Fatigue Damage Evaluation Using Electron Backscatter Diffraction

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It is important to identify residual strength against fatigue damage to ensure the structural integrity of plant components subjected to cyclic loading. In this study, the degree of fatigue damage induced in Type 316 stainless steel was estimated from the local change in crystal orientation (local misorientation) measured by electron backscatter diffraction (EBSD). Crystal orientations were identified by scanning the surfaces of samples that had been subjected to a fatigue test or a tensile test. The magnitude of the averaged local misorientation, \( M_{\text{ave}} \), increased as the strain amplitude and the number of cycles increased, and the relationship between strain amplitude, number of cycles and \( M_{\text{ave}} \) was obtained. Moreover, from the correlation between \( M_{\text{ave}} \) and another parameter, \( \text{MCMD} \), which reflects the average of the orientation spread within the grains, it was shown to be possible to distinguish whether damage was caused by cyclic loading or monotonic loading. Further investigation of the local misorientation revealed that the amplitude of fatigue loading could be estimated from the statistical distribution of local misorientation averaged for each grain. It was concluded that fatigue damage (residual fatigue strength) could be quantified by EBSD measurements using the estimated strain amplitude and the relationship between strain amplitude, number of cycles and \( M_{\text{ave}} \). [doi:10.2320/matertrans.M2011014]

Keywords: electron backscatter diffraction (EBSD), low-cycle fatigue, stainless steel, local misorientation, fatigue damage

1. Introduction

In the structural design of nuclear power plant components, the damage due to cyclic loading (fatigue damage) is taken into account by ensuring that the cumulative fatigue damage during plant operation does not exceed the fatigue strength of the material. The fatigue damage is evaluated using the amplitude of cyclic loading and the number of cycles estimated during plant operation. The main source of cyclic loading is the thermal stress arising during transition of operating conditions such as start-up and shut-down of the plant. However, due to the difficulty of evaluating the thermal stress, uncertain factors are considered conservatively in the design for the sake of simplicity. Moreover, a high safety margin is set for the fatigue strength of the material used in the design. Accordingly, damaged components can often continue to be used without taking countermeasures even if the evaluated fatigue damage exceeds the design criterion. Therefore, it is important to measure the true fatigue damage induced in the material.

In order to measure the fatigue damage, it is necessary to clarify the process by which the material is damaged. There are two main kinds of fatigue damage: surface cracking and bulk damage. Murakami and Miller showed, through low-cycle fatigue tests of S45C, that the fatigue life was dominated by surface crack growth and that the Manson-Coffin relation could be regarded as an alternative expression for the crack growth law, which is determined by the crack length and applied stress/strain. This concept was confirmed by experimental results, in which fatigue life was extended when the surface layer of specimens was removed during the fatigue test. On the other hand, it was pointed out that not only surface cracking but also additional damage (hereafter called “bulk damage”) accumulates in the material due to cyclic loading and reduces the fatigue life. In a previous study by one of the authors, it was shown that the local change in crystal orientation (local misorientation), which was caused by the bulk damage, was closely correlated with crack initiation in a low-cycle fatigue test of Type 316 stainless steel.

Once a crack is initiated in a component, the degree of fatigue damage and the residual strength can be evaluated based on the size of the crack. However, the resolution and accuracy of crack detection are insufficient for evaluating the fatigue damage at an early stage, which dominates the fatigue life of components. On the other hand, the bulk damage accumulates continuously from the start of plant operation. The degree of bulk damage can be evaluated by observing the evolution of defects using transmission electron microscopy or by measuring the density of defects using positron annihilation or ultrasonic attenuation. In particular, electron backscatter diffraction (EBSD) is one of the most promising techniques for assessing crystal lattice defects over a large range from several tens of nanometers to several millimeters. Cyclic plastic strain leads to the occurrence of geometrically necessary dislocations by crystallographic slip, and local misorientation develops as a result. EBSD in conjunction with scanning electron microscopy (SEM) can quantify the degree of local misorientation from measured crystal orientations.

