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Low cycle fatigue behaviors of type 316LN austenitic stainless steel in 310 ◦C deaerated water–fatigue life and dislocation structure development

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Abstract

The low cycle fatigue tests for type 316LN stainless steel were performed in 310° C deaerated water at various strain amplitudes and strain rates. The primary hardening was observed in all test conditions. It was found that the increase of bulk dislocation density and the interaction between mobile dislocations and atoms, which occur under the dynamic strain aging condition, were responsible for the primary hardening. On the other hand, the secondary hardening occurred distinctly for 0.4%/s and 0.4%. For that specific condition, the corduroy contrast resulted from pinned dislocations was observed at the crack tip. The secondary hardening may be associated with the corduroy contrast. The dislocation structure and fatigue surface revealed the characteristic feature of the hydrogen-induced cracking. Thus, we concluded the hydrogen-induced cracking contributed to the reduction in fatigue life and the enhancement of decreasing fatigue life with a decreasing strain rate in 310 ◦C deaerated water. © 2007 Elsevier B.V. All rights reserved.

Keywords: Low cycle fatigue; Cyclic hardening; Dynamic strain aging; Dislocation structure; Fatigue life; Hydrogen-induced cracking

1. Introduction

Many degradation mechanisms occur during the service period of a nuclear power plant. As the onset and the progress of degradation phenomena are closely related to the safety and integrity of nuclear power plants, studies that focus on degradation mechanisms are very important and are carried out continually. Because nuclear power plant components are subjected to cyclic stress caused by a variety of transients during operation, fatigue is one of the most significant degradation mechanisms of these components. Furthermore, as nuclear power plants are operated in high-temperature corrosive aqueous environments, fatigue damage can be worsened by corrosive environments. Thus, environmental fatigue is one of the most significant degradation mechanisms in nuclear power plants [\[1–4\].](#page--1-0) Research on the low cycle fatigue (LCF) behaviors of

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structural materials in high-temperature water is needed in order to assess the safety and integrity of these plants. As austenitic stainless steels (SSs) are used widely as structural materials in nuclear power plants, understanding their LCF behaviors are also critical to integrity assessments.

For austenitic SSs, various dislocation structures, such as cell, tangled dislocation, and wall-and-channels, were observed depending on the LCF loading conditions and test temperature [\[5–7\].](#page--1-0) The cyclic stress response is associated with the dislocation structure developed during LCF deformation. Thus far, studies on the LCF behaviors of austenitic SSs have been reported in various environments [\[1–10\].](#page--1-0) However, studies on the relationship between the cyclic behaviors and the bulk dislocation structure of austenitic SSs have been reported mainly in a high-temperature vacuum [\[5,9\]](#page--1-0) and air conditions [\[7,10\].](#page--1-0) Therefore, an investigation of the relationship between the cyclic hardening behaviors and the dislocation structure at the crack tip in high-temperature water is nonetheless limited.

It has been reported that the fatigue life of austenitic SSs in high-temperature water is shorter than that in air due to

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the onset of environmentally assisted cracking (EAC) mechanisms [\[1,4,11,12\].](#page--1-0) Hydrogen-induced cracking (HIC) is an EAC mechanism that explains the reduction in the fatigue life of austenitic SSs in high-temperature water [\[11,12\].](#page--1-0) Hydrogen produced through a corrosion reaction is absorbed into the metal at or near the crack tip. The absorbed hydrogen induces weakening of the atomic bonds, localized deformation, or emission of dislocation [\[13,14\].](#page--1-0) Therefore, the crack growth rate increases as a result of the absorbed hydrogen. Compared with carbon steels or low-alloy steels, austenitic SSs are less susceptible to the HIC due to the low diffusivity and high solubility of hydrogen in f.c.c. materials. However, austenitic SSs are not entirely free from hydrogen-induced damage; for example, certain evidence of the HIC, such as a decrease in the distance between dislocations, a reduction in ductility, and a brittle fracture mode, have been observed for austenitic SSs in room temperature (RT) slow strain rate tests and tensile tests [\[15–17\].](#page--1-0) Although studies regarding HIC are performed continually, evidence of HIC for austenitic SSs that occur under LCF loading in high-temperature water is lacking.

