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Reactive ECR-Sputter-Deposition of Ni-Zn Ferrite Thin-Films for Backlayer of PMR Media

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SUMMARY A reactive sputtering method using an Electron-Cyclotron-Resonance (ECR) microwave plasma was used to deposit Ni-Zn ferrite thin-films for a soft magnetic backlayer of Co-containing spinel ferrite thin-film perpendicular magnetic recording (PMR) media. The Ni-Zn spinel ferrite thin-films with a preferential orientation of (100) and a relatively low coercivity of 15 Oe were obtained at a high deposition rate of 14 nm/min and at a temperature below 200 degrees C. Although post–annealing treatment in air at 200 degrees C was effective to decrease the coercivity of the Ni-Zn ferrite thin-films, the saturation magnetization and initial permeability decreased and the surface smoothness was deteriorated simultaneously. The Ni-Zn ferrite thin-films prepared by ECR sputtering are promising as the backlayer of the perpendicular magnetic recording medium, but further improvement is required in terms of the soft magnetic properties, the grain size and the surface roughness.

key words: *Ni-Zn ferrite thin-film, reactive ECR sputtering, perpendicular magnetic recording, recording media, backlayer*

1. Introduction

It has been already reported that a novel sputtering method using an Electron-Cyclotron-Resonance (ECR) microwave plasma, in short, ECR sputtering, is effective to deposit Co-Cr thin-films whose micro magnetic structure in grains are well controlled [1], [2]. The ECR sputtering method has advantages over conventional diode or magnetron sputtering method [3], [4]: Firstly, independent and precise process control is possible with respect to plasma generation, sputtering and film deposition. Secondly, the ECR sputtering is suitable for low temperature deposition of oxide or nitride thin-films which need chemical reaction during the film growth because the ECR microwave plasma is dense and contains many energetically excited ions. We already proved that the Co-containing spinel ferrite thin-films with a high coercivity of 3000 Oe for perpendicular magnetic recording (PMR) media application can be deposited at a temperature lower than 200 degrees C using the reactive ECR sputtering method [5]. In that work, the media has a single layer structure. In order to use a single-pole type recording head to achieve higher recording densities, it is inevitable to develop double layered media with a soft magnetic backlayer. In the combination of the recording layer of a Co-containing spinel ferrite thin-film, spinel ferrite thin-films such as Ni-Zn ferrite or Mn-Zn ferrite thin-films are suitable for the backlayer because the hetero-epitaxial growth of the Co-containing spinel ferrite thin-film is expected on the soft magnetic spinel thin-film. To apply soft magnetic ferrite thin-films to the backlayer of PMR media, low temperature deposition is preferable to obtain smooth surface media composed of fine grains.

In this study, low-temperature deposition of soft magnetic Ni-Zn spinel ferrite thin-films using the reactive ECR sputtering and the post-annealing effect of the films were investigated.

2. Experimental

Figure 1 shows the configuration of the ECR sputtering apparatus (SHIMADZ corporation, SLC-75ES) used in this experiment. Plasma was generated by the combination of a 2.45 GHz microwave and a 875 Oe magnetic field which satisfied ECR condition. Dense and active plasma is generated by ECR phenomenon. The process gas was introduced in the following two ways: firstly, argon and oxygen mixture gas was introduced in the plasma generation chamber, secondly, argon gas and oxygen gas were separately introduced in the plasma generation chamber and near the substrate in the film deposition chamber, respectively. Ni-Zn ferrite thin-films were deposited without substrate heating. However, the substrate temperature rose up to 200 degree C

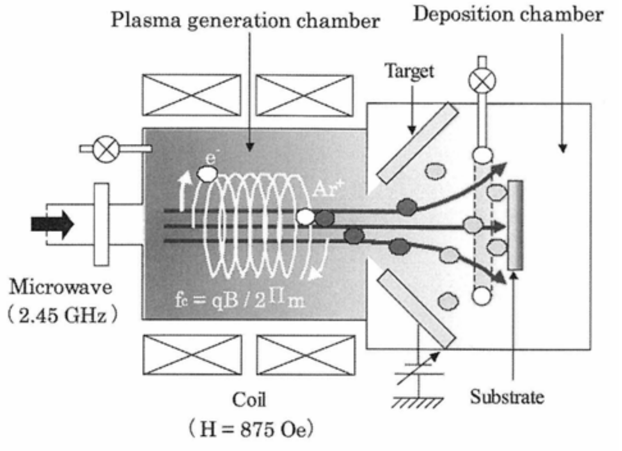

Fig. 1 ECR Sputtering apparatus.

