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Low Temperature Sputter-Deposition of Ni-Zn Ferrite Thin-Films Using Electron-Cyclotron-Resonance Microwave Plasma

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A novel sputtering method using an Electron-Cyclotron-Resonance (ECR) microwave plasma was used to perform reactive sputter-deposition of Ni-Zn ferrite thin-films. Ni-Zn spinel ferrite thin-films with preferential orientation of (400), relatively low coercivity less than 3 Oe and saturation magnetization of about 220 emu/cc were successfully obtained at a high deposition rate of 12-24 nm/min and at a temperature lower than 200 degrees C. To achieve high deposition rate, configuration of ECR sputtering apparatus and processing parameters such as microwave input power, target voltage, oxygen partial pressure were carefully optimized. The reactive ECR sputtering is one of the most suitable preparation methods of ferrite thin-films applicable to magnetic devices such as MMIC's, isolators and circulators.

Key words: Ni-Zn ferrite thin films, reactive ECR sputtering, high-rate deposition, low temperature

1. INTRODUCTION

We have already reported on metallic thin-film deposition by a novel sputtering method using an Electron-Cyclotron-Resonance (ECR) microwave plasma, in short, ECR sputtering [1,2]. The ECR sputtering method has two advantages over conventional diode or magnetron sputtering method [3, 4]: firstly, independent process control 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 film growth because the ECR microwave plasma is dense and contains many energetically excited ions. We have already proved that the Co-containing spinel ferrite thin-films with a high coercivity of 3000 Oe for magnetic recording media application can be deposited at a temperature lower than 200 degrees C using the reactive ECR sputtering method [5].

To apply soft magnetic ferrite thin-films to advanced magnetic devices such as a backlayer of perpendicular magnetic recording media, MMIC (Monolithic Microwave Integrated Circuit), thin-film isolator and circulator, the highest acceptable deposition temperature is at most 300 degrees C, and a high deposition rate is necessary to meet the demand of relatively large film thickness over micrometer.

In this study, high rate and low temperature deposition of soft magnetic Ni-Zn spinel ferrite thin-films was investigated using the reactive ECR sputtering method .

2. EXPERIMENTAL

Fig. 1 shows the configuration of the ECR sputtering apparatus used in this experiment. Plasma was generated by the combination of 2.45 GHz microwave and 875 Gauss magnetic field which satisfied ECR condition. A 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 in the near space of 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 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 placed in the vicinity of plasma extraction window, and the angles between the sputtering targets and substrate was tuned to form so-called " on-axis configuration " to achieve a high deposition rate.

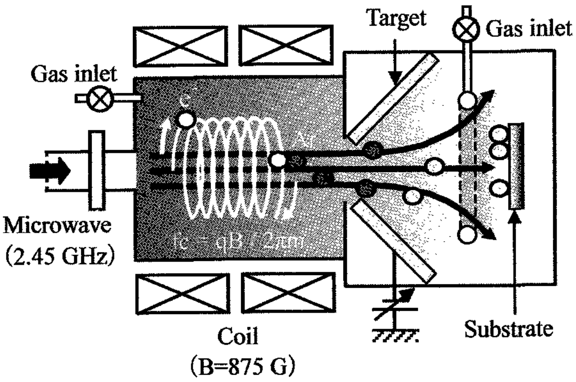

Fig. 1 Sputtering apparatus.

To achieve a high rate and low temperature deposition, the followings were realized in the ECR sputtering appratus. To utilize a high density and active plasma, plasma should be generated by ECR phenomenon, and microwave power supply with high output power of 500 and 600 W was prepared. Target was placed in the vicinity of plasma extraction window to utilize high density plasma, and was placed to make so-called " on-axis configuration " to the substrate. To achieve a operation of the target surface in " metal mode ". oxygen gas should be introduced in proper position not to oxidize the target but to oxidize the deposited film effectively. We expected that it is the best way to introduce the oxygen gas near the substrate, because the target might not be oxidized easily and the oxygen gas excited by ECR plasma stream could oxidize the deposited film effectively.

3. RESULTS AND DISCUSSIONS

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 oxygen partial pressure from 0 to 10 %. The oxygen partial pressure was defined as the percentage of oxygen pressure to total gas (argon and oxygen).

As shown in Fig. 2, the saturation magnetization of the deposited film gradually decreased with increasing oxygen partial pressure and dropped suddenly at a oxygen partial pressure of 4 %. However, high deposition rate over 10 nm/min was still maintained up to oxygen partial pressure of 7 %. At a lower oxygen gas pressure lower than 7 %, target surface works in " metal mode ". Introduction of oxygen gas in the vicinity of the substrate in the film deposition chamber is effective to expand the oxygen gas pressure margin in " metal mode ".

