Fatigue Behavior of Cold-Worked 304 Stainless Steels under In-Situ Irradiation at 300°C

Yoshiharu Murase\textsuperscript{1}, Johsei Nagakawa\textsuperscript{1,2} and Norikazu Yamamoto\textsuperscript{1}

\textsuperscript{1}National Institute for Materials Science, Tsukuba 305-0047, Japan
\textsuperscript{2}Interdisciplinary Graduate School of Engineering Sciences, Kyushu University, Kasuga 816-8580, Japan

Stress-controlled fatigue tests were performed for 5% and 15% cold-worked 304 stainless steels under in-situ irradiation with 17 MeV protons at 300°C. Increase of fatigue life with prolonged crack propagation length under in-situ irradiation was detected for 5% cold-worked specimens, whereas no significant difference was observed in fatigue life or crack propagation length between in-situ irradiation and unirradiated conditions for 15% cold-worked specimens. Fractographic analysis of the fatigue fracture surface suggested more significant participation of strain-induced martensite in fatigue behavior for 15% cold-worked specimens. The strain-induced martensite in 15% cold-worked specimens would play an important role in reducing the in-situ irradiation effect on fatigue behavior based on the interaction between radiation-induced defect clusters and moving dislocations. [doi:10.2320/matertrans.MAW200806]

\textbf{Keywords}: austenitic stainless steel, proton irradiation, fatigue, in-situ irradiation effect, strain-induced martensite

1. Introduction

Structural materials for fission reactors are subjected to not only severe atomic displacement damage but also various simultaneous external loads. Since such external loads can include cyclic components due to thermal fluctuations and mechanical vibrations, it is very important to understand the material fatigue behavior under in-situ irradiation. In the course of extensive efforts to accumulate the available experimental data on in-situ irradiation fatigue tests, some essential differences in fatigue behavior between in-situ irradiation and post-irradiation conditions have been demonstrated in some works.\textsuperscript{1,4} In a previous paper,\textsuperscript{4} stress-controlled fatigue tests were conducted for 20% cold-worked 316 stainless steel under 17 MeV proton irradiation at 300°C. Substantial increase of fatigue life with prolonged crack propagation was detected in the in-situ irradiation condition. The in-situ irradiation effect on fatigue behavior has been discussed based on the interaction between radiation-induced defect (RID) clusters and moving dislocations.

In addition to type 316 stainless steels, type 304 stainless steels are also commonly used as structural materials for fission reactors. However, with respect to phase stability against cold-working, the austenite of type 304 steels is less stable than that of type 316 due to a lower ratio of austenite stabilizing elements (nickel, molybdenum) in type 304. It is well known that the strain-induced transition from austenite phase (α') to martensite phase (α) can be induced by cold-working at room temperature for type 304 steels. Although the fatigue behavior of cold-worked 304 stainless steels has been well examined in terms of the relationship between crack paths and underlying microstructures,\textsuperscript{5,6} experimental data on in-situ irradiation fatigue behavior of cold-worked 304 stainless steels is not available.

In the present study, stress-controlled fatigue tests were conducted for 5% and 15% cold-worked 304 stainless steels in the in-situ irradiation condition with 17 MeV protons at 300°C. The objective of this study was to investigate the role of strain-induced martensite in in-situ irradiation fatigue behavior.

Table 1 Chemical composition of SUS304 stainless steel (mass%).

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Ni</th>
<th>Mn</th>
<th>P</th>
<th>Si</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>SUS304</td>
<td>0.06</td>
<td>18.18</td>
<td>8.41</td>
<td>1.23</td>
<td>0.031</td>
<td>0.46</td>
<td>0.007 balance</td>
</tr>
</tbody>
</table>

2. Experimental Procedure

The material used in the present study was commercial SUS304 stainless steel. The chemical composition of the steel is shown in Table 1. As-received sheets of steel with a thickness of 0.20 mm were annealed for 1 h at 925°C to control the grain size to about 12 μm in diameter. The annealed sheets were rolled to 5% and 15% reduction in thickness at room temperature. The cold-worked sheets were mechanically ground to 0.15 mm in thickness and then punched out into the specimen type shown in Fig. 1. A fatigue crack starter side-notch was introduced into the specimen gauge by means of spark erosion (see Fig. 1) to prepare the 5% and 15% cold-worked (CW) specimens. Cyclic loading mode was tension-tension in stress control under a constant loading rate of 50 MPa/s with a loading ratio (minimum stress/maximum stress) of 0.2. The maximum-minimum stresses for 5% and 15% CW specimens were set in order for the fatigue lifetime to be around 24 h in the unirradiated condition, namely 301.2–60.2 MPa for 5% CW specimens and 326.4–65.3 MPa for 15% CW specimens. The maximum stress corresponds to 98% and 69% of the yield stress at the notched ligament for 5% and 15% CW specimens, respectively. Figure 2 shows the depth dependence of the displacement damage rate for 304 stainless steel calculated by the SRIM 2006 code under the present 17 MeV proton irradiation condition. As shown in Fig. 2, the atomic displacement rate for the specimen with a thickness of 150 μm was estimated as 1 × 10\textsuperscript{-7} dpa/s with an error range of ±11%. The specimen was cooled by circulating helium gas in order to compensate for beam heating. The specimen temperature was controlled at 300 ± 3°C by adjusting the output of the helium gas heater during the in-situ irradiation and unirradiated fatigue tests. Experimental details of...
specimen preparation, irradiation condition and \textit{in-situ} irradiation fatigue testing machine are described elsewhere.\textsuperscript{7)}