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during film deposition by the plasma irradiation to the substrate. Three $Ni_{0.3}$-$Zn_{0.5}$-$Fe_{1.4}$ (wt%) alloy platelet targets were used.

To achieve a high rate deposition without a severe temperature rise, the following setup was realized in the ECR sputtering apparatus. To obtain a high density and active plasma, a microwave power supply with a high output power of 900 W was used for the plasma volume of 5100 cm^3. Targets were placed in the vicinity of the plasma extraction window to utilize the high density plasma, and were placed to make so-called "On-axis configuration" to the substrate. To achieve a sputtering operation with the target surface in "metal mode", oxygen gas was introduced at a position not to oxidize the target surface but to oxidize the deposited film effectively. It was the best way to introduce the oxygen gas near the substrate, because it was expected that the targets are not oxidized easily and the oxygen gas excited by ECR plasma stream oxidizes the deposited film effectively.

3. Results and Discussion

3.1 As-Deposited Ni-Zn Ferrite Films

At first, 200 nm thick Ni-Zn ferrite thin-films were deposited at a microwave input power of 500 W, target-voltage of −300 V, with varying the oxygen partial pressure ratio from 0 to 10%. The oxygen partial pressure ratio was defined as the percentage of oxygen pressure to total gas (argon and oxygen) pressure.

As shown in Fig. 2, the saturation magnetization of the deposited film gradually decreased with increasing the oxygen partial pressure ratio and dropped suddenly at an oxygen partial pressure ratio of 4%. However, high deposition rate over 10 nm/min was still maintained up to the oxygen partial pressure ratio of 7%. XRD diagrams of the deposited films shown in Fig. 3 indicated that the Ni-Zn spinel ferrite thin-films with a preferential orientation of (100) were obtained at oxygen partial pressure ratios ranging from 5 to 7%, where spinel ferrite films were obtained although the targets were operated in "metal mode", and consequently high deposition rates were achieved.

The microwave input power is a primal factor determining the deposition rate because plasma density is proportional to the microwave input power. Therefore the optimum value of oxygen partial pressure shifted upward at higher microwave input power because more oxygen atoms are needed to sufficiently oxidize the increased depositing atoms.

The following three deposition conditions were selected, and deposition of 400 nm thick Ni-Zn ferrite thin-films was carried out to investigate the influence of target-voltage: The experimental "Condition I" was with a microwave input power of 500 W and an oxygen partial pressure ratio of 5%. The Condition II was with an increased microwave input power of 600 W and an oxygen partial pressure ratio of 8%. In these two conditions, oxygen gas was introduced in the plasma generation chamber with argon gas. In the Condition III, the microwave input power and the oxygen partial pressure ratio were the same as Condition I, but the oxygen gas was introduced in the vicinity of the substrate in the film deposition chamber to minimize oxidizing the sputtering target. The target voltage was varied from −200 V to −700 V in these three conditions.

As shown in Fig. 4, in Condition I, the minimum coercivity of 65 Oe was obtained at a target voltage of −300 V. In Condition II, the minimum coercivity of 67 Oe was obtained at a target voltage of −400 V. In Condition III, the lowest coercivity of 15 Oe was obtained at a target voltage of −350 V.

In Fig. 5, the coercivity of the Ni-Zn ferrite thin-

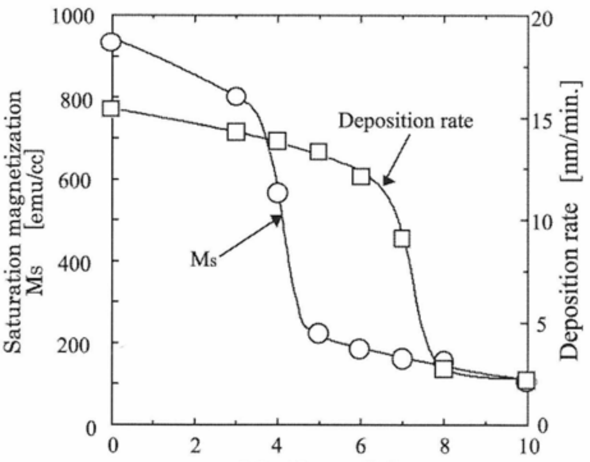

Fig. 2 Oxygen partial pressure ratio dependence of the saturation magnetization and deposition rate.

