# THE EFFECT OF OXYGEN GAS PRESSURE ON THE PROPERTIES OF Pb ADDED Ba-FERRITE SPUTTERED FILMS

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Abstract-BaM films have a lot of advantage of chemical stability and mechanical stability as compared with a metallic thin film. In this paper, (Ba·Pb)M films have been prepared by using dc magnetron sputtering system and the dependences of their crystallographic characteristics and magnetic properties on oxygen pressure(Po<sub>2</sub>) were studied. The films prepared at Po<sub>2</sub> of around 0.02mTorr exhibit a fine particle-like structure and  $\Delta\theta_{50}$  is as small as 1°. Hc<sub>+</sub>, Hc<sub>//</sub> and Ms of (Ba·Pb)M films are 700-800Oe, 200Oe and 180-230emu/cc, respectively.

#### I INTRODUCTION

Perpendicular recording proposed by Prof. S.Iwasaki et al[1] is substantially preferable for ultra high density recording under the geometrical conditions of the head-medium configuration. Many researchers have been focused on perpendicular magnetic recording technologies and there are various candidates for the perpendicular recording materials such as Co-Cr thin films[2], Fe and Co plated films on alminium oxide substrate[3], and Ba-ferrite particulate medium[4]. A chemical stability and mechanical durability as well as excellent magnetic properties are essential for recording medium. Oxide films have a lot of advantage of chemical stability and mechanical stability as compared with a metallic thin film. The authors have been studying a magnetoplumbite type of hexagonal Ba-ferrite (BaM) thin films prepared by a sputtering[5] and a sol-gel method[6] and reported that BaM sputtered rigid disks with small caxis dispersion angle $(\Delta\theta_{50})$  possess an excellent recording characteristics[7]. According to their reports, the recording density(D<sub>50</sub>) depends on  $\Delta\theta_{50}$  and the smaller  $\Delta\theta_{50}$ results in the higher D<sub>50</sub>. In order to prepare BaM films with c-axis orientation, the substrate temperature during the deposition should be elevated up to about 620°C [5]. The authors have already reported that the addition of Pb to BaM films is very effective to lower the crystallization temperature of hexagonal ferrite and the substrate temperature can be reduced to 540°C . Pb added  ${\rm BaM}(({\rm Ba \cdot Pb}){\rm M})$  films exhibited very small  $\Delta \theta_{50}$  of about 1° [8]. In reactive sputtering for the synthesis of oxide films, oxygen gas will play very important role in a crystallization. In this paper, (Ba-Pb)M films have been prepared by using dc magnetron sputtering system and the dependences of their crystallographic characteristics and magnetic properties on oxygen pressure(Po<sub>2</sub>) were studied.

# II EXPERIMENTAL PROCEDURES

(Ba·Pb)M films have been prepared by using dc magnetron sputtering system and the target used in this study

was a sintered ferrite disk with stoichiometric composition of BaM(i.e. n=6 in BaO·nFe<sub>2</sub>O<sub>x</sub>), where Pb chips of 5mm square were placed on this disk. This sintered ferrite target was reduced in nitrogen gas atmosphere at about 800°C in order to be able to discharge in a dc electric field. In this study, 10 pieces of Pb chips were placed on the target[8]. The substrate is a thermally oxidized silicon wafer and the substrate temperature(Ts) is measured with a thermocouple in contact with the substrate surface and Ts was set at  $540^{\circ}\mathrm{C}$  . After evacuating the chamber to a pressure below  $3.0 \times 10^{-6}$  Torr, argon and oxygen gas were introduced and working gas pressure(P<sub>Total</sub>) was set 2mTorr. The thickness of (Ba·Pb)M films was measured with a stylus apparatus and was about 1000Å. The crystal structure of the films was determined by X-ray diffactometry(Cu K $\alpha$ ) and magnetic properties were measured with a vibrating sample magnetometer at room temperature in the maximum field of 10kOe. The composition of the films was measured by electron probe micro analysis(EPMA).

