Microcharacterization of Creep Damage in Energy Structural Materials Using a Magnetic Force Microscope

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Microcharacterization of creep damage in structural materials of austenitic stainless steel has been performed using a magnetic force microscope in order to understand the degradation mechanism of structural materials in power plants such as advanced fast breeder reactors. Magnetic phases of δ-ferrite are observed in the weld metal of the as-received specimen, and these magnetic phases are interconnected. Magnetic phases of δ-ferrite are also observed in the weld metal of a creep-damaged specimen. However, in this case, the amount of magnetic phases decreases and they are not interconnected. Image processing analyses of the magnetic force microscope images indicate that the degradation of creep damage is related to the area fraction of magnetic phases and the standard deviation of magnetic force microscope images. The degree of degradation can thus be evaluated by microcharacterization using a magnetic force microscope.

Keywords: energy structural material, austenitic stainless steel, creep damage, microcharacterization, magnetic force microscope.

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1. Introduction

To ensure the structural integrity of power plants, it is necessary to understand the degradation mechanism of the structural materials. In advanced fast breeder reactors in nuclear power plants, the structures, which contain many weldments, are designed to be used at high temperatures of around 550°C for long periods of time [1]. The weld zone (Fig. 1) consists of weld metal, heat affected zone (HAZ) and base metal, and its degradation mechanism is very complicated due to differences in the mechanical and physical characteristics of each phase. Some research [2, 3] has been performed to obtain the durability characteristics of weld zones in the structural materials. However, the degradation mechanism has not been fully explored. Hence, it becomes necessary to perform durability tests such as creep tests and creep-fatigue tests on the weld zones of the structural materials and to understand their degradation mechanism.

The magnetic force microscope is a scanning probe microscope that can measure micromagnetic information. Recently, it has been commonly used to evaluate the density of magnetic materials such as magnetic recording media [4, 5]. Our group has applied a magnetic force microscope technique to evaluate various kinds of structural and functional materials, and we found that this technique was useful in understanding the degradation mechanism of these materials [6-9].

In this research, we performed creep tests of weld zones of austenitic stainless steel SUS316FR, used as a structural material for advanced fast breeder reactors. We also performed various kinds of macrocharacterization and microcharacterization in order to understand the degradation mechanism during creep. By microcharacterization, the micromagnetic characteristics around the weld zone were investigated using a magnetic force microscope, and the relationship between creep degradation and the micromagnetic characteristics was evaluated.

2. Specimens and Experiment Procedure

2.1 Materials and specimen

The material used in this study was austenitic stainless steel SUS316FR, the chemical composition of which is given in Table 1.

A weld specimen containing double U grooves was used for the creep test. Figure 2 gives the shape and dimensions of the SUS316FR weld specimen along with the details of groove geometry. SUS316L1 was used for the weld metal, and the weld current was maintained at 120 to 150A. Post weld heat treatment was not performed.
Table 1 Chemical composition of SUS316FR.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>N</th>
<th>Al</th>
<th>O</th>
</tr>
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<tbody>
<tr>
<td>0.0013</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>0.028</td>
<td>-0.0000</td>
<td>16.17</td>
<td>18.08</td>
<td>2.17</td>
<td>0.0009</td>
<td>&lt;0.005</td>
<td>0.0216</td>
</tr>
</tbody>
</table>

following which macro- and microcharacterization were conducted around the weld zone. In this study the as-received specimen is called Specimen A, and the creep-damaged specimen is called Specimen B.

2.3 Macro- and microcharacterization

After the creep test, 20×15×5mm macro- and microcharacterization samples of Specimen 316-A and Specimen 316-B were fabricated from around the weld zone and polished with water-proof abrasive papers and high-purity alumina abrasives. Their microvickers hardness was measured at a load of 5N for 15 seconds. Their remanent magnetic flux density was measured after the samples were magnetized by a Nd-Fe-B magnet. The longitudinal magnetic flux density \( B_x \) was measured using a Hall sensor at a lift-off height of 1mm.

Microcharacterization was performed using a magnetic characterization system based on a scanning probe microscope, consisting of a scanner, a controller, and a computer (Fig. 4). Microscopic measurement of magnetic force was carried out in phase detection mode at a lift-off height of 80nm. In phase detection mode, micromagnetic information and topographic information can be obtained simultaneously. The scanning size of image was about 80×80 μm².

Some of the samples were also examined by a transmission electron microscope for which thin foil samples were fabricated by electropolishing. Bright field images and electron diffraction images were taken at an accelerating voltage of 200kV.

3. Experiment Results and Discussion

3.1 Macrocharacterization

3.1.1 Vickers hardness measurement

Figure 5 plots the microvickers hardness around the weld zones of Specimen 316-A and Specimen 316-B. In Specimen 316-A, the microvickers hardness at the weld metal and HAZ is greater than that at the base metal. The maximum microvickers hardness is observed at the boundary between the weld metal and HAZ.

In Specimen 316-B, the hardness distribution exhibits almost the same trend, and the microvickers hardness at the weld metal is found to be somewhat greater than that at the base metal. However, the difference of microvickers hardness between the weld metal and the base metal in Specimen 316-B is less than that in...
Specimen 316-A, and the microvickers hardness at the weld metal in Specimen 316-B seems to decrease during creep.

3.1.2 Remanent magnetic flux density

Figure 6 plots the remanent magnetic flux density $B_y$ around the weld zones of Specimen 316-A and Specimen 316-B. In Specimen 316-A, the magnetic flux density is maximum ($0.6 \times 10^{-6}$ T) at the center of the weld metal. In Specimen 316-B, the magnetic flux density is also maximum at the center of the weld metal. However, the maximum value in this case is about $0.3 \times 10^{-6}$ T, which is much smaller than that of Specimen 316-A.

