LETTER

## Microstructural effect on near-neutral pH stress corrosion cracking resistance of pipeline steels

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Near-neutral pH stress corrosion cracking (SCC) of pipelines occurs in a dilute, deaerated bicarbonate environment with pH ranging from 6 to 7. This environment is generated underneath the polyethylene tape coatings that disbonded from the external pipe surface and shield the pipe from the cathodic protection [1, 2]. The analysis indicated the crack growth rates are some 30% higher in the heataffected zone next to the pipe weld [3], indicating that the steel microstructure may affect the near-neutral pH SCC development of pipelines. Unfortunately, the microstructural effect is yet poorly understood. One of the important features of this transgranular SCC is that there are substantial anodic dissolution at the crack enclaves [1, 2]. Therefore, the anodic dissolution is likely to play a role in SCC. An investigation on the microstructural effect on the anodic dissolution is helpful to understand the SCC mechanism.

Test materials were API X–70 and AISI 1018 steels. Their compositions are given in Table 1. Various heat treatments were used to achieve various microstructures and a wide range of hardness, as listed in Table 2. It is noted that the samples cut from the edge and center of X70 steel skelp have different hardness because of their different grain size. The average grain size of the former is about 5.8  $\mu$ m and that of the latter is 7.4  $\mu$ m. Typical microstructures are illustrated in Figs. 1 and 2. The polarization resistance measurements were conducted in the simulated Canadian groundwater, NS4 solution, with a composition of (g/L) 0.122KCl, 0.483NaHCO<sub>3</sub>, 0.137CaCl, 0.131MgSO<sub>4</sub>·7H<sub>2</sub>O.

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Department of Chemical and Materials Engineering, University of Alberta, T6G 2G6 Edmonton, AB, Canada e-mail: Jingli.luo@ualberta.ca During testing, the solution was bubbled with  $95\%N_2/$ 5%CO<sub>2</sub> to create an anaerobic environment and a pH of 6.7. The tests were started after the solution was deaerated for approximately 4 h. Polarization resistance was measured by scanning potential from -10 mV to +10 mV relative to open circuit potential (OCP) and the scanning rate was 0.125 mV/s. The test results were confirmed with the electrochemical impedance spectroscopy (EIS) which were run from 100 kHz to 0.002 Hz with an amplitude of 10 mV AC about OCP. The SCC tests were carried out with the slow strain rate tensile (SSRT) technique. The specimens had a gauge length of 25 mm and a diameter of 3 mm. The strain rate adopted was  $3 \times 10^{-7}$ . According to Parkins [1, 2], the SCC resistance defined as the ratio of reduction in area ( $RA_{SCC}/RA_{air}$ ), where  $RA_{SCC}$  and  $RA_{air}$  are the reduction in area measured in the corrosive medium and air, respectively.

As summarized in Table 2, the microstructures of steels can be roughly divided into two groups. One has, basically, a single phased structure, such as X70 steel under as-rolled, quenched conditions and quenched 1018 steel. Another has a bi-phased ferrite + pearlite structure, such as the steels under annealed and normalized conditions. The SCC data in Fig. 3 show that such a difference in microstructure can affect the SCC resistance. Generally, the SCC resistance decreases with the yield strength of X70 steel if the steel has the same microstructure. However, the relationship of the SCC resistance and the yield strength is microstructure-dependent. When steels possess the same yield strength, those with uniform bainitic ferrite or polygonal ferrite structure are more resistant to SCC than those having ferrite + pearlite structure. This agrees with the SSRT test results [4] and the threshold cyclic stress to initiate crack from notches [5], which were collected from a range of pipelines from X52 to X100 **Table 1** Nominal compositionsof the tested steels

Steel	Weight percent (wt%)						
	С	Mn	Р	S	Others	Fe	
X70	0.04	1.45	0.008	0.003	<1	Bal.	

 Table 2
 Heat-treatment and microstructure of test materials

Material	ID	Heat-treatment	$3 \times Microstructure$	Hv
X70	X70E	As-rolled, edge of skelp	Bainitic ferrite	228
	X70C	As-rolled, center of skelp	Bainitic ferrite	217
	X70A	950 °C/1 h/furnace cooled	Ferrite + pearlite	133
	X70N	950 °C/1 h/air cooled	Ferrite + pearlite	156
	X70O	950 °C/1 h/water guenched	Bainitic ferrite	231
	X70QT	950 °C/1 h/water quenched + 650 °C tempered	Polygonal ferrite	180
1018	1018A2	1200 °C/1 h/ furnace cooled	Ferrite + pearlite	148
	1018A1	890 °C/1 h/furnace cooled	Ferrite + pearlite	178
	1018N	890 °C/1 h/air cooled	Ferrite + pearlite	205
	1018O	890 °C/1 h/water guenched	Martensite	278
	1018QT1	890 °C/1 h/water quenched + 475 °C tempered	Tempered martensite + fine carbide	265
	1018OT2	890 °C/1 h/water guenched+540 °C tempered	Tempered martensite + carbide	230
	1018QT3	890 °C/1 h/water quenched + 675 °C tempered	Spheroidite	200