## III RESULTS AND DISCUSSION

Figure 1 shows the x-ray diffraction diagrams for the films prepared at various oxygen partial gas pressure(Po<sub>2</sub>). Weak diffraction lines from c-plane of hexagonal M phase are observed even though oxygen gas is not introduced(Po<sub>2</sub>=0mTorr). When Po<sub>2</sub> is increased from 0.012 to 0.03 mTorr, the diffraction lines from both c-plane of hexagonal structure and (222) plane of cubic structure of spinel ferrite are observed. It is found that the hexagonal ferrite is not synthesized for further increase of Po<sub>2</sub>.4 Figure 2 shows the dependence of the intensity of x-ray diffraction lines both from (008) plane for hexagonal structure and (222) plane for cubic structure on Po<sub>2</sub>. Each intensity of diffraction line increase with increase of Po<sub>2</sub> up to 0.02 mTorr and further increase of Po<sub>2</sub> results in the decrease of each intensity.

Figure 3 shows the dependence of the lattice constant c of hexagonal ferrite on Po<sub>2</sub>. The c increases and closes to the value of bulk BaM with increase of Po<sub>2</sub>. This suggests that the oxygen content is increased in the deposited

films with increase of Po<sub>2</sub> because the oxygen ion has a large ionic radius to expand the hexagonal crystallite.

Figure 4 shows the dependence of  $\Delta\theta_{50}$  on Po<sub>2</sub> which was evaluated from the rocking curve of (008) plane. The films prepared without introducing the oxygen gas possesses relatively large value of 3.5°. The  $\Delta\theta_{50}$  decreases rapidly to about 1° with increasing of Po<sub>2</sub>. These small

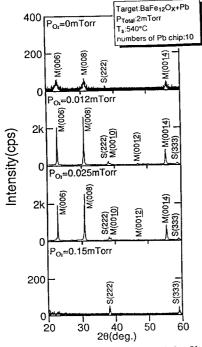


Fig.1 X-ray diffraction diagrams of the films prepared at various  $Po_2$ .

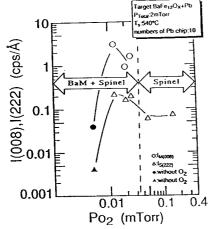


Fig. 2 Dependence of intensity of diffraction line from (008) and (222)plane on Po<sub>2</sub>.

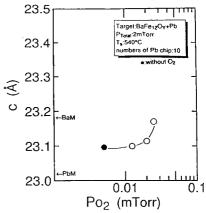


Fig.3 Dependence of lattice constant c on Po2.

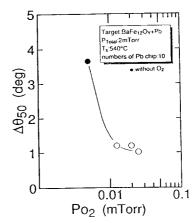


Fig.4 Dependence of  $\Delta\theta_{50}$  on Po<sub>2</sub>.

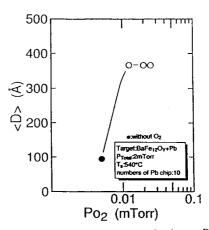


Fig.5 Dependence of average grain size on Po<sub>2</sub>.

values of  $\Delta\theta_{50}$  are suitable for the high density recording media, where the ring type head is used in perpendicular magnetic recording[7].

Figure 5 shows the dependence of average grain size on Po<sub>2</sub> which was evaluated from the x-ray diffraction line of (008) plane for hexagonal crystallite. The average grain size increases rapidly with introducing oxygen gas and is almost constant of 370Å.

Figure 6 shows the dependence of saturation magnetization(Ms) on  $Po_2$ . The Ms increases from 200 to 240 emu/cc with increase of  $Po_2$  up to 0.012mTorr and decreases for further increase of  $Po_2$ .

Figure 7 shows the dependence of coercivities on  $Po_2$ . The  $Hc_{\perp}$  and  $Hc_{//}$  for the film prepared without introducing  $Po_2$  are 1.1kOe and 0.3kOe, respectively, while those prepared at  $Po_2$  in the range from 0.02 to 0.03 mTorr decrease to about 0.8kOe and 0.1kOe, respectively.

