A Comparison of Low-cycle Fatigue Properties in Stainless Steel Types 347N and 316N

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The low-cycle fatigue (LCF) properties of an Nb-stabilized grade, type 347N, and an unstabilized grade, type 316N, of austenitic stainless steels containing approximately 0.1 mass% nitrogen were evaluated at ambient temperature and at the operating temperature of a nuclear power plant (330 °C). Type 347N exhibited a shorter fatigue life than type 316N. The difference in the fatigue lives of the two grades of steel was greater at ambient temperature than at 330 °C. The inferior LCF resistance of type 347N compared to that of type 316N was largely attributed to the presence of carbo-nitride particles in type 347N, which exhibited a bimodal size distribution. Fine particles induced strong secondary cyclic hardening by pinning dislocation cell walls at ambient temperature, whereas no secondary hardening occurred at 330 °C. The hardening caused a higher stress concentration at the tip of the fatigue crack at constant strain amplitude, which resulted in a lower fatigue life at ambient temperature. Coarse particles acted as material defects that deteriorated the fatigue resistance by creating voids on the surface of the propagating fatigue crack, regardless of the testing temperature. An observation of the outer surface showed that the two types of steel exhibited different crack initiation modes on the surface. In type 316N, the crack initiation mode was predominantly intergranular at a high strain amplitude but transgranular at lower strain amplitude. In type 347N, the crack initiation mode was always intergranular, regardless of the strain amplitude.

1. Introduction

Coolant piping in a nuclear power plant (NPP) experiences repeated, severe thermal stress because the temperature gradients along the pipe depend on the mass flow rate change and temperature transients during start-up and shut-down. The flow stability in this component is therefore of considerable concern. Thus, at the design stage, the methodology of elastic-plastic fracture mechanics, called the leak-before-break analysis, has been adopted for some pipes.1,2) The coolant pipes are generally fabricated from unstabilized grades of austenitic stainless steel, such as types 304 and 316. Some pipes, including a pressurized surge line, have been fabricated using a stabilized grade, type 347, based on the fact that at a high temperature, it is stronger than other grades of austenitic stainless steel. The niobium in type 347 improves the yield strength by means of a solid solution and precipitation hardening.3) Moreover, this grade of steel is well known for its excellent resistance to intergranular corrosion and intergranular stress corrosion cracking.4–6) Recent reports, however, have indicated its inferior fracture toughness compared with that of unstabilized grades.7–11) Using fractographic observation, such inferior fracture resistance was found to be mainly caused by the presence of coarse carbide particles that form voids or cracks, which contribute to the growth of the main crack.

Furthermore, one of the main ageing degradation mechanisms of the coolant pipes in NPPs is associated with repeated thermal strain.5,12–16) Thus, the evaluation of the low-cycle fatigue (LCF) property of piping materials is critical. A considerable amount of LCF data has been accumulated over the last several decades. However, evaluations on stabilized grades of austenitic stainless steel are relatively limited. The stabilizing element (Nb) is detrimental for the fracture toughness in type 347 and the LCF behavior may also be influenced by the same element; yet, no systematic evaluation on this subject has been reported. In this article, the LCF properties of type 347N, a stabilized grade of stainless steel, and type 316N, an unstabilized grade of stainless steel, are compared. Both types of steel contained optimum nitrogen content for additional strengthening effect.7,10) This article especially describes the effect of niobium on dislocation substructure, fatigue crack initiation, propagation behavior, and fatigue life by comparing it with unstabilized austenitic stainless steel.

2. Experimental Procedure

Tables 1 and 2 list the chemical compositions and tensile properties of the steels used in this experiment, respectively. Both types of steels had similar carbon content of approximately 0.04 mass%, and contained approximately 0.1 mass% nitrogen, which improved resistance to corrosion and permitted higher stress levels, as compared to other standard grades of austenitic stainless steel, at the reactor temperature. The steels were solution-annealed at 1050 °C for one hour after they were rolled into 30-mm thick plates. The resulting grain size appeared to be finer in type 347N than in type 316N due to the presence of the niobium carbide particles that suppress grain growth in the former. Low-cycle fatigue tests were performed on hollow cylindrical specimens with a gauge length of 8 mm and outer and inner gauge diameters of 7 mm and 4 mm, respectively. The tests were carried out in air by employing saw-tooth waves, using an

Table 1 Chemical composition of the steels (mass%).

