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Stress corrosion cracking behavior of cold-drawn 316 austenitic stainless steels in simulated PWR environment

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ABSTRACT

The stress corrosion cracking (SCC) behavior of solution-treated and cold-drawn 316 austenitic stainless steels was investigated in simulated pressurized water reactor environment by slow strain rate tensile test. Both cold drawing and Cl addition in simulated primary environment can observably increase SCC susceptibility of 316 austenitic stainless steels. The oxides containing higher Cr concentration initially nucleate on crack tip, and then react with dissolved oxygen and H_2O to form the Fe-rich oxides on crack flank during the subsequent crack propagation in abnormal water containing Cl. The deformation twins activated by cold-drawing provide paths to accelerate the oxygen diffusion during SCC.

1. Introduction

316 austenitic stainless steel is the important structural material for pressurized water reactor (PWR) because of its excellent weldability, high mechanical property and reasonable corrosion resistance [1–3]. During the long-term service of the nuclear materials, the accident of environment degradation might occur. Especially, stress corrosion cracking (SCC) behavior of austenitic stainless steels has become a serious problem in the nuclear energy industry for many decades [4–7].

SCC behavior of austenitic stainless steels is significantly influenced by water chemistry [8,9]. The dissolved oxygen is particularly significant in the SCC behavior of the forged 316L stainless steel in PWR primary water, since it could influence the oxide film formed near the crack tip [10]. Lu et al. also suggested that the oxygen content in high temperature water plays a key role in nucleation and propagation of the transgranular stress corrosion cracking (TGSCC) in 304L stainless steel [11]. In addition, the abnormal variations of water chemistry may periodically or occasionally occur in PWR, e.g., the concentration of chloride or oxygen ions may increase during the ion exchanger intrusions or condenser leakages [12]. The SCC susceptibility of austenitic stainless steels can be obviously raised by a certain concentration of chloride ions in primary circuit of PWR [13–16].

Cold-working introduces dislocations, deformation twins and favourable residual stresses, and it is recognized as an effective processing technique to increase the strength of austenitic stainless steels, accompanied by reduced ductility. Cold-working has a great influence on the SCC behavior of stainless steels. Féron et al. reported that the SCC susceptibility of the cold-worked 316 stainless steels in hydrogenated primary water increases with increasing the extent of cold working, and they found that the discontinuous inner Cr-rich oxides play an important role on SCC resistance of cold-worked 316 steels [17]. García et al. demonstrated that TGSCC of the cold-worked 304 stainless steels is caused by increasing anodic dissolution at slip bands and at martensite platelets [18]. Perez et al. also proposed that the TGSCC initiation in the cold-rolled 304 stainless steels is governed by the oxygen diffusion along the twin deformation bands formed during prior cold working, and the oxygen diffusion is accelerated by the stress concentration because of the twin deformation bands [19,20].

In order to raise strength and improve fatigue resistance of the nuclear reactor components made of 316 austenitic stainless steels, they are usually subjected to cold-working. In addition to dislocations, deformation twins are common defects in cold-worked austenitic stainless steels. The influence of deformation twins introduced by prior cold-working on SCC behavior of the cold-worked 316 austenitic stainless steel has rarely been reported. Therefore,

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Table 1
Chemical composition of 316 austenitic stainless steels.

Elements	C	Mn	P	Si	Cr	Ni	Mo	N	Fe
wt.%	0.04	1.25	0.03	0.54	16.65	11.45	2.08	0.07	Bal.

SCC susceptibility of the cold-worked 316 austenitic stainless steels needs to be evaluated. The slow strain rate tensile (SSRT) test provides not only a useful information on SCC behavior of materials, but also a relatively short experimental time to evaluate SCC susceptibility of materials in simulated PWR environment. In this work, the SSRT tests of the solution-treated and cold-drawn 316 austenitic stainless steels were conducted in both a simulated primary water and an abnormal water containing chloride ions at 300 °C and 11.5 MPa. The fractography of the fractured specimens was observed by scanning electron microscope (SEM). The microstructure of TGSCC crack formed on the cold-drawn specimen during SSRT tests in abnormal water was analyzed by transmission electron microscope (TEM). The effect of water environment and cold-drawing on the SCC susceptibility of 316 austenitic stainless steels was discussed.

2. Materials and experiments

The materials used in this work are 316 austenitic stainless steels. Table 1 shows the chemical composition of 316 austenitic stainless steels. The round bars of type 316 stainless steels were solid solution treated at 1050 ° C for 1 h then water quenched. The cold worked steels used as baffle bolts in primary circuit of PWR were cold drawn by 30% reduction in cross-sectional area. The cylindrical specimens with 25.4 mm in gauge length and 5 mm in gauge diameter were machined from the cold-drawn steels along the longitudinal direction. The specimen surface was finally polished by using 2000 grit SiC emery paper to remove surface defects. The geometry schematic of specimens was shown in Fig. 1.

