Nondestructive Evaluation by Reversible Magnetic Permeability of the Residual Life of Ferritic 9Cr Steel Subjected to Creep-Fatigue Damage

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This work presents a nondestructive and magnetic technique for evaluating the residual life of ferritic 9Cr steel using the peak interval of reversible magnetic permeability (PIRMP). The ferritic 9Cr steel was exposed to creep-fatigue damage, and samples were prepared with different damage levels using the interrupt test. The PIRMP decreased continuously with the fraction of creep-fatigue damage. The hardness (HV) also decreased until failure. The rates of decrease of PIRMP and HV were 18.2% and 11.8%, respectively, compared with the as-aged sample. The hardness and PIRMP decreased as a function of the Larson-Miller parameter (LMP) and showed a good linear relationship with the LMP. Mechanical softening was caused by microstructural degradation during creep-fatigue damage. The microstructural features were analyzed and shown to support the variation in hardness and PIRMP. The results are of interest because mechanical softening of structural materials can be evaluated nondestructively by measuring the reversible magnetic permeability. Consequently, the PIRMP offers a useful tool for estimating the residual life of structural materials in a nondestructive and reliable manner.

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Keywords: nondestructive evaluation, creep-fatigue, degradation, permeability, residual life

1. Introduction

Ferritic steels are key materials in fossil fuel and nuclear power plant components such as boilers, steam pipes, and steam turbines. The high chromium content of these steels increases their oxidation resistance so that they can be used at higher temperatures and pressures. Although these materials are designed for superior performance at high temperature, their mechanical strength inevitably decreases, or softens, during exposure to creep and fatigue at elevated temperatures.1–3) Throughout the lifetime of a plant component, the structural material is exposed to various types of loadings and temperatures, and the interaction of various influences such as cyclic loading and thermal history can change the microstructure and mechanical properties.

Using conventional destructive methods, the variation in physical properties cannot be detected during operation because it is impossible to collect specimens for microstructural examination and mechanical tests. Therefore, to ensure the integrity and service life of structural components, it is essential to understand their mechanical properties on a microstructural level. Many researchers have conducted microstructural characterization using certain nondestructive evaluation techniques, such as acoustic, magnetic, electrical, and radiation methods.4–7) Specifically, acoustic methods such as wave velocity and acoustic nonlinearity can be applied to evaluate the material degradation of power plant components. However, these acoustic techniques are limited to the investigation of material degradation because the variation in the elastic property of structural materials is small during the entire lifetime.

However, the magnetic technique is highly sensitive to microstructural features, including grain boundaries, precipitates, voids, and dislocations, which are the principal microstructures that contribute to the micromechanical properties of materials.8,9) Magnetic properties such as coercivity and permeability are well known for their structural sensitivity characteristics. These magnetic properties are closely related to lattice defects of structural materials that evolve from creep and fatigue damages. The reversible permeability is based on the theory that the value of reversible permeability has the same differential value as the hysteresis loop. In principle, this approach is based on the foundation of a harmonics voltage induced in a sensing coil using a lock-in amplifier tuned to the frequency of the excitation.10) Especially, the reversible permeability (RP) is the limiting value of the incremental permeability when the alternating field strength approaches to zero.

In this study, creep-fatigue damaged 9Cr steel was prepared using the interrupt test. The Vickers hardness test was performed to evaluate the mechanical strength of the damaged samples. In addition, the variation in the microstructural features was investigated. The results showed that the peak interval of the reversible magnetic permeability (PIRMP) could be used to nondestructively evaluate the residual life during the service of structural materials in power plants. The reversible magnetic permeability signal was measured using a surface-type yoke probe with a ferrite core.

