Magnetic Properties and Structure of FeN/AlN Multilayered Films

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Magnetic properties and structure of FeN/AlN multilayered films deposited by r.f. magnetron sputtering were studied. $H_c$ decreased as the ratio of a FeN layer thickness to a AlN one became small. $H_c$ also decreased with decreasing film thickness of FeN/AlN bilayers. $H_c$ and 4 $\pi M_s$ in a 22Å/11Å multilayered film were 1 Oe and 8.5 kG, respectively. The film had high permeability, $\mu'$, which was 800 at a 5 MHz and 650 even at a 100 MHz because of high resistivity. SEM observation and $\mu$-AES analysis revealed the film had a layered structure. X-ray diffraction made it clear that high permeability was ascribed to fine grains and smaller lattice mismatch between layers compared to the thick layered films.

**Key words:** soft magnetic film, FeN/AlN multilayered films, high frequency, high resistivity, permeability

1. Introduction

Nitrogen content Fe films are extensively studied because of high saturation magnetization ($4 \pi M_s$). Fe$_x$N$_{2-x}$ phase is said to have a extremely high $4 \pi M_s$ more than 28 kG$^1$ but its magnetic properties have not been fully understood. Moreover films of the phase aren't fabricated by conventional sputter deposition techniques.$^{25-29}$ On the other hand, a few percent N$_2$ content Fe (defined as FeN below) films are easy to get by means of the sputtering techniques.$^{4,6}$ They show good soft magnetic properties with low coercive force and high permeability as well as high $4 \pi M_s$ (20 kG). For magnetic devices used at high frequency, single layered FeN films deteriorate permeability at high frequency because of eddy current loss. Multilayered FeN films were proposed.$^{6,10-12}$ Especially films with ceramic spacers such as FeN/SiN$^{30}$ will be expected to reduce eddy current loss. But soft magnetic properties have been rarely reported in multilayers based on FeN films with several tens of Å in thickness.$^6$ In this paper FeN/AlN multilayered films which bilayer thickness is below 100Å were studied in respect of magnetic properties and structure.

2. Experiment

Films were deposited with a r.f. planar magnetron sputtering apparatus. Sputtering were tried under the conditions listed in Table 1. FeN and AlN films were deposited alternately when substrates were channeled above each target gun. The ratio of nitrogen partial pressure to total gas pressure was expressed as a flow ratio which means the volume fraction in percentage of N$_2$ gas to total gas in a chamber. The each flow ratio was selected as the best condition of soft magnetic properties for FeN and of getting h.c.p. crystalline for AlN.

Coercive force ($H_c$) and 4 $\pi M_s$ were measured by a V.S.M.. Permeability was measured by an eight-figured coil method. X-ray diffraction (Cu radiation) was used for the analysis of film structure. Grain diameters were derived from the half width of FeN d(110) peaks with a Scherrer equation. Resistivity was measured with a four proves method. A cross sectional view was observed by a FE-SEM (Hitachi S-900) with a 15 kV acceleration. Depth profiles on constituent elements were analyzed by a $\mu$-AES.

3. Results and Discussion

It was reported that $H_c$ was influenced by the thickness ratio of magnetic layers to nonmagnetic layers.$^6$ Change in magnetic properties ($H_c$, 4 $\pi M_s$) due to the thickness ratio was