In this regard, the present study investigates the relationship between the cyclic hardening behaviors and the dislocation structure at the crack tip of type 316LN SS from LCF tests performed in 310 ◦C deaerated water. Additionally, evidence of HIC for type 316LN SS in 310 ◦C deaerated water were investigated by the microstructure observation.

2. Experimental details

2.1. Test material and LCF test specimen

The test material used in this study was ASME SA312 type 316LN SS. The chemical composition of the test material is given in Table 1. The test material was provided as solutionannealed at $1065\,^{\circ}\text{C}$ for 1 h, followed by quenching in water. The mechanical properties measured at RT and 310 °C are summarized inTable 2. The LCF test specimen was of round bar type, with a gauge section of 9.63 mm in diameter and 19.05 mm in length [\[4\].](#page--1-0) Prior to the test, the surface of the specimen was polished with 1μ m emery paper and the specimen was then rinsed with acetone in an ultrasonic cleaner.

Table 1 Chemical composition of type 316LN SS (wt.%)

C	0.018	
Mn	1.84	
P	0.022	
S	0.016	
Si	0.46	
Cr	16.37	
Ni	11.30	
Mo	2.11	
N	0.096	
B	0.0015	
Co	0.10	
Cu	0.28	

Table 2 Mechanical properties of type 316LN SS

	Y.S. (MPa)	U.T.S. (MPa)	Elongation $(\%)$
RT	294.7	577.1	67.7
310 °C	174.2	478.2	44.3

2.2. LCF test system

The system for the LCF test in high-temperature water is shown in [Fig. 1.](#page--1-0) The LCF test system was comprised of a servo-electric fatigue test machine, an autoclave, and a water circulation loop. For convenience and efficiency, two water columns installed in the water circulation loop were used to control the dissolved oxygen (DO) level of the test water. The DO concentration was controlled in the two water columns by the purging of argon gas. Mass flow controllers were attached to the water columns to inject argon gas at a constant flow rate. A DO meter and two conductivity meters were installed in the water circulation loop to monitor the water chemistry during LCF testing. The DO concentration and conductivity were measured on the feed water line as well as on the return water line. The temperature in the autoclave was controlled by a pre-heater and a main heater. Temperature fluctuations were maintained within a range of $\pm 2^{\circ}$ C during the test. The strain was measured by two linear position transducers mounted on the specimen.

2.3. Test conditions

The LCF tests were carried out in strain control mode with a fully reversed triangular waveform in 310 ◦C deaerated water. The system pressure was maintained at 15 MPa. The strain rates were 0.008, 0.04, and 0.4%/s, and the strain amplitude varied from 0.4 to 1.0%. Prior to the test, the test water was deionized and the DO concentration was kept under 1 ppb. Additionally, the conductivity was maintained under $0.1 \mu S/cm$ during the test. The LCF tests were also conducted in air for comparison. Fatigue life, N_{25} , was defined as the number of cycles at which the tensile stress drops 25% from its peak value.

After the LCF tests were completed, the fatigue surface was examined by a JEOL JSM-6460 scanning electron microscope (SEM). In addition, the bulk dislocation structure and the dislocation structure at the crack tip were observed using a Technai G2 F30 S-TWIN field emission transmission electron microscope (TEM) operating at an accelerating voltage of 300 kV. In order to investigate the dislocation structure at the crack tip, TEM samples were obtained from just below the fatigue surface using a NOVA 200 dual beam-focused ion beam (FIB). Thin foils for the observation of the bulk dislocation structure were also obtained at a position 1 mm away from the fatigue surface.

3. Results

3.1. Cyclic stress response and fatigue life of type 316LN SS

In [Fig. 2, t](#page--1-0)he cyclic stress response in 310° C deaerated water was compared with that in RT, and in 310° C air for a strain rate

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