## Corrosion Science 53 (2011) 2558-2565

Contents lists available at ScienceDirect

# **Corrosion Science**



# Stress corrosion cracking of uni-directionally cold worked 316NG stainless steel in simulated PWR primary water with various dissolved hydrogen concentrations

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## ARTICLE INFO

Article history: Received 19 January 2011 Accepted 19 April 2011 Available online 22 April 2011

Keywords: A. Stainless steel B. SEM C. Stress corrosion C. Hardening C. Potential parameters

# ABSTRACT

Stress corrosion cracking growth rate of uni-directionally cold-rolled 316L stainless steel was monitored in simulated PWR primary water with different dissolved hydrogen (DH) concentrations at 320 °C. Crack growth rate at a DH of 0.16 cm<sup>3</sup> (STP) H<sub>2</sub>/kg H<sub>2</sub>O is close to that at 5 cm<sup>3</sup> (STP) H<sub>2</sub>/kg H<sub>2</sub>O. Crack growth rate at 30 cm<sup>3</sup> (STP) H<sub>2</sub>/kg H<sub>2</sub>O is about one fourth of that at 5 cm<sup>3</sup> (STP) H<sub>2</sub>/kg H<sub>2</sub>O or two times of that at 50 cm<sup>3</sup> (STP) H<sub>2</sub>/kg H<sub>2</sub>O. Electron back scattering diffraction results show typical intergranular SCC along high angle boundaries with high levels of deformation.

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sile stress of about 571 MPa were found on the inner surface, which were thought to be caused by surface mechanical fabrication. High hardness/strength and high residual stress are thought to be important factors for causing stress corrosion in the stainless steel safe end. Irradiation assisted stress corrosion cracking of austenitic stainless steels has been identified in PWR components, where materials are hardened after irradiation during plant operation.

The above plant experiences showed that strain hardening such as cold work would play an important role in stress corrosion cracking of austenitic stainless steels in PWR primary water environments. Laboratory tests also showed that cold work could significantly enhance the crack growth of austenitic stainless steels in simulated PWR primary water environments [4-9] and in hydrogenated pure water at high temperatures [10]. Shoji et al. [4] have reported the crack growth behavior of cold worked austenitic stainless steels such as 304L SS, 316L SS, and 348 SS in simulated PWR primary water environments, emphasizing the effect of material yield strength. Arioka et al. [5,6] have reported the effects of many factors such as temperature, water chemistry, and material orientation on stress corrosion crack growth rates of cold worked SUS 316 SS in simulated PWR primary water environments. Andresen et al. have reported the effect of water chemistry on SCC growth rates of cold worked stainless steels [10]. In the present work, the effect of dissolved hydrogen (DH) concentration in the range of 0.16–50 cm<sup>3</sup> (STP)  $H_2/kg H_2O$  on crack growth rate (CGR) of uni-directionally cold worked 316 stainless steel in simulated PWR primary water environments is investigated and analyzed.

## 1. Introduction

There are many austenitic stainless steel components in contact with primary water in pressurized water reactors (PWR). Recently several cases of cracking in austenitic stainless steel components in PWR plants have been reported [1–3]. Couvant et al. [1] reported cases of intergranular stress corrosion cracking (IGSCC) of heaters of PWR pressurizers. The heaters were fabricated from low carbon SUS 304L and SUS 316L stainless steels (SS). Stress corrosion cracking (SCC) in these heaters occurred in highly strained-hardened areas. In addition, scratches or metal folds have been identified in the area where SCC initiated. Strain-localization associated with cold working is thought to be crucial for SCC of austenitic stainless steels in PWR plants. Recently, nondestructive and destructive examinations showed that stress corrosion cracking occurred in primary water inlet nozzle welds of steam generators in several PWR plants [2]. Most cracks were identified in the nickel-base weld metals (including weld buttering), with one exception that cracking also penetrated into the stainless steel safe end. The maximum crack depth (intergranular type) in SUS 316 stainless steel safe end is about 0.9 mm. Microstructural analysis showed that there was no indication of Cr-depletion related to sensitization, which was supported by electrochemical potentiodynamic reactivation (EPR) measurements. Clear indications of mechanical fabrication were observed on the inner surface. Slip lines, about 0.1 mm thick hardened surface layer, and a maximum circumferential ten-





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# 2. Experimental methods

#### 2.1. Material and specimen preparation

316NG SS block was cold-rolled uni-directionally and then used for preparing stress corrosion cracking specimens. The 316NG SS was solution-annealed (SA) at  $1050 \,^{\circ}$ C for 4 h followed by a water quench. The chemical composition of 316NG SS is shown in Table 1.

The solution-annealed 316NG was uni-directionally (1D) coldrolled at RT by 28% reduction in thickness, which is called 1DCR 316NG. Multiple pass rolling was used to get relatively uniform deformation along the plate thickness direction. Fischer FERIT-SCOPE Ferritescope showed a reading of 0.30% of martensite, indicating a low (<1%) martensite content in the cold-rolled 316NG.

The mechanical properties of solution annealed (SA) 316NG SS and 28% 1DCR 316NG at room temperature (RT) and 300 °C are shown in Table 2. The data of mechanical properties at 300 °C are used as reference data at the SCC test temperature of 320 °C.

