# The Characteristics of Corrosion Fatigue Crack Growth Rate (CFCGR) for the Petroleum Refining Pressure Vessel Materials (2.25Cr-1Mo Steel) –The effect of Hydrogen Embrittlement–

Shinya Taketomi, <sup>1)</sup> A.Toshimitsu Yokobori, Jr, <sup>1)</sup> Kenichi Takei, <sup>1)</sup> Yoru Wada, <sup>2)</sup> Yasuhiko Tanaka, <sup>2)</sup> Tadao Iwadate, <sup>2)</sup> <sup>1</sup>Graduate School of Engineering Department of Nanomechanics, Tohoku University, Aoba 01 Aramaki Aoba-ku #980-8579, Japan

<sup>2</sup>The Japan Steel Works, LTD, 4 Chatsu-mashi Muroran, #051-8505, Japan

#### ABSTRACT

2.25Cr-1Mo steel is used as a pressure vessel component, which is sometimes suffered from hydrogen embrittlement. Therefore, it is important to investigate the effect of hydrogen on the embrittlement for 2.25Cr-1Mo steel.

Under corrosion fatigue condition, the effect of hydrogen embrittlement (HE) on corrosion fatigue crack growth rate (CFCGR) appears in different manner depending on applied stress wave forms and it was clarified by previous theoretical and experimental analysis.

In this paper, based on the theoretical analyses mentioned above, the effect of stress wave form on CFCGR for 2.25Cr-1Mo steel was investigated and the sensitivity of HE was discussed for this material.

## 1.INTRODUCTION

Industrial plants such as desulfurization equipment for petroleum refining, is a pressure vessel operated under high temperature and high pressure hydrogen environment. Since this structure is directly suffered from hydrogen environment, the problems of embrittlement of the material for this structure occurs due to the absorption of hydrogen. Actually many cases of fracture by hydrogen embrittlement were found out. However, since the mechanism and effective factors of hydrogen embrittlement have not yet been clarified, so clarification of these matters are needed.

On the other hand, under the wet corrosive condition, hydrogen is induced near a crack tip by chemical reaction[1]. These hydrogen diffuse and concentrate due to the gradient of hydrostatic stress under applied load. Theoretical analyses have been conducted [2-4] and it shows that the characteristic of hydrogen diffusion shows different behaviors depending on yield stress and stress wave form[5].

In this paper, on the basis of these results, the effects of yield stress, temperature and J-factor on CFCGR were investigated.

### 2.MATERIAL AND SPECIMEN

The material used in this experiment was 2.25Cr-1Mo with full bainite hardening and tempering steel. Three types of these steels were prepared as shown in Table.1. J100H and J100L materials were manufactured by recent technique with high purity. The yield stress of these steels were 587MPa and 426MPa respectively. These materials were non-brittled materials and the values of J-factor take 100 respectively, J300H is a brittled material, which is manufactured by a simulated technique in the 1960's. That is, solution treatment was conducted for steel added by Si, Mn, P, Sn by 8 ton ingot and then forged to the thickness of 30mm. After that, heat treatments of QT+PWHT (690 × 8hr) and step cooling were conducted. The J-factor of this material is 300, and yield stress is 590MPa.Mechanical properties and chemical compositions of these materials were shown in Table.1. The specimen used in this experiment were Compact Tension (CT) specimens and the shape of this specimen was shown in Fig.1.

	С	Mn	Р	S	Ni	Cr	Mo	V	Al	Sn	J-factor
J100	0.14	0.55	0.005	0.0007	0.21	2.42	1.08	< 0.01	0.019	0.010	95
J300	0.14	0.56	0.0014	0.0009	0.16	2.43	1.05	< 0.01	0.025	0.022	292

	0.2% Y.S.	T.S.
J100H	587MPa	711MPa
J100L	426MPa	588MPa
J300H	590MPa	726MPa

Table.1 Chemical composition and mechanical property



Fig.1 Shape of the CT specimen

# 3. EXPERIMENTAL EQUIPMENT AND PROCEDURE

The autograph testing machine designed for a small specimen[6] was used to conduct the test of CFCGR under load controlled condition. Schematic illustration of this testing machine was shown in Fig.2. Environmental container was made of acrylic acid resin, in order to optically observe crack length. A water bath controller was equipped to circulate the 3.5% NaCl solution through the environmental container under specified temperature. Crack length was measured by optical microscope with the precision of 0.01mm.

The amplitude of stress intensity factor is given by equation(1) [7].

$$\Delta K = \frac{\Delta P}{B\sqrt{W}} F\left(\frac{a}{W}\right)$$

$$F\left(\frac{a}{W}\right) = 4.55 - 40.32\left(\frac{a}{W}\right) + 414.7\left(\frac{a}{W}\right)^2 - 1698\left(\frac{a}{W}\right)^3 + 3781\left(\frac{a}{W}\right)^4 - 4287\left(\frac{a}{W}\right)^5 + 2017\left(\frac{a}{W}\right)^6$$

$$for \qquad 0.2 \le \left(\frac{a}{W}\right) \le 0.8$$

$$(1)$$

where

W : width of the specimen B : thickness of the specimen A : crack length  $\Delta P$  : amplitude of the applied load Stress ratio, R equals to 0.1. A fatigue pre-crack was induced by step decreasing method. It was decreased to the level of the value of  $\Delta K$  with 15.5MPa m.

