Proc. Schl. Eng. Tokai Univ., Ser. E
33（2008）29-34

Oblique Deposition of Giant magnetostrictive Thin Film Formed by Ion-plating Process

by

Kiyoshi SHINOBE[*1], Masahide MORITA[*1] and Yoshihito MATSUMURA[*2]

(Received on Mar. 31, 2008 and accepted on Jul. 9, 2008)

Abstract

The effects of oblique deposition to induce high magnetostrictive susceptibility and huge magnetostriction were studied. The Giant magnetostrictive Tb-Fe films were prepared by an ion plating process with inclined vapor flux using substrates angled with 0, 30, 45, and 60 degrees. Increasing the substrate angle induced strongly in-plane magnetic anisotropy of the films by the oblique deposition. The magnetic and magnetostrictive characteristics of Tb-Fe films were effected by an oblique anisotropy. This oblique anisotropy of the Tb-Fe films may be induced by the shape anisotropy connected with columnar structure morphology. Magnetostriction of samples increased with increasing substrate angle. The maximum value of magnetostriction for film sample was 2000 ppm at the deposition angle of 60°.

Keywords: *Giant magnetostriction materials, Thin Film, Tb-Fe, Oblique deposition, Ion-Plating*

1. Introduction

The giant magnetostrictive (GM) materials have exhibit huge magnetostriction over 1000 ppm[1)]. The GM bulk compounds are applied to devices such as powerful sonars, linear actuators and oscillation dampers[2, 3)]. Recently, we reported the GM films prepared by several deposition processes such as vacuum flash evaporation[4-6)], ion plating[7)], magnetron sputtering[8)] and ion beam sputtering processes[9, 10)]. The preparation conditions of $TbFe_2$ films such as partial pressures of residual gases, deposition rate, substrate temperature and deposition processes were found dominant factors to determine magnetostriction and magnetostrictive susceptibility at low magnetic field[4-6)]. Remarkably, the ion plating (IP) is a most effective process for the GM film due to high energy flux and a high deposition rate and high adhesion of films to substrate[7)]. However the $TbFe_2$ film shows low magne- tostriction at low applied fields corresponding to its perpendicular anisotropy.

A unique structure and properties of oblique deposited films have been extensively invest- tigated since the end of the 1950's[11)]. These properties of oblique deposited films have controlled, and applied to industry, for example, magnetic recording tapes[12)] and film for optical retardation plate[13)]. Oblique deposition is expected to improve magnetostrictive susceptibility because in-plane anisotropy of magne- tization is enhanced by magnetic shape anisotropy of columnar grains inclined in plane[14, 15)]. In this study, preparation of the giant magne- tostrictive Tb-Fe films by the IP process was investigated to induce high magnetostrictive susceptibility and high magnetostriction at low applied magnetic fields, especially in respect to the effects of incidence of the vapor flux by geometrical arrangement of the tilted substrate.

*1 Graduate Student, Course of Applied Science,
*2 Professor, Department of Energy Engineering,

2. Experimental

2.1. Ion plating system

Fig.1 shows the schematic diagram of the IP system. This process produces variety of film character. Because dense plasma flux (~2.5A) of a source material can be dosed and deposited on a substrate. The flux of the source material evaporated by an electron beam is activated by thermal electrons accelerated between a surface of the source and a probe. To achieve oblique deposition, several substrates were fixed to substrate holders with several angles from θ=0° to 60° as shown in Fig.2.

2.2. Film preparation

Bulk Tb-Fe alloy samples for evaporation were prepared by arc melting of Tb and Tb-Fe eutectic alloy (the purity of each element was 99.9%) in an Ar gas atmosphere. Table 1 shows a deposition condition of the film formed by IP process. We measured substrate temperature by thermocouple. And substrate temperature was kept at 400K.

