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# Effect of pre-charged hydrogen on fatigue crack growth of low alloy steel at 288 °C

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#### Abstract

Hydrogen effects on mechanical strength and crack growth were studied at high temperatures. The study was motivated by the fact that the environmentally assisted cracking (EAC) of pressure vessel steel SA508 Cl.3 in 288 °C water was suspected to be related to hydrogen embrittlement. Fatigue crack growth rate and tensile tests were performed with hydrogen pre-charged specimens at high temperatures. At 288 °C the fatigue crack growth rate of the hydrogen pre-charged specimen was faster than that of as-received; the fatigue fracture surface of hydrogen pre-charged specimen correspondingly showed EAC like feature. Meanwhile, ductile striation was evident for the case of as-received in both air and argon gas environments. In the dynamic strain aging (DSA) loading condition at 288 °C during tensile tests, the pre-charged hydrogen induced a marked softening (decrease in ultimate tensile strength; UTS) as well as a little ductility loss; this was accompanied by the macrocracks grown from microvoids/microcracks promoted by DSA and hydrogen. These experiments showed that hydrogen embrittlement is an effective mechanism of EAC not only at low temperature but also at the high temperature.

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# 1. Introduction

It has been reported [1–5] that the crack growth rate of pressure vessel steel in 288 °C aerated water environments is enhanced by a electrochemical potential gradient between crack mouth and crack tip, which can result in concentrating sulfide ions at the crack tip to cause environmentally assisted crack-ing (EAC) [6]. The concentrated sulfur at the tip enhances the crack growth rate through increasing not only dissolution rate but hydrogen absorption rate.

The former favors models based on anodic dissolution [7]; the latter favors models based on hydrogen embrittlement [1–5]. For the model studies, numerical models mainly based on anodic dissolution have been developed to predict crack growth rate in various systems with reasonable accuracy. Meanwhile, the models based on hydrogen embrittlement well explain fracture features although few numerical models have been established due to its complexity.

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0921-5093/\$ - see front matter © 2006 Elsevier B.V. All rights reserved. doi:10.1016/j.msea.2006.01.080 It was found that the cracking in an aerated high temperature was associated with microcracks formed by strain localization on slip bands and the microcracks were linked to the main crack in brittle manners [8]. This would hardly be explained by the mechanism based on anodic dissolution alone. Based on observations of the fracture surface [1–4] and of the sectioned area [8] of the fatigue specimens tested in the 288 °C water environment, hydrogen embrittlement aided by strain localization in the aerated high temperature water environments is conceivable as a cracking mechanism [3,5,8]. Of course, metal dissolution at the crack tip should be considered basically involved in crack growth.

However, the proposed mechanism for EAC is not conclusive. Moreover, few experiments have been reported to directly show hydrogen effect on cracking of low alloy steel at high temperatures since the role of hydrogen in cracking at high temperature has often been ignored because hydrogen is assumed to rapidly escape from low alloy steels. However, thermal desorption spectroscopy (TDS) study with a deformed steel [9] showed hydrogen evolution peaks at high temperatures up to 445 °C depending on the hydrogen trapping defects. Recently it was reported that hydrogen could affect tensile properties at high temperature test with hydrogen pre-charged specimens [8,10].

The aim of the present study is to evaluate internal hydrogen effects on cracking at high temperatures. Since it had to be assumed that hydrogen exists in the material, internal hydrogen was simulated with pre-charging hydrogen. This simulation should be realistic in the sense that the corrosion reactions at the crack tip are a source of hydrogen [11]; however, it would be difficult to realize the amount of hydrogen in real situations. In the present work, we therefore had to limit our concerns to only observing hydrogen effect on cracking at high temperature.

### 2. Test procedures

#### 2.1. Material

The test material was forged pressure vessel steel, 10 in. thick, made of SA508 Cl.3. In as-received condition, the steel was austenized at 880  $^{\circ}$ C for 7 h, water quenched, and then tempered at 655  $^{\circ}$ C for 9 h followed by air cooling.

The chemical composition of the steel is given in Table 1. The microstructure is an upper bainite with well developed laths, as shown in Fig. 1.

# 2.2. Tensile test

Tensile tests of as-received and hydrogen pre-charged (Hprecharged) specimens were carried out in air at controlled temperatures and strain rates. The sample was a cylindrical type of which gauge length and diameter were 24 and 4 mm, respectively. These samples were pre-charged with hydrogen, using  $5 \text{ mA/cm}^2$  of the cathodic current for 10 min in a solution of  $1 \text{ N H}_2 \text{SO}_4 + 250 \text{ mg/l As}_2 \text{O}_3$ , with a platinum wire being used



Fig. 1. Microstructure of SA508 Cl.3.

as the anode. This cathodic charging condition was optimized to avoid any surface damage. The concentration of pre-charged hydrogen with this charging method was measured to be about 2.8 ppm by a fusion extraction method (LECO RH404).

After pre-charging, the sample surface was ground with SiC paper up to 2400 grit and then electrochemically coated with Cu to minimize hydrogen loss during mechanical tests. After each test, the fracture surface was observed with scanning electron microscope (SEM), and some specimens were longitudinally sectioned, polished, etched, and examined.

#### 2.3. Fatigue crack growth rate (FCGR) test

Fatigue crack growth rate test was performed in various environments: room temperature (RT) and 288 °C in air and Ar gas. Samples were exposed to air in the laboratory, while the argon gas environment was obtained by purging high purity argon at the pressure of 400 kPa in an autoclave. The fatigue test was carried out in accordance with the procedure of ASTM E 647 [12]. The fatigue specimens were a 1 in. thick standard compact tension type. All the samples were pre-cracked in RT air with the loading frequency of 10 Hz; the load ratio in sinusoidal cycle (*R*-ratio:  $P_{\rm min}/P_{\rm max}$ ) was fixed at 0.5; crack length was in situ monitored by a reversing direct current potential drop (DCPD) method.

The pre-cracked specimen was hydrogen pre-charged, and then fatigue crack growth rate was performed at 288 °C in argon gas. For this sample preparation, the same H-precharging method as for the tensile test was used; hydrogen was electrochemically pre-charged for 10 h. The charging time was chosen to make the mean diffusion distance of hydrogen equivalent to half of the sample thickness, assuming the hydrogen diffusivity to be  $10^{-6}$  cm<sup>2</sup>/s at room temperature [13]. The H-precharged surface was ground with SiC paper up to 2400 grit and rinsed with distilled water, and copper was then electrochemically coated on the whole sample surface. The specimen installed in the fatigue loading system was immediately pre-loaded to much less than a minimum load of the test loading cycle, which could minimize the hydrogen loss during heating. After each test, the specimen was broken in liquid nitrogen, and the fracture surface was observed under scanning electron microscope; then, the crack length measured by the DCPD method was calibrated.

### 3. Results and discussion

#### 3.1. Fatigue crack growth rates of SA508 Cl.3

As shown in Fig. 2, the crack growth rate of SA508 Cl.3 in air environment was increased by two to three times faster than that at RT. The crack growth rate in the argon gas showed a little effect of loading frequency, which could be attributed to

Table 1	
Composition	of SA508 Cl.3

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C (wt%)	Si (wt%)	Mn (wt%)	S (wt%)	P (wt%)	Ni (wt%)	Cr (wt%)	Mo (wt%)	Al (wt%)	Cu (wt%)	V (wt%)	
0.21	0.25	1.24	0.002	0.007	0.88	0.21	0.47	0.008	0.03	0.004	

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