This experiment was conducted under 3.5% NaCl solution, and the applied load frequency was changed ranging from 0.1~1Hz. Furthermore, to investigate the mechanism of CFCGR, the experiment of the effect of stress wave form on CFCGR was conducted. Applied load frequency, f was kept at 0.25Hz, and experiments were conducted under the condition of Slow-Fast wave form ( $t_R > t_D$ ) and Fast-Slow wave form ( $t_R < t_D$ ) conditions. Where  $t_R$  and  $t_D$  are load increasing and decreasing time between minimum and maximum applied load under fatigue condition. Experimental conditions were shown in Table.2.



Fig.2 Schematic illustration of corrosion fatigue testing system

	Fast - Slow	Symmetry	Slow - Fast
Stress shape form	$\sim$	$\wedge$	$\overline{\ }$
Stress rising time t <sub>R</sub> [sec]	0.8	2	3.2
Stress falling time t <sub>D</sub> [sec]	3.2	2	0.8
frequency [Hz]	0.25	0.25	0.25

Table.2 Experimental conditions



# Figs.4 The effect of temperature on CFCGR under various stress wave form (a) 4 (b) 20 (c) 80

To investigate the effect of temperature on CFCGR/CFCGR was measured for cases of temperature of NaCl solution with 4, 20, and 80 which circulate through environmental container. For the cases of 4, typical plateau characteristic of CFCGR which takes constant CFCGR was observed, which is in good agreement with the characteristic dominated by the mechanism of hydrogen embrittlement. On the other hand, for the case of 80, CFCGR is accelerated in parallel manner to that under air atmospheric condition. This characteristic is in good agreement with the characteristic dominated by the mechanism of agreement with the characteristic is in good agreement with the characteristic dominated by the mechanism of arodic dissolvent

chemical reaction. The typical effect of stress wave form on CFCGR was found out under the condition of 20  $\ .$ 



4.3 The effect of J-factor on CFCGR

(a) (b) Figs.5 The effect of J-factor on CFCGR under various stress wave form (a) J=100 of current materials (b) J=300 of 60's steels

To compare the CFCGR of current steels with that of 60's steels, two types of specimen (J-factor =100, 300) were used for the corrosion fatigue experiment. The experimental result are shown in Figs.5. These results show that, CFCGR for J300 material is ten times higher than that for J100 material in parallel manner, however yield stress for both materials take the same value.

# **5.CONSIDERATION**

Stress wave forms were classified into Slow-Fast and Fast-Slow forms which promote anodic dissolvent and hydrogen embrittlement mechanisms respectively. The effect of stress wave form on CFCGR remarkably appeares under the temperature condition of 20  $\,$ . This means CFCGR is very sensitive to the stress wave form under the condition of 20  $\,$ . Under the temperature condition of 4  $\,$ , CFCGR is dominated by hydrogen embrittlement mechanism. Therefore with decrease in temperature from 20 to 4  $\,$ , CFCGR under Fast-Slow condition increases and CGCGR under Slow-Fast condition decreases as compared with those under 20  $\,$ , which result in the same CFCGR dominated by hydrogen embrittlement mechanism. On the other hand, under the temperature condition of 80  $\,$ , CFCGR is dominated by the mechanism of anodic dissolvent chemical reaction which is time dependent mechanism of stress application.

Therefore, value of CFCGR under both of slow-fast and fast-slow conditions take the same value, because applied load area plotted against applied loading time equals between them.

The effect of J-factor on CFCGR does not affect the dominant mechanism of CFCGR and only accelerate CFCGR in parallel manner, which will be related to the deterioration of material structure such as decrease in bounding stress at grain boundary. From these results mentioned above, yield stress, temperature are dominant factor to the occurrence of mechanisms of CFCGR such as anodic dissolvent chemical reaction and hydrogen embrittlement mechanisms. The effect

of J-factor corresponding to the effect of time sequential deterioration on CFCGR contribute to the acceleration of CFCGR in parallel manner and it does not affect the transition of dominant mechanism of CFCGR.

# **6.CONCLUSIONS**

- 1) The effect of stress wave form on CFCGR was typically found out for materials with higher yield stress. This is caused by the effect of stress wave form on hydrogen embrittlement mechanism.
- 2) From the experimental results under the condition of temperature with 4 , plateau characteristic of CFCGR plotted against  $\Delta K$  was observed. This characteristic was in good agreement with the characteristic dominated by the mechanism of hydrogen embrittlement. Under the condition of temperature with 80 , CFCGR is dominated by time dependent mechanism, that is anodic dissolvent chemical reaction. On the other hand, under the condition of temperature with 20 , CFCGR was found to be sensitive to the effects of both hydrogen embrittlement and anodic dissolvent chemical reaction.
- 3) The effect of J-factor on CFCGR does not affect the dominant mechanism of CFCGR and only accelerate CFCGR in parallel manner, which will be related to the deterioration of material structure such as decrease in bounding stress at grain boundary.

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