In order to accelerate experiments and induce SCC behavior in days rather than years, the severity of the corrosion environment was increased. The SCC susceptibility of the solution-treated and cold-drawn 316 austenitic stainless steels was evaluated by SSRT test with a strain rate of 4.2×10^{-6} s^{-1} in a simulated primary water (1200 ppm B as boric acid and 2 ppm Li as lithium hydroxide) at 300 °C and 11.5 MPa. In order to improve the understanding of the SCC behavior of 316 austenitic stainless steel in an abnormal water, 30 ppm Cl as sodium chloride was added in the simulated primary water. In addition, the concentration of dissolved oxygen in simulated primary water is 8 ppm. The conductivity of the deionized water used for preparing corrosion solution is 0.1 μS/cm.

According to ASTM G129, the relative plasticity loss, I_δ, is defined as the SCC susceptibility. The I_δ can be expressed as

$$I_\delta = \left[1 - \frac{\delta_w}{\delta_n}\right] \times 100\% \quad (1)$$

Where δ_w and δ_n are the elongation in simulated primary water and in air at 300 °C, respectively. Obviously, increasing I_δ indicates raising SCC susceptibility.

The metallographic examination was conducted by Axiocam MRc5 optical microscope (OM). The sample for OM examination was chemical etched in aqua regia after being mechanically grinded and polished. The fractography and the gauge surface of specimens after fracture were observed by JSM-7600F SEM. The initial microstructure of specimens was observed by JEM-2100F TEM operating at 200 kV. TEM sample was mechanically grinded to 50 μm in thickness on 2000 grit SiC paper and then was prepared to 3 mm disk in diameter. The disk sample was twin-jet electropolished in a mixed solution of 5 vol.% $HClO_4$ and 95 vol.% C_2H_5OH with a DC voltage of 55 V and a current of 30 mA at −30 °C. For purpose of further investigating the microstructure of crack tip in the cold-drawn steel, focused ion beam (FIB) was used to prepare the cross-sectional sample of crack. Before ion beam was applied, the Pt layer with 1 μm in thickness was deposited by thermal evaporation on the gauge surfaces to protect the oxide films on crack. The lamella for TEM observation was exactly thinned after being transferred onto a special grid. Energy dispersive X-ray spectroscopy (EDS) and electron energy loss spectroscopy (EELS) analysis for the crack were carried out by a JEM-ARM200F TEM.

3. Results and discussion

3.1. Initial microstructure

The initial microstructures of the solution-treated and cold-drawn 316 austenitic stainless steels were presented in Fig. 2a and b, respectively. The equiaxed and nearly-equiaxed austenitic grains were found in the solution-treated and cold-drawn steels, respectively. The average grain size of the solution-treated and cold-drawn steels was determined to be nearly equal to about 45 μm through the mean linear intercept method. A small volume fraction of delta-ferrites were found inside the austenitic grains of the solution-treated steels. Also, the distribution of delta-ferrites along the longitudinal direction and the lamella structures oriented at about 45° to the longitudinal direction were observed in the austenitic grains of the cold-drawn steels, as shown in Fig. 2b.

Fig. 3 shows the substructures of the cold-drawn 316 austenitic stainless steels observed by TEM. The lamella structures previously

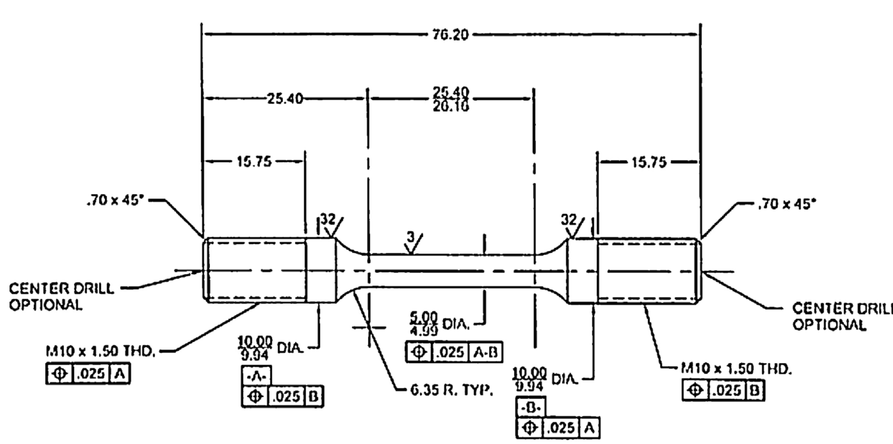

Fig. 1. The geometry schematic of specimen (mm).

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