After completing the fatigue tests, fractographic analysis of fracture surface was performed by a scanning electron microscope (SEM, JSM-5310) for all specimens. Tensile tests were also conducted at 300°C for the irradiated 5% and 15% CW specimens without a side-notch. Pre-irradiation dose levels ranged up to 0.022 dpa. Changes in tensile stress were measured as a function of dose level for both specimens.

3. Results

Table 2 summarizes the number of cycles to fracture (Nf) and the crack propagation length ($a_{cr}$) for all specimens. Larger Nf with prolonged $a_{cr}$ under \textit{in-situ} irradiation was detected for 5% CW specimens, whereas no significant difference was observed in Nf or $a_{cr}$ between \textit{in-situ} and unirradiated conditions for 15% CW specimens. These results indicate not only the existence of an \textit{in-situ} irradiation effect on fatigue behavior for 5% CW specimens but also the reduction of the \textit{in-situ} irradiation effect for 15% CW specimens. Figure 3 shows SEM photos of the typical fracture surface and its magnified morphology in the vicinity of the notch tip in the \textit{in-situ} irradiation condition for (a) 5% and (b) 15% CW specimens. The fractographic morphology of the fracture surface was quite similar between \textit{in-situ} irradiation and unirradiated conditions, and is characterized by glide plane decohesion, fatigue striations, dimples and quasi-cleavage (QC) facets, with an indication of transgranular cracking in the mixed mode of ductile and brittle fractures for both 5% and 15% CW specimens. The crack propagation length ($a_{cr}$) shown in Table 2 is designated as the distance from notch tip to unstable fracture point where fatigue striations disappear on the fracture surface. In comparing the fractographic morphology between 5% and 15% CW specimens, as shown in Fig. 3, the ratio of QC facets to other fracture surface markings was higher for 15% CW specimens. Table 3 presents the changes in tensile properties as a function of atomic displacement damage for 5% and 15% CW specimens. Since the respective dose level integrated during the \textit{in-situ} fatigue tests corresponds to 0.014 and 0.011 dpa on average, the increase of 0.2% offset yield stress (0.2% YS) for the \textit{in-situ} 5% and 15% CW specimens was estimated as 47 and 62 MPa, respectively.

4. Discussion

The rearrangement of dislocation structures under fatigue loading is closely associated with the progress of fatigue processes in the ductile fracture mode. Several studies\textsuperscript{8–10)} have dealt with the evolution of inhomogeneous dislocation structures such as cells and wall-channels at the plastic deformed zone in the vicinity of the crack tip for type 316 stainless steels. The evolution of inhomogeneous dislocation structures accommodates the plastic strain accumulated during fatigue loading. The saturation of inhomogeneous dislocation structures results in the emergence of planar slip leading to the initiation and growth of fatigue cracks.\textsuperscript{8)} In the process of fatigue crack propagation, phenomena such as the formation of striations have been explained by the plastic-blinking process addressing the dislocation slip on duplex slip planes oriented roughly at 45° to the plane of cracking.\textsuperscript{11)}

Thus, the fatigue processes of crack initiation and propag-
tion in ductile fracture mode are governed by dislocation slip. Such fatigue processes can be strongly modified by the introduction of radiation-induced defect (RID) clusters. Since RID clusters can act as obstacles to dislocation slips, continuous introduction of RID clusters under in-situ irradiation would effectively retard the development of inhomogeneous dislocation structures in the process of crack initiation. The enhanced resistance to dislocation slip is also responsible for the delay of the plastic-blunting process as well as the prolongation of crack propagation due to the increase of fracture toughness in the process of crack propagation. Therefore, substantial increase of fatigue life accompanied by prolonged crack propagation under in-situ irradiation could be explained based on the interaction between RID clusters and moving dislocations.\(^1\),\(^4\)

Strain-induced martensite is also considered to significantly influence the fatigue behavior of metastable austenitic stainless steels. Since the strain-induced martensitic phase consists of twin lamellae with higher dislocation density in the austenitic matrix,\(^12\) preferential QC cracking along specific crystallographic planes related to martensitic microstructures could deflect the fatigue process from ductile fracture mode. The formation of QC facets along \([100]_g\) and other unidentified planes on the fracture surface has been reported for 25\% cold-rolled 304 stainless steel fatigued in air at room temperature.\(^6\) Furthermore, the stability of strain-induced martensitic microstructures during fatigue loading and its persistent influence on fatigue behavior have been demonstrated for deep-rolled AISI 304 stainless steel fatigued in the temperature range of 25\textdegree{C}–600\textdegree{C}.\(^13\)