In this study, EBSD was used to quantitatively evaluate the fatigue damage induced in Type 316 stainless steel, which is commonly used in nuclear power plant components. Firstly, low-cycle fatigue tests were conducted and fatigue damage was induced in the material. Then, the change in crystal orientations was measured using EBSD. Finally, by characterizing the microstructural change of the damaged sample, the magnitude of fatigue damage was correlated with parameters derived from measured crystal orientations.

2. Experimental Procedure

The material used for the fatigue tests was solution heat-
treated Type 316 austenitic stainless steel; its alloying constituents and mechanical properties are given in Tables 1 and 2, respectively. The material had an approximately equiaxed grain structure with an average grain diameter of 50 \( \mu \text{m} \). Round bar specimens (gauge length = 20 mm and diameter = 10 mm) were subjected to fully reversed axial strain-controlled fatigue tests, which were conducted in ambient air at room temperature under constant nominal strain. The obtained crystal orientations were processed by in

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Table 1 Chemical content of test material (mass%).

<table>
<thead>
<tr>
<th></th>
<th>Fe</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bal.</td>
<td>0.05</td>
<td>0.26</td>
<td>1.29</td>
<td>0.035</td>
<td>0.028</td>
<td>10.00</td>
<td>16.81</td>
<td>2.00</td>
<td></td>
</tr>
</tbody>
</table>

Table 2 Mechanical properties of test material.

<table>
<thead>
<tr>
<th>0.2% proof strength</th>
<th>Tensile strength</th>
<th>Elongation</th>
<th>Reduction of area</th>
</tr>
</thead>
<tbody>
<tr>
<td>278 MPa</td>
<td>602 MPa</td>
<td>0.58</td>
<td>0.74</td>
</tr>
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</table>

3. Results and Discussion

3.1 Fatigue tests

The obtained fatigue life curve is shown in Fig. 1 and the test results are summarized in Table 3. The number of cycles to failure, \( N_f \), was defined as the number when the maximum stress during the cycle dropped below 75% of the peak stress at half of the number of cycles. The regression curve is represented by:

\[
\epsilon_a = 14.1N_f^{-0.422}
\]

where \( N_f \) and \( \epsilon_a \) denote the fatigue life and the strain amplitude in percent, respectively. The change in the variation of the peak stress during the fatigue tests is shown in Fig. 2. A rapid cyclic hardening occurred at the beginning of each test, followed by a continuous increase in the stress. The degree of hardening increased with the applied strain, whereas the stress remained nearly constant at less than \( \epsilon_a = 0.7\% \).
3.2 Local misorientation map

Based on the crystal orientations obtained by the EBSD measurement, the local misorientation, $M_L$, was calculated by the following equation:

$$M_L(p_o) = \frac{1}{4} \left( \beta(p_o, p_1) + \beta(p_o, p_2) + \beta(p_o, p_3) + \beta(p_o, p_4) \right)$$

where $\beta(p_o, p)$ denotes the misorientation between a fixed point $p_o$ and neighboring points $p$ in the same grain as depicted in Fig. 3. A line was drawn between two adjacent points when the misorientation was larger than $0.8^\circ$. If a series of the lines formed a closed region, the lines were defined as a grain boundary, and the misorientation between the points of different grains was not included in the calculation of $M_L$.

Mapping data of the local misorientations for one sample obtained under $a = 1.0\%$ are shown in Fig. 4. Although the EBSD measurement was performed carefully in order to obtain accurate crystal orientations, the distribution of local misorientations was not clear. The local misorientations in Fig. 4(a) were mostly less than $0.5^\circ$, which was smaller than the typical $0.5–1^\circ$ error in crystal orientation measurements by EBSD. Therefore, it was deduced that the local misorientation map contained significant errors. In order to reduce the error in local misorientations and to obtain a clear and smooth map, the following filtering process was adopted. To remove the error in the crystal orientations obtained, the average of the crystal orientations of surrounding points (up to nine points) was calculated for each point as schematically shown in Fig. 5. Then, the local misorientations were calculated using the filtered crystal orientations. Since this filtering process includes the repetition of averaging of measured crystal orientation, the resultant local
misorientation may blur the characteristics of the distribution. However, unmeaning error can be reduced significantly. Figure 4(b) shows the local misorientation map after applying the filtering process. The error in the local misorientations was successfully reduced and the map became clearer. This filtering process was applied to all data in the following analyses.

