

MAGNETIC ALUMITE DISC FOR PERPENDICULAR RECORDING

N. Tsuya, T. Tokushima*, M. Shiraki*, Y. Wakui*,

Y. Saito, H. Nakamura and Y. Katsumata

College of Engineering, Hosei Univ. Koganei Tokyo 184.

*YAMAHA R&D Lab. Nippon Gakki Co. Ltd. Hamamatsu 430, Japan

ABSTRACT

Details of making are given of perpendicular magnetic alumite discs obtained by an aid of anodization with the additional new process named "pore widening" which was effective to control the coercive force. The electro-deposited fine iron needles were in single crystalline state. The perpendicular orientation of magnetization was confirmed. The magnetic recording characteristics of rigid discs indicated a high potential for use as a perpendicular recording medium.

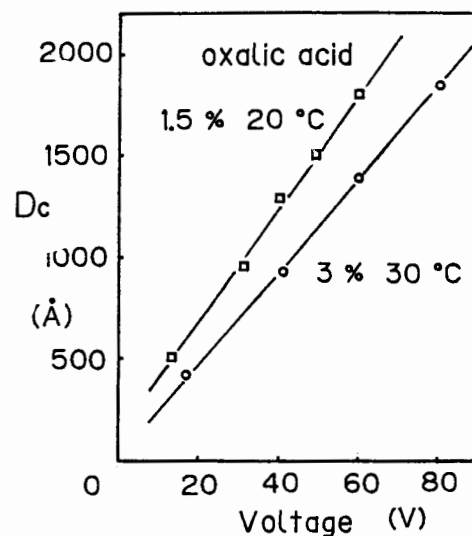
INTRODUCTION

Much effort has been devoted to developing the perpendicular recording techniques proposed by Iwasaki [1], while the investigation of perpendicular recording media has centered on sputtered Co-Cr films [2]. A number of other films such as sputtered Co-Cr-Rh [3], evaporated Co-Cr [4] and electroless-deposited Co-Ni-Mn-Re-P [5] have also been reported as possible candidates for perpendicular recording media. Ba-ferrite films have been investigated, using both coating [6] and sputtering [7] techniques. Recently, in alumite films containing electro-deposited ferromagnetic metals and alloys, we found a new way of increasing the diameter of the pores and lowering the coercive force to a value suitable [8] [9] for perpendicular recording while previous alumite films exhibited too high coercive force [10]. This paper describes the details of the preparation and the magnetic properties of magnetic alumite film, mostly hard discs, made by this new anodic oxidation method, from the view point of its use as a perpendicular recording medium.

INITIAL SUBSTRATE MATERIAL

As the substrate material of the alumite hard disc, 4-weight % Mg-Al alloy was used to ensure sufficient hardness for the surface finishing. Special attention was given to seeing that the aluminium starting material was at least 99.99% pure in order to form an anodic oxidized layer with regularly developed hexagonal cells. Each cell was accompanied by ultra-fine micropores. 1.9mm thick circular plates were prepared. The surface of these substrates was finished by diamond turning, and mirror-face with a surface roughness of far less than $0.01 \mu\text{m}$

was obtained. To obtain an extremely homogeneous oxide layer, which could only be formed on the contamination-free surface of the substrates without a naturally oxidized top layer, the substrates were subjected to an alkali etching. This process was typically performed for 20 second in an aquatic solution containing 5% NaOH at 20 - 70 °C. Substrates thus obtained were neutralized in 6% HNO_3 for one minute and soaked.