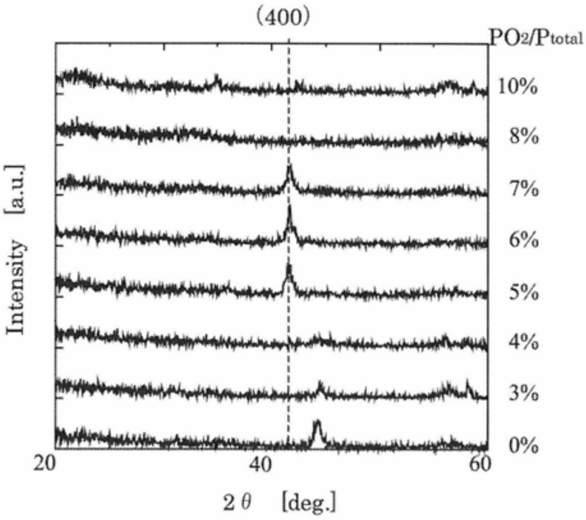

Fig. 3 X-ray diffraction diagram of Ni-Zn ferrite films.

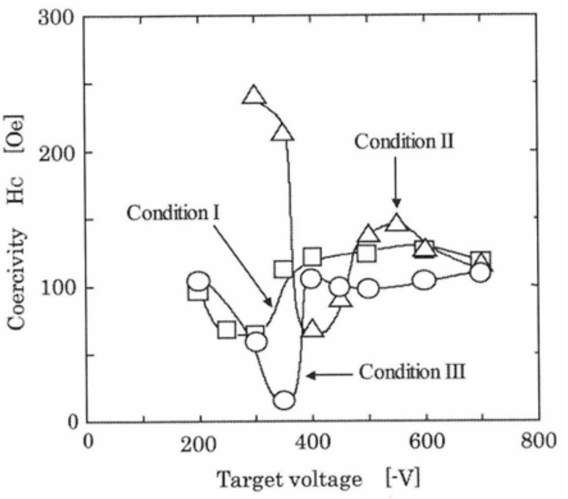

Fig. 4 Target voltage dependence of the coercivity.

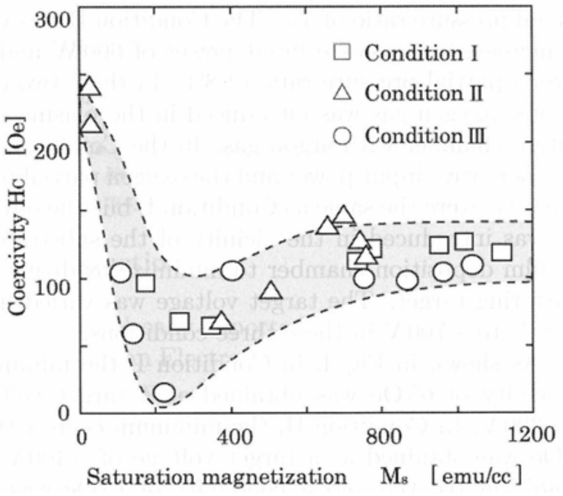

Fig. 5 Saturation magnetization dependence of the coercivity.

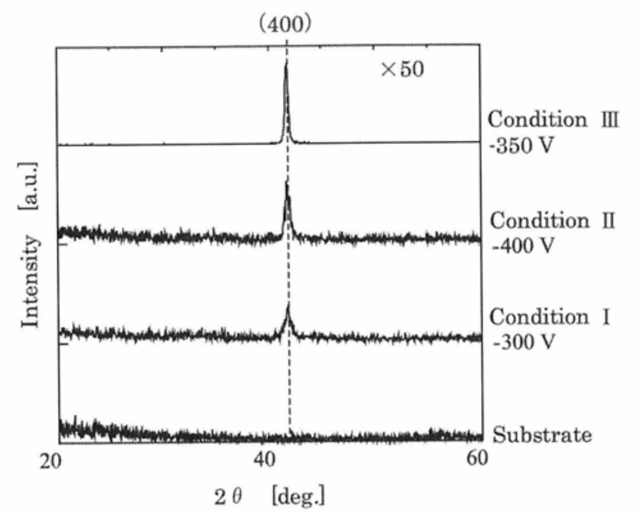

Fig. 6 X-ray diffraction diagram of Ni-Zn ferrite films with the lowest coercivity for each condition.

