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Iron Thin Films Deposited by Low Voltage Sputtering

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Iron thin films were deposited using a low voltage sputtering technique and the effects of different sputtering voltages and current on the structure and magnetic properties of the film were investigated. An rf-dc coupled sputtering system with planar magnetron sputtering sources was used to realize sputtering as low as 100 V. A decrease in the sputtering voltage led to a significant improvement in (110) orientation and crystallinity of the films along with a remarkable reduction of compressive film stress. As a result, coercive force of the film decreased as the sputtering voltage decreased. However, an increase in sputtering current caused a significant increase in the crystallite size of the film, which resulted in a remarkable decrease in the coercive force of the film. The (110) orientation, however, degrades when the sputtering current is increased.

Key words: Low voltage sputtering, iron film, effect of sputtering voltage, effect of plasma

1. INTRODUCTION

Control of the high-energy particle bombardment of the film surface is important when using the sputtering technique for the deposition of thin films [1,2]. Low voltage sputtering is one of the most useful techniques for suppressing high energy particle bombardment when making the film[3-5]. However, it is still not clear how reducing the sputtering voltage affects the structure and properties of the films. Moreover, since the amount of activated particles incident to the film surface increases with changes in plasma density, changes in the space between the target and substrate leads to remarkable transformations in the film structure.

Iron and iron-base alloy thin films with soft magnetic properties are well known attractive materials for a magnetic recording head. Control of the microstructure and crystal orientation is necessary in these systems to realize the soft magnetic properties. In our previous paper, we reported that low temperature deposition combined with ion bombardment was effective in producing iron film with a small crystallite size and high orientation [6]. In the current study, a low voltage sputtering technique utilizing rf-dc coupled sputtering [7] was used to deposit the iron thin films. The effects of sputtering voltage and sputtering current on the structure and magnetic properties of the film were investigated in detail.

2. EXPERIMENTAL

Figure 1 shows the low voltage magnetron sputtering system used for film preparation. Sputtering was performed with rf-dc coupled sputtering using a 54.24 MHz rf source at a voltage as low as 100 V. The maximum sputtering current at 100 V and 2 mTorr was about 800 mA. The substrate was mounted on a substrate holder, which was kept at room temperature. The base pressure prior to deposition was below 5×10-7 Torr.

Sputtering was performed in pure Ar, and films with about 100-nm thickness were deposited on the glass slide substrates at room temperature Troom. Typical film preparation conditions are listed in Table I . The sputtering voltage and sputtering current were changed in the range from 100 V to 300 V, and from 100 mA to 600 mA. Sputter-cleaning of the substrate surface was necessary before the film deposition started to obtain a film with enough adhesion. Film thickness was measured with the Decktak 3030 surface profile measuring system. The structure and surface morphology of the prepared films were evaluated using X-ray diffractometry and atomic force microscopy (AFM). Magnetic properties of the films were measured with a vibrating sample magnetometer (VSM).

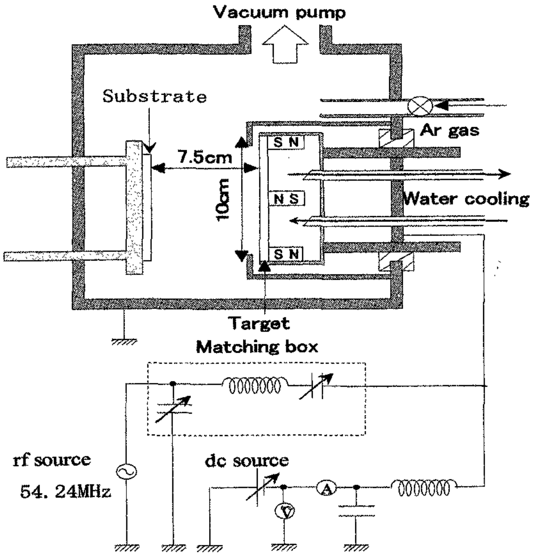


Fig.1. rf-dc coupled sputtering system with planar magnetron sputtering sources, as used in this study.

Table I . Typical film preparation conditions.