Fig.1. Cell diameter D_c against electrolytic voltage V

ANODIC OXIDATION

The alumite films with cells accompanying ultra fine pores into which iron was electro-deposited were obtained by an anodic oxidation process. This oxidation was performed in 3-weight % oxalic acid or a sulfuric acid N_2 bubbled bath at between 20 to 30°C. By this process regularly developed hexagonal close-packed uniform thick columnar Al_2O_3 cell structure was produced as a top layer of the aluminium anode. In each cell a pore with a diameter D_p of a few hundred angstroms was formed along the central axis of the cell. As shown in Fig 1 the cell diameter D_c is proportional to the electrolytic voltage V . According to this rule, keeping the electrolytic voltage V to a certain constant value between 15-65 volts, the anodization was performed and results in an oxide layer with a certain cell diameter D_c . This size was determined by Scanning Electron Microscope SEM as described later. The amount of charge

was monitored and the electrolysis was completed after satisfaction of a scheduled charge amount which was proportional to the thickness of the oxide layer.

PORE WIDENING

When alumite substrate was subjected to electro-deposition without any special treatment for pore size, the coercive force of the magnetic films formed by the deposition showed value too high compared with the head-field. We succeeded in reducing the coercive force to less than around one thousand oersteds by introducing a new process called pore widening, in which the anodized substrate was treated in a 1-% phosphoric-acid at 20-40 °C to dissolve the inner wall of each cell from the pore side. A special temperature control was employed because the pore size D_p was sensitive to the temperature of the solution. D_p could be enlarged from its initial size, which is about $D_c/3$, up to the cell size D_c as shown in Fig.2. Coercive force, shown in Table I,

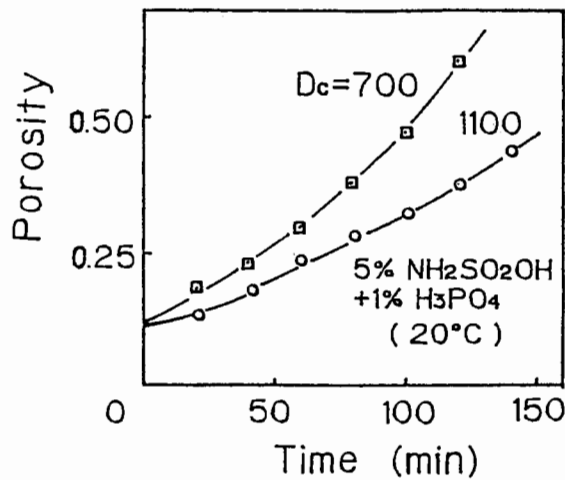


Fig.2. Porosity $(D_p/D_c)^2$ vs. pore widening time

of the magnetic alumite film formed after electro-deposition depends only upon D_p and not D_c as reported [10]. The remarkable effect of the pore widening process is not only control of the coercive force, but also the increase of porosity, which is really proportional to the magnetic flux density of the films as shown in Fig.3. The widened pore diameters produced in the oxidized Al plates were determined by the pore filling method of Nagayama[12]. In this method, the oxide layer was again anodized in an insoluble neutral solution, 0.5 M H_3BO_3 -0.05 M $Na_2B_2O_7$ at 20°C.

Table I. Coercive force H_c vs. pore diameter D_p

Pore diameter D_p (Å)	180	250	400	600	920
$H_c(0e) \perp$	2100	1780	1000	480	250
$H_c(0e) \parallel$	500	520	320	200	200

