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Texture, local misorientation, grain boundary and recrystallization fraction in pipeline steels related to hydrogen induced cracking

M.A. Mohtadi-Bonab*, M. Eskandari, J.A. Szpunar

Department of Mechanical Engineering, University of Saskatchewan, 57 Campus Drive, S7N5A9 Saskatoon, Saskatchewan, Canada

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ABSTRACT

In the present study, API X60 and X60SS pipeline steels were cathodically charged by hydrogen for 8 h using 0.2 M sulfuric acid and 3 g/l ammonium thiocyanate. After charging, SEM observations showed that the hydrogen induced cracking (HIC) appeared at the center of cross section in the X60 specimen. However, HIC did not appear in the X60SS steel. Therefore, electron backscatter diffraction (EBSD) technique was used to analyze the center of cross section of as-received X60SS, X60 and HIC tested X60 specimens. The results showed that the HIC crack not only can propagate through ⟨100⟩||ND oriented grains but also its growth may happen in various orientations. In HIC tested X60 specimen, an accumulation of low angle grain boundaries around the crack path documented that full recrystallization was not achieved during hot rolling. Kernel Average Misorientaion (KAM) histogram illustrated that the deformation is more concentrated in as-received and HIC tested X60 specimens rather than in as-received X60SS specimen. Moreover, the concentration of coincidence site lattice (CSL) boundary in HIC tested X60 specimen was very low compared with other samples. The recrystallization area fraction in X60SS steel was very high. This high amount of recrystallization fraction with no stored energy is one of the main reasons for high HIC resistance of this steel to HIC. The orientation distribution function (ODF) of the recrystallized, substructured and deformed fractions in as-received X60SS and X60 steel showed relative close orientations in both as-received specimens.

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1. Introduction

Hydrogen induced cracking (HIC) in pipeline steel has been recognized as one of the most important modes of failure in sour environments. Hydrogen atoms can diffuse through the steel and cause cracking. Atomic hydrogen can be produced in various ways in pipelines. The main source of hydrogen comes from the surface corrosion of steel. The steel surface can be corroded in an acidic environment and hydrogen is generated by the reduction of hydrogen ions in this corrosion reaction. Several other possibilities, such as heat treatment, welding and certain service environments, can be additional ways that hydrogen can be generated and may enter the pipeline steel. It is worth-mentioning that hydrogen in its diatomic state cannot diffuse through the steel. Therefore, atomic hydrogen is adsorbed on the surface of steel and enters the body of metal. When hydrogen atoms accumulate at the structural defects, they may combine to form hydrogen gas. This process creates a high amount of pressure and eventually will cause cracking. Hydrogen cracks can be generated in the hydrogen environment even without external stress. However, when hydrogen is diffused through the steel, the elongation and reduction in the cross sectional area in HIC tested pipeline steel specimens are decreased. This phenomenon is called ductility loss which happens due to the presence of hydrogen. It has been shown that fracture during tensile testing in the presence of hydrogen occurs at stresses much lower than the ultimate tensile strength, sometimes even below the yield stress. Besides the HIC phenomenon, several theories were developed to explain the mechanism of hydrogen damage such as decohesion model, hydrogen enhanced localized plasticity model, hydride formation, internal pressure theory and surface adsorption theory [1–4]. Based on the above, the internal pressure theory is the most acceptable theory to explain the HIC phenomenon. Based on this theory, hydrogen atoms are accumulated between different structural defects, such as mixed oxide inclusions, manganese sulfide inclusions, carbides and nitrides, and defects in the metal matrix. When the pressure of hydrogen increases up to the yield stress of the metal by hydrogen molecule formation, cracks initiate. There are various factors affecting HIC phenomenon in sour environment. The microstructure of steel plays a key role in HIC susceptibility. Acicular ferrite is recognized as the most beneficial to HIC while the martensite structure makes steel highly susceptible to HIC

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* Corresponding author.
E-mail address: m.mohtadi@usask.ca (M.A. Mohtadi-Bonab).

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[5,6]. In other words, while acicular ferrite is a soft phase and can resist against the deformation developed by hydrogen, the martensite structure is very hard and brittle and therefore is prone to HIC crack propagation. The chemical composition of steel is another factor which affects HIC susceptibility. Pipeline steels do not have a uniform distribution of chemical elements through the thickness of the pipe body. During the casting process, the outer surfaces of steel plates solidify and elements with low melting points are rejected to the center of the thickness. Therefore, the center segregation zone which is a result of an inhomogeneous distribution of elements through the cross section has a higher hardness than other regions and is prone to HIC cracking. Due to the center segregation zone and high density of inclusions at the center of the cross section, all of HIC cracks in the pipeline steels nucleate and propagate in this area. Moon et al. also investigated HIC in pipeline steel and found out that centerline segregation zone with higher hardness values than other regions is very prone to cracking [12,13]. In addition, Matsumoto et al. studied the HIC susceptibility on high strength pipeline steel and showed that the hardness of the segregation zone in steel plate is very important factor in increasing HIC susceptibility [14]. Tehemiro et al. also investigated HIC in pipeline steel and found out that the effect of center segregation zone on HIC susceptibility can be removed by using thermo-mechanical control processing (TMCP) [16].

