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著者	土井　正晶
journal or publication title	Journal of applied physics
volume	76
number	10
page range	6642-6647
year	1994
URL	http://hdl.handle.net/10097/35504

doi: 10.1063/1.358431

Magnetic moment of α''-$Fe_{16}N_2$ films (invited)

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In order to determine the value of the intrinsic magnetic moment of the α'' phase, the films of nitrogen-martensite with various N content were fabricated under various reactive sputtering conditions. The magnetic moment of $(\alpha''+\alpha')$-$Fe_{16}N_2$ films is discussed in connection with the change of the unit-cell volume of the bct structure and the degree of N site ordering in nitrogen-martensite. As a result, it is found that (1) the same structure as bulk α''-$Fe_{16}N_2$ is realized in the present films, (2) the saturation magnetization σ_s of the α' phase increases about 4% with increasing unit-cell volume of the α' phase, (3) the degree of N site ordering from α' to α''-$Fe_{16}N_2$ does not much affect σ_s, and (4) the experimentally obtained maximum value of σ_s for the $(\alpha''+\alpha')$-$Fe_{16}N_2$ film was 232 emu/g. The intrinsic value of σ_s in the α'' phase (in the perfectly ordered state) is proposed to be no more than 240 emu/g at 300 K.

I. INTRODUCTION

Recently, the present authors have established synthesizing processes for α''-$Fe_{16}N_2$ compound films on MgO (100) single-crystal substrates by using both reactive sputtering and plasma evaporation methods.[1,2]

However, the magnitude of the saturation magnetization σ_s for $(\alpha''+\alpha')$-$Fe_{16}N_2$ films with stoichiometric N content showed 226 emu/g for sputtered films and 232 emu/g for plasma evaporated films, respectively. The films thus fabricated did not show any giant magnetic moment, even though clear formation of $(\alpha''+\alpha')$-$Fe_{16}N_2$ phase was achieved. These values of σ_s in $(\alpha''+\alpha')$-$Fe_{16}N_2$ films agree well with the result of the recent theoretical band calculation,[3] and are completely different from the earlier results reported by various groups.[4–7]

Up to now, reported values of σ_s of the α'' phase (about 257–315 emu/g) were estimated ones (not directly measured) except for the films synthesized by MBE[7] (2.9 T directly measured, α'' single phase). The estimation of the value of σ_s in the α'' phase was carried out by using experimentally determined volume-averaged values of σ_s (240–260 emu/g)[4–6] after fixing the volume fraction of the α'' phase in whole films which consist of phase mixtures of α-Fe$+\alpha''$-$Fe_{16}N_2$ and/or $\alpha+\alpha'+\alpha''+\gamma'$-$Fe_4N+\cdots$.

There still exist some physical problems concerning σ_s of the α''-$Fe_{16}N_2$ phase, especially for the multiphase films, namely, (1) the big difference among reported values of σ_s in the α'' phase, from 257 to 315 emu/g at RT, and (2) quantitative evaluation for fixing the volume fraction of the α'' phase in whole film. These physical situations lead to a conclusion that the intrinsic magnetic moment of σ_s in the α'' phase is still unknown.

On the other hand, for the single-crystal films with a single α'' phase prepared by the MBE method,[7] there also exists some physically contradictive problems: (1) No clear superlattice lines from the lattice planes including the a axis in the α'' phase were detected, even though a perfect site ordering of N atoms was realized;[8] (2) the hyperfine field Hi of the α'' phase was nearly equal to that of α-Fe (330 kOe) and no splitting of Hi due to three different Fe sites in the α''-$Fe_{16}N_2$ structure was observed,[8] even though σ_s showed 2.9 T ($\simeq$315 emu/g, assuming ρ=7.4 g/cm^3 for α''-$Fe_{16}N_2$); (3) σ_s of the fully ordered α''-$Fe_{16}N_2$ films changed reversibly within the temperature range up to 400 °C and an irreversible change of σ_s due to the phase transformation from α'' to $\alpha+\gamma'$ was not observed.[8] According to the experiment by Jack using powder, the α'' phase is metastable and it must decompose into $\alpha+\gamma'$ phases at about 200 °C.[10]

These experimental results lead to a physical conclusion that the appearance of the giant magnetic moment in α''-$Fe_{16}N_2$ films proposed by Kim and Takahashi and Komuro *et al.* are not simply attributable to the conventional α''-$Fe_{16}N_2$ structure. Therefore the origin of the giant magnetic moment arising from the nitrogen-martensite structure is still under question.

