Structure and magnetic properties of Fe–Cr–N sputter-deposited films

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Structure and magnetic properties of Fe–Cr–N sputter-deposited films


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Abstract

Structure and magnetic properties of Fe–Cr–N ternary films prepared by DC magnetron facing-target sputtering have been investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), vibrating sample magnetometry (VSM) and Mössbauer effect. These films exhibit perpendicular magnetic anisotropy. We found that N₂-to-Ar flow ratio, Fe–Cr target area ratio and substrate temperature during film preparation are the factors influencing the anisotropy. Adjusting the chemical composition and deposition parameters, we obtained saturation magnetization of 300–400 emu/cm³ and perpendicular coercivity of 80–100 Oe. XRD measurements show that the films generally consist of the α-Fe(Cr) and γ′-(Fe,Cr)₄Nₓ (x < 1) phases, and that the enhancement of perpendicular anisotropy is always accompanied by a decrease in the grain size of α-Fe(Cr) phase and growth of the γ′-(Fe,Cr)₄Nₓ phase with a pronounced (2 0 0) texture. The Mössbauer spectra show that the γ′-(Fe,Cr)₄Nₓ phase is nonmagnetic at room temperature. Using the SEM and TEM, we found that the nonmagnetic γ′-(Fe,Cr)₄Nₓ phase displays columnar growth and that the small ferromagnetic α-Fe(Cr) grains of about 2–20 nm in diameter are located at grain boundaries of γ′-(Fe,Cr)₄Nₓ grains. Shape anisotropy seems to play an important part in the perpendicular magnetic anisotropy in these Fe–Cr–N films.

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Keywords: Perpendicular magnetic anisotropy; Fe–Cr–N film; Sputter-deposition; Structure and morphology; Mössbauer effect

1. Introduction

In the past few years, there has been a great deal of interest in the study of ternary Fe–X–N (X = Zr, Hf, Nb, Ta, Ti) systems [1–8]. This is because the third element X has strong affinity for nitrogen and ability of amorphous formation, and thus cause good soft magnetic properties, high saturation flux density, small magnetostriction and better thermal stability than binary Fe–N film. However, perpendicular magnetic anisotropy in Fe-based ternary nitride films has not been reported so far. Recently,
Honda et al. reported that the addition of a nitrogen gas during the deposition of Co–Cr films by sputtering has a remarkable effect on increasing perpendicular coercivity, $H_{c\perp}$ [9]. We reported that ternary Fe–Cr–N films deposited by DC reactive sputtering also show perpendicular magnetic anisotropy [10], and described detailed X-ray diffraction studies and X-ray photoelectron spectra of these films [11].

Perpendicular magnetic anisotropy has been observed in binary Fe–Cr films [12, 13]. In sputtered Fe$_{1-x}$Cr$_x$ ($x = 0.33–0.4$) films, coexistence of Cr-rich and Fe-rich phases was detected by SEM and X-ray diffraction. The Fe-rich and Cr-rich BCC phase regions grow in the direction normal to the film plane and the nonmagnetic Cr-rich grains separate the ferromagnetic Fe-rich grains. The perpendicular magnetic anisotropy is attributed to the shape anisotropy of the isolated ferromagnetic regions.

In this paper, we report in detail microstructure and magnetic properties of the Fe–Cr–N ternary films prepared by DC reactive sputtering. We show that the structure of the films and thereby the perpendicular magnetic anisotropy of these films depends sensitively on preparation conditions including nitrogen flow ratio, chemical composition and the substrate temperature.

2. Experimental

Fe–Cr–N films were deposited on glass and aluminium foil substrates by facing-target-type DC magnetron sputtering in mixed Ar + N$_2$ plasma, using a composite target consisting of pure Fe (99.9%) and Cr (99.9%) plates. The alloy composition was adjusted by changing the Cr target area ratio, $A_{Cr}$, defined as $A_{Cr} = \frac{\text{the area of Cr}}{\text{the total area of Fe and Cr targets}}$. In order to obtain Fe–Cr–N films with various nitrogen contents, a N$_2$ flow ratio, $R(N_2) = \frac{\text{N}_2\text{-flow rate}}{\text{[Ar-flow rate + N}_2\text{-flow rate]}}$, was changed with mass flow controllers. The substrate temperature, $T_s$, during the sputtering deposition was kept at about 50°C by water cooling and at 150, 250 and 350°C by indirect resistive heating. The detail of sputtering conditions were described previously [10]. The chemical composition of deposited films was determined by inductively coupled plasma (ICP) optical emission spectrometry and helium carrier fusion-thermal conductivity methods.

