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Magnetic NDE for Sensitization of Inconel 600 Alloy

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Inconel 600 alloy, Ni base alloy, is widely used for steam generator tubings where sensitization occurs at grain boundaries and sensitization will induce tubing failures. This alloy has usually paramagnetic property, however, it transforms into ferromagnetic property along grain boundaries when sensitization occurs: this means NDE using magnetism for sensitization is possible. Therefore, in this study, Inconel 600 alloys were heat treated at 873 K from 0 to 400 hours so as to generate sensitization and their magnetic properties were investigated in detail. The saturation and the residual magnetization increase with increasing heat treatment time and take a maximum. On the other hand, the coercive force decreases with the increase in time of heat treatment. We confirmed that characteristics at only grain boundaries change into ferromagnetic phase by a MFM observation. As a trial for industrial application, heat treated Inconel 600 alloy was scanned by a magnetic field sensor, and the variations in magnetization were obtained nondestructively. The results indicate a feasibility of magnetic NDE for sensitization of Inconel 600 alloy.

Keywords : nondestructive evaluation, sensitization, chromium depletion, magnetism

1. Introduction

Inconel 600 alloy, Ni base alloy, has been widely used as steam generator tubings for pressurized water reactors (PWRs) in nuclear power plants due to its mechanical strength and high corrosion resistance. However, chromium carbides precipitate along grain boundaries consequent to an effect of heat treatment. The formation of chromium carbides leads to a depletion of chromium, which progresses sensitization of the material. The sensitization causes a degradation of corrosion resistance and generates an intergranular stress corrosion cracking (IGSCC) [1-4] which will induce tubing failures during normal operation on plants. Therefore nondestructive evaluation (NDE) technique for sensitization of Inconel 600 alloy is necessity so as to keep the integrity of plants.

It is well known that Inconel 600 alloy shows usually paramagnetic [5, 6], however, it is transformed to ferromagnetic phase along grain boundaries when sensitization occurs. Hence, NDE using magnetic or electromagnetic technique for sensitization of Inconel 600 alloy was proposed and several fundamental studies have been done [6-8]. In this paper, the changes in magnetic properties of Inconel 600 alloy with heat treatment were investigated to develop a magnetic NDE for sensitization. Then, as a feasibility study for industrial applications, the variations in magnetic properties of heat treated Inconel 600 alloy were detected nondestructively using a hall sensor.

2. Magnetic Properties and Chromium Depletion

The Inconel 600 alloy has chemical compositions listed in Table 1. The magnetic properties of ternary $Ni_{76+x}Cr_{16-x}$-Fe_8 alloy were well investigated in Ref. [6]. According to Ref. [6], when $x = 0$, that is, compositions of ternary alloy is similar to that of Inconel 600 alloy, Curie temperature, T_c, is nearly 110 K. Consequently, this alloy is paramagnetic at room temperature. The Curie temperature increases with decreasing the value of x, and it becomes above room temperature (T_c = 300 K at x = 6) when the value of

Table 1. Chemical composition of Inconel 600 alloy (wt. %).

Ni	Cr	Fe	Mn	Si	C
76	15.5	7.8	0.5	0.2	0.02

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x becomes more than 6 (Cr content < 10 wt. %): this means that the alloy with $x > 6\%$ shows ferromagnetic at room temperature.

Since the chromium concentration at the grain boundaries is equal to that of the matrix before sensitization, the grain boundaries show paramagnetic as well as the matrix at room temperature. When sensitization occurs due to heat treatment, chromium carbides, M_7C_3, are formed along the grain boundaries [2, 4]. The formation of carbides absorbs chromium elements from the vicinity of the grain boundaries, which results in the generation of chromium depletion zones near the grain boundaries. Firstly, the chromium concentration around the grain boundaries decreases monotonically due to the formation of carbides and spontaneous magnetization appears in the chromium depletion zone at room temperature when the concentration reaches a critical chromium concentration (10% for Inconel 600 alloy). As heat treatment elapses, the carbides become coarser with absorbing chromium elements from the vicinity of the grain boundaries, while chromium in the matrix diffuses to the chromium depletion zone. As a result, the chromium concentration in the chromium depletion zone recovers and spontaneous magnetization around the grain boundaries disappears when it becomes above 10 wt. % with longer heat treatment.