In the present study, in order to determine the value of the intrinsic magnetic moment of the α'' phase, nitrogen-martensites with various N content were systematically fabricated under various sputtering conditions. Also, the magnetic moment of $(\alpha''+\alpha')$-$Fe_{16}N_2$ film is discussed in connection with the change of the unit-cell volume of the body-centered-tetragonal (bct) structure and the degree of N site ordering in nitrogen-martensites.

II. EXPERIMENTAL PROCEDURE

Fe-N films were fabricated by a facing target-type dc sputtering system under the selected plasma condition (Te =0.2 eV, Ne$\simeq 1\times10^{10}$ cm^{-3}). The base pressure of the sputtering chamber was below 3×10^{-7} Torr. An Ar-N_2 mixture was introduced to the sputtering chamber at 5 sccm (standard cc/min) with controlling N_2 flow ratio (0%–30%) under a fixed total pressure (1–10 mTorr).

MgO (100) and (110) single-crystal substrates were used. Before the film fabrication, substrates were baked at 200 °C for 2 h and cooled down to RT. This heat treatment was carried out in the evacuated sputtering chamber ($\simeq 2\times10^{-6}$ Torr).

Prior to the fabrication of Fe-N films, an α-Fe underlayer with thickness of 50 Å was deposited on MgO (deposition rate=33 Å/min; Ar pressure P_{Ar}=5 mTorr). Successively, an

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Fe-N film with a thickness of 3000 Å was deposited onto the α-Fe underlayer (deposition rate=33–240 Å/min; pressure of Ar-N_2 mixture P_{total}=1–10 mTorr).

Annealing of the films after an air exposure was carried out at 150 °C for 2–20 h in a vacuum atmosphere below 5×10^{-6} Torr.

Values of the saturation magnetization σ_s of the films were determined by a vibrating sample magnetometer (VSM) measurement. Conversion electron Mössbauer (CEM) spectra were obtained at RT. The velocity was referred to the pure α-Fe.

Structure analysis of the films were made with a Co $K\alpha$ x-ray diffractometer (XRD) equipped with a graphite monochrometer and a pole figure attachment. Schultz's reflection method was used for the determination of lattice constants and preferred orientation of grains. Contents of nitrogen atoms in the films were determined by electron spectroscopy for chemical analysis (ESCA). Calibration of nitrogen contents were made by using the value obtained from ϵ-$Fe_{2-3}N$ foil (24.2 at. % N) for a standard sample.

N contents in the films increased with increasing N_2 flow ratio. In the case of the film deposited at P_{total} of 5 mTorr and N_2 flow ratio of 18%, the N content was found to be about 11 at. %, which is the same value as the stoichiometric N content of the α''-$Fe_{16}N_2$ phase.

III. RESULTS AND DISCUSSION

A. Structure

According to Jack,[10,11] in the α' phase, N atoms occupy randomly the octahedral interstices at the midpoints of the c edges of the bct cell (0, 0, $\frac{1}{2}$), and the centers of the C faces, ($\frac{1}{2}$, $\frac{1}{2}$, 0). As a result, the lattice constant c of the α' phase is elongated from 2.866 to 3.195 Å and the lattice constant a is shortened from 2.866 to 2.832 Å, respectively, depending on the N content.

The α'' phase has an ordered N site location of the octahedral interstices. The unit cell of the α'' phase contains eight of the expanded bct pseudo-unit cells and has dimensions $a'=2a$ and $c'=2c$, where a and c are the lattice constants of the pseudocell. In the larger true unit cell, the symmetry is also bct, since the α'' phase can be identified by observing reflections from this larger true unit cell for which $(h+k+l)$, Miller's index, is even. Based on this structural knowledge, phase identification is carried out in the present films.

