Effect of tempering temperature and inclusions on hydrogen-assisted fracture behaviors of a low alloy steel

Xinfeng Li [a,*], Jin Zhang [b], Sicong Shen [a], Yanfei Wang [c], Xiaolong Song [a,*]

[a] State Key Laboratory for Mechanical Behavior of Materials, School of Materials Science and Engineering, Xi'an Jiaotong University, Xi'an 710049, China
[b] Department of Geosciences, Center for Materials by Design, and Institute for Advanced Computational Science, State University of New York, Stony Brook, NY 11794-2100, USA
[c] School of Chemical Engineering & Technology, China University of Mining and Technology, Xuzhou 221116, China

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ABSTACT

The tensile properties and fracture behaviors of the pre-charged hydrogen low alloy steel subjected to various tempering temperatures were investigated by slow strain rate tensile tests. Hydrogen was introduced into the samples by electrochemical method in 0.5 mol L^{-1} NaOH solution with 1 g L^{-1} CH_4N_2S for 24 h at room temperature (298 K). The results show that with an increase in tempering temperature, the resistance to hydrogen embrittlement increases. For the sample tempered at 200 °C, hydrogen-assisted microcracks initiate at the lath boundaries and the interfaces between inclusions and matrix. Moreover, the void around the inclusion initiates at the matrix-inclusion interface and grows in the form of the interface decohesion for hydrogen free sample, while the void also starts at the interfaces and propagates into the steel matrix along vertical tensile stress direction in the presence of hydrogen. Fractography reveals that for hydrogen-charged samples, the crack originates from mixed O-Si-Al-Ca inclusions in the samples tempered at 200 °C and 400 °C, while it is not located at inclusions in the sample tempered at 650 °C.

1. Introduction

In the presence of hydrogen, the mechanical properties degradation of the steel is known as hydrogen embrittlement. The sources of hydrogen invasion into steel contain smelting, weld, electroplating, corrosion and hydrogen environment in service. Once the hydrogen atoms invade the steel, they interact with crystal defects, such as grain boundaries, dislocations and vacancies, and induce the premature failure of the steel. At present, the acceptable hydrogen embrittlement mechanisms include hydrogen-enhanced decohesion (HEDE) mechanism, hydrogen-enhanced localized plasticity (HELP) mechanism and hydrogen-enhanced strain-induced vacancies (HESIV) mechanism. The HEDE mechanism supposes that hydrogen decreases the cohesion strength across cleavage planes and grain boundaries. For HELP mechanism, the failure occurs by locally ductile process and hydrogen enhances the mobility of dislocations. Moreover, HESIV mechanism maintains that hydrogen enhances deformation-induced creation and agglomeration of vacancies, resulting in accelerating fracture process.

The suscepibility to hydrogen embrittlement of the steel depends on microstructure. Kim et al. [1] revealed that at the same tensile strength level ~1600 MPa, the steel with a fully pearlitic structure was more beneficial to suppresss hydrogen delayed fracture in comparison

with a tempered martensitic structure steel. Fracture surface observation showed that the tempered martensitic steel was fractured in an intergranular brittle manner while the fully pearlitic steel exhibited the dimple ductile fracture mode. Li et al. [2] suggested that an increasing in austenite content can reduce hydrogen embrittlment sensibility. Kang et al. [3] reported that the resistance to hydrogen embrittlement was improved with increasing the spheroidization rate of fine carbides. The spheroidization of carbides acted as a barrier to the initiation and propagation of microcracks, whereas needle-like and long shape of carbides facilitated the microcrack propagation. Moreover, it is feasible that nano-scale precipates in steel can enhance the resistance to hydrogen embrittlement, such as Cu-rich precipates [4] and Cr_2O_3 particles of 10 nm [5].

Inclusions, which act as irreversible hydrogen traps, play the critical role in hydrogen embrittlement. Gojić et al. [6] reported that the initiation of fracture occurred at complex MnS inclusions/matrix interfaces and the steel with lower mechanical properties and content of non-metallic inclusions exhibited better resistance to hydrogen embrittlement. However, Schiapparelli et al. [7] revealed that the crack initiation in hydrogenated specimens was associated with undeformable inclusions, such as Al_2O_3, whereas interfaces of deformable inclusions were never associated with crack initiation. They suggested

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* Corresponding author.
E-mail addresses: lixinfeng@stu.xjtu.edu.cn (X. Li), songxl@mail.xjtu.edu.cn (X. Song).

that a decrease in the hard inclusions content rather than MnS inclusion content was important to improve the susceptibility of the steel to hydrogen damage. Li et al. [8] also demostrated that the hydrogen-assisted crack initiated from the inclusions enriching O-Al-Ca-Si in PSB1080 steel during slow strain rate tensile tests. In addition, hydrogen-assisted cracks were also found in Ti(N, C) inclusions, indicating that this type inclusions served as another crack origin sites [9–11]. Thus, it is necessary to further investigate the relationship between inclusions and hydrogen-assisted crack.