Figure 6 shows the local misorientation maps for two samples of different strain amplitude. The magnitude of the local misorientation increased with strain amplitude. In particular, in the case of $\varepsilon_a = 1.5\%$, the local misorientation increased significantly. In these maps, grains consisting of less than 10 points are indicated by white pixels. If the crystal orientation was not identified correctly in the measurement, such a point constituted an isolated small grain and it was indicated by a white pixel. Some clusters of white pixels were observed in the maps of large strain amplitude. The diffraction pattern suggested that most of the white pixels corresponded to bcc crystal structure (martensite structure). The positions of the martensite structure correlated well with large local misorientations. In all cases, the development of local misorientation was not homogeneous but it was localized. Even in the case of $\varepsilon_a = 0.5\%$, the local misorientation became large locally (hereafter called a “localized region”). The spacing between localized regions ranged from 50 to 100 $\mu$m.

Figure 7 shows the maps for $\varepsilon_a = 1.0\%$ for different numbers of cycles. The local misorientation increased as the number of cycles increased. In the map of F-0.5%100$N_f$, some localized regions could be found, and such regions were considered to be nuclei for localization of the local misorientation in the following cycles. In the low-cycle fatigue test in the previous paper, microstructurally small cracks were preferentially initiated at the localized regions, suggesting that the local misorientation was a kind of bulk damage. Therefore, by evaluating the change in the magnitude of the local misorientation, the degree of fatigue damage can be estimated.

Local misorientation also developed due to plastic strain under monotonic loading as shown in Fig. 8. However, the characteristic of the distribution of local misorientation was slightly different from that of the fatigue samples. In the tensile samples, the degree of localization was relatively low and spread over the whole area, and the local misorientation tended to be large near grain boundaries or their junction points. As pointed out in the previous study, in tensile tests, the local misorientation easily develops near grain boundaries due to the pile-up of dislocations. No localization was found in the sample of T-2.2%.

### 3.3 Parameters $M_{\text{ave}}$ and $M_{\text{CD}}$

For a quantitative evaluation two parameters were calculated from obtained crystal orientations: the averaged local misorientation $M_{\text{ave}}$ and the modified crystal deformation $M_{\text{CD}}$. $M_{\text{ave}}$ was calculated as the log-normal mean of the local misorientation by the following equation:

$$M_{\text{ave}} = \exp \left[ \frac{1}{n} \sum_{i=1}^{n} \ln[M_L(p_i)] \right]$$

where $n$ is the number of data. It was shown that $M_{\text{ave}}$ correlated well with the degree of plastic strain induced by tensile tests. On the other hand, $M_{\text{CD}}$ was defined by the following equation:

$$M_{\text{CD}}$$
Again, \( \beta(m_k, p_i) \) denotes the misorientation between the central orientation, \( m \), of the \( k \)th grain and the point \( i \) that belongs to the \( k \)th grain, and \( n_k \) is the number of points included in the grain. \( n_g \) is the number of grains. The central orientation was determined as the averaged crystal orientation of all points included in the grain (see Fig. 9). The parameter \( MCD \) was also shown to be linearly correlated with plastic strain up to 15%. These parameters were calculated for each map and the mean value of the five maps was as summarized in Table 3.

The change in \( M_{ave} \) of fatigue and tensile samples is shown in Fig. 10, in which the mean value and variations in five maps are indicated. \( M_{ave} \) was 0.037° for the undamaged sample and increased with strain amplitude and number of cycles, although it was almost the same under \( \varepsilon_a = 0.5\% \). Therefore, it is possible to estimate the degree of fatigue damage by evaluating \( M_{ave} \). However, in order to determine the residual fatigue strength of the damaged material based on the fatigue life curve, it is necessary to identify two independent parameters; the strain amplitude and the number of cycles. Therefore, to estimate the fatigue damage from Fig. 10(a), the strain amplitude or the number of cycles must be specified.