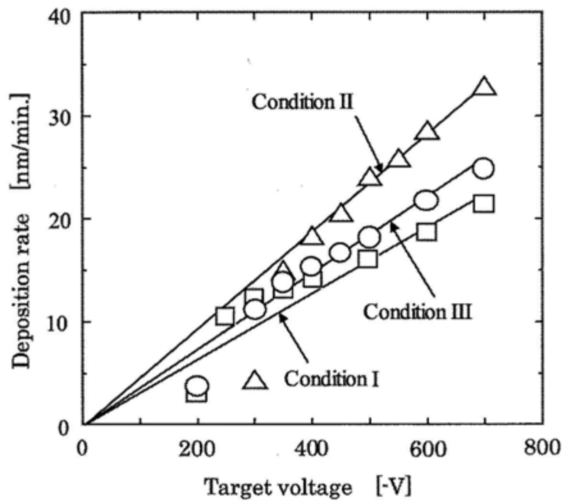

Fig. 7 Target voltage dependence of deposition rate.

films deposited in Condition I, II and III was plotted against the saturation magnetization. The expected saturation magnetization for the present film deposited using the $Ni_{0.3}$-$Zn_{0.5}$-$Fe_{1.4}$ (wt%) target and completely oxidized was 280 emu/cm^3. It was found that the coercivity has a relationships with saturation magnetization, and low coercivities were only achieved at narrow saturation magnetization range of about 150–350 emu/cm^3. This experimental result shows that the precise control of oxidation is necessary to achieve low coercivity Ni-Zn ferrite thin-films.

Figure 6 shows the XRD diagrams for the Ni-Zn ferrite thin-films deposited at the target voltage wich produced lowest coercivity for each condition. The highest diffraction peak from (400) plane was observed for the Ni-Zn film deposited in Condition III. The result proves that superior crystallinity was achieved in this film.

The target voltage dependence of the deposition rate in the ECR sputtering is shown in Fig. 7. In a conventional sputtering system, target voltage concerns to both plasma generation and collection of argon ions to the target. On the contrary, in the ECR sputtering system, the target voltage plays a role to collect and accelerate argon ions to the target from the plasma flow. Therefore, the deposition rate was proportional to the target voltage as shown in Fig. 7. The maximum deposition rate could reach 33 nm/min at a target voltage of −700 V in Condition II. This value is about 20 times larger than that obtained using our prototype ECR sputtering apparatus [6].

At the best condition where minimum coercivity was obtained, the deposition rate was 12.3 nm/min, 18.1 nm/min and 13.8 nm/min in Condition I, II and III, respectively.

3.2 Post-Annealed Ni-Zn Ferrite Films

In order to change the magnetic properties of the Ni-Zn ferrite films, post-annealing effect was tried. The Ni-Zn ferrite thin-films deposited under the above described optimal condition where minimum coercivity was obtainable in Condition III were heated up, and kept in air at temperatures ranging from 150 to 350 degrees C for 60 minutes.

Annealing temperature dependence of magnetic properties of the Ni-Zn ferrite thin-films is shown in Fig. 8. At annealing temperatures from 150 to 200 degrees C, the coercivity of the Ni-Zn ferrite thin-films became lower than that of as-deposited film and reached to the lowest value of 8 Oe. The decrease in coercivity is probably due to the increase in grain size as shown in Table 1. However, the coercivity gradually increased with increasing the annealing temperature higher than 250 degrees C. The saturation magnetization gradually decreased with elevating post-annealing temperature.