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Sputtering conditions</th>
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<tr>
<td>method</td>
<td>r.f. planar magnetron</td>
</tr>
<tr>
<td>back pressure</td>
<td>$\leq 5 \times 10^{-7}$ torr</td>
</tr>
<tr>
<td>substrates</td>
<td>Corning 7059</td>
</tr>
<tr>
<td>substrate temperature</td>
<td>150°C</td>
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<tr>
<td>total gas pressure</td>
<td>5 mtorr (N$_2$ flow ratio:3% for FeN,20% for AlN)</td>
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<tr>
<td>deposition rate</td>
<td>11 A/sec (FeN)</td>
</tr>
<tr>
<td>applied field</td>
<td>130 Oe</td>
</tr>
<tr>
<td>total film thickness</td>
<td>$\sim 2 \mu$m</td>
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investigated when AIN layer was kept in a constant thickness (11 Å) (Fig. 1). The thickness of FeN and AIN layers in one sequence (period) was expressed as t(FeN) and t(AIN), respectively. Circles denote $H_c$ and triangles 4 $\pi M_s$. $H_c$ decreases with decreasing t(FeN) and reaches below 1 Oe. This suggests magnetic interaction between FeN films was weakened as FeN volume in a period decreases. 4 $\pi M_s$ also decreases with decreasing FeN film thickness because of the decrease in total FeN volume. From this result the thickness ratio in a period, t(FeN)/t(AIN), was kept 2 in a later discussion.

Change in magnetic properties due to FeN film thickness in a period is shown in Fig. 2. When t(FeN) is around 100 Å, films peeled off from substrates. That’s why data around 100 Å is absent. The cause of this exfoliation will be described hereafter. $H_c$ is less than 5 Oe in the range beyond 200 Å (region 1) and has a minimum value, 2 Oe, at a t(FeN)=500 Å. Below 100 Å (region 2), $H_c$ decreases as t(FeN) decreases and reaches about 1 Oe at t(FeN)=11 and 22 Å. 4 $\pi M_s$ is almost constant in the region 1 but reduces drastically in the region 2 in spite of having the same film thickness ratio. This suggests the crystal structure or something of FeN films in the region 2 is different from that in the region 1. From the above results, films near t(FeN)=100 Å were thought to be subjected stronger internal stress compared to other region and peeled off from the substrates. This interpretation will be supported by the results of X-ray diffraction analysis discussed in Fig. 8. Note that low $H_c$ (∼1 Oe) was reached when t(FeN) were below 22 Å, which is superior to the region 1 reported so far.

To clarify the soft magnetic characteristics of films in the region 2, permeability was also measured (Fig. 3). Here permeability means real part of permeability, $\mu'$. Open and solid circles denote the value of permeability at a 5 MHz and a 50 MHz frequency, respectively. The $\mu'$ at a 5 MHz decreases in the range 1 and increases in the range 2 with decreasing t(FeN). The $\mu'$ is 800 at a t(FeN)=22 Å and 900 at a 11 Å. It is noteworthy that the deterioration in $\mu'$ (50 MHz)/$\mu'$ (5 MHz) of these thin t(FeN) films is smaller that of thick ones. Fig. 4 shows the dependence of permeability on frequency about t(FeN)/t(AIN)=1000 Å/500 Å and 22 Å/11 Å films. Permeability are expressed by two parts. One is real part, $\mu'$, and the other imaginary part, $\mu''$. Circles and triangles denote $\mu'$ and $\mu''$, respectively. Solid marks and open ones mean data on a 1000 Å/500 Å and a 22 Å/11 Å films, respectively. The $\mu'$ of the 1000 Å/500 Å film deteriorates at high frequency while $\mu'$ of 22 Å/11 Å one doesn’t change so much. The $\mu'$ of the thin film at a 100 MHz is superior to that of the thick one. The $\mu''$ of a 22 Å/11 Å film is much smaller than that of a 1000 Å/500 Å film at any frequency. This low value of $\mu''$ is the large advantage from the viewpoint of view of application for micro-magnetic devices in high frequency range, because the quality factor (Q) can be expressed as $Q=\mu'/\mu''$. So the 22 Å/11 Å film would be one of the candidates.
Fig. 4 Dependence of permeability on frequency.

![Graph showing permeability vs. frequency for FeN/AIN multilayers](image)

(a) cross sectional view

(b) μ-AES depth profiles

**Fig. 5** FeN(22Å)/AIN(11Å) multilayered film (a) cross sectional view (b) depth profile.

profiles for constituent elements (b) show that films stood for a layered structure even if the thickness of a period is 22Å/11Å. There was no distinct interdiffusion between layers.