\( M_{ave} \) was also dependent on the plastic strain as shown in Fig. 10(b). The variation in the five maps seemed to be less than that of the fatigue samples. This suggests that the scanning range of 250 × 250 \( \mu m \) is large enough to disregard the microstructural inhomogeneity formed by the tensile test. The change in \( MCD \) exhibited a similar tendency with the number of cycles and plastic strain.

Figure 11 shows the relationship between the averaged local misorientation \( M_{ave} \) and the parameter \( MCD \). The results from five maps for each specimen are plotted in Fig. 11. \( M_{ave} \) and \( MCD \) were closely correlated and increased

\[
MCD = \exp \left[ \frac{\sum_{k=1}^{n_g} \left( \sum_{i=1}^{n_k} \ln(\beta(m_k, p_i)) \right)}{\sum_{k=1}^{n_g} n_k} \right].
\]
as the strain amplitude increased. The inclination of the regression line was different for the fatigue and tensile samples. Accordingly, it is possible to discern whether the source of damage is cyclic loading or monotonic loading by evaluating the correlation between $M_{\text{ave}}$ and $MCD$.

### 3.4 Rotation axis of misorientation

In order to identify the reason for the difference in the correlation of $M_{\text{ave}}$ and $MCD$ between the fatigue and tensile samples, misorientation axis was examined. When the misorientation angles are calculated from two crystal orientations, the rotation axis of misorientation is identified as well. The change in the rotation axis was visualized by assigning colors so that the contrast was maximized. Figure 12 shows the rotation axis of the misorientation from the central orientation, which is the averaged crystal orientation evaluated for each grain (Fig. 9). The color notation was determined so that the maximum value of the horizontal component of the vector was assigned the maximum intensity of red, while the minimum value was assigned to the minimum intensity of red, and similarly the vertical and normal components were assigned maximum and minimum intensities of green and blue. The vector of rotation axis was then transferred into red-green-blue (RGB) space. It should be stressed that each grain is given a different color code and that comparisons between color and intensity of different data points are only valid when they belong to the same grain. This visualization allows examination of how the crystal orientation fluctuated inside the grain. The fatigue sample showed a frequent change of the rotation axis, whereas that of the tensile sample changed monotonically. In the tensile samples, due to relatively large strain, the whole of the specimen was deformed and exhibited a monotonous change of the rotation axis inside the grain. On the contrary, in the fatigue samples, the magnitude of the strain was not enough to develop the local misorientation spread over the sample as shown in Fig. 7(a), which indicated only a few localized regions. The fluctuation of the rotation axis was attributed to the repetition of small strain.
Figure 13 schematically shows the change in crystal orientation in the fatigue and tensile samples. The fatigue sample exhibited relatively large fluctuation in the crystal orientation. Since the parameter $M_{\text{ave}}$ is the average of the misorientation between neighboring points, the fluctuation in the crystal orientation made $M_{\text{ave}}$ larger. On the other hand, $M_{\text{CD}}$ was not influenced by the local change in the crystal orientation because it is the misorientation from the central orientation. Namely, in Fig. 13, $M_{\text{ave}}$ corresponds to differentiation of the crystal orientation curve, while $M_{\text{CD}}$ corresponds to integration of the curve. Therefore, $M_{\text{CD}}$ reflects the deformation of the whole grain and does not take into account the local change in the crystal orientation at each point. It is worth mentioning that the magnitude of $M_{\text{CD}}$ does not depend on the step size in the EBSD measurement. Accordingly, even if $M_{\text{CD}}$ was the same, $M_{\text{ave}}$ of the fatigue samples tended to be larger than that of the tensile samples.  

