Detection of Sensitization for 600 Alloy and Austenitic Stainless Steel by Magnetic Field Sensor

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Abstract. The Ni base alloy and austenitic stainless steel show usually paramagnetic property, whereas they transform into ferromagnetic along grain boundaries where sensitization occurs; this indicates a potential of non-destructive evaluation using magnetic measurement for sensitization. Here, as a feasibility study for application of non-destructive evaluation for sensitization using magnetic measurement, heat treated Ni base alloy and austenitic stainless steel were scanned by a magnetic field sensor and the changes in magnetization were obtained non-destructively. When the specimens were magnetized with the direction perpendicular to their surface before measurement, behaviour of changes in the magnetic flux density perpendicular to the surface are consistent with the results of magnetization obtained by the Vibrating Sample Magnetometer. The change in the flux density near the surface of specimen is related to the degree of chromium carbides progressing sensitization. The obtained results suggest that scanning with magnetic field sensor is effective to assess sensitization due to heat treatment for Ni base alloy and austenitic stainless steel.

1. Introduction

The Ni base alloy and austenitic stainless steel are widely used for steam generators tubing because of its high strength and high corrosion resistance. However sensitization occurs along grain boundaries during long-term heat treatment, which degrades the corrosion resistance of the materials and generates stress corrosion cracking (SCC). The SCC induces tubing failures in steam generators. The sensitization mechanism has been thoroughly investigated [1-8], and the eddy current testing detects micro cracks caused by sensitization in practical use [9, 10]. However, it is preferable to detect suspicious sensitized areas prior to the formation of cracks for keeping the integrity of the tubes.

The Ni base alloy and austenitic stainless steel show typically paramagnetic characteristics, while they transform into ferromagnetic phase along grain boundaries where sensitization occurs. Thus non-destructive evaluation (NDE) using magnetic measurement for sensitization can be possible. Several fundamental studies have been performed about these phenomena [11-13] and we also confirmed by a Vibrating Sample Magnetometer (VSM) that the alloys and steels showed ferromagnetic properties during heat treatment [14, 15]; however, the measurement using VSM is not NDE technique. Here, as a feasibility study of NDE application using magnetic measurement for sensitization, Inconel 600, Ni base alloy, and SUS304, austenitic stainless steel, were heat treated and scanned by a magnetic
field sensor and the changes in magnetization were examined non-destructively. The behaviours of the flux density near the surface of specimen depend on the combination of magnetizing direction, sensing direction and scanning direction. On the basis of obtaining results, a possibility of non-destructive evaluation (NDE) for sensitization is discussed.

2. Sensitization and Magnetization

Generally, Ni base alloy and austenitic stainless steel include chromium to enhance corrosion resistance. Sensitization is a phenomenon that chromium concentration decreases along grain boundaries and the chromium depletion zone is formed, consequently, corrosion resistance becomes weaker. The chromium depletion generates by the formation of chromium rich carbide: the carbon absorbs chromium when it forms carbide. When chromium concentration decreases to less than 10%, the Curie temperature of 600 alloy rises above the room temperature. On the other hand, the martensitic transition temperature of SUS304 shifts to the higher temperature, although typical temperature is very row temperature. These temperature shifts are attributed to the transformation into ferromagnetic phase in those materials. The carbides become coarser with absorbing chromium elements from the vicinity of grain boundaries, while chromium in the matrix diffuses to the chromium depletion zone; this causes the recovery of chromium concentration in the chromium depletion zone and the magnetization decreases for 600 alloy with longer heat treatment. Since the formation of martensitic phase contributes to the transformation into ferromagnetic for SUS304, the magnetization is kept higher during heat treatment.

3. Experimental Procedure

3.1 Preparation of Sample

The Inconel 600 alloy manufactured by the Nilaco Co. (Japan) and SUS304 stainless steel were used here. The chemical compositions of the samples are listed in Table 1. The three types of SUS304, have different carbon content, were prepared and named as H, S and L, respectively. The as-received alloy and steel were cut out to plate specimens using an electro-discharge machining, and those plates have 5 mm width, 10 mm length and 1 mm thickness. The specimens were heat treated in vacuum (less than 10^{-3} Pa) at 873 K with 1 to 400 hours for 600 alloy, at 973K with 1 to 330 hours for SUS304, respectively, followed by air-cooling. After heat treatment, the specimens were polished by an electrochemical polishing to remove an oxide layer of the specimen surface.

<p>| Table 1. Chemical compositions of (a) Inconel 600 and (b) SUS304. |
|-----------------|---------|---------|---------|---------|---------|</p>
<table>
<thead>
<tr>
<th>(a) Inconel 600</th>
<th>Ni</th>
<th>Cr</th>
<th>Fe</th>
<th>Mn</th>
<th>Si</th>
<th>C</th>
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<td></td>
<td>76</td>
<td>15.5</td>
<td>7.8</td>
<td>0.5</td>
<td>0.2</td>
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<tr>
<td>(b) SUS304</td>
<td>Ni</td>
<td>Cr</td>
<td>P</td>
<td>S</td>
<td>Mn</td>
<td>Si</td>
</tr>
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<td></td>
<td></td>
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<tr>
<td>H</td>
<td>8.2</td>
<td>18.7</td>
<td>0.023</td>
<td>0.001</td>
<td>1.63</td>
<td>0.48</td>
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<tr>
<td>S</td>
<td>9.2</td>
<td>18.5</td>
<td>0.023</td>
<td>0.001</td>
<td>1.63</td>
<td>0.51</td>
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<tr>
<td>L</td>
<td>10.0</td>
<td>18.4</td>
<td>0.025</td>
<td>0.001</td>
<td>1.65</td>
<td>0.51</td>
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3.2 Measurements

A magnetic field sensor scanned over the specimens after magnetization to investigate an applicability of the technique for a practical application. A hall sensor (Arepec HHP-S series), was adopted to detect surface magnetic flux density, which reflects the amount of magnetization inside a specimen, and to evaluate the degree of chromium depletion of specimens. Fig. 1 shows the measurement setup to scan a hall sensor over a specimen. The active area of hall sensor has dimensions of 20 $\mu$m x 20 $\mu$m and detects the component of magnetic flux density perpendicular to the surface of the specimen. The origin is the center of the specimen and width, longitudinal and perpendicular directions are defined as the $x$-, $y$- and $z$- directions, respectively. Before measurement, the specimens were magnetized with the particular direction using VSM and the sensor scanned along with $x$- or $y$- direction. The combination of magnetizing and scanning direction is summarized in Table 2.