From several experiments as described above, it was found that the microwave input power is a primal factor determining the deposition rate because plasma density is proportional to the microwave input power, and that, at higher microwave input power, optimum value of oxygen partial pressure shifts upward, because the more oxygen atoms are needed to oxidize sufficiently when the number of atom is increased.

The following three deposition condition was selected, and deposition of 400 nm thick Ni-Zn ferrite thin-films was carried out to investigate the influence of target-voltage. One is a microwave input power of 500 W and oxygen partial pressure of 5 %. This experimental condition is expressed as " Condition I ". The other (Condition II) is a microwave input power of 600 W and oxygen partial pressure of 8 %. In these two conditions, oxygen gas was introduced in the plasma generation chamber. In Condition II, microwave input power and oxygen partial pressure were increased as compared with that in Condition I. The next one (Condition III) is a microwave input power of 500 W, oxygen partial pressure of 5 %. The other (Condition IV) is a microwave input power of 600 W and oxygen partial pressure of 8 %. In these two conditions, oxygen gas was introduced in the vicinity of the substrate in the film deposition chamber not to easily oxidized the sputtering target. In Condition IV, microwave input power and oxygen partial pressure were increased as compared with that in Condition III. Target voltage was varied from 200 V to 700 V.

In Fig. 3, target voltage dependence of coercivity of Ni-Zn ferrite thin-films is shown. 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 minimum coercivity of 15 Oe was obtained at a target voltage of –350 V. In condition IV, the minimum coercivity of 3 Oe was obtained at a target voltage of –500 V. Introduction of oxygen gas in the vicinity of the substrate in the film deposition chamber achieved superior soft magnetic properties.

In Fig. 4, coercivity of the Ni-Zn ferrite thin-film deposited varing target voltage in condition I, II, III and IV was plotted against their saturation magnetization of the thin-films. 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 strongly depend on

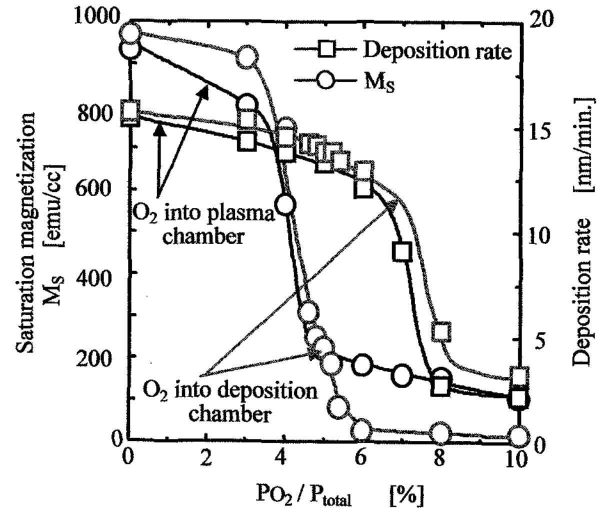

Fig. 2 Oxygen partial pressure dependence of saturation magnetization and deposition rate.

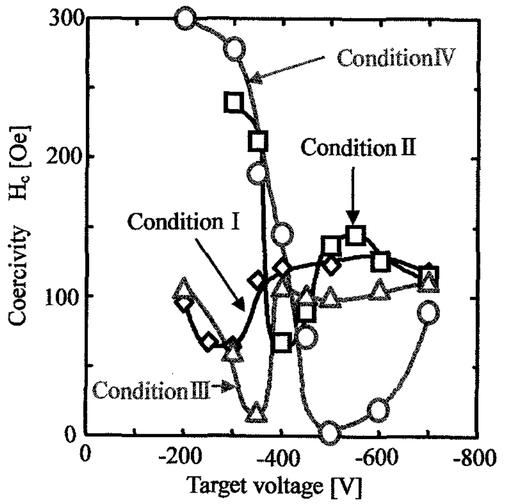

Fig. 3 Target-voltage dependence of coercivity.

saturation magnetization, and that low coercivity was achieved at the saturation magnetization of about 150-350 emu/cc. Generally, saturation magnetization of ferrite decreases as the oxidation of the film is enhanced. This experimental result shows that the precise control of oxidation is necessary to achieve low coercivity Ni-Zn ferrite thin-films.

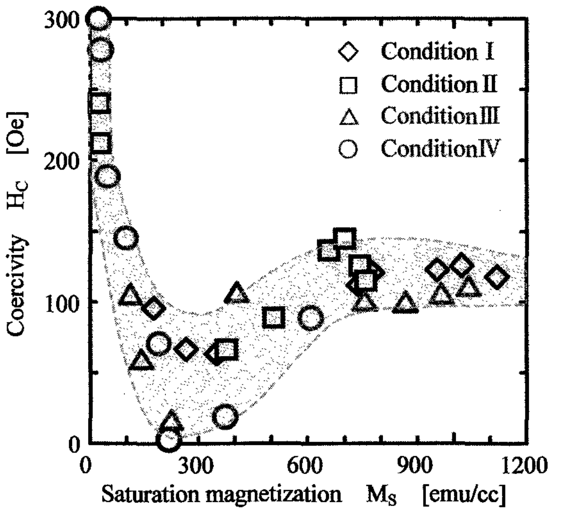

Fig. 4 Relationship between saturation magnetization and coercivity.