Structures of the films were analyzed by XRD. We used Rigaku 4037 XRD apparatus in the Bragg–Brentano geometry with Cu K$_\alpha$ radiation monochromatized by a graphite crystal for the XRD study, while we employed Hitachi HF-2000 operating at 200 kV for the TEM study. The fractured cross-section morphology of the sputter-deposited films was observed by a SEM. Mössbauer spectra were measured by a conventional multichannel spectrometer in a transmission geometry with the incident $\gamma$-rays perpendicular to the film planes. We used $^{57}$Co source in Rh matrix and calibrated the velocity scale using pure $\alpha$-Fe. Magnetic properties of the films were measured by a VSM in a magnetic field up to 16 kOe applied parallel or perpendicular to the plane of the films.

3. Results

3.1. Magnetic properties and structure

3.1.1. Effects of nitrogen flow ratio

Fig. 1 shows (a) the saturation magnetization, $M_S$, in-plane coercivity, $H_{c\parallel}$, and perpendicular coercivity, $H_{c\perp}$, and (b) the nitrogen content, $C_N$, of the Fe–Cr–N films sputter-deposited at $T_s = 50^\circ$C and $A_{Cr} = 33\%$ as a function of $R(N_2)$. The ICP measurement showed that the ratios of Fe and Cr atoms in these films are not influenced by $R(N_2)$, being constant at about 77 : 23. The $M_S$ value of the films decreases monotonically with increasing $R(N_2)$, but $H_{c\parallel}$ first increases and then decreases. Thus, there is a maximum in $H_{c\perp}$ near $R(N_2) = 35\%$. Moreover, $H_{c\perp}$ is always larger than $H_{c\parallel}$ and it is about twice as large as $H_{c\parallel}$ for $R(N_2) = 35\%$. As seen from Fig. 1b, $C_N$ increases as $R(N_2)$ increases and is 18 at% at $R(N_2) = 40\%$ for $A_{Cr} = 33\%$. This value is slightly smaller than the theoretical nitrogen content, 20 at%, of stoichiometric $\gamma'$-Fe$_4$N.

The XRD patterns of these films are shown in Fig. 2. The films consist of $\alpha$-Fe(Cr) and $\gamma'$-(Fe$_{1-x}$Cr$_x$)$_4$N$_x$ ($x < 1$) phases [11]. For the $\alpha$-Fe(Cr) phase, (1 1 0) peak broadens and becomes smaller.
as \( R(N_2) \) increases. This indicates that the size of \( \alpha \)-Fe(Cr) grains becomes smaller as \( R(N_2) \) increases. For the \( \gamma' \)-(Fe,Cr)\(_2\)N\(_x\) phase, the intensity of the (2 0 0) peak becomes larger and the (2 0 0) texture becomes more pronounced as \( R(N_2) \) increases [11]. At \( R(N_2) = 40\% \), the \( \alpha \)-Fe(Cr)(1 1 0) peak is negligibly small, indicating that the film mainly consists of the \( \gamma' \)-(Fe,Cr)\(_2\)N\(_x\) phase.

In order to further explain the change in the saturation magnetization described in Fig. 1, we measured Mössbauer spectra of these films at room temperature as a function of \( R(N_2) \). Fig. 3 shows that the Mössbauer spectra of the films consist of a ferromagnetic sextet and a paramagnetic doublet, corresponding to the \( \alpha \)-Fe(Cr) and the \( \gamma' \)-(Fe,Cr)\(_2\)N\(_x\) phases, respectively. As shown here, the peak area of the sextet becomes smaller, while that of the doublet becomes larger with increasing \( R(N_2) \). For the film made at \( R(N_2) = 40\% \), the sextet is almost undetectable. These changes in the Mössbauer spectra agree with the XRD results shown in Fig. 2. In addition, the peaks at about ±2.5 mm/s for the films deposited at \( R(N_2) = 30\% \) and 35\% disappear almost completely. This also indicates that the films have strong perpendicular magnetic anisotropy.
3.1.2. Effects of Cr contents

