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## MAGNETIC PROPERTIES OF Fe ELECTRODEPOSITED ALUMITE FILMS

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**Abstract**—We investigated the magnetic properties such as the coercive force and the magnetic anisotropy energy of alumite magnetic films containing Fe particles electrodeposited in the pores. These properties change with respect to the particle diameter, particle length and distance between the particles. It is found that the magnetic anisotropy energy of the film is originated in shape anisotropy and in magnetostatic interaction among the particles. It is also found from the relation between coercive force and pore diameter that the magnetization reversal of this films is caused by curling mode. By using the Fe electrodeposited alumite films with the perpendicular magnetization, we developed a new magnetic rotary encoder with twice high angular resolution as conventional one with longitudinal magnetization.

## INTRODUCTION

It is well known that alumite films (anodic oxidized aluminium films) have excellent wear and corrosion resistance. The film has small pores perpendicularly to the film. The diameter of the pore and the distance between pores can be controlled by selecting the anodizing voltage and the solution.

The Fe particles size is determined by the pore diameter since the Fe can be fully electrodeposited in the pores of the film. The Fe electrodeposited alumite films have unique characteristics as follows:

- (1) The needle-shaped Fe particles stand perpendicularly to the film plane.
- (2) Each particle is separated by a non-magnetic material, that is, the oxidized aluminium.
- (3) The particle diameter, the particle length and the distance between the particles can be changed easily and widely.

Kawai et al.[1] studied the magnetic properties of alumite films containing such particles as Fe, Co, Ni and their alloys and suggested that the films have great possibilities as perpendicular magnetic recording media. Tsuya et al.[2] found a method to widen the pores before electrodeposition of magnetic particles by dissolving the side wall surface of the pores. With this method, the coercive force becomes low and the packing density (the surface ratio of magnetic particles to the film) becomes large. This means that the magnetic alumite films can be used for magnetic recording media. However, the media using the alumite magnetic film has not been realized yet, and further there is not enough theoretical research on the magnetic properties of the alumite magnetic film.

In this work we measured the magnetic anisotropy energy and the coercive force of Fe electrodeposited alumite films while changing the geometrical structure of Fe particles. In order to show that the origin of the perpendicular magnetic anisotropy and the mode of the magnetization reversal of the alumite magnetic films, the measured values of the magnetic anisotropy and the coercive force were compared with the theoretical values. Moreover we made a new type magnetic rotary encoder with high angular resolution utilizing the alumite film.

## EXPERIMENTAL PROCEDURE

Anodic oxidization of the aluminium plates

The aluminium plates (99.99 % in purity and 95  $\mu\text{m}$  in thickness) were used as substrates after being etched

in 1.2 mol/l NaOH solution and being neutralized in 0.8 mol/l  $\text{HNO}_3$  solution. Then, the substrates were anodized in solution (20  $^\circ\text{C}$ ) of either  $\text{H}_2\text{SO}_4$  (1.6 mol/l),  $\text{HOCOCOOH}$  (0.34mol/l) or  $\text{H}_3\text{PO}_4$  (0.52mol/l). The solution and anodizing voltage are chosen to obtain the desired distance between the pores. The pore diameter was widened by dissolving the pore wall surface in a 0.1 mol/l  $\text{H}_3\text{PO}_4$  solution at 30  $^\circ\text{C}$ .

Thinning and improving the uniformity of the barrier layer

In the alumite film, a semiconductive barrier layer is formed between bottom of the pores and aluminium substrate during the anodizing oxidation process, and the thickness of the barrier layer is proportional to the anodizing voltage. This thickness is very important for electrodeposition. In the film just only anodized, it is difficult to electrodeposit since the barrier layer is too thick (several hundreds angstrom). In order to thin the barrier layer, the film was anodized again at a constant current density in the pore widening solution. After this treatment, the thickness of the barrier layer becomes about 100  $\text{\AA}$ .

However, at this state, the thickness of the barrier layer is not uniform, which affects the electrodeposition speed of each particle. To unify the barrier layer, additional anodic oxidation is carried out at a constant voltage in a mixed neutral solution of  $\text{H}_3\text{BO}_3$  (0.50mol/l) and  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$  (0.05mol/l).

Electrodeposition and measuring

After the anodic oxidation and controlling the thickness and uniforming of the barrier layer, Fe was electrodeposited into pores in a mixed solution containing 0.48 mol/l of  $\text{H}_3\text{BO}_3$  and 0.2mol/l of  $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ .

The saturation magnetization was measured by a VSM, and the magnetic anisotropy by a torque meter, respectively. The crystalline structure was analyzed by an X-ray diffraction method. The electrodeposited Fe particles were liberated by dissolving the aluminium oxide in a mixed solution of  $\text{H}_3\text{PO}_4$  (0.31mol/l) and  $\text{CrO}_3$  (0.30mol/l) and observed by a TEM.