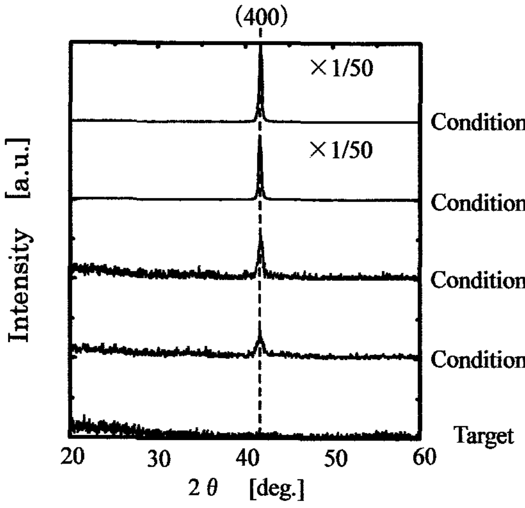

Fig. 5 X-ray diffraction diagrams of Ni-Zn ferrite films.

Fig. 5 shows the XRD diagrams for the Ni-Zn ferrite thin-films with lowest coercivity in in condition I, II, III and IV. A diffraction peak from only spinel (400) plane was observed in all samples. The high diffraction peaks was observed for the Ni-Zn film deposited in condition III and IV. The result proves that superior crystallinity was achieved in these films showing low coercivity

The deposition rate was proportional to the target voltage as shown in Fig 6. This results means that the target voltage is secondary factor to determine the deposition rate in this ECR sputtering method. In this system, target voltage plays a role only to collect and accelerate argon ions to the target from the plasma flow. In a conventional sputtering system, target voltage concerns to both plasma generation and collection of argon ions. The maximum deposition rate reached to over 30nm/min at a target voltage of -700 V in Condition II and IV. 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. 13.8 nm/min. and 24.3 nm/min. in Condition I, II, III and IV, respectively.

In Table 1, magnetic properties, grain size, average surface roughness (R_a), and deposition rate of the

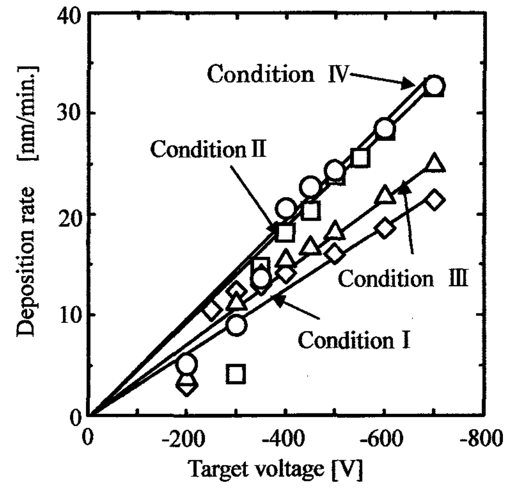

Fig. 6 Target-voltage dependence of deposition rate.

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

Deposition condition	Target-voltage	Oxygen gas inlet position	H_c [Oe]	M_s [emu/cc]	Grain size [nm]	R_a [nm]	Deposition-rate [nm/min]
Condition I	-300 V	Plasma chamber	65	348	33.6	0.89	12.3
Condition II	-400 V	Plasma chamber	67	375	37.1	1.14	18.1
Condition III	-350 V	Deposition chamber	15	224	42.9	1.36	13.8
Condition IV	-500 V	Deposition chamber	3	218	74.2	2.34	24.3

Ni-Zn films deposited under the above described minimum coercivity condition are summarized. The ferrite thin films deposited in condition III and IV had a larger grain size and a smoother surface than those in condition III and IV. This may be one of the reasons of lower coercivity of the films deposited in condition III and IV.

The residual stress of the Ni-Zn thin-films is less than 1×10^{10} dyne/cm^2 which is relatively small value. This is convenient in deposition of thick ferrite films because this ensures the difficulty in peeling off from the substrate.

4. CONCLUSIONS

A novel reactive sputtering method using an Electron-Cyclotron-Resonance (ECR) microwave plasma was firstly used to deposit Ni-Zn ferrite thin-films. The Ni-Zn spinel ferrite thin-films with preferential orientation of (400), good crrystallinity and relatively low coercivity less than 3 Oe were successfully obtained at a high deposition rate of 24 nm/min. and at a temperatures lower than 200 degrees C. The reactive ECR sputtering is one of the most suitable reparation methods of ferrite thin-films applicable to the backlayer of ferrite thin-film perpendicular magnetic recording media and other magnetic thin-film devices.

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