4. Experimental Results and Discussion

Fig. 2 shows the magnetization curves for 600 alloy and SUS304H measured by VSM with maximum applied field of 20 kOe (1.6 MA/m), when specimens were heat treated. These curves were evaluated at room temperature. The magnetization curves show paramagnetic property for both materials before heat treatment (0 h). The susceptibilities are $4.9 \times 10^{-3}$ and $2.8 \times 10^{-3}$ for 600 alloy and SUS304H, respectively. The magnetization curves become nonlinear with increasing heat treatment time; the maximum magnetization increases up to 200 hours heat treatment and then decreases for 600 alloy, whereas it increases monotonically up to 330 hours for SUS304H. For SUS304S&L, the apparent changes in magnetization were not observed during heat treatment.

Fig. 3 shows the images of ferromagnetic phase distribution obtained by a magnetic
force microscopy (MFM) for 600 alloy. No ferromagnetic area appears on the image of the specimen before heat treatment (0 h), while the image for 100 hours heat treatment shows clear ferromagnetic phases which correspond to grain boundaries. MFM measurement may be a strong tool for evaluation of chromium depletion zone, i.e., sensitization, however, it is not applicable to the practical use. Although a transformation into magnetism occurs in quite local area, it is reflected on the averaging magnetic properties of the specimens as shown in the magnetization curves obtained by VSM measurement. Therefore, a magnetic field sensor is an expedient tool for evaluating magnetic properties of heat treated specimens.

Fig. 4 (a) – (f) show the magnetic flux density distribution obtained by scanning the sensor over the surface of the specimen for Inconel 600 alloy before and after heat treatment with the combination of magnetization direction and scanning direction in Table 2. The magnetic flux density distribution is almost zero for the specimens before heat treatment in the case of all combinations. This is consistent with the fact that the specimen is paramagnetic. In the case of (a) and (d), i.e., the scanning direction is parallel to the magnetizing direction, the intensity of magnetic flux density shows peaks only near the edges and becomes nearly zero at the centre of the specimen. This is because magnetic pole exists at the edge of the specimen. In the case of (b) and (c), the scanning direction is perpendicular to the magnetizing direction and the coordinates of the perpendicular axis to the scanning axis is zero; the magnetic flux density is zero and has no distribution. The scanning directions are parallel to in-plane where the magnetization exists in the cases of (a) – (d). When the specimens were magnetized with the direction perpendicular to their surface before measurement, i.e. cases (e) and (f), the magnetic flux density is almost constant above the specimen, though it has slight distribution near edges of specimens. From the results mentioned above, it is suitable to magnetize specimen perpendicular to its surface.

Fig. 5 shows the results of scanning the sensor over the surface of the specimen magnetized perpendicular to the surface of specimens (z-direction) with heat treatments. The scanning direction is parallel to the longitudinal direction (y-direction). Fig. 6 plots the changes in the magnetic flux density obtained from the results in Fig. 5 against the heat treatment time for Inconel 600 and SUS304H. As a reference, the saturation magnetization

<table>
<thead>
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<tbody>
<tr>
<td>(a)</td>
<td>x</td>
</tr>
<tr>
<td>(b)</td>
<td>x</td>
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<td>(e)</td>
<td>z</td>
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<td>(f)</td>
<td>z</td>
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(a) before heat treatment (b) 100 hours

Fig. 3. MFM images for Inconel 600 before and after heat treatment of 100 hours at 873K.
obtained from the VSM measurement is also plotted. The saturation magnetization is obtained by the subtraction of paramagnetic component from maximum magnetization. The behaviors of changes in the magnetic flux density detected by hall sensor are consistent with the results of magnetization obtained by the VSM. For example, the magnetic flux density increases with elapsing heat treatment time up to 200 hours and then decreases, followed by disappearance of ferromagnetic properties for Inconel 600. On the other hand, the magnetic flux density increases monotonically with heat treatment time for SUS304H, and both saturation magnetization and leakage magnetic flux density is constant before and after heat treatment with 330 hours. Consequently, the combination mentioned above is optimized for the detection of sensitization using a magnetic field sensor. In a practical use, steam generator tubing usually does not include the edge parts, therefore we can expect the uniform distribution of magnetic flux density reflecting the degree of transformation into

![Fig. 4. Distribution of magnetic flux density on surface of Inconel 600 alloy with heat treatment.](image)
A hall sensor scanned over the specimens and successfully detected the change in magnetic flux density on the surface of the specimen. The changes in the magnetic flux density are consistent with the saturation magnetization evaluated by the VSM. This change is related to the degree of chromium depletion. Thus, a potential of magnetic NDE for sensitization due to heat treatment of Inconel 600 alloy and SUS304 was revealed.

Acknowledgement

This research was partially supported by a Grant-in-Aid for Scientific Research (B), Grant No. 20360416, from Japan Society for the Promotion of Science and carried out in part at the International Research Center for Nuclear Materials Science, Institute for Materials Research, Tohoku University.

References


