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Effect of stress on electrochemical characteristics of pre-cracked ultrahigh strength stainless steel in acid sodium sulphate solution



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ABSTRACT

The electrochemical behaviors of ultrahigh strength stainless steel Cr12Ni5MoCo14 at crack tip under applied stress were studied by micro-electrochemical measurements as well as finite element analysis. The non-uniform distribution of the stress and strain induces a higher electrochemical activity at crack tip and promotes the anodic dissolution rate. The corrosion rate increases with increasing applied stress. In the elastic stress range, the effect of the applied stress on the electrochemical behaviors of Cr12Ni4Mo2Co14 steel is small. In the plastic stress range, the plastic deformation has a dramatic effect on the mechanical–electrochemical interaction and enhances the anodic activity.

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1. Introduction

Ultrahigh strength stainless (UHSS) steels are combined with the characteristics of high strength and high corrosion resistance, developed at the base of ultrahigh strength steel, such as 4340, 300M, AerMet 100 steel [1–4]. UHSS steels are always used as structure materials under complex stress/strain conditions in the curse of using. The additional stress and strain would enhance the corrosion process of UHSS steels, contributing to the decrease of service life and even safety accidents [5]. Some studies [6–10] show that the applied stress can promote the diffusion and accumulation of hydrogen at the defects and micro-cracks, and then leads to localized corrosion and expand of micro-cracks.

The electrochemistry and stress state at crack tip are very different from the region far away from the crack tip, and it is quite difficult to investigate in situ the corrosion behaviors of the crack tip due to the incapability of conventional electrochemical measurements [11–13]. So the electrochemical and corrosion behaviors of the specimens with a pre-crack by micro-electrochemical techniques have been attracted more and more notice. Sheng and co-workers [14] focused on the localized electrochemical characterization of 2024 aluminum alloy at crack tip in 3.5% NaCl by scanning Kelvin probe (SKP). Researchers found that, in addition to increasing the electrochemical activity at crack tip, the passive film was less stable than that on other region of the specimen

* Corresponding author. *E-mail addresses:* min.sun@139.com (M. Sun), lixiaogang@ustb.edu.cn (X. Li). surface. SKP results showed a non-uniform distribution of Volta potential on the pre-cracked wedge-open loading (WOL) specimen surface, indicating the preferential anodic dissolution and the accumulation of the corrosion product at crack tip. Cheng and the co-workers [15–18] have studied the occurrence of stress corrosion cracking of the pre-cracked pipelines, including X65, X70 and X100 steels, under a near-neutral pH condition. They found that the pre-strain enhanced both anodic and cathodic reactions and furthermore, the non-uniform distribution of plastic stress on specimens would initiate preferential localized corrosion and even cracks. There was a highest dissolution current density measured at crack tip. For the pre-cracked specimen, there was no detectable effect of the static elastic stress on electrochemical corrosion potential of the steel, but it becomes significant under plastic deformation.

The studies of the electrochemical corrosion behavior by microelectrochemical technology are mainly focused on pipelines and aluminum alloys, and the SCC mechanism is mainly hydrogen cracking and anodic dissolution [17,18]. The results showed that scanning vibrating electrode technique (SVET) and local electrochemical impedance spectroscope (LEIS) have advantages to study the electrochemical behaviors at crack tip on a pre-cracked specimen [19,20]. But there has been very little research on the studies of stainless steel, especially UHSS steels.

In this paper, the electrochemical behaviors of the pre-cracked UHSS steel Cr12Ni4Mo2Co14 steel were studied by LEIS and SVET, with respect to the difference of the current and impedance between the crack tip and the substrate. The finite element



analysis was used to study the distribution of the stress and stain around the specimen. The effect of the applied stress, including elastic and plastic stress, on the corrosion behavior of the crack tip was studied.

2. Experimental

2.1. Material and solution

Experimental materials in this work were cut from a sheet of UHSS steel, with the main chemical compositions (wt%): C 2.06, Cr 12.1, Mo 2.01, Co 13.9, Ni 4.27, Mo 2.01, V 0.51, W 0.72, Fe balanced. The UHSS steel was named Cr12Ni5Mo2Co14 for short. Cr12Ni5Mo2Co14 steel is heated by austenitizing at 1080 °C, oil quenching to room temperature followed by 1 h at 73 °C to transform retained austenite and finally tempered at 540 °C. The resulting yield strength and tensile stress are 1500 MPa and 1900 MPa. The microstructure of Cr12Ni5Mo2Co14 steel consists of lath/plant martensite with carbides distributed within the matrix and at the boundaries [21,22].