<table>
<thead>
<tr>
<th>Alloys</th>
<th>Cr</th>
<th>Ni</th>
<th>Mn</th>
<th>Si</th>
<th>Co</th>
<th>Mo</th>
<th>C</th>
<th>N</th>
<th>Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type 347N</td>
<td>17.22</td>
<td>9.14</td>
<td>1.58</td>
<td>0.48</td>
<td>0.102</td>
<td>0.30</td>
<td>0.040</td>
<td>0.10</td>
<td>0.44</td>
</tr>
<tr>
<td>Type 316N</td>
<td>17.46</td>
<td>12.36</td>
<td>1.00</td>
<td>0.59</td>
<td>—</td>
<td>0.30</td>
<td>0.037</td>
<td>0.11</td>
<td>—</td>
</tr>
</tbody>
</table>

Table 2 Tensile properties of the steels at 330 °C

<table>
<thead>
<tr>
<th></th>
<th>YS (MPa)</th>
<th>UTS (MPa)</th>
<th>El (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type 347N</td>
<td>202</td>
<td>469</td>
<td>38</td>
</tr>
<tr>
<td>Type 316N</td>
<td>193</td>
<td>513</td>
<td>46</td>
</tr>
</tbody>
</table>
Instron 8801 servohydraulic testing machine equipped with an induction heating system. The fatigue tests were performed under strain-controlled and fully reversed conditions at ambient temperature and 330°C, at a strain rate of $4 \times 10^{-3} \text{s}^{-1}$. The specimens were heated on an induction coil, and the temperature was controlled within an accuracy of ±2°C during the tests. Testing at 330°C began within 20 min after reaching a desired temperature. To reveal the fractographic morphologies, the outer surfaces, the fatigue crack surfaces, and the longitudinal sections of the failed samples were examined using a scanning electron microscope (SEM). The dislocation substructure of the fatigued samples was investigated using a transmission electron microscope (TEM). The composition of the precipitates was analyzed using an energy dispersive spectrum (EDS) analyzer.

3. Results

3.1 Microstructure before LCF and tensile properties

The stabilized steel grade, type 347N, is known to have a bimodal distribution of coarse and fine carbo-nitride particles due to the combination of C and N with a stabilizing element. Figure 1 shows the typical appearance of the particles in type 347N. After further analysis using the TEM, it was found that the fine particles comprised a mixture of Nb(CN) and NbCr(CN), while the coarse particles were primarily composed of Nb(CN), as shown in Fig. 2. Table 2 shows the tensile data for types 316N and 347N at 330°C. Compared to type 316N, type 347N exhibited higher yield stress but lower tensile stress and elongation.

3.2 Fatigue life

Figure 3 depicts the plot of the total strain range versus the number of cycles to failure. The number of cycles to failure ($N_f$) was defined as the number of cycles at which the load dropped to 80% of the saturated value. At the same testing temperature, the fatigue life of type 347N was shorter than that of type 316N. An increase in testing temperature decreased the fatigue life of the two steel grades, which is in accordance with the general behavior of austenitic stainless steels. However, it should be noted that the temperature effect on LCF resistance is less significant in type 347N than in type 316N, as shown in Fig. 4. In other words, when compared to type 316N, the LCF resistance of type 347N is inferior, and the difference is more distinctive at ambient temperature than at 330°C.

3.3 Cyclic stress response

Cyclic stress response represents the locus of the tensile peak stress of the hysteresis loop as a function of the number of fatigue cycles. Figure 5 shows the responses for types 316N and 347N at ambient temperature and 330°C. Type 316N typically exhibited cyclic softening after slight initial cyclic hardening, except in the case of a total strain range of ±2.0% being tested at ambient temperature, where it showed...
no initial cyclic hardening. On the other hand, the cyclic responses of type 347N, tested at ambient temperature, were characterized by strong secondary hardening after the stages of initial hardening, saturation, and slight softening in the low-strain range. The extent of the secondary hardening increased with the cyclic strain range. Furthermore, at a high strain range \((\Delta \varepsilon_t = \pm 2.0\%)\), strong, continuous hardening took place without the softening and saturation stages. However, at 330°C, the cyclic responses of type 347N exhibited no secondary hardening and decreased gradually after reaching the peak at the middle stages of their fatigue lives, except in the case of a total strain range of \(\pm 2.0\%\), for which the peak stress was saturated.