2. Experimental Procedure

The chemical composition in mass% of the 9Cr ferritic steel used in this study is shown in Table 1. The test materials were normalized at 1050°C and tempered at 760°C. The creep-fatigue test was conducted at 550°C under load control using a servo-hydraulic fatigue test machine with a resistance-heating furnace. A trapezoidal wave was applied and the loading rate was 2.94 kN/s. The maximum tensile load amplitude was 14.7 kN and hold time at a tensile load was 600 seconds. Cylindrical specimens were prepared with a gauge length of 16 mm, the diameter of 10 mm and shoulder radius of 20 mm. The interruption time was determined such that the ratios of fatigue cycles to the total fatigue life were
0.1, 0.2, 0.4, 0.6, 0.8, and 1. Table 2 depicts the determination of hold duration at the peak load, the number of cycles and the Larson-Miller parameter (LMP) for the interrupted creep-fatigue test. Therefore, seven types of specimens with different microstructures were prepared. The microstructural observations were performed using field emission scanning electron microscopy (FESEM) after chemical etching with Vilella’s reagent. To observe the dislocation substructures, thin foils were prepared using twin-jet polishing. Carbon replication was conducted to analyze the composition and structure of precipitates using transmission electron microscopy (TEM). The measured mechanical properties and variations in the microstructural features such as the number and size of precipitates, lath width and dislocation density of the samples are shown in Table 3. The cyclic softening was found to be the deformation characteristic of ferritic 9Cr steel subjected to creep-fatigue at the temperature of 550°C. This cyclic softening occurred to the microstructural evaluation such as a decrease in dislocation density, the growth of subgrains, coarsening of secondary phases, and generation of microvoids during creep-fatigue damage. As already shown in Table 3, the Vickers hardness decrease with creep-fatigue cycles. The martensite lath width was quantitatively measured from the TEM micrographs, using the line intersection method, from approximately 150 laths for each specimen. The X-ray diffraction patterns were obtained using a high-resolution X-ray diffractometer, with monochromatic Cu Kα1 radiation. The dislocation density was measured using the Williamson-Hall (WH) method through the XRD peak width and strain relations.\(^{11,12}\)

A surface-type probe with a ferritic core\(^{13}\) was used in the nondestructive evaluation of the reversible permeability on the surface of specimens. The ferritic yoke probe was wound with pick-up coils, i.e., AC perturbing coils for modulating the magnetic field and DC magnetizing coils for magnetizing the test specimens.\(^{13}\) Plate-shaped specimens with a length of 16 mm, the width of 5 mm, and thickness of 1 mm were magnetized by a maximum magnetic field of 12 kA/m with a sinusoidal waveform of 0.05 Hz. A perturbation field of 80 A/m was applied as a reference signal at 40 Hz. The induced first harmonic voltage in the pick-up coil was obtained using a lock-in amplifier. The reversible magnetic permeability (\(\mu_r\)) is the limiting value of the incremental permeability (\(\mu_i\)) when the alternating field strength approaches zero.\(^{13}\) The incremental permeability is obtained as \(\mu_i = \mu_0 \cdot \Delta B/\Delta H\), where \(\mu_0\) is a magnetic constant, \(\Delta B\) is the incremental magnetic induction, and \(\Delta H\) is the incremental magnetic field.

### Table 1. The chemical composition of ferritic 9Cr steel (mass%).

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>P</th>
<th>S</th>
<th>V</th>
<th>Nb</th>
<th>B</th>
<th>N</th>
<th>Co</th>
<th>Cu</th>
<th>Al</th>
<th>Fe</th>
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<tr>
<td>0.09</td>
<td>0.23</td>
<td>0.38</td>
<td>0.065</td>
<td>8.66</td>
<td>0.90</td>
<td>0.015</td>
<td>0.013</td>
<td>0.21</td>
<td>0.07</td>
<td>0.01</td>
<td>0.035</td>
<td>2.97</td>
<td>0.04</td>
<td>0.01</td>
<td>bal.</td>
</tr>
</tbody>
</table>

### Table 2. Determination of hold duration and LMP for interrupt creep-fatigue test.

<table>
<thead>
<tr>
<th>Creep-fatigue</th>
<th>10% N</th>
<th>20% N</th>
<th>40% N</th>
<th>60% N</th>
<th>80% N</th>
<th>100% N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of cycles (N)</td>
<td>95</td>
<td>190</td>
<td>380</td>
<td>570</td>
<td>760</td>
<td>950</td>
</tr>
<tr>
<td>Hold duration (h)</td>
<td>15.8</td>
<td>31.6</td>
<td>63.3</td>
<td>95</td>
<td>126.6</td>
<td>158.3</td>
</tr>
<tr>
<td>LMP</td>
<td>17.446</td>
<td>17.694</td>
<td>17.942</td>
<td>18.087</td>
<td>18.190</td>
<td>18.270</td>
</tr>
</tbody>
</table>