Figure 9 shows the X-ray diffraction diagrams of the as-deposited and post-annealed Ni-Zn ferrite thin-films. The peak intensity from (400) plane of the Ni-Zn ferrite thin-films annealed below 200 degrees C is as the same as that of the as-deposited sample. This result suggests that the good crystallinity was maintained even in the samples post-annealed under 200 degrees Celsius. However, in the Ni-Zn ferrite thin-films annealed at temperatures above 250 degrees Celsius, the peak intensity from (400) plane gradually decreased showing the deterioration of the crystallinty or crystal orientation.

In Table 1, magnetic properties, grain size, average surface roughness (R_a) measured from AFM surface images, and deposition rate of the Ni-Zn films deposited under the above described minimum coercivity condition are summarized. Sample I, II and III are as-deposited Ni-Zn ferrite thin-films. Sample IV is a post-annealed film at 200 degrees C after deposition under the Condition III. The as-deposited ferrite thin films prepared in Condition I, II and III had a relatively small grain size and a smooth surface which are necessary for the media application. However, the Ni-Zn ferrite thin-film post-annealed at 200 degrees C (Sample IV) had a larger grain size of 80.3 nm and a larger R_a of 2.03 nm as compared with the other three samples. Post-annealing at 200 degrees C caused considerable increase both in the grain size and the surface roughness. Although the Sample IV had the lowest coercivity of 8 Oe, initial permeability becomes lower than Sample III (as-deposited sample). This is supposed to be due to

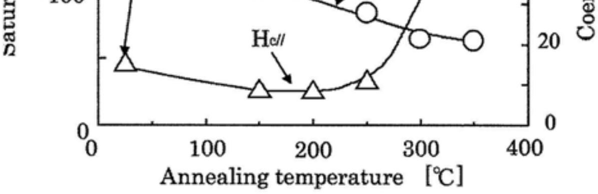

Fig. 8 Annealing temperature dependence of M_s and $H_{c/\!/}$.

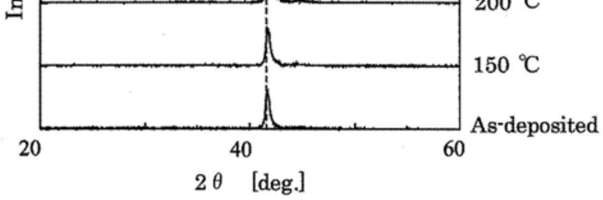

Fig. 9 X-ray diffraction diagram of Ni-Zn ferrite films.

Table 1 Magnetic properties, surface roughness and deposition rate of Ni-Zn ferrite thin-films.

	Deposition condition	Target voltage	Post-anneal	Oxygen gas inlet	H_c [Oe]	M_s [emu/cm^3]	μ_i	Grain size [nm]	R_a [nm]	Deposition -rate [nm/min]
Sample I	Condition I	-300 V	none	Plasma chamber	65	348	1.3	33.6	0.89	12.3
Sample II	Condition II	-400 V	none	Plasma chamber	67	375	1.2	37.1	1.14	18.1
Sample III	Condition III	-350 V	none	Deposition chamber	15	224	46.7	42.9	1.36	13.8
Sample IV	Condition III	-350 V	at 200℃	Deposition chamber	8	105	5.4	80.3	2.03	13.8

decreased magnetization saturation by post-annealing.

As the backlayer of PMR media, high permeability, large saturation magnetization, smooth surface and small grain size are required. Further improvement is necessary on the Ni-Zn films to be used as the backlayer.

4. Conclusions

A reactive sputtering method using an Electron-Cyclotron-Resonance microwave plasma was used to deposit Ni-Zn ferrite thin-films for a soft magnetic backlayer of Co-containing spinel ferrite thin-film perpendicular magnetic recording media. Ni-Zn spinel ferrite thin-films with a preferential orientation of (100) and a relatively low coercivity of 15 Oe were obtained at a high deposition rate of 14 nm/min and at a temperature below 200 degrees C. Although post–annealing treatment in air at 200 degrees C was effective to decrease the coercivity of the Ni-Zn ferrite thin-films, the saturation magnetization and initial permeability decreased and the surface smoothness was deteriorated simultaneously.

The Ni-Zn ferrite thin-films prepared by ECR sputtering are promising as the backlayer of the perpendicular magnetic recording medium, but further improvement is required in terms of the soft magnetic properties, the grain size and the surface roughness.

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