### 3.4 Dislocation substructure after fatigue failure

To compare the cyclic deformation behavior in types 316N and 347N, a TEM observation of the fatigued samples was conducted. Figure 6 shows the typical substructures at the end of the fatigue life. Overall, dislocations appeared in the cell structures after fatigue cycling. The type 316N samples exhibited sharper cell walls and lower dislocation density within the cells at 330°C than those at ambient temperature, as shown in Fig. 6(b). These outcomes indicate the occurrence of thermal recovery during the cycling. In type 347N, the dislocation substructures were developed in the form of highly tangled structures around the precipitates and the cell structures. It was noted that the cell spacing of type 347N was much smaller than that of type 316N at ambient temperature. At 330°C, however, the spacing was found to be similar to that of type 316N.

### 3.5 Fatigue crack initiation mode on the outer surface

The appearance of fatigue cracks on the outer surface of the specimens was examined using an SEM. The grain structures of the steel grades before the fatigue test are shown in the optical micrographs in Fig. 7. The typical developments of small cracks on the outer surface of the samples fatigued at 330°C under different total strain ranges are shown in Fig. 8. In Fig. 8(a), it can be seen that when type
316N was tested at a total strain range of ±1.2%, small, straight cracks, the length of which approximately corresponded to the grain size of the steel, were formed around a main, large crack perpendicular to the loading direction. Moreover, the main, large crack seemingly consisted of small, straight regions corresponding to the grain size, which supported the transgranular mode of crack formation. However, at a strain range of ±2.0%, the cracks formed intergranularly in the shape of the grain boundary, and some of them linked with each other to form large cracks, as shown in Fig. 8(b). In type 347N, fatigue cracks were formed intergranularly, regardless of the total strain range, as shown in Figs. 8(c) and (d). The number of small cracks increased with the strain range.

4. Discussion

4.1 Cyclic stress response: the effect of the microstructure

The cyclic response of type 347N stainless steel exhibits distinctive behavior with respect to testing temperature: strong secondary cyclic hardening till the final failure at ambient temperature and almost saturated behavior for most of its fatigue life after slight initial hardening at 330°C. This is in contrast to the cyclic response of type 316N, which did not exhibit secondary hardening behavior at both temperatures. To investigate the microstructure responsible for the secondary hardening in type 347N, the dislocation substructure was examined in the specimens that were fatigued to different cycle numbers at ambient temperature. At the earliest hardening stage, the dislocations developed into a loosely tangled structure due to the dislocation interaction, as shown in Fig. 9(a). The fine carbo-nitride particles in the matrix are found to contribute to the entangled structure, as shown in Fig. 9(b). As cyclic deformation proceeded beyond the early hardening stage, the dislocations were rearranged into a veining structure (or weak cell structure) of low energy, as shown in Fig. 5(c). This process corresponds to the slight softening stage, as shown in Fig. 5(c). At this stage, small carbo-nitride particles were largely found on dislocation vein walls, where a greater number of dislocations were prone to entanglement. The last stage in the evolution of dislocation structures in fatigue cycling is shown in Fig. 9(d). When Fig. 9(d) and Fig. 9(c) are compared, it can be seen that the dislocation substructure was transformed into a distinct cell structure with lower spacing. Laird et al. have indicated that cell formation during fatigue cycling is triggered by the onset of multiple glides. To accommodate fatiguing strain without additional hardening, cell dislocations should be mobile and somewhat transparent to glide dislocations. However, in this study, the movement of the wall dislocations was rather limited in type 347N, because almost all the fine carbo-nitride particles were located on the vein or cell walls. In constant strain amplitude fatigue, this could enhance multiple-slip and induce subsequent generation of additional cell walls through the interaction between the gliding dislocations on different slip planes. It can thus be concluded that the secondary hardening observed in Fig. 5(c) is attributed to the fine...
precipitates in type 347N that increase the dislocation cell density after the early softening stage. A comparison of the cell structures in types 347N and 316N after fatigue failure supports this claim. At the same cyclic strain range, the cell structure at ambient temperature is much finer in type 347N than in type 316N, which has no carbide particles, as shown in Fig. 6.