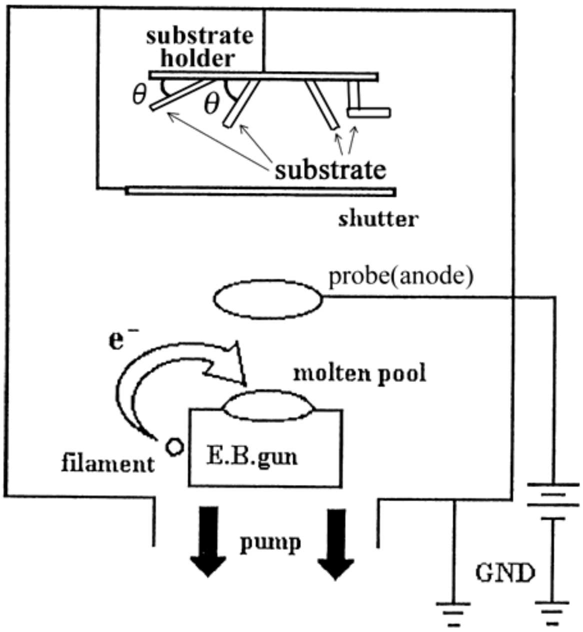


Fig.1. Schematic diagram of IP system

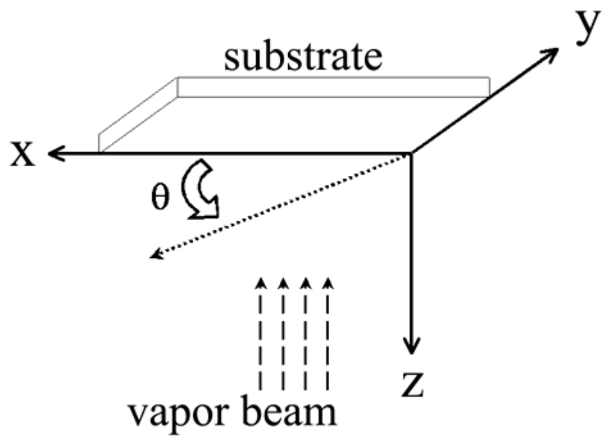


Fig.2. Schematic representation of oblique deposition and directions of substrate angle

Table 1.Deposition Condition of films by IP process

Base pressure	$5.5x10^{-5}$ ～1.0 x 10^{-4} Pa
Deposition rate	4 ~ 10 nm/s
Substrate temperature	400 K
Deposit time	120~180 s
Film thickness	1.2~0.5 m
Anode potential	100V
Discharge current	2.0 ~ 2.5A
Substrate angle	0, 30, 60 degrees
Substrate	Si (100)

2.3. Sample analysis

Fracture cross section of the film samples were observed by scanning electron microscope (SEM). The composition of the film samples was determined using energy dispersive X-ray spectroscopy (EDX). The film structures were analyzed by X-ray diffraction (XRD) using (CuKα) and transmission electron microscope (TEM). The magnetization of formed film samples was measured as a function of magnetic field using vibrating sample magnetometer (VSM) in the range from -1200 KA/m to +1200 KA/m. In magnetization measurements, directions of sample measurement were x, y, and z axes (in Fig.2.) The magnetostriction of the films along with x axes was measured by using a cantilever method described elsewhere[2)]. The equation of the magnetostriction is as follows[16)]:

$$\Delta\lambda_{//} = \frac{D \cdot t_s^2 \cdot E_s(1+\nu_f)}{3l^2 \cdot t_f \cdot Ef(1-\nu_s)}$$

Here E_f=76 GPa modulus Poisson's ratio of Tb-Fe_2 film [pa]; ν_f=0.4 Poisson's ratio of Tb-Fe_2 film; E_s=130 GPa modulus Poisson's ratio of Si (100) substrate [pa]; ν_s=0.28 Poisson's ratio of Si (100) substrate[17)].

3. Resulst and discussion

3.1. Deposition rate

Fig.3 shows dependence of the deposition angles to deposition rate of the films. Increasing substrate angle caused the films thin because increasing substrate angle leads to increase shadow area. However, the larger the angle θ increased, the more ratio of deposition rate disagreed with random cosine distribution.