3. Experimental Procedure

The Inconel 600 alloy manufactured by the Nilaco Co. (Japan) was used here. The as-received alloy was 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 at 873 K in vacuum (less than 10^{-3} Pa) with 1, 3, 10, 24, 50, 100, 200 and 400 hours followed by air-cooling. After heat treatment, the specimens were polished by an electrochemical polishing to remove an oxide layer of surface in the specimen.

The magnetization curves were measured by a vibrating sample magnetometer, VSM (Toei VSM P-7). The behaviors of sensitization along the grain boundaries due to chromium depletion were evaluated by a magnetic force microscopy, MFM (Seiko SPA400). Additionally, a hall sensor (Arepoc HHP-S series) scanned over the specimens after magnetization to investigate an applicability of the technique for practical applications.

4. Experimental Results and Discussion

4.1. Magnetization Curve

Fig. 1 and Fig. 2 show the magnetization curves measured by the VSM with maximum applied field of 1.6 MA/m (20 kOe) and 8 kA/m (100 Oe), respectively. Before heat treatment (0 h), the curve shows a linear series, i.e. paramagnetic property. One can see that the magnetization curves become nonlinear with increasing heat treatment time, and the maximum magnetization increases up to 200 hours heat treatment. The reduction of magnetization is confirmed above 200 hours heat treatment.

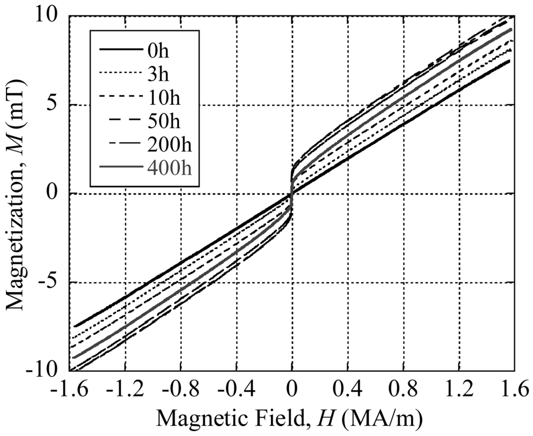


Fig. 1. Magnetization curves of heat treated Inconel 600 alloy with wide measurement range.

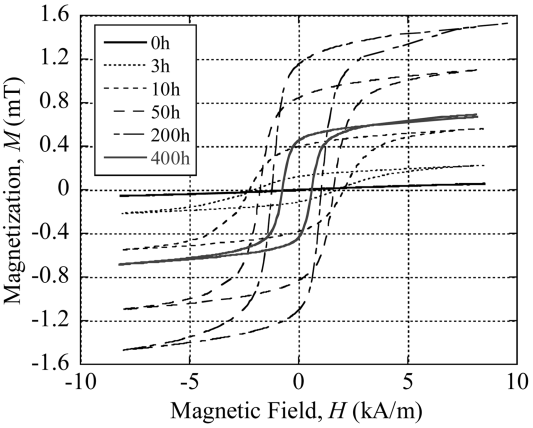


Fig. 2. Magnetization curves of heat treated Inconel 600 alloy with narrow measurement range.

Fig. 3 shows the dependence of magnetic parameters obtained from the magnetization curves on heat treatment time. The coercive force and the residual magnetization were calculated from the curves shown in Fig. 2. The saturation magnetization was calculated by the subtraction of paramagnetic component from each curve in Fig. 1.

