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SOFT MAGNETIC PROPERTIES OF FeTaNC NANOCRYSTALLINE FILMS

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ABSTRACT

Soft magnetic properties and microstructural evolution of FeTaNC films were investigated, and compared with FeTaN and FeTaC films. Effects of substrate species (glass vs. $CaTiO_3$) on the magnetic properties were also investigated. Co-addition of N and C significantly enhance the grain refinments and magnetism, compared with N or C addition only. Good soft magnetic characteristics of coercivity of 0.17 Oe, permeability of 4000 (5MHz), and saturation flux density of 17 kG can be obtained in the FeTaNC in the relatively wide process windows. While these values appears to be similar to those of FeTaN on glass substrate, most distinctive difference between FeTaNC and FeTaN(or C) is in the effects of substrate. Whereas FeTaNC films show good magnetic characteristics for both glass and $CaTiO_3$ substrates, FeTaN(or C) films show significant degradation on the $CaTiO_3$ substrate.

INTRODUCTION

MIG type magnetic recording heads require the soft magnetic films with high magnetic flux density. Recently, Fe-based nanocrystalline films have been reported to have high saturation magnetization as well as good soft magnetic properties[1,2]. Various Fe-M-X(M =Mo, Ti, Zr, Hf, Ta; X=C, N) systems have been investigated for these applications[3,4]. So far, there has been little reports on the systematic comparison between Fe-M-N and Fe-M-C system. Generally, good soft magnetism can be obtained in both Fe-M-N and Fe-M-C, while Fe-M-C are known to show higher thermal stability and Fe-M-N films have superior wear and corrosion properties[5]. Therefore, in order to achieve the Fe-based films with high thermal stabilities as well as wear and corrosion resistance, Fe-M-N-C quaternary system is worthy to be investigated. In this study, FeTaNC system was selected and investigated, considering that both FeTaN and FeTaC are known to have good soft magnetism and that Ta forms strong compounds with N and C.

EXPERIMENTAL PROCEDURE

FeTa(C and/or N) films with 1㎛ thickness were deposited by DC magnetron reactive sputtering system. Ta composition in the film was varied by Ta chip area on the 4″ Fe target, and N or C contents were controlled by N_2(or CH_4)/Ar flow ratio. Initial vacuum was 1×10^{-6} Torr and total working pressure was fixed to 15 mTorr. DC power of 200 W was used and their deposition rates were 500 ~600 Å/min, which slightly depended on N_2 and/or CH_4 flow ratio.

Glass (Corning #7059) and $CaTiO_3$ were used as substrates which were water-cooled during deposition. Deposited samples were va-cuum-annealed (5×10^{-5} Torr) for 30 min at 500℃. The film composition and their depth distribution were analyzed by Auger electron spectroscopy after calibration with a Rutherford Back Scattering method. Film microstructures after heat treatments were investigated by X-ray diffraction (XRD) and transmission electron microscopy (TEM). Coercivity (H_C) and saturation magnetization (B_s) were measured using a B-H loop tracer and a vibrating sample magnetometer (VSM), and effective permeability (μ') was measured using the figure-8 coil method.

RESULT AND DISCUSSION

Films on glass substrake

Under the fixed working pressure (1.5 mTorr) and Ta chip area, Ta compositions in the films prepared in this work were 8.0~8.5 at.%, which slightly depended on reactive gas species and reactive gas species and reactive gas partial pressures[6].

Fig. 1 shows the measured H_c values as a function of reactive gas partial pressure(P_r) for various CH_4 to N_2 mixing ratios ($P_m=P_{CH4}/P_{CH4}+P_{N2}$). For $P_m=0\%$ (i.e., FeTaN), H_c value gradually decreases with increasing P_r and reaches the minimum value of 0.23 Oe at P_r of 15%. The FeTaC films woth $P_m=30$ and 50% show more rapid reduction of H_c value with P_r, and the minimum values (~0.17 Oe) are observed in the relatively wide P_r range of 5~10%. For $P_m=100\%$ (i.e., FeTaC), minimum H_c value of 0.28 Oe are observed at 15%, although relatively low values were observed in 5~15%. These results indicate thar co-addition of N and C is more effective in obtaining low H_c than N or C addition only. Also, process window for obtaining low H_c values appears to be wider.