Fig. 4 shows (a) \( M_S \), \( H_{c\parallel} \), and \( H_{c\perp} \) and (b) the Cr content, \( C_{Cr} \), of the films deposited at \( T_S = 50^\circ C \) and \( R(N_2) = 30\% \) as a function of \( A_{Cr} \). The ICP measurement showed that the nitrogen contents in these films are about 11 at\%. \( M_S \) decreases rapidly with increasing \( A_{Cr} \) and becomes almost zero at \( A_{Cr} = 40\% \). On the other hand, \( H_{c\perp} \) first increases and then decreases with increasing \( A_{Cr} \). Note that, for the film deposited at \( A_{Cr} = 35\% \) with Cr content \( C_{Cr} = 25\% \), \( H_{c\perp} \) is much larger than \( H_{c\parallel} \), displaying perpendicular magnetic anisotropy.

The XRD patterns of the films are shown in Fig. 5. As can be seen here, the films deposited at \( A_{Cr} = 33 \) and 35\% exhibit broad \( \alpha\)-Fe(Cr)(1 1 0) peak and strong \( \gamma\prime\)-(Fe, Cr)\(_4\)N\(_x\)(2 0 0) peak, indicating that these films are composed of a fine grain \( \alpha\)-Fe(Cr) phase and the \( \gamma\prime\)-(Fe, Cr)\(_4\)N\(_x\) phase with a pronounced (2 0 0) texture. On the other hand, the film deposited at \( A_{Cr} = 40\% \) which has the \( M_S \) value of about zero (Fig. 4) is almost entirely composed of the single \( \gamma\prime\)-(Fe, Cr)\(_4\)N\(_x\) phase. Therefore, we can consider that the \( \gamma\prime\)-(Fe, Cr)\(_4\)N\(_x\) phase is paramagnetic. This result is also in agreement with the results of the Mössbauer spectra.

3.1.3. Effects of substrate temperature

Fig. 6 shows the \( T_S \) dependence of \( M_S \), \( H_{c\parallel} \), and \( H_{c\perp} \) of the films deposited at \( R(N_2) = 35\% \) and \( A_{Cr} = 33\% \). The magnetic properties of the films change drastically around \( T_S = 250^\circ C \). \( H_{c\perp} \) and \( H_{c\parallel} \) increase and \( M_S \) decreases with increasing \( T_S \) up to \( T_S \approx 250^\circ C \). At \( T_S = 250^\circ C \), \( H_{c\perp} \) is about three times as large as \( H_{c\parallel} \). However, above \( T_S \approx 250^\circ C \), \( H_{c\parallel} \) decreases and \( M_S \) increases rapidly.

Fig. 7 shows XRD patterns of the films deposited at different \( T_S \). The films deposited below \( T_S = 250^\circ C \) show weak and broad \( \alpha\)-Fe(Cr)(1 1 0) peaks combined with strong \( \gamma\prime\)-(Fe, Cr)\(_4\)N\(_x\)(2 0 0) peaks, indicating that the grain size of the \( \alpha\)-Fe(Cr) phase is small. At \( T_S = 350^\circ C \), however, the (1 1 0) peak of \( \alpha\)-Fe(Cr) phase becomes larger and sharper,
while the (2 0 0) peak of the $\gamma$-(Fe,Cr)$_4$N$_x$ phase becomes smaller.

These results indicate that the amount and grain size of the $\alpha$-Fe(Cr) phase increase with substrate temperatures above 250°C.

3.1.4. Effect of nitrogen flow ratio at $T_s = 250^\circ$C

Thus, the substrate temperature of 250°C seems to be optimum for depositing films with large perpendicular anisotropy. We therefore further examined the effect of N$_2$ flow ratio on the magnetic properties of the films deposited at $T_s = 250^\circ$C. Fig. 8 shows the $R$(N$_2$) dependence of $M_s$, $H_{c\perp}$ and $H_{c\parallel}$ for the Fe–Cr–N films prepared at $T_s = 250^\circ$C and $A_{Cr} = 33\%$. With increasing $R$(N$_2$) from 20 to 35%, $M_s$ decreases and $H_c$ increases. These features can be ascribed to the increase in the amount of the nonmagnetic $\gamma$-(Fe,Cr)$_4$N$_x$ phase with increasing $R$(N$_2$). Moreover, $M_s$ and $H_{c\perp}$ change drastically around $R$(N$_2$) = 25%, while the change in $H_{c\parallel}$ is only gradual, resulting in the strong perpendicular anisotropy above $R$(N$_2$) = 25%.