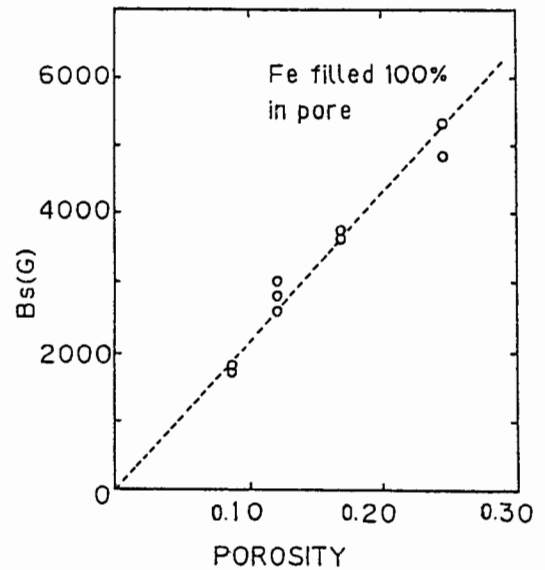


Fig.3. Porosity dependence of saturation flux density B_s

ELECTRO-DEPOSITION

The bottom of the pore mentioned above and the Al base were separated by a dense layer called a barrier layer which was electronically conductive. To realize a uniform deposition rate among pores, the thickness of the barrier layer at the base of each pore should be homogenized. For this purpose the same 1 % phosphoric-acid solution as for the pore widening was used. Typically, the current density of 30mA/dm² from the Al side was first flowed for several minutes, then the voltage was raised to 10-20 volts for a few minutes, and the thickness of the barrier layer became uniform.

Electro-deposition was carried out in an iron sulphate bath at 20-60°C with the aid of a special current consisting of a direct current with a suitable super imposed alternating current. Typically, the peak current densities were 1.6 Amp/dm² and -0.4 Amp/dm². Effectively 0.9 Amp/dm² was the average current. It should be noted that observations were made of the voltage-current Lissajous figure to monitor a reasonably proper deposition process. Typically the deposition was performed in 0.1-1mol/l solution of a pH 4-5 iron sulfate bath added

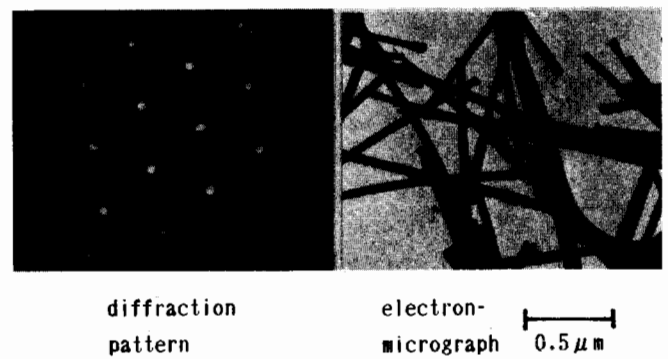


Fig.4. Single crystal iron needles

with 30 gr/l H_3BO_3 at 20-60°C. When stability was requested, a small amount of stabilizing agent, such as $MgSO_4$, glycerin or others was introduced. An overdeposition was easily detected the lightening of the surface' color. It is noted that the electro-deposited needles are pure electrolytic iron as shown in Fig.4.

POLISHING AND OVER-COATING

After electro-deposition, the magnetic film was subjected to special buff polishing using silica-sol for few minutes. The total indicated run-out of the discs was less than $10\mu m$. Surface roughness of far less than $0.01\mu m$ was attained. The Newtonian ring inspection was sometimes convenient for the quick testing of the surface status. A differential-interferometric microscope was used for observation of the local surface roughness of the discs. The film thickness under test was prepared from $1.5\mu m$ to $5\mu m$.

Coating of the surface polished discs by sputtered SiO_2 for $0.02\mu m$ was effective not only for surface protection against erroneous touching but also for corrosion against long term working environmental atmosphere. The small amount of lubricant, appropriate for the present pore structure, typically fluoro-carbon, was applied to the surface of the discs for testing.

RECORDING CHARACTERISTICS

The rigid alumite discs were measured by a Mn-Zn ring head with $0.5\mu m$ gap, $20\mu m$ track width driven at $10m/s$. High recording density D_{50} with high output as well as its envelope were satisfactorily detected. Concerning the recording properties with their dependability are shown in Table II

Table II. Recording characteristics

Output voltage(at 20 kBPI)	1.2Vpp/m T
Recording density	50 kBPI
Signal to noise ratio	29.2 db
Overwrite modulation	less than -30 db
Life test	40°C 90% RH,1000 hr pass OK
(Over coated by SiO_2 200A)	3ppm H_2S , 10ppm SO_2 , 40°C 75% RH,72 hr OK
	contact start stop 30000 OK
Missing (55 % threshold)	5/surface unchanged

SUMMARY

The details of the fabrication of alumite rigid discs by introducing the pore widening method were given. Satisfactory recording characteristics with dependability in life tests were confirmed.

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