HIP SINTERING AND SATURATION MAGNETIZATION OF Fe₄N

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Abstract—Fe₄N powder has been sintered at below the decomposition temperature applying a hiping pressure, and the influence of oxygen content on saturation magnetization of hiped Fe₄N has been studied. The hiping temperature was restricted below 500°C, because Fe₄N was decomposed at 550°C. The maximum relative density of 97% was obtained for the Fe₄N hiped at 500°C under 200MPa. The Fe₄N hiped at 450°C under 200MPa revealed the maximum saturation magnetization of 182emu/g with a high relative density of 95%. It was confirmed that the presence of oxygen decreased saturation magnetization of Fe₄N. The Vickers hardness was measured as Hv5.5 × 10², and the corrosion resistance was higher than that of steel.

I. INTRODUCTION

Several stable compounds such as γ -Fe₄N, ϵ -Fe_xN (2 < x ≤ 1), ζ -Fe₂N are known in Fe-N system. Among them, Fe₄N powder exhibits high saturation magnetization of 186emu/g [1] at room temperature. This saturation magnetization is slightly lower than that of pure iron, which is calculated as 218emu/g. However, the electric resistance of Fe₄N is ten times larger than that of pure iron [2]. In addition, Fe₄N reveals excellent corrosion resistance and wear resistance, compared to pure iron. Therefore, the preparation of Fe₄N powder and the deposition of thin film have been widely studied [3-5]. The thin film is applicable to the magnetic recording head [6]. On the other hand, Fe₄N decomposes to iron and nitrogen at 670°C under atmospheric pressure [7]. Accordingly, it is impossible to sinter Fe₄N powder under atmospheric pressure. Thus, its shape has been limited to a powder or to a thin film. If the sintering is possible, it will be expected to use the sintered Fe₄N as a magnetic recording material.

We previously prepared HIP sintering of Fe₄N applying a hiping pressure at below the decomposition temperature [8]. The saturation magnetization of the specimen was 150emu/g, which was lower than that of Fe₄N powder (186 emu/g).

The purpose of this study is to increase the saturation magnetization of hiped Fe₄N. In order to obtain such a high saturation magnetization, the influence of oxygen content on saturation magnetization of hiped Fe₄N has been studied. In addition, the hiped Fe₄N has been characterized by Vickers hardness, three point bending strength and corrosion resistance.

II. EXPERIMENTAL

Iron powder with average particle size of 5 μ m, which was prepared from iron carbonyl, was nitrided at 500°C for 3 hours in a mixed gas of NH₃/H₂ (NH₃, 210ml/min H₂, 140ml/min) to obtain Fe₄N powder. The Fe₄N powder (10g) was charged into an aluminum capsule (35mm, ϕ 11mm) applying uniaxial pressure of 160 MPa. The process was carried out in a dried nitrogen atmosphere. Then, the sealed capsule was hiped at 450-550°C for 2 hours under 50-200 MPa.

The hiped Fe₄N was identified by XRD. The relative density of hiped Fe₄N was measured by Archimedes' method. The saturation magnetization of hiped Fe₄N was measured by a vibrating sample magnetometer in an applied field up to 7kOe at room temperature. The Vickers hardness was measured with 10gf. The three point bending strength was measured for the specimen with a size of 5mm × 5mm × 20mm. The corrosion resistance was evaluated by measuring a corrosion current density in water.

III. RESULTS AND DISCUSSION

Figure 1 shows the XRD patterns for hiped Fe₄N. The hiped specimens obtained at 450°C and 500°C under 50MPa were identified as single-phase Fe₄N. Small iron peaks were detected in the XRD pattern of the specimen hiped at 550°C. However, by the application of 200MPa, the iron peaks were slightly suppressed even at 550°C. Therefore, hiping temperature was limited at below 500°C to prevent the decomposition of Fe₄N.

Figure 2 shows the relative densities of hiped Fe₄N.

Fig. 1. XRD patterns for hipped Fe_4N .
○ Fe_4N □ Fe

Hipping pressure largely affected the sintering densities of Fe_4N . The sintering densities of Fe_4N obtained at 450°C were slightly smaller than those obtained at 500°C in the whole range of hipping pressure (50–200MPa). The maximum relative density of 97% was obtained in the Fe_4N hipped at 500°C under 200 MPa. The dense microstructure was also confirmed by scanning electron microscope.

Figure 3 shows the saturation magnetizations of Fe_4N . The Fe_4N obtained at 450°C exhibited higher saturation magnetizations than those obtained at 500°C. The Fe_4N specimens obtained under 200MPa showed high saturation magnetizations compared to those obtained under lower pressure. The maximum saturation magnetization 182emu/g

Fig.2 Relative densities of hipped Fe_4N .

Fig.3 Saturation magnetizations of hipped Fe_4N measured at room temperature.

was obtained for the Fe_4N hipped at 450°C under 200MPa. This saturation magnetization is higher than that in the previous paper (150emu/g), and is approximately same as that of Fe_4N powder (186emu/g).

Nakajima et al. reported that in a hot pressed Fe_4N with relative density of 79.9%, low saturation magnetization of Fe_4N was caused by Fe_3O_4 , Fe_2N and austenite in Fe_4N [9]. Therefore, H_2O was added to oxidize the Fe_4N powder, and the influence of oxygen content was studied. The hipped specimens including oxygen were determined as the single phase of Fe_4N by XRD. The dependence of oxygen content on the saturation magnetization is shown in Fig. 4. By the addition of small amount of oxygen (0.33%) to Fe_4N raw powder, the saturation magnetization was decreased to 157emu/g. It was confirmed that the presence of oxygen decreased saturation magnetization of Fe_4N . The low satu-

Fig.4 Dependence of oxygen content on the saturation magnetization of hipped Fe_4N under 200MPa.

Fig.5 Polarization curves of hipped Fe₄N and steel(SS400). (in water, pH 7)

ration magnetization in the previous paper may be due to the presence of oxygen.

The Vickers hardness of Fe₄N hipped at 450°C under 200MPa was Hv 5.5×10^2 , which is higher than that of pure iron (2×10^2). The three point bending strength of the Fe₄N hipped at 450°C under 200MPa was $3 \times 10 \text{ kg/mm}^2$. This strength is comparable to that of Sialon ($3.3 \times 10 \text{ kg/mm}^2$)[10] sintered under atmospheric pressure. Figure 5 shows polarization curves of hipped Fe₄N and SS400. The hipped Fe₄N revealed excellent corrosion resistance, compared to steel(SS400). The strength and corrosion resistance of hipped Fe₄N may be enough to use as a magnetic recording material.

IV. CONCLUSIONS

The Fe₄N hipped at 450°C under 200MPa revealed the maximum saturation magnetization of 182emu/g with a high relative density of 95%. The oxygen content of Fe₄N largely influenced on the saturation magnetization of Fe₄N.

Vickers hardness of the hipped Fe₄N was Hv 5.5×10^2 , and the corrosion resistance was higher than that of steel.

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