As shown in Fig. 9, corresponding to the change in $M_s$ and $H_c$ with $R$(N$_2$), the XRD patterns for the samples with $R$(N$_2$) < 25% clearly show the (2 0 0) peak of the $\alpha$-Fe(Cr) phase and only a moderate (2 0 0) texture of the nonmagnetic $\gamma$-(Fe,Cr)$_4$N$_x$ phase. In addition, a diffraction peak at around

Fig. 4. (a) $M_s$, $H_{c\parallel}$ and $H_{c\perp}$ and (b) the Cr content of the Fe–Cr–N films deposited at $T_s = 50^\circ$C and $R$(N$_2$) = 30% as a function of $A_{Cr}$.

\[ 372x311 \]

\[ 40x279 \]
$\theta = 61^\circ$, which is not that of the fine-crystalline $\alpha$-Fe(Cr) and the $\gamma'$-(Fe,Cr)$_4$N$_x$ phases, indicates that the grain texture is inhomogeneous. However, when $R(N_2) > 25\%$, the XRD patterns show the strong $(200)$ texture of the $\gamma'$-(Fe,Cr)$_4$N$_x$ phase and almost no $\alpha$-Fe(Cr)(200) peak. These results suggest that the appearance of the large perpendicular magnetic anisotropy in the Fe–Cr–N films is

Fig. 5. X-ray diffraction patterns of the Fe–Cr–N films deposited at $T_s = 50^\circ$C and $R(N_2) = 30\%$ at different $A_{Cr}$.

Fig. 6. $M_s$, $H_{c//}$ and $H_{c\perp}$ of the Fe–Cr–N films deposited at $R(N_2) = 35\%$ and $A_{Cr} = 33\%$ as a function of $T_s$.

Fig. 7. X-ray diffraction patterns of the Fe–Cr–N films deposited at $R(N_2) = 35\%$ and $A_{Cr} = 33\%$ as a function of $T_s$. 
Fig. 8. $M_s$, $H_{c//}$ and $H_{c\perp}$ of the Fe–Cr–N films deposited at $A_{Cr} = 33\%$ and $T_s = 250^\circ C$ as a function of $R(N_2)$.

closely related to the amount of the (2 0 0) textured $\gamma'-(Fe,Cr)_{4}N_2$ phase.

Fig. 10 shows the magnetization curves of the Fe–Cr–N films deposited at (a) $T_s = 50\degree C$ and (b) $T_s = 250^\circ C$ as a function of $R(N_2) = 35\%$ and $A_{Cr} = 33\%$ at (a) $T_s = 50^\circ C$ and (b) $T_s = 250^\circ C$.
(b) 250°C with \(R(N_2) = 35\%\) and \(A_{Cr} = 33\%\). For the Fe–Cr–N film deposited at \(T_S = 50°C\) (Fig. 10a), \(H_{c\perp}\) is twice as large as \(H_{c\parallel}\) but the field dependence of residual magnetization in the perpendicular direction \(M_{r\perp}\) is the same as that in the in-plane \(M_{r\parallel}\). In Fig. 10b, however, the magnetization curve more easily saturates in the perpendicular direction than in the in-plane direction and \(H_{c\perp}\) is much larger than \(H_{c\parallel}\) for the Fe–Cr–N film deposited at \(T_S = 250°C\).

3.2. Structural observation by electron microscopy

In order to examine the origin of perpendicular magnetic anisotropy, we observed microstructures of the Fe–Cr–N films with large perpendicular magnetic anisotropy using SEM and TEM. Fig. 11 shows a fractured cross-sectional SEM image of the Fe\(_{70}\)Cr\(_{30}\)N\(_9\) film deposited at \(T_S = 250°C\), \(A_{Cr} = 33\%\) and \(R(N_2) = 25\%\). This picture clearly shows the columnar structure of the film.