In Fig. 2, B_s values are plotted against P_r for various P_m. For all P_m, B_s value slowly decreases with P_r, when P_r is lower than 15%. Since the P_r range showing the lowest H_c value in the FeTaNC is lower than that of either FeTaN or FeTaC, the B_s values of films with lowest H_c appears to be 16~17 kG for FeTaNC and 14~15 kG for FeTaN or FeTaC.

Permeability(μ') is also plotted against P_r in Fig. 3. Permeability displays clear relationship with H_c results in that a film with low H_c shows high μ'. While highest μ' values are observed at 15% of P_r in both FeTaN and FeTaC, the value of FeTaN(~4000) is higher than that of FeTaC(2700). Compositions of FeTaN and FeTaC films with highest μ' values are $Fe_{78.8}Ta_{8.5}N_{12.7}$ and $Fe_{75.6}Ta_{8.1}C_{16.3}$, respectively. In the case of FeTaNC, P_r range

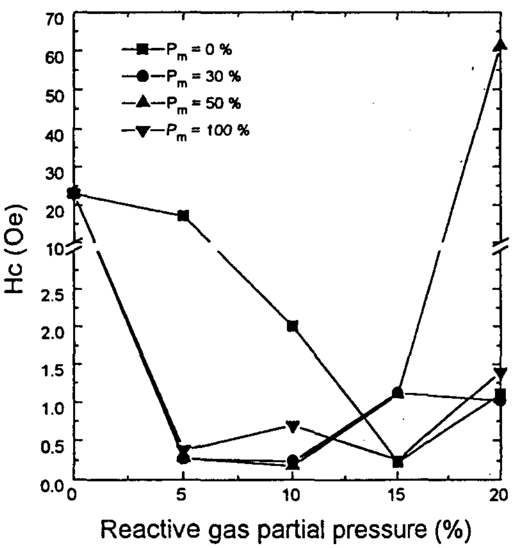

Fig. 1 Changes of coercivity as a function of reactive gas partial pressure for various P_m.

of the highest μ' is 5∼10% and their values are 3000∼4000.

For the P_r range showing the best soft magnetic properties of FeTaNC(P_r=5∼10%), changes of H_c are plotted as a function of CH_4 to N_2 mixing ratio, P_m, in Fig. 4. FeTaNC films show much lower H_c values than those of unmixed FeTaN or FeTaC films. Also, those low values can be obtained in the wide mixing range of 20∼80%. Similar trend can be seen in the permea-bility values plotted against P_r (Fig. 5). FeTaNC shows superior μ' characteristics to either FeTaN or FeTaC. The highest μ' values of approximately 4000 are observed at P_m=0.2 and P_m=0.5 for P_r=5% and P_r=10%, repe-ctively.

Microstructures of FeTaNC films were investigated by XRD analysis for various mixing ratios. Fig. 6 shows the XRD patterns for P_r=5%. The FeTaN film(P_m=0%) shows the sharp Fe_3N Peak and α-Fe peak with very low intensity. This result reveals that most of Fe atoms react with N to form Fe_3N. Takeshima et. al.[7] have reported that formation of FeN phases such as Fe_4N and Fe_3N degrade the soft magnetic properties of FeTaN,

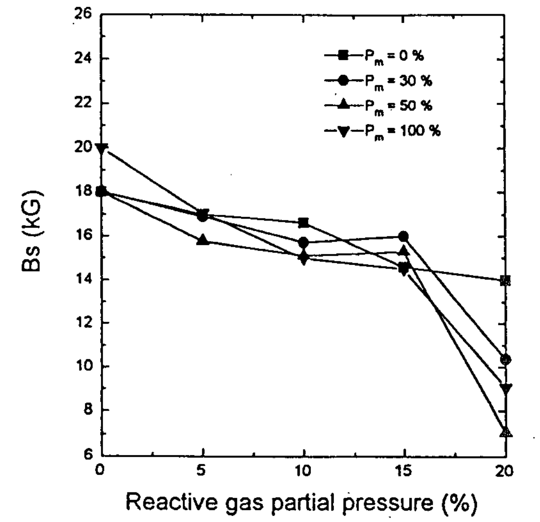

Fig. 2 Changes of saturation flux density plotted nst against reactive gas partial pressure for various P_m.

