Magnetic Behavior and Properties of Electrodeposited Iron Films Undergoing Flash Annealing

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フラッシュ焼鈍による鉄電析膜の磁気挙動と物性

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Introduction

The soft magnetic properties of iron are poor because the coercive force of iron is rather large. Soft magnetic materials, which have lower coercive force and a higher saturated magnetic flux density, were studied by preparing Fe-based alloys and/or compounds¹⁾. To find new applications for electrodeposited iron film, we studied its crystal structure and properties^{2), 3)}. We found that these films showed stronger soft magnetic properties after flash annealing, a method used to improve the soft magnetic properties of Fe-based amorphous alloys. P. Gorria⁴⁾ and T. Jagielinski et al.^{5), 6)}, have shown that the soft magnetic properties of nanocrystalline alloys obtained from Fe-rich and/or Co-rich amorphous metallic glass could also be improved by flash annealing. Flash annealing is handy because it can be performed in air, without appreciable oxidation of the sample, thanks to the short heating time, which ranges from some milliseconds to a few seconds.

This paper explores the effects of flash annealing on the magnetic behavior and properties of electrodeposited iron films.

Experiment

The solution (solution volume ; 100dm³) for electrodeposition contained 1.57mol/dm³ FeCl₂ and 2.04mol/dm³ CaCl₂, and the pH was adjusted using HCl solution. The substrate used for deposition was a 99.9% copper wire 0.080mm in diameter which had been annealed beforehand in a furnace, electrodeposited in NaOH solution,

and etched in HCl solution. The anode was a high-purity iron. Electrolysis was performed under an applied current density of 10A/dm², a solution temperature of 60°C, and pH2. The thickness of the deposited iron was 0.8 μm. Flash annealing and conventional annealing of iron films were performed using an electric furnace 2m long in flowing nitrogen gas. The flash annealing time was determined by the speed of the Fe-plated Cu wire passing through the furnace. The annealing temperature in this study was calculated from heat conduction theory⁷⁾, which implies the actual applied temperature. Magnetic properties were measured using a vibrating-sample magnetometer (Tamagawa : TM-VSM1550HGC). The crystal grain size *D* was calculated by Sherrer's expression by measuring the half-life width of the diffraction peak of the (110) plane which was obtained by X-ray diffraction (Rigaku, Cu-Kα, θ : 86-40°). The residual stress σ of the deposits was measured by X-ray stress measurement (Rigaku : PSPC-MSF-2).

Results and Discussion

Fig. 1 shows B-H loops of iron films used in as-deposited and flash annealing, the coercive force (*H_c*) for as-deposited was approximately 30 Oe. *H_c* for flash annealing at 700 °C and 2 seconds decreased markedly and the magnetic flux density saturated at a lower magnetic field.

Fig. 2 compares flash annealing and conventional annealing and the effects of annealing temperature on the coercive force, crystal grain size and residual stress in iron films. In conventional annealing, the decreasing temperature of *H_c* agreed with the decreasing temperature of σ and the increasing temperature of *D*. *D* increased markedly above 600 °C, and was 1200 Å at

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Fig. 1 B-H loops of iron films used in as-deposited and flash annealing (700°C, 2s).

Fig. 2 Comparison of flash annealing (○: 2s) and conventional annealing (●: 2h) and the effects of annealing temperature on the coercive force (H_c), crystal grain size (D), and residual stress (σ) in iron films.

800°C. In flash annealing, the decreasing temperature of H_c contrasted with the decreasing temperature of σ . D was markedly small compared to that in conventional annealing. Thus, flash annealing can be used to obtain iron films with a low H_c and without a marked increase in D .

Fig. 3 compares flash annealing and conventional annealing and the effects of annealing time on the above properties at an annealing temperature of 500°C. In conventional annealing, some properties of iron films hardly varied and included a low H_c , a low σ , and a large D . In flash annealing, H_c decreased with the in-

Fig. 3 Comparison of flash annealing and conventional annealing and the effects of annealing time on coercive force (H_c), crystal grain size (D), and residual stress (σ) in iron films at an annealing temperature of 500°C.

crease of annealing time. σ decreased markedly above 1 second, while D hardly varied.

As indicated above, flash annealing provides certain advantages over conventional annealing. It was of particular interest to find that flash annealing could be used to produce iron films with a low H_c and without a marked increase in crystal grain size. This phenomenon is not found in the case of conventional annealing. We surmise that the separation of the behavior of residual stress and crystal grain size in flash annealing leads to the mechanism of the soft magnetization of iron films.

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References

- 1) K. Murakami; *Iron and Steel Inst. Jpn.*, **73**, 1485 (1987)
- 2) S. Yoshimura, S. Yoshihara, T. Shirakashi, and E. Sato; *Electrochim. Acta*, **39**, 589 (1994)
- 3) S. Yoshimura, S. Yoshihara, T. Shirakashi, E. Sato, and K. Ishii; *J. Magn. Soc. Jpn.*, **18**, 281 (1994)
- 4) P. Gorria, I. Orue, F. Plazaola, and J. M. Barandiaran; *J. Appl. Phys.*, **73**, 6600 (1993)
- 5) T. Jagielinski; *IEEE Trans. Magn.*, **MAG-19**, 1925 (1983)
- 6) J. M. Barandiaran, A. Hernando, and O. V. Nielsen; *IEEE Trans. Magn.*, **MAG-22**, 1864 (1986)
- 7) S. Awano and T. Kuzuoka; *Dennetsu Kogaku*, 2nd ed. (Maruzen, Tokyo, 1961)