Acid sodium sulphate solution was used as the test solution. The composition was $0.05 \text{ M} \text{ Na}_2 \text{SO}_4$, and the solution pH was adjusted to 5 by sulfuric acid. All solutions were made from analytical grade $98\% \text{ H}_2 \text{SO}_4$ and ultra-pure water, and all the tests were carried out at room temperature (about 25 °C), at open to air.

2.2. LEIS measurement

The impendence characteristic of pre-cracked specimen was investigated by LEIS measurement. The micro-electrochemical measurements are performed at open-circuit potential of the constant-tensile specimen through a PAR Model 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. The tensile stress applied on the specimen was 0, 750 (50% $\sigma_{0,2}$) and 1500 MPa (100% $\sigma_{0.2}$) by a compressed spring force loader, as shown in Fig. 1, calculated based on the spring constant and the length change of the spring, as shown in Fig. 1(a). The schematic diagram of the tensile specimen is shown in Fig. 1(b). The thickness of the specimen is 1 mm and a notch with 1 mm length is cutted in the center of the tensile specimens, used as the start of a prefatigue cracks. The specimen is polished to 2000# and washed by deionized water and alcohol. After that a fatigue crack with 3 mm length is produced on the low cycle fatigue machine. The three-electrode system was used for the micro-electrochemical measurements, the pre-cracked specimen of Cr12Ni5Mo2Co14 steel as the working electrode, a saturated calomel electrode (SCE) as a reference electrode and the platinum microprobe as a counter electrode.

The LEIS scanning microprobe was operated in two modes: one mode was used for point-to-point local impedance measurements. The platinum microprobe with a 10 μ m tip was set directly above the specimen to measure the impedance responses at individual points. The distance between specimen surface and the probe tip was 50 μ m. During LEIS measurements, and an AC disturbance signal of 20 mV peak to peak potential was applied on the electrode. The measuring frequency ranged from 100 kHz to 0.1 Hz. The point-to-point LEIS measurements were carried on the pre-cracked specimen at point A (the crack tip) and B (the substrate, far away from crack tip).

The other mode of LEIS measurement was used for local impedance mapping, and the microprobe was stepped over a designated line (or area) of the specimen. The excitation frequency for impedance mapping measurements was fixed at 5 Hz. The impedance



Fig. 1. Schematic diagram of constant-strain tensile device (a) and the positions for electrochemical measurement (b).

distribution was determined within line A (at the crack tip) and line B (2500 μ m away from line A) with a stepped distance of 50 μ m. All the LEIS measurements were conducted at corrosion potential.

2.3. SVET measurement

The SVET measurements were performed in a PAR Model 370 Scanning Electrochemical Workstation. A Pt–Ir microelectrode probe with a 10 μ m micro-tip immersed in the solution was used and vibrated perpendicular to the working electrode surface. The SVET measurements were conducted to investigate the current distribution at crack tip, vibrating 100 μ m above the specimen surface at the amplitude of 30 μ m, with a vibrating frequency 80 Hz. The probe was stepped across a testet area of 3000 μ m \times 1500 μ m in the dashed-line frame above the specimen surface, as shown in Fig. 1(b). The step size was 50 μ m in the *X* and *Y* direction.

The potential of the micro-electrode was proportional to its position in the vibrating plane. The difference of potentials $\Delta \phi$, when the micro-electrode was located at the vibrating peak and valley, was measured by an electrometer incorporated in M370. The solution resistance *R* between the vibrating peak and valley is determined by R = d/k, where *k* is the solution conductivity, *d* is vibrating amplitude (30 µm). The SVET current density *i* was then obtained by Eq. (1) [23,24]:

$$i = -\frac{\kappa \cdot \Delta \phi}{d} \tag{1}$$

The current density plotted in the figures is actually a value based on the calculation show above. A flat SVET current density diagram means that there is a uniform electrochemical activity of the test electrode, while a fluctuating SVET diagram is associated with an electrode with non-uniform electrochemical activity [25]. Download English Version:

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