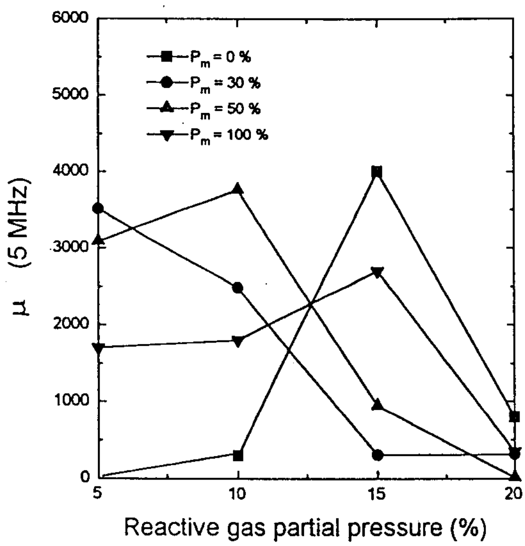

Fig. 3 Changes of effective permeability plotted against reactive gas partial pressure.

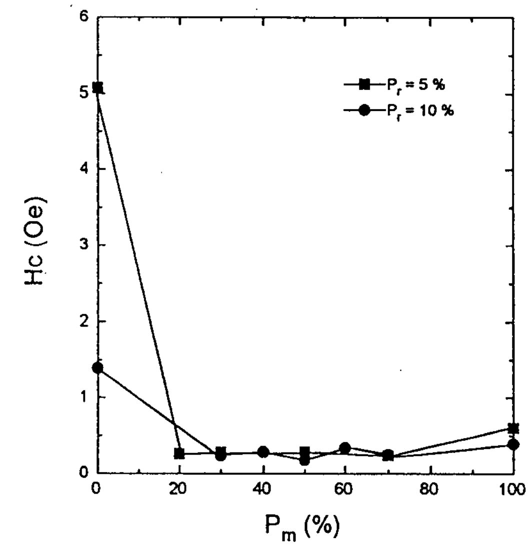

Fig. 4. Changes of coercivity plotted against mixing gas for various P_m. ratio for partial pressure.

which is in agreement of our result. Probably, insufficient N incorporation leads to Fe_3N phases during deposition or heat treatment. The Fe_3N phase was not detected in the FeTaN of P_r=15%. Similar behavior is ob served in the FeTaC film (P_m=100%).FeTaC film shows Fe_3C peak with an absence of α-Fe peak.

In the case of FeTaNC films, strong α-Fe (110) peaks and broadened TaN(or TaC) peaks are observed for all P_m. These XRD patterns have been are typically observed in the films with good soft magnetism[5]. When compared with FeTaN (or C) results, co-addition of N and C is considered to enhance the efficiency in realizing the microstructures with good soft magnetic characteristics. It is difficult to distinguish TaN and TaC peaks because of close lattice dimensions between two phases and broadened peak patterns. However, Ta is considered to preferentially react with C to form TaC phase from the results that after heat treatments at higher temperature (700℃), only TaC phases are observed.

Grain sizes, which were determined from FWHM (Full Width at Half Maximum) of XRD peaks, are shown in Fig. 7. For FeTaNC films, grain size was determined from of α-Fe (110) peaks. Since main peaks of FeTaN and FeTaC films were Fe_3N and Fe_3C, respectively, these peaks were used for grain size calculation.

While FeTaN and FeTaC films have large grains of 110Å and 80Å, respectively, FeTaNC films have finer grains with sizes of 40~50 Å. The fine grain size in the FeTaNC films can be explained by enhanced formation of TaN(or C) particles observed in these films. At this stage, it is not clear why co-addition

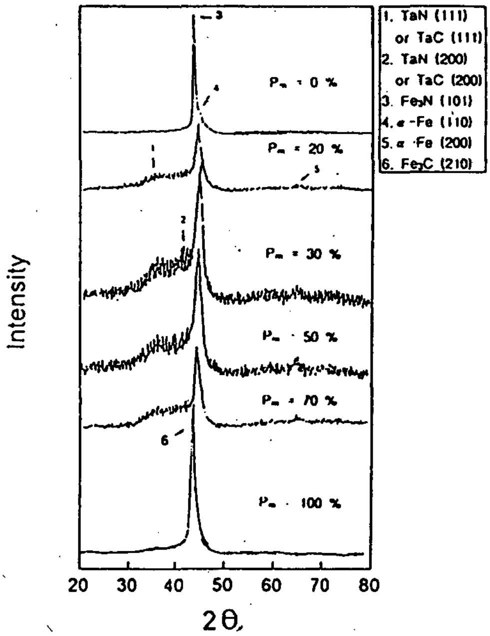

Fig. 6 XRD diffraction patterns for partial pressure of 5% after anneals at 500℃.