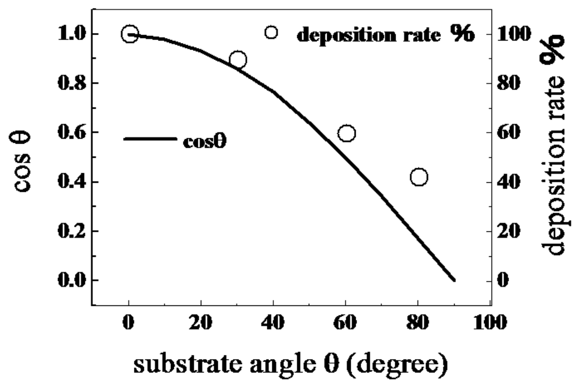
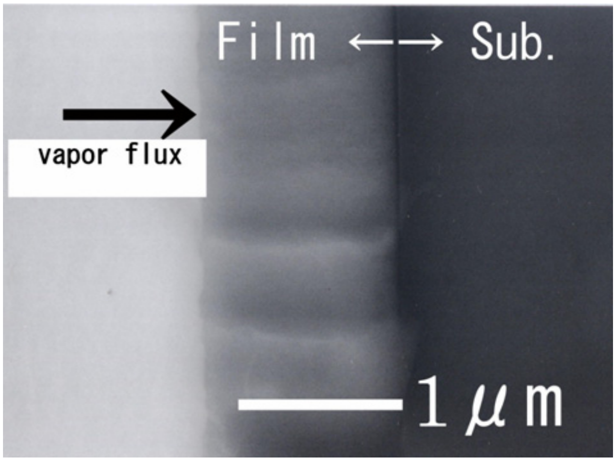


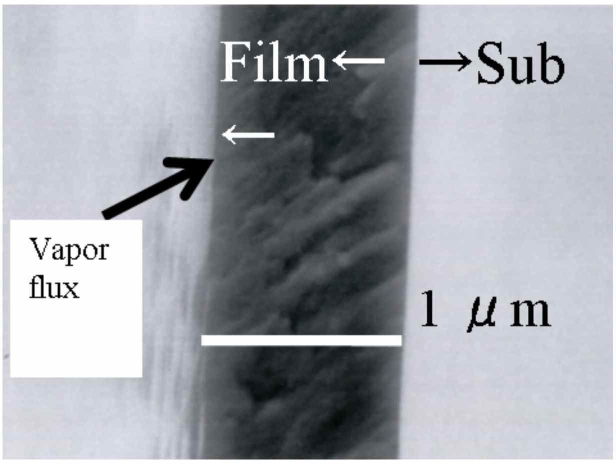
Fig.3. Deposition rate of the films prepared by IP process under different substrate angles

3.2. Morphology of the film

Fig.4 shows the SEM fracture cross section micrographs of the films prepared by a normal deposition at θ=0°(Fig.4(a)) and by an oblique deposition at θ=60°(Fig.4(b)). Columnar grains were found to grow in the direction of the incident vapor flux. When the vapor flux is oblique incident, atoms in the growing films shadow unoccupied sites from the direct sticking of incidence atoms. Moreover, owing to limited mobility, the unoccupied sites are not filled. This supports the result of film thickness as shown in Fig.3. As a result, oblique columns grew in the direction of the incident vapor flux.



(a)



(b)

Fig.4. SEM images of the films (fracture cross section): (a) substrate angle θ=0° and (b) substrate angle θ=60°.

3.3. Crystal structure of the film

XRD result showed no distinct diffraction peak was observed for each sample. In previous work, we found that the crystal structure of thin film samples was found dependent on deposition conditions such as deposition rates and substrate temperatures[7]. In this study, the substrate temperature was from 293 K to 333 K, and the deposition rate was 10nm/s. The crystal structure of film samples by oblique deposition was independent of incidence angles.

Fig.5 shows a TEM image of the $TbFe_2$ film sample prepared by IP process at room tempera- ture. An amorphous phase was found to coexist with a nanocrystalline phases (<5 nm). Therefore the SEM and TEM observations and XRD analyses suggest that the $TbFe_2$ films were composed of columnar grains with amorphous and nanocrystalline phases.

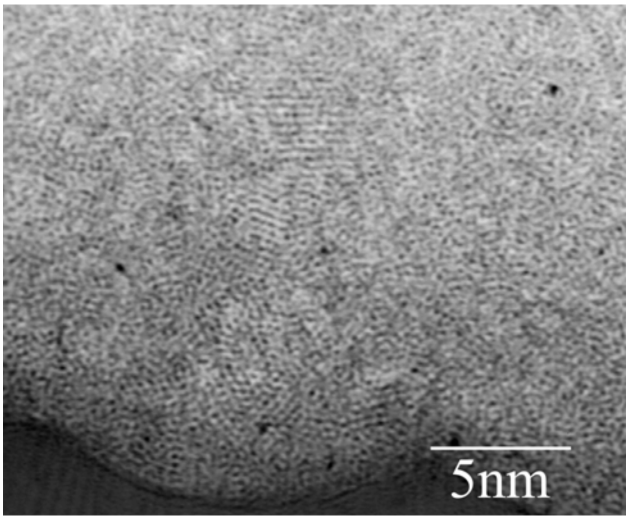


Fig.5 TEM images of the film sample by IP process at room temperature.