In the case of a MgO(100) single-crystal substrate, a diffraction line from the (002) plane of the α'' phase, α''(002), which is expected to appear around 33° for 2θ, was not observed in an as-deposited state. Only α'(002) was clearly observed in the high angle region. The peak position of α'(002) shifted from 75° to 68° for 2θ with increasing N_2 flow ratio. This shift to lower angle of 2θ is simply explained by the elongation of the c axis of the bct structure of the α' phase. By taking into account the N concentration dependence on lattice constants a and c in nitrogen-martensite,[11] the N content of the α' phase in the present films was found to increase with increasing N_2 flow ratio. This result agrees well with that of ESCA measurements.

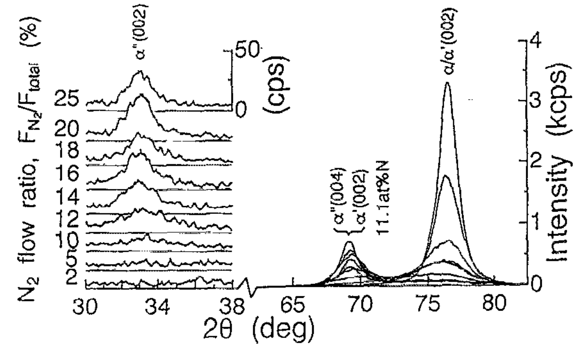

FIG. 1. X-ray diffraction patterns for the films fabricated under P_{total}=5 mTorr, deposition rate=240 Å/min after annealing at 150 °C for 2 h.

In Fig. 1, typical changes of XRD patterns of the films after annealing are shown. After annealing, α'(002), which had been observed in an as-deposited state, split into two diffraction lines. One corresponds to α''(004) and/or a'(002) with satisfying stoichiometric N content of α''-$Fe_{16}N_2$ (11 at. % N). The other corresponds to α(002) of slightly deformed α-Fe. Furthermore, simultaneously at around 33° for 2θ, α''(002), which is the diffraction line from the larger true unit cell of the α'' phase, came to be clearly observed. This fact means that the ordering of N atoms was promoted by annealing while retaining the bct structure and the α'' phase with stoichiometric N content was synthesized.

The unique diffraction patterns from (103), (105), (112), (114), and (213) planes of the α'' phase, including the a axis for $(\alpha''+\alpha')$-$Fe_{16}N_2$ film, are shown in Fig. 2. As seen in the

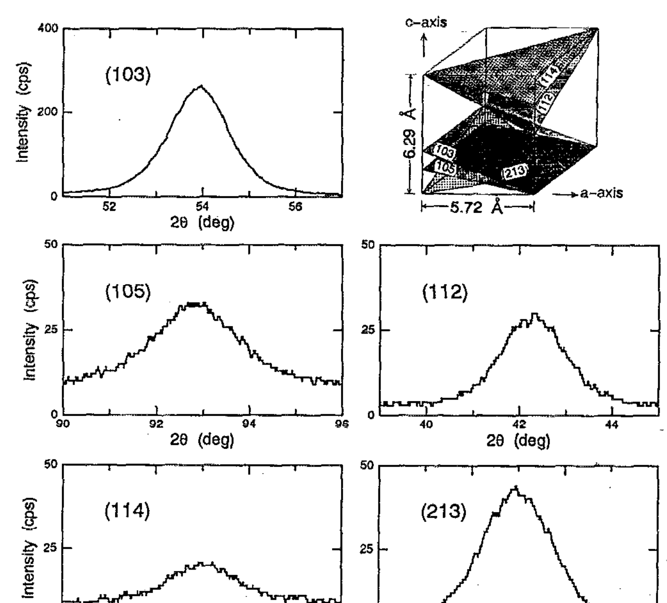

FIG. 2. The unique diffraction patterns from (103), (105), (112), (114), and (213) planes of the α'' phase including the a axis for $(\alpha''+\alpha')$-$Fe_{16}N_2$ film fabricated under P_{total}=10 mTorr, F_{N_2}=12%, deposition rate=33 Å/min, and annealed at 150 °C for 2 h.