Crystallographic texture is considered as one of the main factors that plays a very important role in HIC crack propagation. It is well-accepted that a {111} dominant texture makes steel highly resistant to HIC while a {100} dominant texture increases HIC susceptibility by increasing the number of easy paths for HIC crack propagation. Not only, the role of texture is important in HIC related failure, but it also plays a key role in stress corrosion cracking (SCC) in pipeline steels. In an interesting study, it was concluded that the boundaries linked to the grains with ⟨110⟩||ND and, to some extent, ⟨111⟩||ND orientations provided high resistance paths to intergranular SCC. However, boundaries linked to the grains with ⟨100⟩||ND orientations make pipeline steel highly susceptible to HIC [7]. There are also several studies in the literature that focused on the role of texture on HIC in pipeline steels. It has been implied that the ⟨111⟩||ND oriented grains decrease the number of trans and intergranular cleavage paths. Additionally, coincidence site lattice (CSL) boundaries and low angle grain boundaries (LAGBs) between grains with the dominant ⟨100⟩||ND orientations can improve HIC resistance [8–10]. In a recent study, several pipeline steel specimens with similar microstructure but different crystallographic texture were subjected to the HIC test [11]. The results of this study showed that the warm rolled samples with a final rolling temperature at 600 °C and 800 °C, with the {111} dominant fiber texture, had very high resistance to HIC and no HIC cracks were observed after the HIC test. In conclusion, crystallographic texture, beyond the traditional methods, can increase the resistance of pipeline steel to HIC.

The effect of different microstructural parameters such as the local misorientation of grains, CSL boundaries and the role of the recrystallization fraction in HIC related failure has been less considered in the literature. The current study focused on investigating the effect of different microstructural parameters on HIC susceptibility. Therefore, two types of pipeline steels with almost the same mechanical properties but different chemical composition and texture were selected to investigate the microstructural and textural differences between susceptible (X60) and non-susceptible (X60SS) steels to HIC phenomenon. The effect of the grain orientations, Kernel Average Misorientation (KAM) angle, CSL boundaries and recrystallized, substructured and deformed fractions in HIC crack propagation was discussed. Specimens were subjected to a hydrogen charging test in an acidic environment and SEM and EBSD techniques were used to analyze the tested specimens.

2. Experimental procedure

2.1. Tested materials

Two different types of pipeline steels, API X60 and X60SS, were examined in this study. The chemical composition of both steels is presented in Table 1. The rolling, transverse and normal directions of steels were named as RD, TD and ND, respectively.

2.2. Electrochemical hydrogen charging

In order to induce hydrogen cracks in both steel specimens, we charged both X60 and X60SS pipeline specimens with hydrogen using 0.2 M sulfuric acid and 3 g/l ammonium thiocyanate. The following reactions are occurred during hydrogen charging to produce hydrogen:

$$\text{Oxidation at anode: } 2H_2O(l) \rightarrow O_2(g) + 4H^+(aq) + 4e^- \quad (1)$$

$$\text{Reduction at cathode: } 4H^+(aq) + 4e^- \rightarrow 4H^\circ \rightarrow 2H_2(g) \quad (2)$$

$$\text{Overall chemical reaction: } 2H_2O(l) \rightarrow 2H_2(g) + O_2(g) \quad (3)$$

It is notable that most of the hydrogen in the form of hydrogen bubbles (molecules) goes out from the solution and does not diffuse through the steel. However, some of the hydrogen atoms are diffused through the steel. Ammonium thiocyanate acts as a hydrogen recombination poison and prevents hydrogen gas formation on the steel surface. In other words, the hydrogen recombination poison increases the amount of hydrogen inside the steel specimens.

Three specimens from both X60 and X60SS steels with dimensions of 130(TD) × 25(RD) × 6(ND) mm and 130(TD) × 25(RD) × 9(ND) mm were cut from pipeline plates. The samples were polished on all surfaces with 600 grit SiC emery paper at the final stage. The specimens were washed with distilled water and then ultrasonically degreased with acetone for 30 min. The steel specimens were placed separately in a glass test vessel and filled with 2 L of charging solution. An Instek type power supply was used to provide a constant current density of 20 mA/cm^2. The charging solution test vessel was firmly covered with Para film to avoid evaporation of the charging solution. All specimens were cathodically charged for 8 h.

2.3. EBSD measurements

Each charged and as-received specimen with the above mentioned dimensions were sectioned to three equal parts from the transverse direction. Then, the cross sections of the specimens were polished with 1 μm diamond paste at the final stage of polishing. After manual polishing, the samples were vibrometery

Table 1
Chemical composition of as-received X60 and X60SS pipeline steels (wt%).

Pipeline steels	C	Mn	Si	Nb	Mo	Ti	Cr	Cu	Ni	V	S	P	N
X60	0.052	1.50	0.15	0.067	0.096	0.022	0.07	0.18	0.19	0.001	0.0027	0.007	0.009
X60SS	0.027	1.26	0.16	0.045	0.016	0.010	0.08	0.12	0.05	0.066	0.0006	0.007	0.0083

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