### 3. Results and Discussion

The reversible magnetic permeability (RMP) is the limiting value of the incremental permeability (\(\mu_i\)) when the alternating field strength approaches zero.\(^{14}\) The RP can be obtained by considering that the value of RMP is the same as the differential value of the hysteresis loop. Incremental permeability refers to the alternating field excitation for a specified static field value. Figure 1 shows the change in the reversible permeability profile for creep-fatigue damaged samples. As shown in this figure, the PIRMP has two peaks of permeability, which was twice the coercivity. The PIRMP of the as-tempered sample was 1.5263, and this value decreased with creep-fatigue damage. This phenomenon can be explained by magnetic softening resulting from microstructural changes on the atomic order during creep-fatigue damage.\(^{15}\)

Figure 2 shows the variation in the normalized values of the as-tempered sample of PIRMP and the Vickers hardness of creep-fatigue damaged samples for the 9Cr ferritic steel. The PIRMP and hardness decreased continuously over the entire lifetime. The rates of decrease for the parameters were 18.2% for PIRMP and 11.8% for Hv. Interestingly, the mechanical softening of structural materials is the primary value for considering the residual life of materials and components in the plant industry. Consequently, the PIRMP could be used to estimate the residual life of structural materials in a nondestructive and reliable manner. As shown in Fig. 3, the martensite lath width increased monotonically over the entire lifetime, but the variation became small when the creep-fatigue damage exceeded 40%. Dislocation is the primary lattice defect and successfully influences the lattice strain. Therefore, the PIRMP could be strongly affected by the dislocation density during creep-fatigue damage. The dislocation density decreased for short creep-fatigue damage up to approximately 40% of the lifetime, with little change observed thereafter. The precipitate was coarsened during the initial creep-fatigue damage of 40% lifetime, and the growth of precipitates was not observed for long-term creep-fatigue damage. This result agrees with the FWHM variation shown in Table 3. The FWHM is sensitive to the variation in the microstructure and stress-strain accumulation in the material. Goto reported a rapid decrease in FWHM towards the end of the fatigue life.
in 17-4PH steel tested at stresses greater than the fatigue limit.16) A decrease in the FWHM response near the end of low-cycle fatigue tests in Cr–Mo–V steel was also observed by Ozdemir and Edwards.17) The FWHM measurement allows researchers to obtain important information on the surface state of the material, which is related to the grain distortion, dislocation density, and residual stresses.

Table 3 Variation in the microstructural features of creep-fatigue damaged samples.

<table>
<thead>
<tr>
<th>Creep-fatigue</th>
<th>0%</th>
<th>10% $N_f$</th>
<th>20% $N_f$</th>
<th>40% $N_f$</th>
<th>60% $N_f$</th>
<th>80% $N_f$</th>
<th>100% $N_f$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vickers hardness (Hv)</td>
<td>302 ± 5</td>
<td>297 ± 4</td>
<td>289 ± 5</td>
<td>285 ± 8</td>
<td>286 ± 3</td>
<td>277 ± 5</td>
<td>267 ± 6</td>
</tr>
<tr>
<td>No. precipitates (count/µm²)</td>
<td>18 ± 5</td>
<td>15 ± 3</td>
<td>17 ± 4</td>
<td>17 ± 4</td>
<td>15 ± 5</td>
<td>14 ± 3</td>
<td>13 ± 4</td>
</tr>
<tr>
<td>Size of precipitates (nm)</td>
<td>85.1 ± 2.5</td>
<td>87.2 ± 2.1</td>
<td>87 ± 2.7</td>
<td>90 ± 3.1</td>
<td>91 ± 3.7</td>
<td>91.7 ± 4.2</td>
<td>91.5 ± 4.5</td>
</tr>
<tr>
<td>Dislocation (10¹⁵ m⁻²)</td>
<td>1.67</td>
<td>1.49</td>
<td>1.35</td>
<td>1.34</td>
<td>1.29</td>
<td>1.27</td>
<td>1.29</td>
</tr>
<tr>
<td>Lath width (nm)</td>
<td>214 ± 10</td>
<td>255 ± 12</td>
<td>304 ± 15</td>
<td>351 ± 17</td>
<td>365 ± 18</td>
<td>374 ± 18</td>
<td>397 ± 19</td>
</tr>
<tr>
<td>FWHM $d_{10}$ (deg.)</td>
<td>0.618</td>
<td>0.586</td>
<td>0.570</td>
<td>0.522</td>
<td>0.516</td>
<td>0.517</td>
<td>0.521</td>
</tr>
</tbody>
</table>

