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## Relationship between microstructure and hydrogen induced cracking behavior in a low alloy pipeline steel

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### ABSTRACT

Hydrogen induced cracking (HIC) behaviors of a high strength pipeline steel with three different microstructures, granular bainite & lath bainite (GB + LB), granular bainite & acicular ferrite (GB + AF), and quasi-polygonal ferrite (QF), were studied by using corrosion experiment based on standard NACE TM 0284. The HIC experiment was conducted in hydrogen sulfide (H<sub>2</sub>S)-saturated solution. The experimental results show that the steel with GB + AF and QF microstructure present excellent corrosion resistance to HIC, whereas the phases of bainite lath and martensite/austenite in LB + GB microstructure are responsible for poor corrosion resistance. Compared with ferrite phase, the bainite microstructure exhibits higher strength and crack susceptibility of HIC. The AF + GB microstructure is believed to have the best combination of mechanical properties and resistance to HIC among the designed steels.

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### 1. Introduction

During transportation of oil and gas, the corrosion problem is very intractable in some cases [1,2]. Pipeline steels are usually used to transport oil and gas from the oil and gas field to the users. The effective control of corrosion is important because the failure of pipeline steel will cause unexpected accidents. H<sub>2</sub>S, together with H<sub>2</sub>O, is the main electrolyte, causing corrosion in oil and gas production [3]. Hydrogen induced cracking (HIC) is clearly known as one of the significant failure modes in the wet corrosive environment [4–6]. The combination of high strength, high toughness at low temperature, and high HIC resistance to sour environments is necessary for the development of pipeline steels. Thus, it is a great challenge for pipeline steels to possess both high strength and excellent corrosion resistance to HIC [7]. So far, some researchers have made efforts to improve the resistance to H<sub>2</sub>S corrosion for pipeline steel [8–10].

In wet H<sub>2</sub>S environment, hydrogen atoms can combine into hydrogen molecule, resulting in high hydrogen pressure in steel. Hydrogen decreases the local atomic cohesive force of steel [7]. Many studies have been conducted concerning the effects of inclusions, chemical compositions and microstructure on the HIC susceptibility [11–14]. HIC resistance depends on several fac-

tors, such as sulfur content, type and morphology of inclusions, microstructure variation, grain size, grain boundary, and precipitates [15]. The crack arrestability in bainite microstructure is not due to the fine grain structure but to the high density of the dislocations, together with the deformation-induced low-angle subgrain boundaries [16]. Defects, such as vacancy, dislocation, grain boundaries and phase interfaces, are considered as trap sites for hydrogen isotopes in iron [17]. The phase interfaces have the largest trapping energy, and then the grain boundaries, single vacancy, dislocation and interstitial atoms. The segregation of hydrogen at the grain boundaries is energetically stable, so the diffusion of hydrogen at the grain boundaries is slower than that in the lattice. In general, with increasing strength level, HIC susceptibility deteriorates high strength pipeline steel [8]. The ferrite and pearlite steels are gradually replaced by low carbon bainitic steel in corrosion environment due to their high strength and toughness [18].

However, the above-mentioned studies do not provide much information about HIC susceptibility on high strength pipeline steel with different phases in various strength levels. In present work, the corrosion behaviors of HIC resistance for three high strength pipeline steels with different kinds of microstructures were studied by using immersion experiment in H<sub>2</sub>S-containing environment.

### 2. Experimental

The steel used in this study was melted in a vacuum induction furnace of 150 kg capacity and forged into slab with cross-section

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**Table 1**  
Chemical composition of the experimental steel (mass fraction, %).

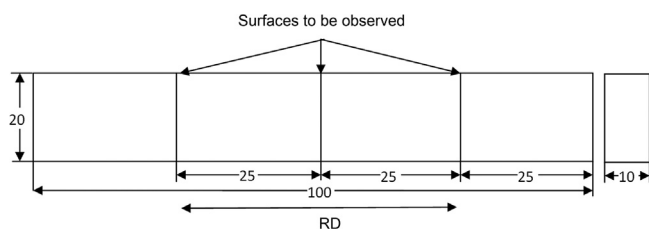
C	Si	Mn	P	S	Ni	Cr	Cu	Mo	Nb	Ti	Al	Fe
0.05	0.20	1.50	0.005	0.001	0.20	0.30	0.30	0.20	0.04	0.01	0.03	Bal.