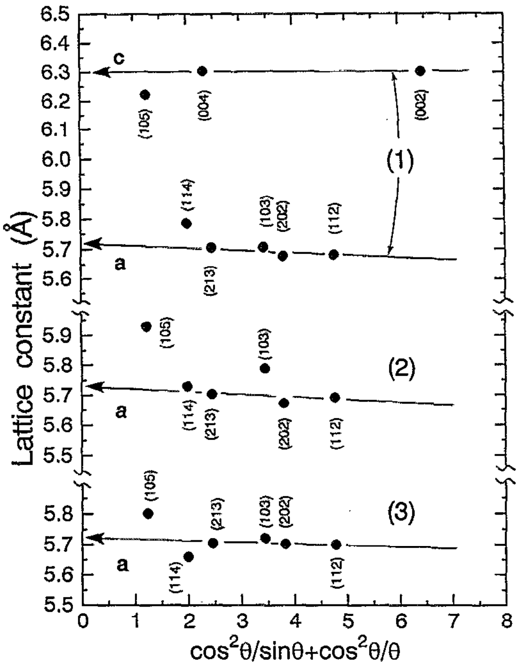

FIG. 3. The lattice constants a and c of the α'' phase, determined by various unique diffraction lines of the α'' phase, plotted against the Nelson–Riley function. Fabrication conditions of the films: (1) $P_{total}=10$ mTorr, $F_{N_2}=16\%$, deposition rate=240 Å/min, annealed at 150 °C for 2 h; (2) the same as (1) annealed for 20 h; (3) $P_{total}=10$ mTorr, $F_{N_2}=12\%$, deposition rate=33 Å/min, annealed at 150 °C for 2 h.

figure, the existence of the α'' phase was reconfirmed by these clear unique diffraction lines. In order to determine accurate lattice constants a and c of the α'' phase, the lattice constants a calculated from each plane are plotted against the Nelson–Riley function[12] ($\cos^2\theta/\sin\theta+\cos^2\theta/\theta$) in Fig. 3. In the figure, the reflections marked (3) correspond to the film shown in Fig. 2, and those marked (1) and (2) correspond to $(\alpha''+\alpha')$-$Fe_{16}N_2$ films prepared by different experimental conditions. As a reference, the lattice constant c calculated from $\alpha''(002)$ and $\alpha''(004)$ is also shown as (1). Each extrapolated value of the lattice constants a and c of the present films coincided with that of the α''-$Fe_{16}N_2$ precipitates in bulk powder reported by Jack.[10] Therefore it is concluded that the α'' phase formed in sputtered films has the same structure as the bulk α'' phase.[13]

While in the case of a MgO(110) single-crystal substrate, a preferred grain orientation of (211) and (112) of the α' phase was found in an as-deposited state. By annealing, unique diffraction lines of the α'' phase, $\alpha''(211)$, and $\alpha''(112)$, were observed. Therefore it was found that the α'' phase with a preferred orientation of (211) of the α'' phase can also be synthesized even on MgO(110) substrates. Through the whole result, relationships concerning the crystal orientation between α''-$Fe_{16}N_2$ structure and MgO are shown in Table I.

B. Magnetic moment

1. Dependence of magnetic moment on N_2 flow ratio

Values of σ_s in an as-deposited state increased slightly with increasing N_2 flow ratio, and took a maximum of 220

TABLE I. Crystal orientation relations between α''-$Fe_{16}N_2$ and MgO.

MgO(100)	MgO(110)
$\alpha''(001)\|\|MgO(001)$	$\alpha''(211)\|\|MgO(101)$
$\alpha''[110]\|\|MgO[100]$	$\alpha''[011]\|\|MgO[001]$

emu/g around a N_2 flow ratio of 15%. The N content of the films, which showed the broad maximum in σ_s, was nearly equal to the stoichiometric N content of $Fe_{16}N_2$ (11 at. %). The values of σ_s for annealed film with stoichiometric N content of $Fe_{16}N_2$ (11 at. %) ranged from 213 to 226 emu/g. The average values of σ_s for Cu-coated films were 228 emu/g for the film consisting of the α' phase and 232 emu/g for the film consisting of $(\alpha''+\alpha')$-$Fe_{16}N_2$ phases. The difference of the value of σ_s between coated and noncoated ones may mainly be caused by the surface oxidation due to the adsorbed oxygen at the film surface introduced by venting the chamber with air. In the case of films deposited on a MgO(110) substrate, the value of σ_s in $(\alpha''+\alpha')$-$Fe_{16}N_2$ was 210 emu/g (non-Cu-coated). As a whole, maximum values of σ_s of about 232 emu/g were obtained for $(\alpha''+\alpha')$-$Fe_{16}N_2$ films in the present study. This experimentally determined value was definitely smaller than the value reported as a giant magnetic moment of 2.9 T.[7]