Fig. 1 RMP profiles of creep-fatigue damaged samples; (a) as-tempered, (b) 0.1, (c) 0.6 and (d) 1.

Fig. 2 Normalized PIRMP and Vickers hardness of as-tempered specimen.

A higher PIRMP is caused by pinning of the domain walls and retardation of wall motion due to the increased amount of lattice defects, fine precipitates and internal residual stress that influence the mechanical strength.7,10,14) As shown in Fig. 2, mechanical softening of the materials is the physical phenomenon linked to the decrease in mechanical strength after long-term degradation by creep, fatigue, and creep-
fatigue. Long-term degradation of structural materials might cause coarsening of intermetallic particles, generation of stable precipitates, depletion of alloy atoms in the matrix, recovery of lattice defects such as dislocation and vacancy, phase transformation of unstable phases, and recovery of martensite lath substructure, among other factors. These microstructural features can possibly soften the material strength. Table 3 and Fig. 3 depict typical microstructural features such as the number and size of precipitates, dislocation density, lath width, and FWHM with creep-fatigue damage.

To evaluate the integrity of structural materials in plant facilities during service, degraded mechanical strength values such as hardness and tensile strength are required. The PIRMP could be used to nondestructively estimate important mechanical properties such as tensile strength and yield strength as measured by destructive methods. The variations of the hardness and PIRMP as a function of LMP are shown in Fig. 4. The hardness and PIRMP are inversely proportional to the LMP. The relationship is described as follows:

$$H_v = A_1 - B_1 \times \text{LMP}$$

$$\text{PIRMP} = A_2 - B_2 \times \text{LMP}$$

where $A_1, A_2, B_1, B_2$ are $8.14 \times 10^2, 2.95 \times 10^{-2}, 5.66, 2.407 \times 10^{-4}$, respectively.

The results indicate that the residual life can be estimated from the existing relationship between the PIRMP and a life-estimation parameter. As shown in Fig. 4, the LMP can be estimated using the hardness measured by a destructive technique. As mentioned in the introduction, a destructive technique for material evaluation is difficult and is challenging to apply to equipment in service because it is difficult to sample materials or specimens without damaging the component and facilities. However, nondestructive evaluation techniques such as PIRMP can be used to estimate the residual life of the components without damaging them.

4. Conclusions

A nondestructive evaluation technique using reversible magnetic permeability was developed for estimating the residual life of ferritic 9Cr steel subjected to creep-fatigue damage at high temperature in power plants. The reversible magnetic permeability signal was measured using a surface-type yoke probe with a ferrite core. The PIRMP decreased continuously over the entire life of creep-fatigue up to failure. The Vickers hardness also decreased monotonically with the fraction of creep-fatigue damage. The rates of decrease of the parameters were 18.2% for PIRMP and 11.8% for $H_v$. The hardness and PIRMP decreased as a function of the Larson-Miller parameter and showed a good linear relationship with the LMP. These results are highly interesting because mechanical softening of structural materials is a primary value for considering the residual life of materials and components in the plant industry. From this point of view, PIRMP could offer a potential tool for estimating the residual life of structural materials in a nondestructive and reliable manner.

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REFERENCES