Target	10 cm ϕ Fe(99.9%) disk
Sputtering gas	Ar
Gas pressure	2 mTorr
Sputtering voltage	100 V – 300 V
Sputtering current	100 – 600 mA
Substrate	Glass slide
Film thickness	≒100 nm
Substrate temperature	Room temperature

3. RESULTS AND DISCUSSION

3.1 Dependence on sputtering voltage

Figure 2 shows typical X-ray diffraction diagrams of the film, deposited at various sputtering voltages. Sputtering current was fixed at 150 mA and substrate temperature was set to room temperature. The figure shows clearly that the film deposited at 100 V had the largest (110) diffraction peak, and (110) orientation improved significantly by the sputtering at 100 V. The full width at half maximum of the locking curve of the (110) diffraction peak of the film was 5.3 °

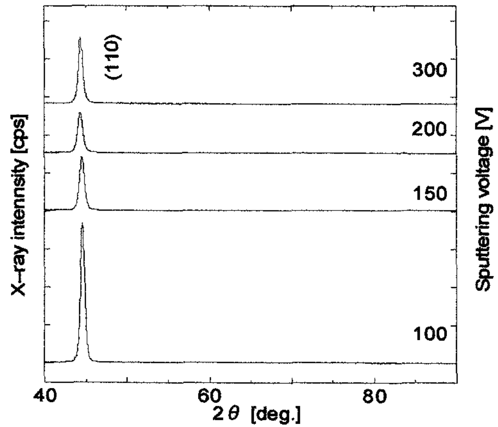


Fig. 2. X-ray diffraction diagrams of the films, deposited at various sputtering voltages.

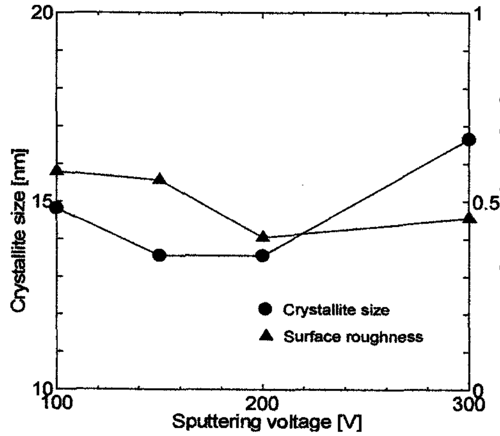


Fig. 3. Changes in crystallite size and surface roughness of the films deposited at various sputtering voltages.

Figure 3 shows changes in the mean crystallite size and surface roughness Ra of the films with sputtering voltage. The crystallite size was evaluated from the full width at half maximum of (110) diffraction peak. The decrease in sputtering voltage from 300 V to 200 V led to a decrease in crystallite size and in surface roughness. On the contrary, further decrease in the sputtering voltage to 100 V caused an increase in crystallite size and surface roughness. These results suggest that the crystallinity and (110) orientation of the film can be improved by the decrease in sputtering voltage as low as 100 V. Figure 4 shows spacing of (110) and film stress of these films deposited at various sputtering voltages. It is evident from the figure that the compressive film stress decreases remarkably as the sputtering voltage decreases. This decrease in film stress is thought to be attributable to the decrease in lattice spacing of the (110) plane in the Fig.4. Taking the fact that the compressive stress in the sputtered film is mainly caused by the high energy particle bombardment of the film during deposition [8,9] into consideration, these decreases in compressive film stress and lattice spacing at a low sputtering voltage suggest that low voltage sputtering is useful for the suppression of the high energy particle bombardment during film deposition.

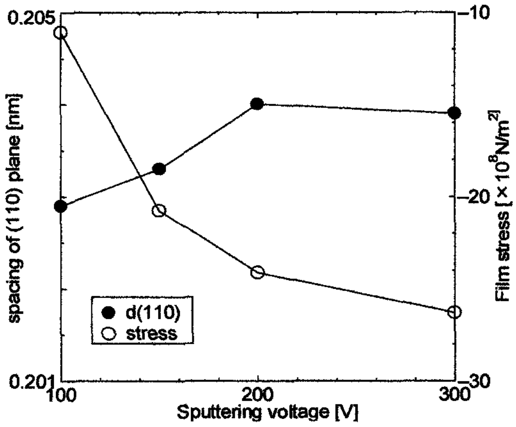
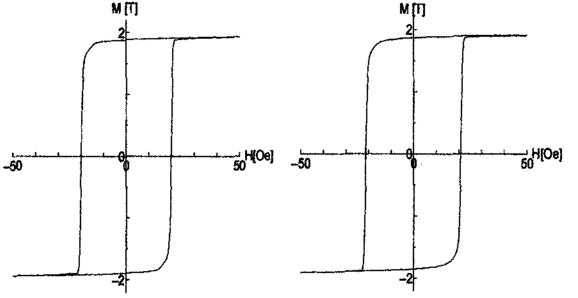


Fig. 4. Changes in spacing of the (110) plane and film stress with sputtering voltage.