The change in resistivity of multilayered films due to FeN film thickness in a period is shown in Fig. 6. Since the top films were AIN layers and non-conductive in these experiments, resistivity couldn't be measured by a four probes method when AIN films were thick. So t(AIN) was kept a constant value, 11Å. Resistivity gradually increases with decreasing t(FeN), steeply increases below 40Å and has the maximum value of about 500 μΩcm around 20 Å of t(FeN). This high value may be one of the reasons why a 22Å/11Å film shows better μ-f response than the case of 1000Å/500Å one. Resistivity of multilayered films depends on grain diameter. The abrupt increase of resistivity is attributed to fine grains. But small decrease below 22Å is unknown.

Permeability is known to closely relate to magnetic anisotropy energy. The energy sums up magnetocrystalline anisotropy energy and magnetoelastic energy. It was reported that actual magnetocrystalline anisotropy decreases as the grain size becomes small. Fig. 7 shows the change in grain diameter due to the thickness of FeN layer in a period, in the case of t(FeN)/t(AIN)=2. Grain diameter (D) was derived from a Scherrer equation. Grains were around 100Å in diameter and became small with decreasing film thickness in the region 1. In the region 2, D was less than 50Å and about 25Å at t(FeN)=22Å and 11Å. This may be another reason why thin layered films had high permeability and low \( H_c \).

Lattice mismatch between two different layers enhances the magnetoelastic energy as the mismatch grows large. So we measured the lattice spacings of FeN and AIN in multilayered films (Fig. 8). FeN and AIN films are supposed to be b.c.c. and h.c.p. structure, respectively. The representative spacing, \( d \) (002)
for AlN and d(110) for FeN, are taken in the figure because of their strong preferred orientation in the film plane. There is no change of FeN d(110) in the region 1. On the other hand AlN d(002) slightly increases as increasing thickness in that region. In the region 2, d(110) increases while d(002) decreases. The d(110) of FeN films was 2.11 Å and the d(002) of AlN films 2.40 Å below 22 Å in \( t(\text{FeN}) \). These changes reduce lattice mismatch between two layers if structural configuration doesn’t change. The reduction in mismatch seems to deteriorate magnetoelastic energy and improve permeability in a thin layer region. Magnetostriction \( \lambda \) of FeN single layer was almost zero and apparent \( \lambda \) of films changed into plus by multilayering. The absolute value could not be obtained because of the lack of any information on mechanical factors for multilayered films.

Lattice spacing of FeN d(110) in thin layered films were distorted by about 3% compared to thick films such as 1000 Å/500 Å. This distortion is fairly large for a Fe–N solid solution. Moreover \( 4 \pi M_s \) in the thin films were deteriorated drastically. If the films which were subjected to strong stress have a f.c.c. phase with (111) orientation in the film plane, calculated lattice constant is \( a = 3.66 \) Å. This is coincide with the data already reported.\(^{13}\) Magneto-crystalline anisotropy of cubic (111) planes is much smaller than that of (110) ones.\(^{14}\) So high permeability in thin multilayered films maybe ascribes to this kind of phase transition. To confirm this transition more detailed study will be needed.

4. Conclusion

FeN/AlN multilayered films were studied for the purpose of applying to micromagnetic devices used at high frequency. Films showed a soft magnetic properties when the thickness of bilayers was as thin as 22 Å/11 Å. \( H_c \) and \( 4 \pi M_s \) of this film was 1 Oe and 8.5 kG, respectively. Resistivity increased as layers became thin and its value was 500 \( \mu \Omega \text{cm} \) on a 22 Å/11 Å film. Its permeability \( \mu' \) was 800 at a 5 MHz and 650 even at a 100 MHz. The films had finer grains and smaller lattice mismatch between FeN layers and AlN ones compared to thick layered films over 200 Å.

References


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