3.4. Magnetostrictive properties

Fig.6 shows an effect of the substrate angles to magnetostriction of films samples. The film sample with oblique deposition showed a larger magnetostriction than the sample with conventional deposition (θ=0°).

Magnetostriction of samples increased with increasing substrate angle. The maximum value of magnetostriction for film sample was 2000 ppm at 60° of deposition angle.

Fig.7 shows a dependence on the deposition angle to an area of magnetostrictive hysteresis of samples. We defined magnetostrictive hysteresis of samples by area surrounded from magnetostrictive curves. Magnetostrictive hysteresis of samples increased with increasing substrate angle. This is because contamination in films increased with decreasing deposition rate due to reduced vapor flux with substrate angle.

3.5. Magnetic properties

Fig.8 shows an influence on the deposition angles to coercitive force of samples. In-plane coercitive force of samples was increased and perpendicular coercitive force of samples was decreased with increasing substrate angle. These are because contamination in films increased with increasing of substrate angle.

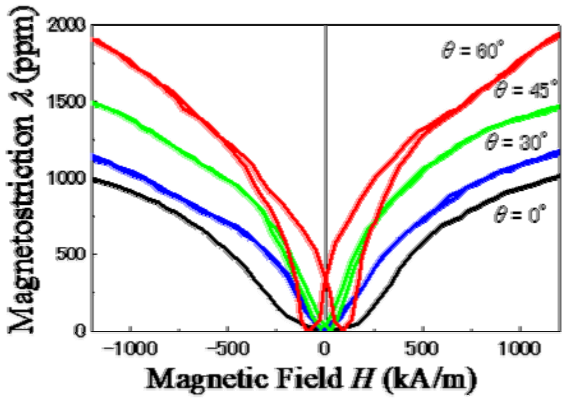


Fig.6. Magnetostriction of film samples prepared by IP process under different substrate angle

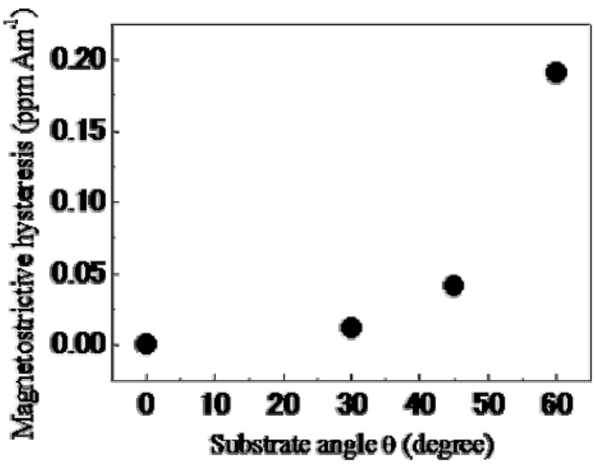


Fig.7. A relative magnetostrictive hysteresis defind as area surrounded by magnetostrictive curve

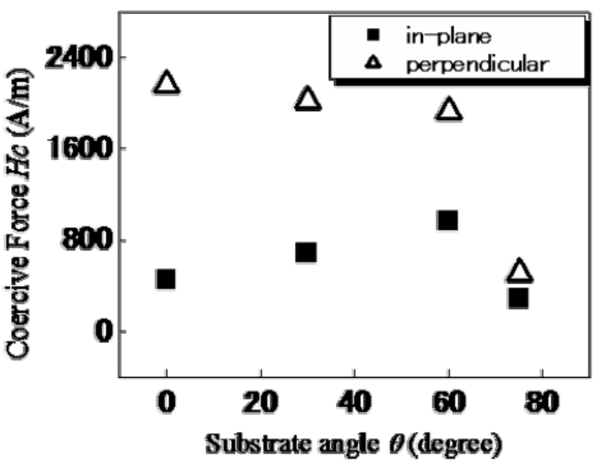


Fig.8 Coercive force of film samples prepared by IP process under different substrate angle

Fig.9 shows a dependence of the deposition angles to saturated magnetization of sample. For X-axis, saturated magnetization in film increased. In contrast, each Y-axis and Z-Axis in film decreased. These are because columnar grins grew in the direction of in-plane in films corresponding to Fig.3 and 4.