Typical M-H curves of the films, deposited at various sputtering voltages, are shown in Fig.5. Figure 6 shows the changes in the coercive force of these films with different sputtering voltages. The coercive force increases as the sputtering voltage increases. This suggests that the films deposited at a higher voltage have a low uniformity.



(a) (b)

Fig. 5. Typical M-H curves of the film deposited at (a) 150V and (b) 200V.

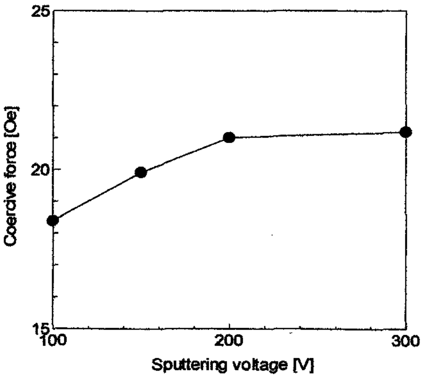


Fig. 6. Changes in coercive force versus sputtering voltage.

Typical M-H curves of the films, deposited at various sputtering voltages, are shown in Fig.5. Figure 6 shows the changes in the coercive force of these films with different sputtering voltages. The coercive force increases as the sputtering voltage increases. This suggests that the films deposited at a higher voltage have a low uniformity.

3.2 Dependence on sputtering current

To clarify the effect of sputtering current on film structure, sputtering voltage was fixed and films were deposited at various sputtering current. Figure 7 shows the X-ray diffraction diagrams of the films deposited at various sputtering currents. Sputtering voltage was fixed at 100 V. It is clear from the figure that the (110) peak intensity decreases monotonically as the sputtering current increases, which suggests that the films deposited at a higher sputtering current have a poor orientation.

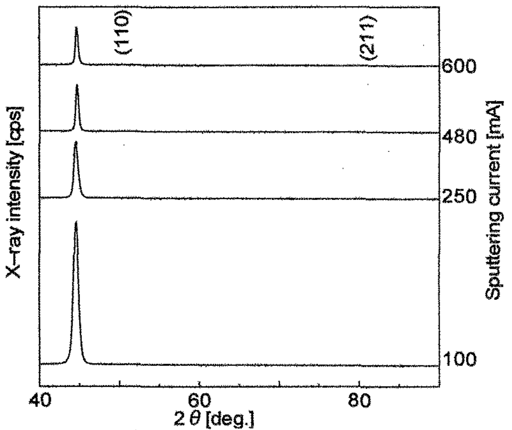


Fig. 7. X-ray diffraction diagrams of the films deposited at various sputtering currents. Sputtering voltage was fixed at 100 V.

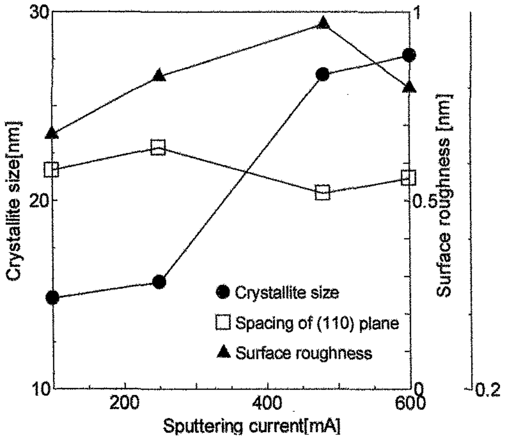
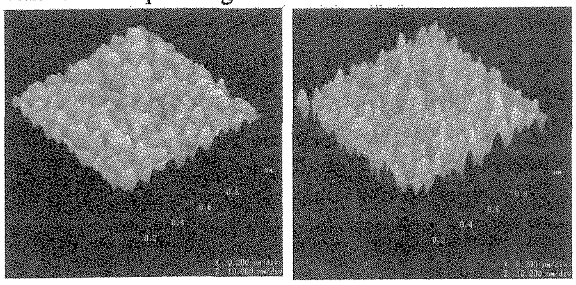
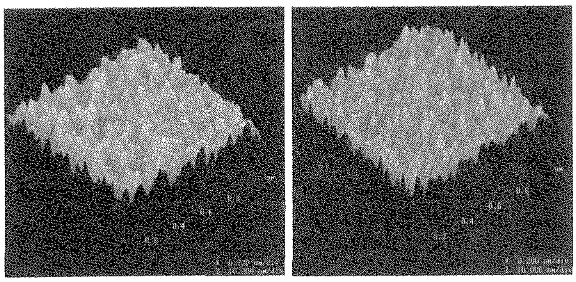