Fig. 1 Typical microstructure of the as-received X-70 pipeline steel, (**a**) annealed, (**b**) normalized, (**c**) quenched, (**d**) quenched and tempered, (**e**) asrolled



Fig. 2 Microstructure of 1018 steel, (a) 1018A2, (b) 1018A1, (c) 1018N, (d) 1018Q, (e) 1018QT1, (f) 1018QT2, (g) 1018QT3





Fig. 3 Relationship between SCC resistance and yield strength of X70 steel

grades under various heat-treatment conditions. Recent experimental research in our laboratory showed that, because of the difference in microstructures, X70 steel was more resistant to crack initiation in the near-neutral pH groundwater, compared with X65 steel which had a ferrite + pearlite structure, although the yield strength of both were very close [6]. The data in Fig. 3 indicate also that the bainitic structure obtained in X70 steel by the waterquench is less resistant to SCC, compared with material in the as-rolled condition, although water-quenching treatment raises the yield strength. In fact, the formation of pearlite in X70 steel is suppressed by the water-quenching and only a small amount of fine carbides precipitate in the microstructure during the temper treatment. As shown in Fig. 3, the temper after the water-quenching can improve the SCC resistance. The same phenomenon was also observed in X80 steel [7]. It suggests that the high susceptibility of steel in the water-quenched condition might result from the high micro-stresses produced by the excess carbon trapped interstitially [8]. X70QT has a coarsegrained polygonal ferrite structure but SCC resistance is little better than that of the same steel with fine-gained bainitic ferrite, implying that the observed poor SCC resistance of steels with ferrite + pearlite structure may relate to the pearlite in the microstructure. It has been observed that small cracks were more likely to initiate and to propagate in the pearlite colonies or along the boundary of pearlite/ferrite [9]. One possible reason is that the quenched + tempered treatment produces a uniform microstructure while pearlite colonies in the ferrite + pearlite structure are likely to promote non-uniform microplastic deformation [5], since pearlite possesses higher strength than ferrite. Recent research of ours indicated that both the metallurgical and environmental factors which enhanced the local plastic deformation would accelerate the development of near-neutral pH SCC of pipeline steels [10]. Beavers et al. [3] pointed out that the micro-hardness in SCC zones on the pipes was slightly higher than that in non-SCC zones. For steel with the ferrite + pearlite structure, SCC zones are likely to contain more pearlite because the pearlite is harder than ferrite.

To confirm this statement, the fast screen test proposed by Lu and Luo [4] is used to further evaluate the effects of metallurgical factors. As indicated by Fig. 4, a linear relation is approximately held between the SCC resistance ( $RA_{SCC}/RA_{AIR}$ ) and the polarization resistance ( $R_p$ ) of pipeline steels measured in an anaerobic NS4 solution. This is due to the fact that around the OCP, both the ingress of hydrogen and anodic dissolution promoted the development of near-neutral pH SCC [10]. The reactions of anodic dissolution and cathodic reduction of hydrogen in the near-neutral pH SCC couple together, any factor enhancing the anodic dissolution may increase the likelihood of hydrogen ingress into the steel. Because the anodic dissolution rate at the open circuit potential is reversed proportional to the polarization resistance, the linear relationship between the RA-ratio and polarization resistance implies that anodic dissolution may play an important role in the near-neutral pH SCC around the open circuit potential. The linear relationship between hardness and yield strength of steels has been well recognized [11]. Therefore, the dependence of near-neutral pH SCC resistance on yield strength can be assessed using the relationship of Hv vs.  $R_p$ .

The data in Fig. 5 show that, the polarization resistance in the deaerated NS4 solution decreases, in general, with hardness and that, when the hardness is same, the steels with uniform single phased structures have higher  $R_{\rm p}$ values than those with a multiphase (ferrite + pearlite) structure. This implies that the conclusion obtained from SCC test data of X70 steel is also applicable to the other pipeline steels in the near-neutral pH groundwater. A martensite structure can be obtained by water quenching and the hardness decreases and polarization resistance increases with rising temper temperature, as indicated by the dashed line in Fig. 5. When the temper temperature is relatively low (475 °C), the carbide precipitates are fine and the relation of polarization resistance vs. the hardness follows the pattern of single phased steels. As temper temperature is raised, both the amount and size of carbides increase, the trend of polarization resistance vs. the hardness coincides with that of steels with ferrite + pearlite structure. The data in Fig. 5 also suggest that the difference in polarization resistance of steels with different microstructures is reduced with carbon content and hardness.

In summary, pipeline steels with a microstructure of fine-grained bainite + ferrite have a better combination



Fig. 4 Relationship between SCC resistance and yield strength of X70 steel



Fig. 5 Relationship between hardness and polarization resistance

of strength and SCC resistance than those with ferrite + pearlite structures.

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