To determine the distribution of the \(\gamma’\)-(Fe,Cr)\(_4\)N\(_x\) and \(\alpha\text{-Fe(Cr)}\) phases, we carried out a TEM study. Fig. 12 show plan-view and cross-sectional images of the film. The bright field images in Fig. 12a and d show that the film is composed mainly of the columnar grains of 20–40 nm in diameter. Fig. 12b and e show dark field (DF) images of the same area obtained with the objective aperture set at the \(\{2 0 0\}\) position of the \(\gamma’\)-(Fe,Cr)\(_4\)N\(_x\) phase. These pictures demonstrate that the columnar grains of about 20–40 nm in diameter are composed of the \(\gamma’\)-(Fe,Cr)\(_4\)N\(_x\) phase, even though their orientations are not fixed throughout a column. Fig. 12c and f show DF images of the same area with the aperture set at the \(\{1 1 0\}\) position of the \(\alpha\text{-Fe(Cr)}\) phase. These pictures show that the ferromagnetic \(\alpha\text{-Fe(Cr)}\) grains have a pin-like shape with a size distribution of about 2–20 nm in diameter, and are distributed or ‘segregated’ at the grain boundaries of the columnar \(\gamma’\)-(Fe,Cr)\(_4\)N\(_x\) phase. Therefore, the shape anisotropy seems to play an important role in the perpendicular anisotropy of the Fe–Cr–N films.

Fig. 13 is diffraction patterns of the same film taken from (a) plan-view and (b) cross-sectional sample. The spots in the latter arises from the Si substrate. All the diffraction rings can be identified with either the \(\gamma’\)-(Fe,Cr)\(_4\)N\(_x\) or the \(\alpha\text{-Fe(Cr)}\) phase. For example, the rings 1, 3 and 5 are \(\{1 1 1\}\), \(\{2 0 0\}\) and \(\{2 2 0\}\) of the \(\gamma’\)-(Fe,Cr)\(_4\)N\(_x\) phase, respectively, while the rings 2 and 4 are \(\{1 1 0\}\) and \(\{2 0 0\}\) of the \(\alpha\text{-Fe(Cr)}\) phase, respectively. The cross-sectional diffraction pattern clearly show that the \(\gamma’\)-(Fe,Cr)\(_4\)N\(_x\) and \(\alpha\text{-Fe(Cr)}\) phases possess pronounced \(\{2 0 0\}\) and \(\{1 1 0\}\) texture, in agreement with the XRD results.

4. Discussion

According to the results described in the previous sections, the quality of the perpendicular magnetic anisotropy of the Fe–Cr–N films strongly depends on the deposition parameters \(R(N_2)\).
The increase of $R(N_2)$, $A_{Cr}$ and $T_S$ (,< 250°C) all causes the increase in the amount of the $\gamma'-(Fe,Cr)_4N_x$ phase and a decrease in the size of the $\alpha$-Fe(Cr) pin-like crystalline grains, and thus also results in the decrease in $M_S$. On the other hand, the change in $H_c$ is complicated because it involves the uninterrupted decreasing of the ferromagnetic $\alpha$-Fe(Cr) grain size with increasing the amount of the $\gamma'-(Fe,Cr)_4N_x$ phase. As described in Fig. 1a, $H_c$ first increases and then decreases, and a maximum in $H_c$ appears around $R(N_2) = 35\%$. The XRD patterns (Fig. 2) show a tendency of the decrease in the size of the $\alpha$-Fe(Cr) crystal grains as $R(N_2)$ increases. The cross-sectional TEM images also show that the size of most of the $\alpha$-Fe(Cr) grains along the in-plane direction is 2–10 nm in the Fe–Cr–N film at $R(N_2) = 25\%$. Therefore, it can be said that very small $\alpha$-Fe(Cr) grains formed in the film deposited at $R(N_2) = 40\%$ have shape anisotropy and show superparamagnetism. Fig. 14 shows the magnetization curves at room temperature and 4.2 K for the films deposited at $T_S \approx 50^\circ C$ and at (a) $R(N_2) = 35\%$ and (b) 40\%, respectively. At room temperature, the coercivity ($H_{c\perp} = 580$ Oe) of the film deposited at $R(N_2) = 35\%$ is larger than that ($H_{c\perp} = 345$ Oe) of the film deposited at $R(N_2) = 40\%$, while the results obtained at 4.2 K are on the contrary ($H_{c\perp} = 700$ Oe for $R(N_2) = 35\%$; $H_{c\perp} = 750$ Oe for $R(N_2) = 40\%$). Moreover, the magnetization at 4.2 K under applied field of 10 kOe is about twice of that at room temperature for the film deposited at $R(N_2) = 40\%$, while the ratio of the magnetization at 4.2 K to that at 300 K is small for the film deposited at $R(N_2) = 35\%$. These results suggest that the fine-crystalline $\alpha$-Fe(Cr) in the Fe–Cr–N film deposited...
Fig. 13. Electron diffraction pattern of the film shown in Fig. 12. (a) Plan-view specimen and (b) cross-sectional specimen. The spots in the latter arise from the Si substrate. The diffraction rings are: 1, 3 and 5 for [1 1 1], [2 0 0] and [2 2 0] of the $\gamma'$(Fe,Cr)$_4$N$_x$ phase, respectively, and 2 and 4 for [1 1 0] and [2 0 0] of the $\alpha$-Fe(Cr) phase, respectively.