PSB1080 high strength steel bars are widely used in the wind turbine foundation, which suffer from failures due to hydrogen embrittlement [12]. Starting from these insights, slow strain rate tensile tests were performed with the objective of researching the effect of tempering temperature and inclusions on tensile fracture behaviors of hydrogen free (HF) or hydrogen charged (HC) low alloy steel bar. The hydrogen embrittlement susceptibility of the samples at various tempering temperature was analyzed. After tensile tests, the samples normal to the fracture surfaces were extracted by wire electrical discharge machining, mechanical polished, etched and then investigated by scanning electron microscopy to unravel underlying crack nucleation and secondary cracks. In addition, fractography of the HF and HC samples were observed and discussed in detail.

2. Experimental procedures

2.1. Materials preparation and microstructure analysis

The chemical composition of the steel bars is shown in Table 1. The steel bars with a diameter of 40 mm were austenitized at 900 °C for 1.8 ks (0.5 h), cooled in water and then tempered at 200 °C, 400 °C and 650 °C, respectively, for 3.6 ks (1 h). The corresponding specimens were designated as the T200, T400 and T650 samples respectively. After heat treatment, the smooth cylindrical tensile specimens with a gauge size of 5 mm diameter ×25 mm length were machined with axial direction paralleling to the longitudinal direction of steel bars. Meanwhile hydrogen permeation specimens with a size of 40 mm diameter ×0.5 mm thickness were also prepared by electrical discharge machining from the steel bar. The thickness direction of hydrogen permeation samples paralleled the steel bar longitudinal direction. Subsequently, all surfaces of the specimens were mechanically ground to obtain a finish equivalent to that achieved using 800 grit carbide silicon. These specimens were rinsed with deionised water and then degreased with alcohol.

To seek microstructural observation, the specimens were ground with carbide silicon paper and polished with 0.5 μm diamond paste. Prior to the etch, the distribution of the inclusions on the non-etched clean steel surfaces was observed. Subsequently, the microstructures of the samples etched with 4% nital solution were characterized by field emission scanning electron microscopy (Hitachi SU6600 FESEM) and energy dispersive spectroscopy (EDS) with the operating voltage 15 kV. In addition, some thin sheet samples with a size of 8 mm diameter and 0.4 mm thickness were cut from the alloy, mechanically grinded to about 80 μm by sand paper and then electrochemically thinned by a twin-jet machine in 10 vol%) perchloric acid and 90vol%) ethanol solution at −30 °C. JEM-200CX transmission electron microscopy (TEM) was used to investigate the evolution of submicrostructure at different tempering temperatures.

2.2. Electrochemical hydrogen charging

One group of tensile specimens at various heat treatment conditions was electrochemically charged with hydrogen in 0.5 mol L^{-1} NaOH solution at current density of 1 mA cm^{-2} at room temperature (298 K). The CH$_4$N$_2$S (1 g L^{-1}) was added in the solution as a hydrogen recombination poison. To ensure an equilibrium and constant hydrogen content throughout the specimen, electrochemical hydrogen charging time was 24 h, which was approximately determined by following equation [13]:

$$t_{0.9} \approx 0.334 \frac{R^2}{D} \tag{1}$$

where R was the radius of a cylinder specimen, D was the diffusion coefficient. The time $t_{0.9}$ can be roughly considered as the time necessary to attain the steady state concentration gradient in the cylinder. Platinum was used as an anode and the specimen was used as a cathode. In order to ensure that only the gauge section of specimens was charged with hydrogen, the surfaces of the other parts were covered with paraffin.

2.3. Electroplated cadmium tests

In order to prevent hydrogen releasing from the hydrogen charged samples, electroplated cadmium on the sample surface was carried out [14]. After charging hydrogen, electroplated cadmium was carried out immediately. Protective cadmium plating was performed in an aqueous solution with 98% oil of vitriol (50 g L^{-1}), CdSO$_4$ powder (50 g L^{-1}), Na$_2$SO$_4$ (45 g L^{-1}), glutin (6 g L^{-1}) and phenol (3 g L^{-1}) at the current density of 25 mA cm^{-2} for 5 min. The cadmium was used as an anode and the specimen was used as a cathode.

2.4. Slow strain rate tensile tests

After electroplated cadmium, tensile tests were immediately conducted on these charged specimens at a crosshead speed of 0.03 mm min^{-1}, corresponding to a normal strain rate of 2×10^{-5} s^{-1}. Meanwhile, corresponding specimens without hydrogen charging were also tensile tested as a reference. Tensile tests were conducted three times for each HF and HC specimen to check the reproducibility. The index of relative susceptibility to hydrogen embrittlement (HEI) of specimens was determined by measuring the relative plasticity loss of the specimens, which can be expressed as:

$$\text{HEI} = \frac{\varphi_0 - \varphi_H}{\varphi_0} \times 100\% \tag{2}$$

$$\phi_0 = \frac{A_0 - A_1}{A_0} \tag{3}$$

$$\phi_H = \frac{A_0 - A_{1H}}{A_0} \tag{4}$$

where φ_0 and φ_H were the reduction of area of HF and HC specimens, respectively. A_0 was the original cross section area of tensile sample, A_1 was the cross section area of HF sample at necking after fracture, A_{1H} was the cross section area of HC sample at necking after fracture.

Table 1
Chemical composition of the steel (wt%).

Element	C	Si	Mn	P	S	Cr	Ni	Cu	Al	Fe
	0.25	1.41	2.19	0.014	0.0049	1.07	0.011	0.018	0.015	Balance