Fig. 8. Changes in mean crystallite size, spacing of (110) plane and surface roughness of the films with sputtering current.

Figure 8 shows the changes in the mean crystallite size, the lattice spacing of the (110) plane, and the surface roughness with sputtering current. It is evident from the figure that the crystallite size increases steeply as sputtering current increases, which results in an increase in surface roughness as shown in Fig.9. This suggests that the increase in plasma density in the space between the target and substrate was effective in promoting crystal growth in the films. The substrate temperature was increased gradually during sputtering, and the substrate temperature at the end of the deposition was about 45 ℃ and changed little with the sputtering current.

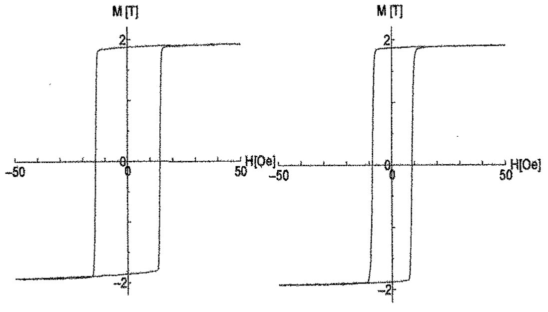


(a)100mA Ra=0.579nm (b)250mA Ra=0.829nm



(c)480mA Ra=0.967nm (d)600mA Ra=0.799nm

Fig. 9. Surface morphology of the films deposited at sputtering current.



(a) (b)

Fig. 10. Typical M-H curves of the film deposited at sputtering current (a) at 250 mA and (b) at 600 mA.

Typical magnetization curves are shown in Fig.10. The coercive force decreases as the sputtering current increases as shown in Fig.11 and takes a value as low as 7 Oe. This decrease in coercive force may be caused by the improvement in the uniformity of the films.

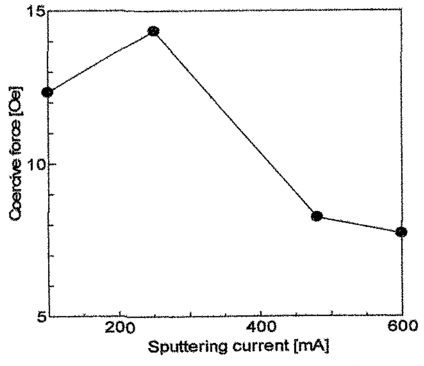


Fig. 11. Changes in coercive force of the film versus sputtering current.

To clarify the differences between the sputtering current and sputtering voltage in the effect on film structure, crystallite size of the films deposited at a fixed sputtering voltage of 100V and a fixed sputtering current 150 mA was plotted against deposition rate in Fig.12.

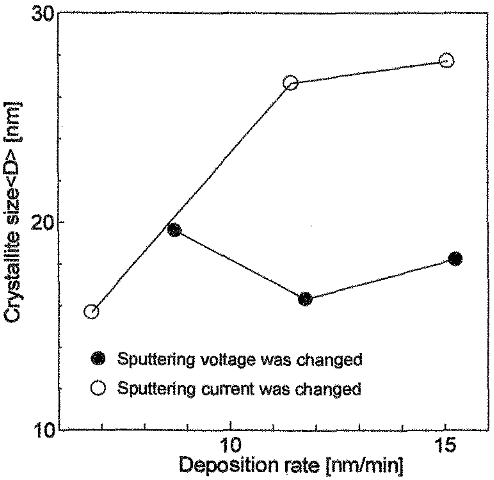


Fig. 12. Crystallite size versus deposition rate of the films deposited at a fixed sputtering current of 150 mA, and fixed sputtering voltage of 100 V.