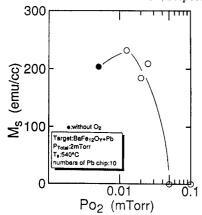


Fig.6 Dependence of Ms on Po<sub>2</sub>.

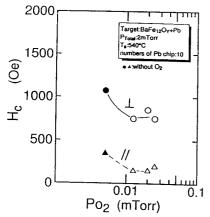


Fig.7 Dependence of coercivities on Po2.

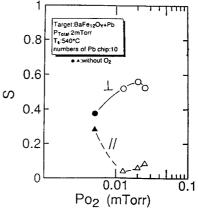


Fig.8 Dependence of squareness ratio on Po2.

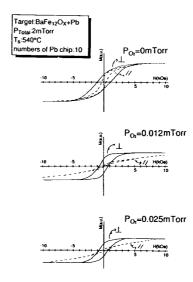


Fig.9 M-H loops for the films prepared at various Po2.

Figure 8 shows the dependence of squareness ratio on Po<sub>2</sub>. The  $S_{\perp}$  increases with increasing Po<sub>2</sub> and takes a maximum value of 0.55 at 0.02mTorr, while the  $S_{//}$  was decreases with increasing Po<sub>2</sub>.

Figure 9 shows the M-H loops for the films prepared at various  $Po_2$ . As indicated in this figure, the solid line and dashed line are M-H loop in perpendicular direction and in-plane direction, respectively. It is found that the remanent magnetization in perpendicular direction is higher than that in in-plane direction. Although the  $Hc_{\perp}$  of these films is less than 1kOe, perpendicular anisotropy field can be estimated from in-plane M-H loop and is more than 10kOe for the film prepared at  $Po_2$  of 0.12mTorr.

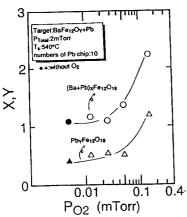


Fig.10 Dependence of (Ba+Pb) and Pb content on Po<sub>2</sub>.

Po <sub>2</sub> =0mTorr	Po <sub>2</sub> =0.012mTorr
1000 A	1000 Å
Po <sub>2</sub> =0.025mTorr	Po <sub>2</sub> =0.15mTorr
J 1000 Å	1000 Å

Fig.11 Surface structure of the films prepared at various Po<sub>2</sub>.

Figure 10 shows the dependence of (Ba+Pb) and Pb content for (Ba·Pb)M films on Po<sub>2</sub>. The value of X which represents (Ba+Pb) content in a formula of (Ba+Pb)<sub>X</sub>Fe<sub>12</sub>O<sub>19</sub>. The X is almost constant at 1.1 at Po<sub>2</sub> below 0.05mTorr. This means that these films have almost stoichiometric composition of hexagonal M-type ferrite. Further increase of Po<sub>2</sub> results in a rapid increase of the X of above 2.0. This increase is mainly due to the increase of Pb content as indicated in this figure. Although the reason of this rapid increase of Pb content in the deposited films at higher Po<sub>2</sub> is not yet clear, it may be caused by an increase of Pb-oxide in the film.

Figure 11 shows the surface of the films prepared at various Po<sub>2</sub>. It is found that all of the films consists of fine particle-like structure of about 200-300Å in diameter. These films can be expected to be a high density recording media with low noise.

#### IV CONCLUSION

(Ba·Pb)M films have been prepared by using a dc magnetron sputtering system and their crystallographic characteristics and magnetic properties were studied. It is found that the crystallinity of hexagonal M phase is improved by introducing oxygen gas up to about 0.03mTorr where  $P_{Total}$  is 2mTorr. The films prepared at  $Po_2$  of around 0.02mTorr exhibit a fine particle-like structure and the  $\Delta\theta_{50}$  is as small as 1°. The Hc $_{\perp}$ , Hc $_{//}$  and Ms of (Ba·Pb)M films are 700-800Oe, 200Oe and 180-230emu/cc, respectively. These values of Hc should be improved to be higher than 1kOe for ultra high density recording media.

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