The saturation and the residual magnetization increase up to around 100-200 hours, and then decrease, whereas the coercive force shows sudden decrease with increasing

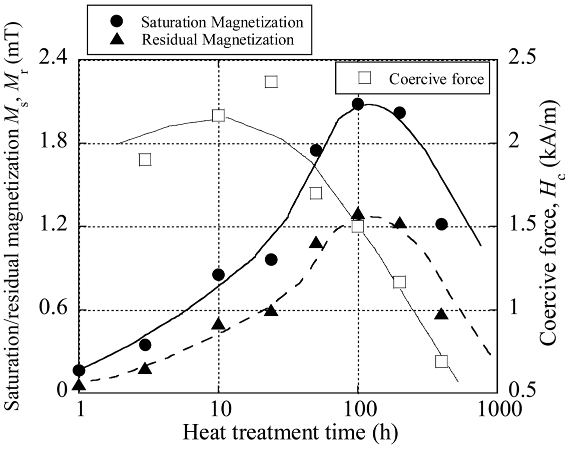


Fig. 3. Relations between magnetic properties and heat treatment time.

heat treatment time. These phenomena are explained by the formation of carbides with chromium depletion around the grain boundaries as described in Section 2.

4.2. MFM observation

Fig. 4 shows the images of ferromagnetic phase distributions on the surface of specimens obtained by the MFM. One can see that no ferromagnetic area appears on the image for the specimen before heat treatment (0 h), and the obvious ferromagnetic area is not confirmed when 1 hour heat treatment, while the images for 3 to 200 hours heat treatment show the apparent ferromagnetic phase which correspond to the grain boundaries. The contrast between ferromagnetic and nonmagnetic phase enhances up to 100 hours, and then weakens slightly. The width of ferromagnetic area also becomes slightly wider with elapsing heat treatment. The ferromagnetic phase disappears at 400 hours heat treatment due to the recovery of chromium content near the grain boundaries. The results are evidence for the increment of magnetization on the VSM results and for the generation and the annihilation of chromium depletion zone around the grain boundaries.

4.3. Scanning with magnetic field sensor

A measurement using MFM is a strong tool for evaluation of chromium depletion zone, i.e. sensitization, however, it is not applicable to the practical uses. Though a transformation of magnetism occurs in quite local areas, it reflects averaged magnetic properties of specimens as shown in the magnetization curves obtained by the VSM measurement. Since the VSM is not NDE technique, a magnetic field sensor, hall senor, was adopted to detect magnetic flux density on the surface of specimen which reflects the amount of magnetization inside the specimen, and to evaluate the degree of chromium depletion of the specimens. Fig. 5 shows the measurement setup to scan a hall sensor over a specimen. The hall sensor has dimensions of 20 μm × 20 μm and detects the component of magnetic flux perpendicular to the surface of specimen.

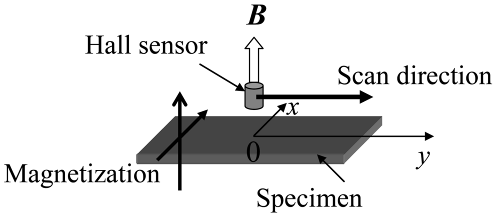
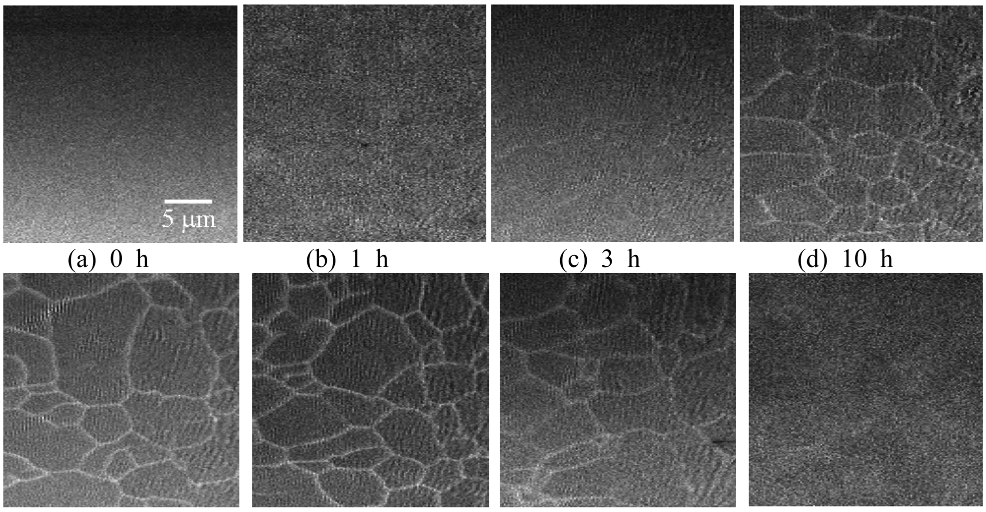


Fig. 5. Measurement system for scanning hall sensor over specimen.