It should be noted that the deposition rate is increased by the increases in both sputtering voltage and sputtering current, and the deposition rate about 15nm/min was obtained at 600 mA for the 100 V, and at 300 V for the 150 mA, respectively. The figure shows that the crystallite size of the film deposited at 100 V increases as the deposition rate increases, although it changes little when sputtering current was fixed. As a result, the film deposited at 100 V and 600 mA has a crystallite size twice as large as the film deposited at 300 V and 150 mA. This leads to a remarkable difference in the coercive force, as shown in Fig.13. The film deposited at a low sputtering voltage and at a large sputtering current has a small coercive force.

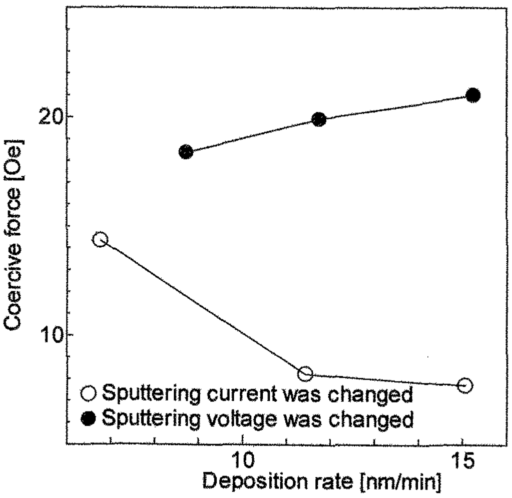


Fig. 13. Coercive forces versus deposition rate of the films deposited at fixed sputtering current of 150 mA and fixed sputtering voltage of 100 V.

These results indicate that films deposited at a low voltage and high plasma density has good crystallinity and a low coercive force. However, (110) orientation is degraded by the increase in plasma density. The increase in plasma density will lead to an increase in the amount of ions, electrons and radicals incident to the film surface, which may promote the crystal growth in the film, although further investigation should be necessary.

4. CONCLUSIONS

Iron thin films were deposited using a low voltage sputtering system and the following results were obtained.

(1) Sputtering as low as 100 V was achieved using an rf-dc coupled sputtering system with a planar magnetron sputtering source.

(2) A decrease in sputtering voltage led to a significant improvement in (110) orientation along with a remarkable reduction in compressive film stress.

(3) Coercive force of the film decreased as the sputtering voltage decreased.

(4) An increase in plasma density in the space between the target and substrate led to a significant increase in crystallite size of the film, which resulted in a remarkable decrease in the coercive force of the film. The (110) orientation, however, was degraded by the increase in plasma density.

These results indicate that a voltage sputtering as low as 100 V and high plasma density seem to be useful for the deposition of iron films with a low coercive force, although further improvements in soft magnetic properties is necessary.

REFERENCES

[1] K. Tominaga, T. Yasuda, M. Kume, O. Tada, Jpn. J. Appl. Phys., 24 (1992) 1718
[2] K. Ishibashi, K. Hirata, N. Hosokawa, J. Vac. Sci. Thechnol., A10(4) (1990) 1399
[3] Y. Hoshi, R. Ohki, Proceedings of NAGANO magel '99, (1999) 411
[4] Y. Hoshi, R. Ohki, Electrochimica Acta, 44 (1999) 3927-3932
[5] S. Ishibashi, Y. Higuchi, Y. Ohta, K. Nakamura, J. Vac. Sci. Technol., A8, (3) (1990) 1403
[6] W. Seiko, Y. Hoshi, and H. Shimizu, J. Magn. Magn. Mater., 235 (2001) 196-200
[7] T. Ohmi, T. Ichikawa, T. Shibata, K. Matsudo, H. Iwabuchi, Appl. Phys. Lett., 53(1988)45
[8] J.A. Thornton and D.W. Hoffman, J. Vac. Sci. Technol., 14(1977)164
[9] J.A. Thornton and D.W. Hoffman, J. Vac. Sci. Technol., 18(1981)203

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