and have remarkable advantages over traditional electrochemical techniques in understanding the role of microstructure and grain size with corrosion [24–30]. Deshpande [25] used SVET to investigate the effects of β phase fraction on corrosion behavior of Mg alloy. Dzib-Pérez et al. [26] used SVET to characterize different electrochemical properties of dissimilar microstructures of Al–La alloy produced by different solidification rates. SVET has proved capable of providing spatially and temporally resolved data on local anodes evolving on the surface of samples. Galicia et al. [27] used LEIS to observe the influence of microstructure on corrosion rate of AZ91 alloy. Lima-Neto et al. [28] showed that the length of the sensitized zone in the heat-affected zone of welded 304 stainless steel was detected by LEIS technique. By comparing the Bode magnitude plot of LEIS with that of EIS for the same electrode system, Lillard et al. [29] concluded that the local ac impedance measurements made with this technique accurately reflected the true electrochemical properties of the electrode under investigation.

The objective of this study was to understand the effect of welding heat input and thermal cycles on the microstructures of API X80 pipeline steel and the relationship between microstructures and corrosion by investigating in situ corrosion behavior of the simulated weld HAZ in an acidic soil environment.

## 2. Experimental

### 2.1. Material and solution

The API X80 high strength low alloy pipeline steel was used as the base metal for thermomechanical controlled processing. The chemical compositions of the steel are (wt.%): 0.036 C, 0.197 Si, 1.771 Mn, 0.012 P, 0.002 S, 0.223 Cr, 0.278 Ni, 0.220 Cu, 0.021 Al, 0.019 Ti, 0.184 Mo, 0.001 V, 0.110 Nb, 0.005 N and balanced by Fe. The microstructure of the steel was observed by Quanta 250 Scanning Electron Microscopy and JEM-100CX II Transmission Electron Microscopy after polishing and etching with 4% nitric acid-alcohol solution. A 10 mm × 10 mm × 80 mm rectangular sample of the API X80 pipeline steel was subjected to thermal cycle in the DSI Gleeble 3500 thermomechanical simulator. Heating and cooling parameters provided in Table 1 were selected to simulate possible HAZ microstructures occurred in real welding production. The experimental sample was heated up to 1250 °C at a rate of 160 °C/s and then cooled down to ambient temperature. Following the thermal cycles, 15 mm × 10 mm section was cut from the middle part of the thermomechanical simulated sample and then polished for microstructure characterization and electrochemical tests. Microstructure observation was performed by Quanta 250 SEM. Micro-hardness tests were conducted to the specimen on a Buehler Micromet Model MII micro-hardness tester by a pyramidal diamond indenter with a load charge of 0.49 N. Each experiment was repeated three times to ensure accuracy and reproducibility of the results.

The electrochemical test solution was the simulation solution of an acidic soil located in southeast China. It was made from analytic grade reagents (Fisher Scientific) and ultra-pure water (18 MΩ cm in resistivity) with the contents: 0.0111 g/L $CaCl_2$, 0.0468 g/L NaCl, 0.0142 g/L $Na_2SO_4$, 0.0197 g/L $MgSO_4 \cdot 7H_2O$, 0.0293 g/L $KNO_3$, 0.0151 g/L $NaHCO_3$. The solution pH was adjusted to 4.6 by adding hydrochloric acid. The conductivity of the solution was measured to be 212 μS/cm. Prior to test, the solution was purged with $N_2$ for 2 h allowing a low concentration of oxygen and the gas flow was maintained throughout the test.

### 2.2. Conventional electrochemistry tests

Open circuit potential (OCP) and potentiodynamic polarization tests were performed at a Princeton 2273 PARSTAT for which a conventional three-electrode electrochemical cell was used, where the counter electrode is a sheet of Pt and potentials were measured against a saturated calomel electrode (SCE). The simulated HAZ specimen cut from the middle part of the thermomechanical simulated sample with the dimension of 15 mm × 10 mm × 5 mm was used for working electrode after coating with epoxy resin. The surface was ground consequentially up to 2000 grit emery paper followed by rinsing with double-distilled water and finally degreased in ethanol. Prior to test, the specimen was sealed with Parafilm M barrier film, leaving an exposure area of 1 mm in diameter for different microstructure regions to do electrochemical tests. OCP was recorded for 1 h to reach a steady-state freely corroding condition before potentiodynamic test. The potentiodynamic polarization curves were measured in the potential range from −0.3 to 0.3 V (vs. OCP) with the scanning rate of 0.1667 mV/s.

### 2.3. LEIS tests

LEIS tests were performed at open-circuit potential of the simulated HAZ specimen through a PAR 370 Scanning Electrochemical Workstation, which was comprised of a 370 scanning control unit, a Princeton M263A potentiostat, a Solartron 1255B frequency response analyzer and a video camera system. A three-electrode test cell was used, with a platinum plate as counter electrode, a saturated calomel electrode (SCE) as reference electrode. A dual-element probe consisted of two platinum electrodes positioned on and in a conical plastic holder was set directly above the specimen surface, one is a platinum wire with a 50 μm tip and the other is a platinum ring placed at a distance of 3 mm from the tip. Fig. 1 shows the experimental setup. The relative position of the microprobe to specimen was monitored by the camera system, and the distance between the probe tip and working electrode was less than 100 μm. An ac disturbance signal of 10 mV peak to peak potential was applied to the electrode in a frequency range of 100 kHz–0.1 Hz.

### 2.4. SVET scanning

The SVET measurements were performed in a PAR Model 370 Scanning Electrochemical Workstation. A Pt-Ir microelectrode probe with a 50 μm micro-tip immersed in the solution was used and vibrated perpendicular to the working electrode surface. The probe was stepped across an area of 12,000 μm × 1000 μm above

**Table 1**
Time intervals for Gleeble thermomechanical simulation cycle parameters.

| Peak temperature | Heating rate | Holding time at peak temperature | Cooling time | | | |
|---|---|---|---|---|---|---|
| | | | $T_{max}$ to 800 °C | 800–500 °C | 500–300 °C | 300 °C to ambient |
| 1250 °C | 160 °C/s | 0 s | 5 s | 8 s | 15 s | 89 s |

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