The orientation of the specimen taken from 28% 1DCR 316NG block for crack growth rate test is defined in Fig. 1. For example, the cold rolling direction is designated as the longitudinal direction, L. It is noted that the notations for orientations for orientations and planes are different. For example, S–L (with a dash between two directions) refers to S–L orientation and SL (without dash between two directions) refers to SL plane. A compact tension (CT) specimen was used for SCC growth rate test of 28% 1DCR 316NG SS, in which the notch direction is parallel to the S–L orientation. The geometry of CT specimen has been described in a previous publication [11]. The length of the mechanical notch is 20 mm and the thickness of the CT specimen 6LBSL1 is 14.0 mm. Based on ASTM E399, small-scale yielding can be satisfied for such a specimen geometry for applied K up to 37 MPa m<sup>0.5</sup> at temperature of about 300 °C.

The grain boundary microstructure of 1DCR 316NG SS was observed by electron backscattering diffraction (EBSD) technique. EBSD was measured with Hitachi S-4300 FE-SEM, TSL solutions camera control system VIT1000, image processing system DSP 2000, and interface controller MSC 2000. The EBSD pattern was analyzed using OIM-Analysis software provided by TSL, Co. Ltd. [12]. Acceleration voltage of SEM beam for the EBSD measurement was 25 kV and the beam current was 15  $\mu$ A. The surface was finally finished by polishing with 0.3  $\mu$ m deagglomerated alpha alumina

#### Table 1

Chemical composition (wt.%) of 316NG SS.

С	Si	Mn	Р	S	Ni	Cr	Мо	Ν	Fe
0.020	0.50	0.81	0.025	0.002	12.89	16.59	2.39	0.094	Bal.

Table	2
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M	ecl	nanic	al	properti	ies o	f a	s-SA	. 3	16N	IG	SS	and	28%	1DCR	31	6N	GS	S.
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Temperature	Material	0.2% Proof stress (YS) (MPa)	Tensile stress (MPa)	Total elongation (%)	Reduction of area (%)
RT	SA 316NG	278	548	58	81
	28% 1DCR 316NG	685	732	24	77
300 °C	SA 316NG	168	426	42	78
	28% 1DCR 316NG	506	627	12	64



Fig. 1. Schematic of the orientation of 28% 1DCR 316NG SS and the alignment of CT specimen 6LBSL1.

powder/water mixture followed by electropolishing using  $HCIO_4 + C_2H_5OH$  electrolyte in order to get relatively smooth surface free from surface hardening caused by the mechanical polishing.

### 2.2. Stress corrosion cracking test procedures

1DCR 316NG SS specimen 6LBSL1 was used for SCC crack growth rate test in simulated PWR primary water environments. The SCC tests were performed in a simulated PWR primary water (B: 1200 ppm (ppm in this paper refers weight percentages) as H<sub>3</sub>BO<sub>3</sub>, Li: 2.0 ppm as LiOH, DO < 5 ppb, Cond.  $\sim$ 20 µS/cm, flow rate:  $\sim$ 5 L/h,  $P \sim$  13 MPa) at 320 °C. The DH levels in the inlet water were controlled by bubbling N<sub>2</sub>, H<sub>2</sub> and mixed H<sub>2</sub>/N<sub>2</sub> or keeping a certain pressure of H<sub>2</sub> in the make-up water tank. The procedures for SCC tests are in the following sequence: fatigue in air for precracking, fatigue cracking in high temperature water for in situ pre-cracking, stress corrosion cracking growth tests under constant loading, and post-test fatigue in air for fracturing specimens to expose the fracture surfaces. The specimens were first fatigue precracked in air under a sine-wave loading at 20 Hz and a maximum stress intensity factor ( $K_{max}$ ) value of less than 15.6 MPa m<sup>0.5</sup>, where the load-ratio  $(K_{\min}/K_{\max})$  was R = 0.2. After pre-cracking in air, the specimen was side-grooved to 5% on each side. The specimen was ultrasonically cleaned in ethanol before the SCC test.

After the water chemistry and temperature had reached their designed values, the specimen was subjected to in-situ fatigue pre-cracking in high temperature water under a series of triangular-wave loadings. The triangular (Tri.) wave loading modes for in-situ pre-cracking are:  $f \sim 0.01$  Hz, R = 0.3 (288 cycles), R = 0.5(576 cycles), and R = 0.7 (864 cycles) respectively, with a  $K_{\text{max}}$  of about 31.3 MPa m<sup>0.5</sup> for specimen 6LBSL1. In situ fatigue precracking was employed to facilitate the transition from the transgranular crack front produced by fatigue precracking in air to intergranular stress corrosion cracking in high temperature water, which has been proposed and used by Andresen et al. [13]. After the in situ pre-cracking, the specimen was subjected to a constant loading during the SCC test periods. Based on the measurement of the SCC length on the fracture surface after the SCC test, K was 31.3 MPa  $m^{0.5}$  in the beginning of the SCC test and changed to 34.4 MPa m<sup>0.5</sup> at the end of the SCC test. The average K for SCC test takes the average of the initial and the final K, which is 32.8 MPa m<sup>0.5</sup>. The test matrix is shown in Table 3. The cracklengths were continuously monitored by using an alternating-current potential-drop (ACPD) probes and machine equipped with a Download English Version:

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