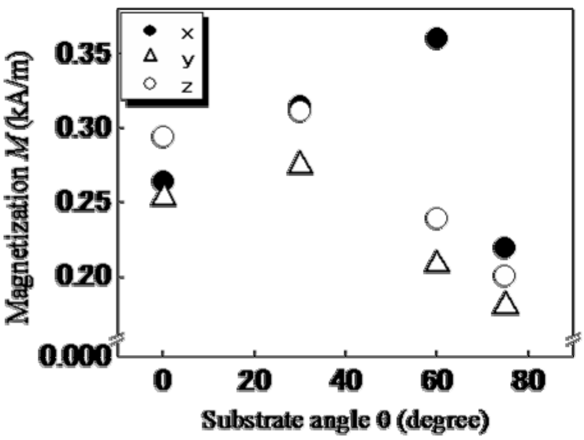


Fig.9 Magnetization M at 1200KA/m of film samples prepared by IP process under different substrate angle

4. Conclusion

Magnetic properties of oblique deposited $TbFe_2$ films prepared by IP process were investigated. The oblique deposition produced a columnar structure where each column was found composed of a mixture of amorphous and nano-crystalline phases. Magnetic properties of the film prepared by IP process were found to depend upon shape magnetic anisotropy, i.e. columnar structure morphology.

The oblique deposition by IP process is effective for preparation of giant magnetostrictive films to induce high magnetostriction.

Acknowledgments

This study was financially supported by the Japan Society for the Promotion of Science (JSPS) for Grant in-Aid for Scientific Research (C) as "Design of Magnetostrictive Susceptibility on Thin Film" with No.19560709. And this study was made in the frame of Development of Advanced Production processes for Energy Conversion Materials, Future Science & Technology Joint Research Center, Tokai University.

5. References

1) *A.E. Clark, E.P. Wohlfahrt*, Ferromagnetic Materials, vol.1, North Holland, Amsterdam, 1980, Chapter 7.
2) *V. Koeninger, Y. Matsumura, T. Noguchi, H. H. Uchida, H. Uchida, H. Funakura, H. Kaneko, T. Kuroki,*

Proceeding of the International symposium on Giant Magnetostrictive Materials and their Applications, 1992, pp. 151-156.
3) *H. Uchida, M. Wada, A. Ichikawa, Y. Matsumura, H.H. Uchida.* Proceeding of the Fifth International Conference On New Actuators, ACTUATOR'96, 1996, pp. 275-278.
4) *M. Wada, H. Uchida, H. Kneko.* J. Alloys Compd. 258(1997) 143-147.
5) *M. Wada, H. Uchida, H. Kaneko*, J. Alloys Compd. 258(1997) 169-173.
6) *M. Wada, H. Uchida, H. Kaneko*, J. Alloys Compd. 258(1997) 174-178.
7) *Y. Matsumura, H. Uchida, M. Ono, H. Kaneko,* Proc. of the 16th Int. Workshop on Rare-Earth Magnets and Their Applications, The Japan Institute of Metals, Sendai , 2000, pp. 985-994.
8) *K. Nakazato, M. Hashimoto, H. Uchida, Y.Matsumura*, Rev. Sci. Instrum. 71 (2000) 996-1001.
9) *M. Wada, H.H. Uchida, Y. Matsumura, H. Uchida, H. kaneko,* Thin Solid Films, 281-282 (1996) 503-506.
10) *H.H. Uchida, V. Koeninger, H. Kaneko,* J. Alloys Compd. 211-212 (1994) 455-459.
11) *R. Messier, A. Lakhtakia,* Mater. Res. Innovations 2 (1999) 217.
12) *E. Kita, M. Kamikubota, A. Tasaki, K. Tagawa.* IEEE Trans. Magn. 17 (6) (1981) 3193-3195.
13) *T. Motohiro, Y. Taga,* Appl. Optics 28 (13) (1989) 2466-2482.
14) T *Otiti* . J Mater Sci, 39, (2) (2004) 477-480.
15) *H.-M. Ho, G. Thomas*. J. Appl. Phys. 65 (8) (1989) 3161-3166.
16) *A.C. Tam, H. Schroeder.* IEEE Trans. Magn. 25 (1989) 2629
17) *J.J. Wortman, R.A. Evans,* J. Appl. Phys. 36 (1965) 153