Macrocharacteristics such as vickers hardness and remanent magnetic flux density at the weld metal are found to differ from those at the base metal, and the difference of macrocharacteristics between the weld metal and the base metal decreases during creep.

3.2 Microcharacterization

3.2.1 Magnetic force microscope images at weld metal

Figure 7 (a) presents magnetic force microscope images at the center of the weld metal of Specimen 316-A. Interconnected or flake-like magnetic phases are clearly observable as bright phases in the weld metal. Figure 7 (b) is a magnified view of Fig. 7 (a) at the weld metal. The bright phase is confirmed to consist of magnetic domains, and no other phases are observed in the bright phase.

Figure 8 compares atomic force microscope and magnetic force microscope images of the electrically etched surface at the weld metal of Specimen 316-A. After etching interconnected and flake-like structures are also seen in the atomic force microscope image. However, the atomic force microscope image (Fig. 8(a)) and the magnetic force microscope image (Fig. 8 (b)) are not quite identical. Some nonmagnetic regions are also etched in the atomic force microscope image. This is because intermetallic compounds such as Laves phases appeared at the weld metal by welding.

Interconnected or flake-like magnetic phases are clearly observable as bright phases in the weld metal.
These interconnected or flake-like magnetic phases can be correctly evaluated only by using a magnetic force microscope.

Figure 9 presents transmission electron micrographs at the weld metal of Specimen 316-A. In Fig. 9 (a), flake-like phases, which are consistent with magnetic phases in Fig. 7, are observed in the weld metal. Figure 9 (b) presents an electron diffraction image and associated crystallographic analysis results. The images confirm that the interconnected or flake-like magnetic phases in Fig. 7 are δ-ferrite and the base metals are γ-phase.

3.2.2 Changes in magnetic force microscope images in weld metal during creep

Figure 10 presents magnetic force microscope images around the weld metal of Specimen 316-A. The specimen surface was not etched. However, many interconnected magnetic phases are observed at the center of the weld metal (Fig. 10(a)). The area fraction of magnetic phase decreases in the weld metal close to HAZ (Fig. 10 (b)) and at the boundary between the weld metal and HAZ (Fig. 10 (c)). No magnetic phases are observed at the base metal (Fig. 10 (d)).

Figure 11 presents magnetic force microscope images around the weld metal of Specimen 316-B. Magnetic phases are observed at the center of the weld metal (Fig. 11 (a)), at the weld metal close to HAZ (Fig. 11 (b)), and at the boundary between the weld metal and HAZ (Fig. 11 (c)). However, almost no magnetic phases are interconnected; these magnetic phases are isolated. There are no magnetic phases at the base metal either (Fig. 11 (d)).

3.2.3 Image processing analysis of magnetic force microscope images

Image processing analyses were performed to quantitatively evaluate the difference in magnetic force microscope images between Specimen 316-A and Specimen 316-B.

Figure 12 (a) presents the image processing analysis results for the area fraction of the magnetic force microscope images of Specimen 316-A and Specimen 316-B. At the weld metal in Specimen 316-A, the area fraction of the magnetic phase of δ-ferrite is about 14%, while in Specimen 316-B it is around 4%.

At the weld metal close to HAZ in Specimen 316-A, the amount of magnetic phase of δ-ferrite is about 4.5%, while in Specimen 316-B, it is 2.5%. At the weld metal, the area fraction of magnetic phase of Specimen 316-B is much smaller than that of Specimen 316-A. At HAZ and the base metal, no magnetic phases are observed in either Specimen 316-A or Specimen 316-B.

Figure 12 (b) presents the image processing analysis results for the standard deviation of the magnetic force microscope images of Specimen 316-A and Specimen 316-B. The standard deviation is calculated from each magnetic force microscope image at a size of 80x80 μm², which contains of 256x128 pixels of magnetic force microscope data.

At the center of the weld metal of Specimen 316-A the standard deviation is about 0.6, while at the center of the weld metal of Specimen 316-B, the standard deviation is about 0.1, which is much smaller than that of Specimen 316-A. At the weld metal close to HAZ, the...
crease during creep deformation, and the morphology related both to the amount of ferrite could not be obtained directly. However, in our metal, the standard deviation of Specimen 316-B is and that of the weld metal is about 0.1. At the weld metal, the standard deviation of Specimen 316-A is about 0.25, and the base metal, the difference in the standard deviation between Specimen 316-A and Specimen 316-B is much smaller than that of Specimen 316-A. At HAZ Close to HAZ and the base metal, the difference between the weld metal and the base metal decreased during creep.

Image processing analyses of the area fraction of magnetic phase and the standard deviation of the magnetic force microscope image reveal that microstructure change in the magnetic phases at the weld metal exhibited during the creep could be explained by microstructure change in the magnetic phases at the weld metal in the creep-damaged specimen by analyzing magnetic force microscope image data.

4. Conclusions

A creep test was conducted and macrocharacterization and microcharacterization were carried out in order to understand the degradation mechanism in a weld metal specimen of austenitic stainless steel SUS316FR.

(1) Based on the macrocharacterization the vickers hardness and remanent magnetic flux density at the weld metal were found to differ from those at the base metal. However, the difference between the weld metal and the base metal decreased during creep.

(2) Microcharacterization using a magnetic force microscope at the weld metal of a creep-damaged specimen indicated that the magnetic phases of δ-ferrite had decreased and the morphology of the δ-ferrite had changed.

(3) The changes in macroscopic characteristics exhibited during the creep could be explained by microstructure change in the magnetic phases at the weld metal in the creep-damaged specimen by analyzing magnetic force microscope image data.

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References