**Table 2**  
Processing parameters in the TMCP for the experimental steel.

Steel	Rough rolling		Finish rolling		Accelerated cooling	
	Start rolling temp. (°C)	Finish rolling temp. (°C)	Start rolling temp. (°C)	Finish rolling temp. (°C)	Finish cooling temp. (°C)	Cooling rate. (°C s <sup>-1</sup> )
QF	1174	1112	819	810	500	24
GB	1112	1022	809	805	450	24
LB	1147	1105	819	807	300	72

**Table 3**  
Mechanical properties of the experimental steels.

Steel	R <sub>t0.5</sub> (MPa)	R <sub>m</sub> (MPa)	R <sub>t0.5</sub> /R <sub>m</sub>	A <sub>50</sub> (%)	A <sub>k</sub> (J) (-20 °C)
QF	557	705	0.79	22	293
GB	600	695	0.87	22	279
LB	680	933	0.73	17	175

**Fig. 1.** Schematic diagram of the specimen for HIC experiment and the surfaces to be observed.

dimension of 110 mm × 100 mm. The chemical composition of this steel is shown in Table 1.

The slab was firstly homogenized at 1200 °C for 1 h, and then hot rolled into plate of 12 mm in thickness after 9 passes in a laboratory rolling mill. The three different plates were obtained by thermo-mechanical controlled processing (TMCP). Those plates were prepared by controlling the deformation temperature, cooling temperature and cooling rate. The processing parameters in the TMCP is shown in Table 2.

Metallographic samples were mechanically polished and etched using 4% nital (nitric acid-ethanol) solution. Microstructures of hot rolled samples were observed by scanning electron microscopy (SEM), electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM). Qualitative microanalysis of inclusions and precipitates were carried out by energy dispersive spectroscopy (EDS).

Tensile specimens with a gage size of 8 mm in diameter and 50 mm in length were cut from the middle section of rolled plates along transverse direction. The tensile experiment was conducted at room temperature with the speed of 3 mm/min in an INSTRON-4206 universal tensile machine. The Charpy V-notch impact toughness was measured at -20 °C and the specimens have a size of 10 mm × 10 mm × 55 mm, which were machined from rolled plates along transverse direction according to standard ASTM A370.

HIC experiment was based on the standard NACE TM 0284-2003. The specimens have a size of 100 mm (Rolling Direction, RD) × 20 mm (Transverse Direction, TD) × 10 mm (Normal Direction, ND) mm and were cut from rolled steel plates, ground with grit paper and ultrasonically degreased with acetone. The specimens were put into glass test vessel filled with corrosion solution of 0.5 mass% CH<sub>3</sub>COOH, 5 mass% NaCl and 94.5 mass% distilled water.

Nitrogen gas was purged into the solution for 1 h to remove oxygen. Then H<sub>2</sub>S was injected into the corrosion solution until the solution was saturated. The specimens were corroded for 96 h. The temperature was at 25 °C. The pH values of the solution at the start and the end of the experiment were 2.9 and 3.8, respectively. Fig. 1 shows the schematic diagram of tested specimen and the surfaces to be observed after experiment. The TD-ND planes of sectioned specimens were polished and etched with 4% nital solution. The obtained surfaces were observed with OM to show HIC cracks. The value of crack sensitivity ratio (CSR), crack length ratio (CLR), and crack thickness ratio (CTR) were calculated according to Eqs. (1)–(3) as follows:

$$CSR = \frac{\sum(a \times b)}{(W \times T)} \times 100\% \quad (1)$$

$$CLR = \frac{\sum a}{W} \times 100\% \quad (2)$$

$$CTR = \frac{\sum b}{T} \times 100\% \quad (3)$$

where  $a$  is the cracking length,  $b$  represents the crack thickness,  $W$  is the specimen width,  $T$  is the specimen thickness.

### 3. Results and discussion

#### 3.1. Morphology and microstructure

The SEM microstructures of the experimental steels were presented in Fig. 2. The microstructure in Steel QF is of quasi-polygonal ferrite (QF) and the grain size is about 5 μm (Fig. 2(a)). Steel GB shows granular bainite (GB) and acicular ferrite (AF) microstructure (Fig. 2(b)). The microstructure in Steel LB consists of granular bainite (GB) and lath bainite (LB) together with a small fraction of acicular ferrite constituents (Fig. 2(c)). The volume fraction of granular bainite is much higher than that of lath bainite in Steel LB. The constituents of martensite and austenite (M/A island) are distributed in ferrite matrix. Fig. 2 also shows the distribution of the M/A islands in these three steels and their volume percentages of the M/A islands are estimated to be 1.5%, 14.9% and 22.6%, respectively, by statistical image analysis.

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