## RESULTS AND DISCUSSION

The Fe electrodeposited films with packing density 0.2 have strong (110) diffraction line regardless of the pore diameters as shown in Fig. 1. Weak (211) lines are also seen at large pore diameters. The half width of the rocking curve of (110) diffraction are 10-18 degrees and are relatively large. These indicate that the direction [100] axis of easy magnetization of Fe does not orient perpendicular to film plane and that the magnetocrystalline anisotropy energy of the particles is relatively small.

Generally, the magnetic anisotropy energy of the magnetic films which having columnar structure such as alumite magnetic films is presented by the sum of the shape anisotropy energy of the columnar particles, the magnetostatic energy caused by an interaction between the particles, and the magnetocrystalline anisotropy energy of the particles.

Figure 2 shows the dependence of the magnetic anisotropy energy upon the packing density. The easy axis of magnetization is perpendicular to the film plane when the packing density is smaller than 0.32, and in larger packing density the easy axis is in

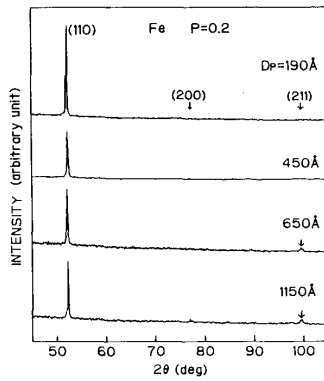


Fig. 1 The relation between the X-ray diffraction intensity and the pore diameter  $D_p$ .

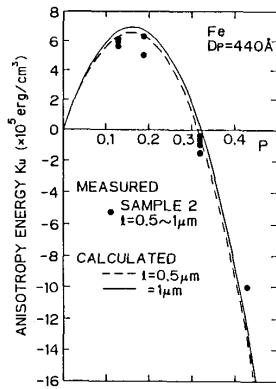


Fig. 2 The packing density  $P$  dependence of the magnetic anisotropy energy  $K_u$ .

plane. Based on the theory of Masuda et al.[3], we calculated the contribution of the sum of shape anisotropy energy and the magnetostatic energy to the total anisotropy energy. The calculated value shown by the solid and broken lines coincides with the experimental values very well. From this result, the magnetocrystalline anisotropy contributes little to the magnetic anisotropy of the Fe electrodeposited alumite films.

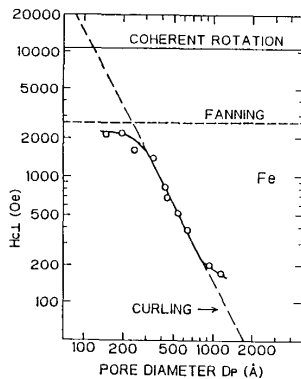


Fig. 3 The pore diameter  $D_p$  dependence of the perpendicular coercive force  $H_{c\perp}$ .

The dependence of coercive force upon the pore diameter  $D_p$  is shown in Fig. 3. The perpendicular coercive force  $H_{c\perp}$  to film plane has high value of 2200 Oe when the pore diameter is 200 Å. Then it rapidly decreases with increasing the pore diameter as a function of  $D_p^{-2}$ . This relation shows that the magnetization reversal is governed by the curling mode[4],[5]. But the coercive force is independent of the pore diameter  $D_p$  when the  $D_p$  is smaller than 200 Å. This relation shows that the mode of magnetization reversal with the small  $D_p$  is fanning mode[6].

Figure 4 shows TEM photographs of the electrodeposited Fe particles when the pore diameters are 200 Å and 440 Å, respectively. Comparing the particles of pore diameter 200 Å with 440 Å, the side surface of particles are not smooth but similar to the chain of spheres. This supports the result that the magnetization reversal of particles is occurred by the fanning mode with the pore diameter less than 200 Å as shown in Fig. 3.

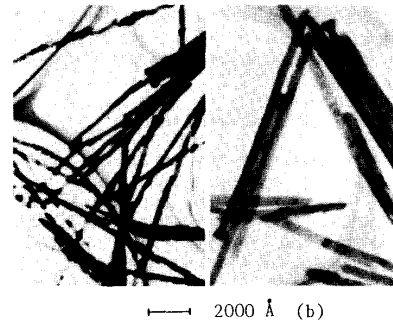


Fig. 4 TEM photographs of electrodeposited Fe particles when the pore diameters are 200 Å(a) and 440 Å(b).

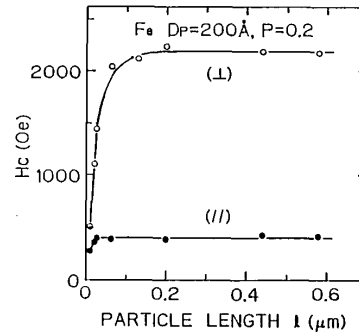


Fig. 5 The particle length  $l$  dependence of the coercive force  $H_c$ .