2. Dependence of magnetic moment on unit-cell volume

Figure 4 shows the changes of σ_s against unit-cell volume of the α' phase with various N contents in an as-deposited state. In the figure, 1/8 of the unit-cell volume of the bulk α''-$Fe_{16}N_2$ is indicated. Filled marks correspond to

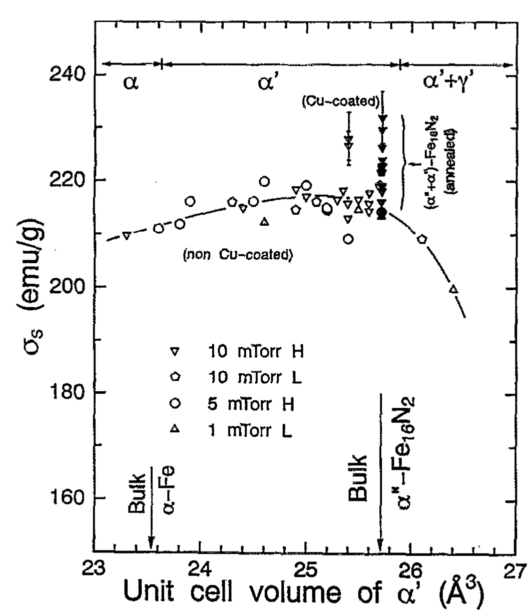

FIG. 4. The changes of σ_s against unit-cell volumes: (1) α' phase with various nitrogen contents in an as-deposited state, and (2) annealed $(\alpha''+\alpha')$-$Fe_{16}N_2$ films with and without Cu coating. H and L correspond to high (240 Å/min) and low (33 Å/min) deposition rates, respectively.

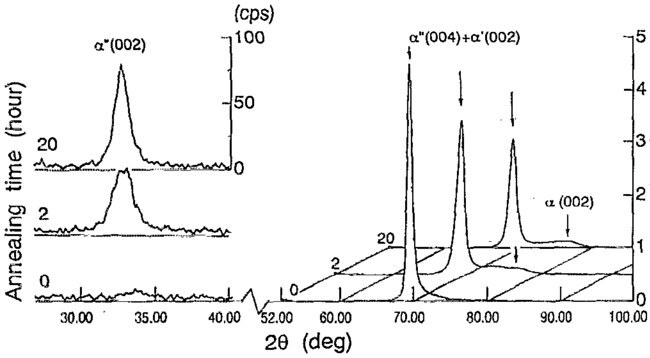

FIG. 5. The change of x-ray profiles against annealing time for the film with stoichiometric N content of the α'' phase (11 at. %) sputtered under $P_{\text{total}}=10$ mTorr, $F_{N_2}=10\%$, and deposition rate=33 Å/min.

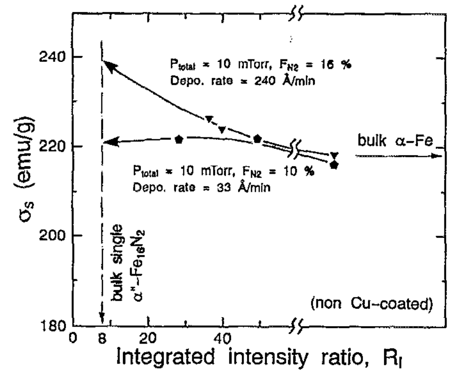

FIG. 6. The change of σ_s against the integrated intensity ratio R_I for the films sputtered under $P_{\text{total}}=10$ mTorr, deposition rate=33 and 240 Å/min, $F_{N_2}=10\%$ and 16%, non-Cu-coated.

the annealed $(\alpha''+\alpha')$-$Fe_{16}N_2$ films with stoichiometric N content. In the same figure, σ_s for the Cu-coated films are also shown.