(a) 0 h (b) 1 h (c) 3 h (d) 10 h

(e) 50 h (f) 100 h (g) 200 h (h) 400 h

Fig. 4. MFM images for Inconel 600 alloy with heat treatment of 0-400 hours at 873 K.

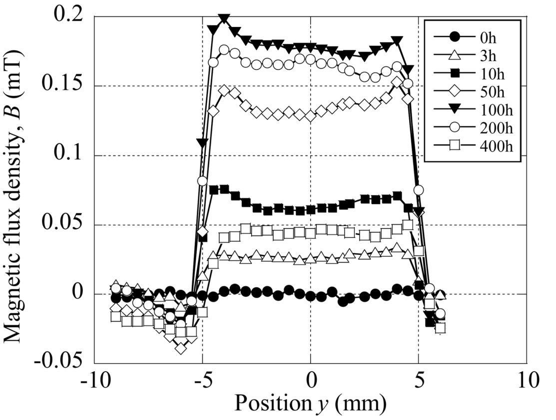


Fig. 6. Distribution of magnetic flux density on surface of Inconel 600 alloy with sensitization (Magnetization perpendicular to in-plane of specimen).

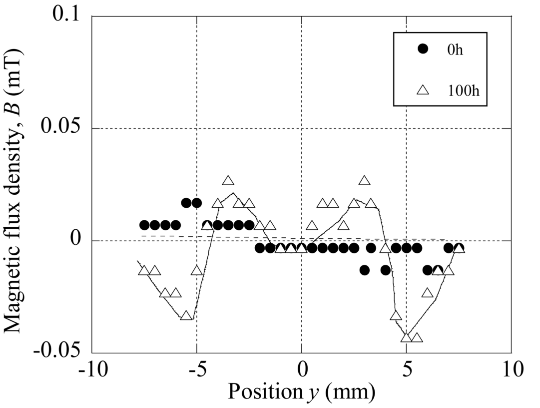


Fig. 7. Distribution of magnetic flux density on surface of Inconel 600 alloy with sensitization (Magnetization parallel to *x*- direction of specimen).

The origin is the center of specimen and longitudinal direction is defined as *y*- direction. Before measurement, specimens were magnetized with the direction perpendicular to the longitudinal axis in-plane (*x*- direction) or the direction perpendicular to the surface of specimen. The sensor scanned along with *y*- direction.

Fig. 6 and Fig. 7 show the results of scanning the sensor over the surface of specimen magnetized along the direction perpendicular to the surface of specimens or along *x*- direction, respectively. The magnetic flux density distribution is nearly constant against *y*- direction on the specimen, and the intensity of flux increases with increasing heat treatment time up to 200 hours then decreases in Fig. 6. On the other hand, magnetic flux density changes at only edge of the specimen in Fig. 7. This is because magnetic pole exists at the edge of specimen. In a practical use, steam generator tubing usually does not include the edge part. Thus, these results indicate that detection of sensitization using magnetic method is effective when a specimen is magnetized perpendicular to in-plane of specimens.

5. Conclusion

The variations in magnetic properties and ferromagnetic phase due to sensitization on Inconel 600 alloy were investigated by the VSM measurement and the MFM observation. The magnetization increases with heat treatment at first, which is attributed to the chromium depletion near the grain boundaries. The chromium depletion zone was confirmed by the observation of ferromagnetic phase formed along the grain boundaries. The ferromagnetic phase disappeared owing to the recovery of chromium content with longer heat treatment: this reflects the decrease in magnetization on the VSM results. A hall sensor scanned over the specimens and successfully detected the change in magnetic flux density on the surface of specimen. This change is related to the degree of chromium depletion. Thus, a potential of magnetic NDE for sensitization of Inconel 600 alloy was revealed.

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