### 3.5 Grain-averaged local misorientation

The change in crystal orientation grain by grain was investigated next; the change within grains was discussed in Fig. 12. Figure 14 shows the local misorientation averaged for each grain (hereafter called “grain-averaged local misorientation ($M_{\text{ave(g)}}$") for samples F-1.0%100N and T-9.7%, whose local misorientation maps are shown in Figs. 4(b) and 8(b), respectively. The variation in $M_{\text{ave(g)}}$ was larger for the fatigue sample, although $M_{\text{ave}}$ of these figures was almost the same: 0.286° and 0.260° for the fatigue and tensile samples, respectively. In the tensile samples, due to relatively large strain, the local misorientation developed throughout the whole map and the scatter in $M_{\text{ave(g)}}$ was suppressed. On the other hand, in the fatigue sample, the
deformation was localized and $M_{\text{ave}(g)}$ was larger for certain grains. The magnitude of $M_{\text{ave}(g)}$ seemed to depend on the grain size; smaller grains showed larger local misorientation. This tendency was also observed in the samples of small strain amplitude as shown in Figs. 6(a) and 7(a), whereas the tensile sample with a similar value of $M_{\text{ave}}$ showed no localized region as shown in Fig. 8(a). Hence, the degree of localization depended on the strain amplitude and it can be evaluated by the variations in $M_{\text{ave}(g)}$.

$M_{\text{ave}(g)}$ was then calculated for each map. Figure 15 shows the frequency distribution obtained from five maps of the F-1.0%80N$_{t}$ sample. The number of data (grains) was typically more than 2000 from five maps, and the distribution was regarded as log-normal. Figure 16 shows the relationship between the log-normal mean and the log-normal standard deviation of $M_{\text{ave}(g)}$. The standard deviation was relatively small for the tensile samples and the fatigue samples of large strain amplitude, and it increased as the number of cycles increased except for samples F-1.0%100N$_{t}$ and F-1.5%100N$_{t}$. In particular, in the case of $e_{a} = 0.5\%$, the standard deviation increased monotonically, although $M_{\text{ave}}$ remained almost constant as shown in Fig. 10(a). Since the inclination of the correlation curve in Fig. 16 depended on the strain amplitude, it was possible to estimate the strain amplitude of the fatigue loading. Namely, large strain amplitude caused a relatively uniform distribution of local misorientation and relatively small standard deviation of $M_{\text{ave}(g)}$. On the other hand, small strain amplitude resulted in inhomogeneous development of the local misorientation and caused a relatively large standard deviation of $M_{\text{ave}(g)}$. The standard deviation of the local misorientation also exhibited a similar dependency on the strain amplitude, although the dependency was not as clear as that of $M_{\text{ave}(g)}$.

As mentioned earlier, in order to measure the residual fatigue strength of a damaged material based on the fatigue life curve, it is necessary to identify the strain amplitude and the number of cycles. Since the strain amplitude can be identified using the correlation of Fig. 16, it is possible to estimate the number of cycles to $N_{f}$ (residual fatigue strength) by evaluating $M_{\text{ave}}$ based on the relation shown in Fig. 10(a).

4. Conclusions

In order to quantify the degree of fatigue damage of Type 316 stainless steel, EBSD observations were made for samples subjected to fatigue or tensile tests. Based on the crystal orientations obtained by scanning the sample surface, mapping data of the local misorientation were obtained. The local misorientation increased as the strain amplitude and the number of cycles increased, and the relationship between the number of cycles and the averaged local misorientation $M_{\text{ave}}$ was quantified. Moreover, from the correlation between $M_{\text{ave}}$ and another parameter, $MCD$, obtained from the crystal orientations, it was demonstrated to be possible to distinguish whether damage was caused by cyclic loading or monotonic loading. Further investigation of the local misorientation revealed that the amplitude of fatigue loading correlated well with the statistical distribution of $M_{\text{ave}(g)}$, and that it was possible to estimate the strain amplitude. It was concluded that the fatigue damage (residual fatigue strength) could be quantified by EBSD measurements using the estimated strain amplitude and the relationship between strain amplitude, number of cycles and $M_{\text{ave}}$.

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