at $R(N_2) = 40\%$ has a size distribution and very small $\alpha$-Fe(Cr) grains display the superparamagnetism. The $\alpha$-Fe(Cr) crystallite size decreases and the amount of the nonmagnetic $\gamma'$(Fe,Cr)$_4$N$_x$ phase increases with increasing $R(N_2)$, accompanied by the decrease of the saturation magnetization. Therefore, we can suggest that the Fe–Cr–N films with large perpendicular anisotropy and large

$M_S$ will be obtained if the size of the $\alpha$-Fe(Cr) crystallites is above the critical value at which superparamagnetic particles become ferromagnetic.

Finally, it is worth mentioning the effect of film thickness on the magnetic properties of the Fe–Cr–N films. Fig. 15 shows $M_S$, $H_c\perp$ and $H_c\parallel$ of the Fe–Cr–N films deposited at $T_S = 250^\circ$C, $A_{Cr} = 32\%$ and $R(N_2) = 30\%$ as a function of film thickness. The film thickness is estimated from the deposition rate and deposition time. This figure indicates that the strong perpendicular magnetic anisotropy appears when films are thicker than about 0.5 $\mu$m. The in-plane and perpendicular coercivities have a same trend with increasing the thickness: $H_c$ gradually increases up to 7 $\mu$m and then becomes constant above 7 $\mu$m.
The crystallized $\gamma'-(Fe,Cr)_4N_x$ phase is essential for induction of the perpendicular anisotropy in the Fe–Cr–N ternary system. This phase is nonmagnetic, cutting off the exchange interaction between the ferromagnetic $\alpha$-Fe(Cr) phases. As can be seen in Fig. 14, the perpendicular magnetic anisotropy is still retained at 4.2 K. Therefore, it can be considered that the $\gamma'-(Fe,Cr)_4N_x$ phase is still nonmagnetic in Fe–Cr–N films deposited under this experimental conditions.

5. Conclusion

Ternary Fe–Cr–N films with large perpendicular magnetic anisotropy have been obtained by a reactive sputtering (Ar + N$_2$ mixture) using a facing target sputtering system. The Fe–Cr–N films consist of a mixture of the $\alpha$-Fe(Cr) and $\gamma'-(Fe,Cr)_4N_x$ ($x < 1$) phases, whose volume fraction depends on the deposition parameters $R(N_2)$, $A_{Cr}$, and $T_s$. In particular, the Fe–Cr–N films with large perpendicular anisotropy are composed of the nonmagnetic $\gamma'-(Fe,Cr)_4N_x$ phase which has a strong (2 0 0) texture and the ferromagnetic $\alpha$-Fe(Cr) phase which has the pin-like shape grains with a size distribution of about 2-20 nm in diameter located at the grains boundaries of the $\gamma'-(Fe,Cr)_4N_x$ phase. The origin of the perpendicular anisotropy is ascribed to the shape anisotropy of the $\alpha$-Fe(Cr) fine grains.

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