In the present fractographic analysis of fracture surface for 5\% and 15\% CW specimens, some evidence of transgranular cracking in the mixed mode of ductile and brittle fracture was detected for all specimens fatigued in both in-situ and unirradiated conditions. The lower ratio of QC facets on the fracture surface for 5\% CW specimens shown in Fig. 3 implies a small influence of strain-induced martensite on fatigue behavior. Larger N\(_f\) with prolonged \(a_{cr}\) for in-situ 5\% CW specimens (see Table 2) indicates the existence of an in-situ irradiation effect on fatigue behavior, and the features of the in-situ irradiation effect are quite similar to those of 20\% CW 316 stainless steels reported in a previous work.\(^4\) Since the increase of 0.2\% YS for the irradiated 5\% CW specimens (see Table 3) suggests irradiation hardening induced by the introduction of RID clusters, the in-situ irradiation effect on the fatigue behavior of 5\% CW specimens can be explained based on the interaction between RID clusters and moving dislocations. However, as for 15\% CW specimens, a less significant difference in N\(_f\) as well as \(a_{cr}\) between in-situ and unirradiated conditions (see Table 2) indicates the reduction of in-situ irradiation effect on fatigue behavior. Irrespective of irradiation hardening for the irradiated 15\% CW specimens (see Table 3), the introduction of RID clusters would not effectively influence fatigue behavior in the in-situ irradiation condition. The higher ratio of QC facets on the fracture surface for 15\% CW specimens shown in Fig. 3 indicates more significant participation of strain-induced martensite in fatigue behavior. As preferential QC cracking along crystallographic planes related to martensitic microstructures leads to a relative decrease in the contribution of dislocation slip to the fatigue process, the strain-induced martensite in 15\% CW specimens would play an important role in reducing the in-situ irradiation effect based on the interaction between RID clusters and moving dislocations.

<table>
<thead>
<tr>
<th>CW condition</th>
<th>Dose (dpa)</th>
<th>0.2% YS (MPa)</th>
<th>UTS (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5%</td>
<td>0</td>
<td>362</td>
<td>510</td>
<td>15.1</td>
</tr>
<tr>
<td></td>
<td>0.011</td>
<td>409</td>
<td>540</td>
<td>9.5</td>
</tr>
<tr>
<td></td>
<td>0.022</td>
<td>411</td>
<td>541</td>
<td>9.3</td>
</tr>
<tr>
<td>15%</td>
<td>0.0054</td>
<td>640</td>
<td>710</td>
<td>3.2</td>
</tr>
<tr>
<td></td>
<td>0.011</td>
<td>642</td>
<td>716</td>
<td>3.2</td>
</tr>
</tbody>
</table>

Fig. 3 SEM photo of the typical fatigue fracture surfaces for (a) 5\% CW and (b) 15\% CW specimens the in-situ irradiation condition.

Table 3 Dose dependence of tensile properties for 5\% CW and 15\% CW specimens.
In high-temperature water and steam environments of a light water nuclear power plant, material susceptibility to corrosion cracking would be one of the most conclusive factors for fatigue behavior of structural materials. In the case of austenitic stainless steels, material degradation in corrosion resistance during neutron irradiation is closely connected with both irradiation hardening and radiation-induced segregation at grain boundaries, leading to irradiation-assisted stress corrosion cracking (IASCC). In such corrosive environments, the stability of austenite in stainless steels may also be related to corrosion cracking due to the higher sensitivity of stain-induced martensite to hydrogen embrittlement. Therefore, the role of strain-induced martensite in the in-situ irradiation fatigue behavior would become more complex in the light water reactor environment. More extensive efforts to understand the combined effects of in-situ irradiation and other influential factors on fatigue behavior are needed for further development of fission reactor materials designed for long life and safe operations.

5. Conclusions

Fatigue behavior of 5% and 15% cold-worked 304 stainless steels was examined under in-situ irradiation conditions with 17 MeV protons at 300°C. From the experimental results, the following conclusions can be drawn:

1) The fractographic morphology of the fracture surface was quite similar between in-situ irradiation and unirradiated conditions, and was characterized by glide plane decohesion, fatigue striations, dimples and quasi-cleavage (QC) facets, with an indication of transgranular cracking in the mixed mode of ductile and brittle fractures for both 5% and 15% CW specimens. Higher ratio of QC facets on the fracture surface for 15% CW specimens implies more significant participation of strain-induced martensite in fatigue behavior.

2) The increase of fatigue life with prolonged crack propagation indicates the existence of an in-situ irradiation effect on fatigue behavior for 5% CW specimens. The in-situ irradiation effect can be explained based on the interaction between RID clusters and moving dislocations.

3) The less significant difference in life as well as ductility between in-situ and unirradiated conditions implies the reduction of the in-situ irradiation effect on fatigue behavior for 15% CW specimens. As preferential QC cracking along crystallographic planes related to martensitic structures leads to a relative decrease in the contribution of dislocation slip to the fatigue process, the strain-induced martensite in 15% CW specimens would play an important role in reducing the in-situ irradiation effect based on the interaction between RID clusters and moving dislocations.

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REFERENCES