For the films consisting of α' single phase (in an as-deposited state), the values of σ_s increased slightly with the increment of the unit-cell volume. At the unit-cell volume of about 25.5 Å^3 (α' phase with 11 at. % N), σ_s showed 228 emu/g on average (Cu-coated) and this value was about 4% higher compared to that of bulk α-Fe.

In the case of annealed $(\alpha''+\alpha')$-$Fe_{16}N_2$ films (Cu-coated), the value of σ_s showed 232 emu/g on average, and were about 2% larger than that of each as-deposited film, while the unit-cell volume of the α'' phase is always constant and coincided with that of the bulk α'' phase (see Fig. 3). The unit-cell volume of the α'' phase with 11 at. % N is equal to that of the α''-$Fe_{16}N_2$ phase within the accuracy of this experiment. Therefore the change of σ_s by annealing in nitrogen-martensite with 11 at. % N content cannot be discussed as a function of the change of unit-cell volume of a bct structure caused by the phase transformation from α' to α'' phase. In the next section, as a second physical factor the degree of N site ordering in nitrogen-martensite will be discussed in connection with the change of σ_s.

3. Dependence of magnetic moment on N site ordering

To evaluate the degree of N site ordering in the bct structure of nitrogen-martensite, two factors should be taken into account. One is the change of the integrated intensity of the $\alpha''(002)$ line which is the unique superlattice diffraction from the α'' phase. Another is the integrated intensity ratio of $\alpha''(004)+\alpha'(002)$ to $\alpha''(002)$, R_I, namely

$$R_I \equiv [I^{\alpha''}{}_{(004)}+I^{\alpha'}{}_{(002)}]/I^{\alpha''}{}_{(002)}.$$

The calculated value of R_I is about 8 for the ideal structure of the α'' phase.[14]

Figure 5 shows the change of XRD profiles against annealing time for the film with stoichiometric N content of the α'' phase (11 at. %). From these profiles, the intensity of $\alpha(200)$ was relatively very weak and any diffraction lines from the γ' phase were not observed. After annealing for 2 h, $\alpha''(002)$ came to be observed clearly. By annealing further for 20 h, the integrated intensity of $\alpha''(002)$ increased about 20% compared to that of 2 h. On the other hand, the experimentally determined value of R_I changed from 50 to 28 with the increase of annealing time.

Therefore, from these experimental results (1) the increase of the integrated intensity of $\alpha''(002)$ and (2) the change of R_I approaching to the ideal value of 8, the increase of degree of N site ordering in nitrogen-martensite, which directly corresponds to the increase of the volume fraction of the α'' phase in the films, is strongly promoted by annealing.

In Fig. 6, the changes of σ_s in $(\alpha''+\alpha')$-$Fe_{16}N_2$ films by isothermal annealing at 150 °C are shown against the integrated intensity ratio R_I. For one film σ_s increases slightly from 218 (as deposited) to 226 emu/g at $R_I=36.4$ (20 h). On the other hand, for another film, σ_s takes the value of about 222 emu/g at $R_I=49$ (2 h) and keeps a constant value even though R_I approaches to the value of 8. From these experimental facts, it was found that the degree of N site ordering in nitrogen-martensite does not much affect the increment of σ_s. The expected values of σ_s at $R_I=8$ (perfect ordered state in α''-$Fe_{16}N_2$) estimated by the simple extrapolation with using the data points of σ_s against R_I are no more than 222–240 emu/g, a value which is definitely smaller than the giant magnetic moment of 2.9 T.