Next, we examined the dependence of the coercive force on the length of Fe electrodeposited particles. In Fig. 5, the perpendicular coercive force  $H_{c\perp}$  increases rapidly with increasing the particle length; it has constant value of 2200 Oe over 0.2 μm long. Thus the variation of the perpendicular coercive force  $H_{c\perp}$  corresponds to the shape anisotropy accompanied with length of the particles. If the ratio of the particle length to its diameter is very large, the coercive force in the direction of the length becomes independent of its length.

APPLICATION TO ROTARY ENCODER

Recently precise mechanical controllers need high angular resolutional magnetic rotary encoders. A conventional magnetic encoder with longitudinal

magnetic recording technique shown in Fig. 6(a) is widely used at present. However in this encoder it is difficult to decrease the magnetizing pitch less than  $80\ \mu\text{m}$  because the demagnetizing field becomes large and the output flux from the surface of the encoder drum becomes very small.

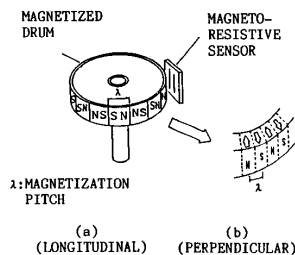


Fig. 6 The basic structure of two types of magnetic rotary encoders.

On the other hand, a new precise perpendicular magnetic rotary encoders can be developed with Fe electrodeposited alumite films because the Fe electrodeposited alumite films have perpendicular magnetic anisotropy and high saturation magnetization. As shown in Fig. 6(b) the alumite magnetic film is formed on the drum and is utilized as the write-once media with large air gap between magnetoresistive sensor and the drum. In order to obtain high resolution of the rotating angle, the media is magnetized perpendicular to magnetic drum surface. By using perpendicular magnetic recording system, the demagnetizing effect becomes very small. A magnetic domain pattern on the film surface observed by using a Bitter method is shown in Fig. 7. In this figure, the pitch of the magnetization is  $40\ \mu\text{m}$ .

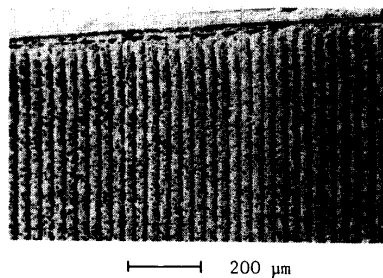


Fig. 7 A magnetic domain pattern observed by using a Bitter method.

Figure 8 shows the relation between the magnetizing pitch and the magnetic outputs with the longitudinal recording system (a) and the perpendicular recording system (b). The maximum gap between drum and sensor to detect the magnetic flux by a MR sensor was employed as a substitutional data for magnetic outputs because a sensor of the gaussmeter is too big to measure the magnetic output directly, and the magnetic output is normalized by the maximum gap detectable magnetic flux from the drum by MR sensor when the magnetizing pitch is  $80\ \mu\text{m}$ . The output of longitudinal drum becomes almost the same as that of perpendicular one when the is  $80\ \mu\text{m}$ , and when the is larger than  $80\ \mu\text{m}$ , the output of the encoder with longitudinal recording system is larger than that with the perpendicular one. However, with decreasing the magnetizing pitch, the magnetic output of longitudinal recording decreases and become smaller than that of perpendicular one since the demagnetizing effect is increased. It means that longitudinal recording media is difficult to use for

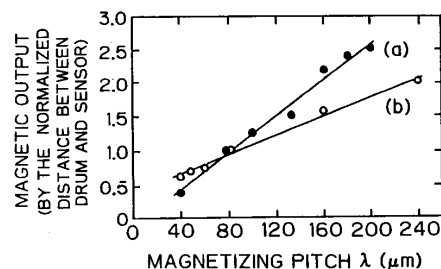


Fig. 8 The relation between the magnetizing pitch and the magnetic outputs.

(a) In the longitudinal recording system.  
(b) In the perpendicular recording system with alumite magnetic film.

the rotary encoder because the output from the longitudinal recording media becomes very small at  $40\ \mu\text{m}$ . Consequently, the rotary encoders with magnetizing pitch  $40\ \mu\text{m}$  adapted the perpendicular magnetic recording were developed and commercialized. This encoder has a small magnetizing pitch as comparing with the longitudinal one which is being marketed and it means the developed encoder has twice as high angular resolution as conventional one with the same diameter of the drum[7].

#### CONCLUSION

The magnetic anisotropy energy of Fe electrodeposited alumite films is originated in shape anisotropy energy of each particle and in magnetostatic interaction energy between the particles. It is almost independent of magnetocrystalline anisotropy energy. The perpendicular coercive force of Fe electrodeposited films is explained by the curling mode. We developed a precise magnetic rotary encoder with  $40\ \mu\text{m}$  pitch of the magnetization by using the Fe electrodeposited film. This encoder has high angular resolution than that of the conventional encoder.

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