With regard to type 347N being fatigued at 330°C, cyclic deformation may activate not only multiple-slip but also recovery due to the annihilation of dislocations. This could prevent the continuous generation of dislocation cells and lead to the early saturation of cyclic stress, as shown in Fig. 5(d).

4.2 Influence of the microstructure on fracture behavior and fatigue life

Fracture behavior and fatigue life are closely related to the underlying microstructure of materials. A characteristic microstructural feature of type 347N is the presence of the carbo-nitride particles that have a bimodal size distribution. These particles may influence the cyclic stress response discussed in the previous section and the failure mechanism of the steel during fatigue cycling.

In this study, it is unclear whether the fatigue crack initiation mode on the outer surface, shown in Fig. 8, was affected by the presence of the carbo-nitride particles. This correlation needs to be explored further. However, irrespective of the correlation, the difference in the initiation mode did not seem to noticeably influence fatigue resistance in this study. In case the initiation mode is relevant for fatigue endurance, the mode change with respect to the strain amplitude is concurrent with the slope change of the strain-life curve.23,24) In Fig. 4, there is no apparent slope change with the cyclic strain range for type 316N at 330°C. This is because although the crack initiates at the grain boundary, it grows transgranularly immediately after advancing by about one grain distance, as shown in Fig. 10 (the images were obtained from the section perpendicular to the loading axis and the direction of crack growth).

Nevertheless, it is almost certain that the carbo-nitride particles were responsible for the inferior fatigue resistance of type 347N. As indicated in the previous section, the fine particles induced the strong secondary cyclic hardening at ambient temperature. Under the condition of constant strain amplitude, this would result in a higher stress concentration at the tip of the fatigue cracks and greater crack propagation. At 330°C, this hardening effect was diminished and the difference in the fatigue lives of types 347N and 316N reduced, as shown in Fig. 4.

The coarse particles also contributed to the inferior LCF resistance of type 347N. In the authors’ previous work on fracture toughness evaluation, it was found that coarse particles were prone to breaking and they reduced fracture toughness by enhancing crack propagation.7) Figure 11 shows the comparison of the fatigue fracture surfaces of types 347N and 316N, which were fatigued at the same conditions. Figures 11(a) and (c) were obtained at a distance of about 100–200 µm from the crack initiation location, and Figs. 11(b) and (d) were obtained at a farther distance of about 1 mm. As for type 347N in Fig. 11(c), the fracture surface over a relatively small distance was fairly irregular,
and a few secondary cracks or voids related to material defects were seen. At a location much farther from the initiation site, fatigue striations were prevalent even at a short distance from the initiation site (Figs. 11(a) and (b)). These features suggest that the coarse particles in type 347N create voids or secondary cracks on the surface of the fatigue crack. Generally, in austenitic steel grades, more than half of the secondary cracks on the surface of the fatigue crack initiated site (Figs. 11(a) and (b)). These features suggest that the coarse particles in type 347N create voids or secondary cracks on the surface of the fatigue crack.

In summary, the fine and coarse carbide precipitates in type 347N steel have detrimental effects on low-cycle fatigue resistance because they induced strong secondary cyclic hardening at lower temperatures and initiated cracks or voids that contributed to the fatigue crack behavior at all testing temperatures.

5. Conclusions

The LCF resistance of type 347N stainless steel that was stabilized with niobium was compared with that of unstabilized type 316N stainless steel at ambient temperature and at 330°C. At both temperatures, type 347N exhibited a shorter fatigue life than type 316N. Furthermore, in type 347N, the fatigue life at ambient temperature was short enough to be similar to that at 330°C, whereas in type 316N, the lower test temperature resulted in a longer fatigue life. These behaviors in type 347N were attributed to the niobium carbide-nitride precipitates that have a bimodal size distribution. The fine particles induced strong secondary cyclic hardening at ambient temperature by pinning dislocation cell walls, while no secondary hardening occurred at 330°C. The hardening caused a higher stress concentration at the tip of the fatigue crack at a constant strain amplitude and resulted in a shorter fatigue life at ambient temperature. The coarse particles acted as material defects, reducing fatigue life by forming voids regardless of the testing temperature. After observing the outer surfaces, it was found that the two steel grades exhibited different surface crack initiation modes: for type 316N, it was predominantly intergranular at a high strain amplitude but transgranular at a low strain amplitude; and for type 347N, it was always intergranular, regardless of the strain amplitude.

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REFERENCES