4. Dependence of magnetic moment on temperature

Figure 7 shows the temperature dependence of σ_s in $(\alpha''+\alpha')$-$Fe_{16}N_2$ films with stoichiometric N content deposited on MgO (100) and (110) substrates, respectively. Heating and cooling were at 60 °C/h. On heating, the value of σ_s gradually decreased with increasing temperature. Around 200 °C a sudden discontinuous decrease of σ_s from 200 to 170 emu/g was observed. With further increasing temperature, σ_s decreased monotonously and reached about 130 emu/g at 400 °C, while on cooling, the change of σ_s with respect to temperature was completely different from that of heating, and no sudden change of σ_s was observed. The sudden discontinuous change of σ_s observed around 200 °C is considered to correspond to the phase change from $\alpha''+\alpha'$ to $\alpha+\gamma'$. Therefore the hysteresis in the σ_s-T curve is caused by this irreversible phase decomposition from

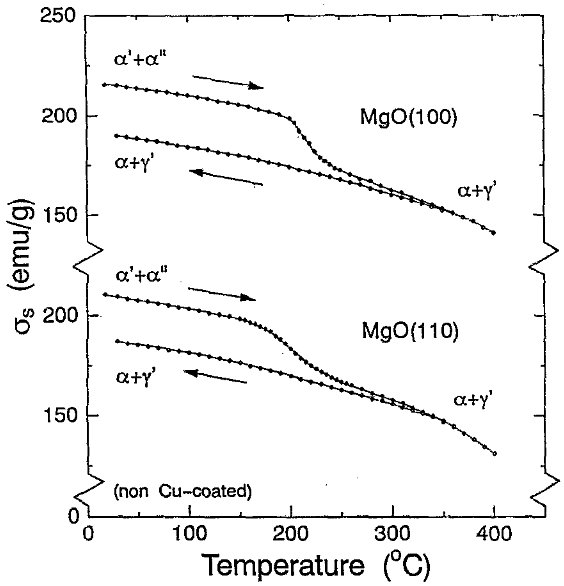

FIG. 7. The temperature dependence of σ_s in the films consisting of the α'' phase deposited on MgO (100) and (110) substrates fabricated under P_{total}=10 mTorr, deposition rate =240 Å/min, and F_{N_2}=16% after an initial anneal at 150 °C for 2 h.

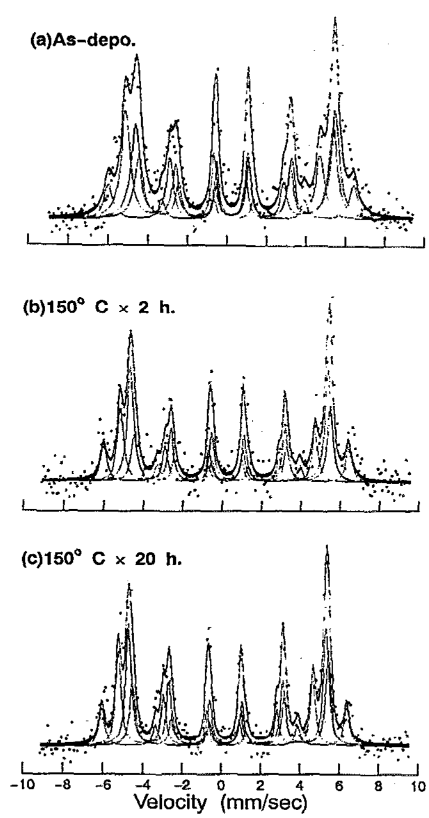

FIG. 8. Mössbauer spectra of $(\alpha''+\alpha')$-$Fe_{16}N_2$ film (non-Cu-coated) measured at R.T. (a) As-deposited, (b) annealed at 150 °C for 2 h, and (c) annealed at 150 °C for 20 h, respectively. The film was deposited under P_{total}=10 mTorr, deposition rate=240 Å/min, and F_{N_2}=16%.

TABLE II. Mössbauer parameters of the film deposited under P_{total}=10 mTorr, F_{N_2}=16%, deposition rate=240 Å/min. *Hi* is the hyperfine field, *I.S.* the isomer shift, *e.q.Q.* the quadrupole splitting, *Hwid* the distribution of *Hi*, and area the relative intensity, respectively.

Site	*Hi* (kOe)	*I.S.* (mm/s)	*e.q.Q.* (mm/s)	*Hwid* (kOe)	Area (%)
Fe(I)	289	0.01	−0.05	7.00	21.3
Fe(II)	316	0.17	0.04	7.00	31.3
Fe(III)	391	0.11	−0.05	7.00	11.2
α-Fe	335	0.02	−0.007	7.00	36.1
(As-deposited)					
Fe(I)	289	0.01	−0.05	4.00	17.8
Fe(II)	316	0.17	0.04	4.00	41.8
Fe(III)	391	0.11	−0.05	4.00	13.1
α-Fe	335	0.02	−0.007	4.00	27.3
(Annealed at 150 °C for 2 h)					
Fe(I)	289	0.01	−0.05	3.00	20.6
Fe(II)	316	0.17	0.04	3.00	37.7
Fe(III)	391	0.11	−0.05	3.00	12.5
α-Fe	335	0.02	−0.007	3.00	29.3
(Annealed at 150 °C for 20 h)					

$\alpha''+\alpha'$ to $\alpha+\gamma'$. The temperatures of this phase decomposition for the present films were good agreement with that of the α''-$Fe_{16}N_2$ precipitates in bulk powder reported by Jack.[10] The temperature dependence of σ_s observed in present experiments is found to be quite different from that of Gao and Komuro.[15,16]

C. Mössbauer spectrum

Figure 8 shows the change of Mössbauer spectra of the $Fe_{16}N_2$ film with stoichiometric N content by annealing. The spectrum in each film can be fitted into four hyperfine field interactions *Hi* of α phase, Fe(I), Fe(II), and Fe(III) of the $(\alpha''+\alpha')$-$Fe_{16}N_2$ phase. The fitted Mössbauer parameters are listed in Table II. As seen in the table, the large value of *Hi*, about 390 kOe, due to the Fe(III) site in nitrogen-martensite was detected in each film. Half widths of the peaks become narrower with increasing annealing time. This result corresponds to the promotion of N site ordering in nitrogen-martensite caused by annealing, and is in good agreement with the change of R_I and also the result of ion-implanted films reported by Nakajima.[14]

For the films examined presently, the average value of *Hi* was about 325 kOe, which was nearly equal to that of *Hi* of α-Fe. Therefore the value of σ_s in $(\alpha''+\alpha')$-$Fe_{16}N_2$ film of about 232 emu/g determined by VSM was consistent with the result of Mössbauer spectrum analysis. Based on the fitted Mössbauer parameters, the volume fraction of α-Fe was estimated to be 27% and 73% for the $(\alpha''+\alpha')$-$Fe_{16}N_2$ phase in annealed films. Using these values, the value of σ_s in the $(\alpha''+\alpha')$-$Fe_{16}N_2$ phase, $\sigma_{Fe_{16}N_2}$, is estimated by the following equation:

$$232^{exp}=\sigma_{Fe_{16}N_2}\times 0.73+\sigma_{\alpha\text{-}Fe}\times 0.27,$$

where $\sigma_{\alpha\text{-}Fe}$ is 218 emu/g. The obtained value of $\sigma_{Fe_{16}N_2}$ is 237 emu/g. Considering this calculated result and the result of the dependence of σ_s on R_I, the value of saturation magnetization of the α'' phase (perfect ordered state) should be no more than 240 emu/g, while the site population of the $(\alpha''+\alpha')$-$Fe_{16}N_2$ phase determined in this study was about 4:9:3, which is slightly different from the ideal ratio of 4:8:4 determined uniquely from the structure of α''-$Fe_{16}N_2$.

IV. SUMMARY

(i) $(\alpha''+\alpha')$-$Fe_{16}N_2$ films were synthesized on MgO single-crystal substrates by the reactive sputtering method. The result of the structural analysis using XRD and CEM revealed that the same structure as bulk α''-$Fe_{16}N_2$ is realized in the present films.

(2) The intrinsic value of saturation magnetization of the α'' phase (perfect ordered state) is proposed to be no more than 240 emu/g ($\simeq$2.4 μ_B per Fe atom in average) at 300 K.

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[13] The values of lattice constant a of the α'' phase reported by the present authors was previously determined only by using $\alpha''(004)$ and $\alpha''(202)$, which are duplicated with $\alpha'(002)$ and $\alpha'(101)$, respectively. Therefore the discrepancy for the value of lattice constant a between the present and earlier results is caused by the difference of the determination methods.

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