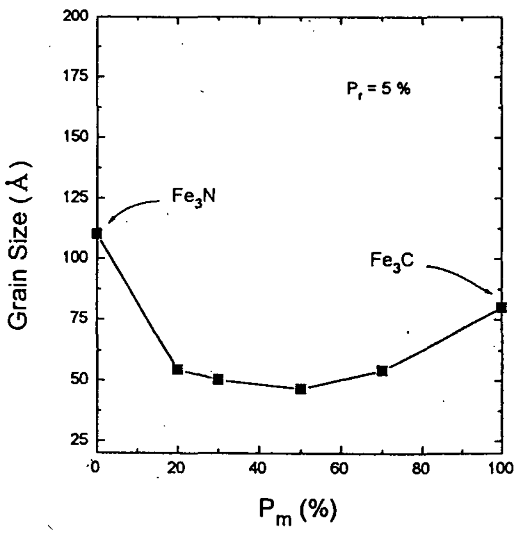

Fig.7 Changes of grain size as a function of mixing gas ratio.

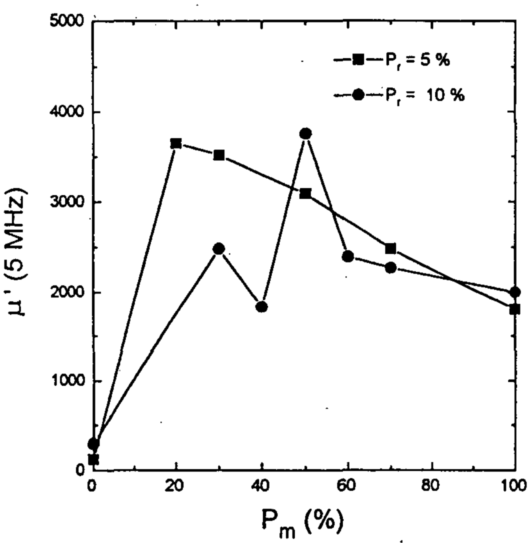

Fig. 5 Changes of effective permeability as a function of mixing gas ratio.

promote the TaN(or C) formation. Probably, co-addi-tion reduce the supers-aturation limit of N or C in α-Fe lattice, and allow more excess N and C atoms to react with Ta.

Fig. 8 shows the interplanar spacing of α-Fe (110) planes, $d_{(110)}$. The spacing is larger than the bulk Fe spacing of 2.207Å indicating the lattice expansion. The lattice expansion slightly decreases with increasing mixing ration in the FeTaNC films. Takahashi and Shimatsu[8] have reported that N incorporation in α-Fe lattice leads to lattice distortion from BCC to BCT structure with a lattice expansion, and that this lattice distortion causes reduction of magnetic anisotropic energy, thus, enhancing soft magnetic properties. At P_m of 20% showing best soft magnetic properties in the FeTaNC, lattice expansion coefficient ($\Delta d/d$) is 0.44%. This value can be compared with 0.88% for the $Fe_{78.8}Ta_{8.5}N_{12.7}$ showing best properties with similar values of H_c and μ'.

Films on $CaTiO_3$ substrate

In many reports, magnetic properties of Fe-based metal films have been evaluated on glass substrate. For the MIG head application, however, metal films are deposited on Ferrite or $CaTiO_3$ substrate. Substrate species

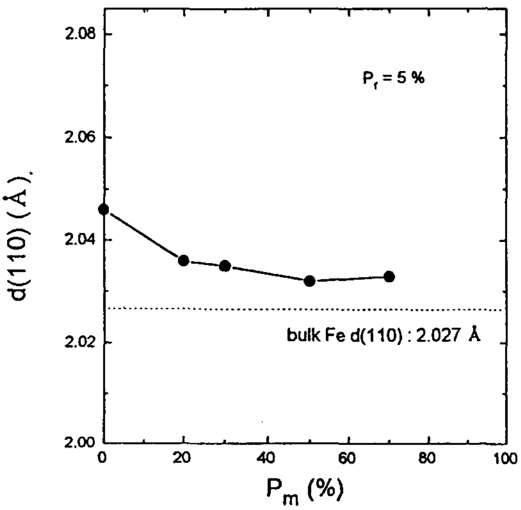

Fig.8 Changes of α-Fe(110) lattice parameter as a function of mixing gas ratio.

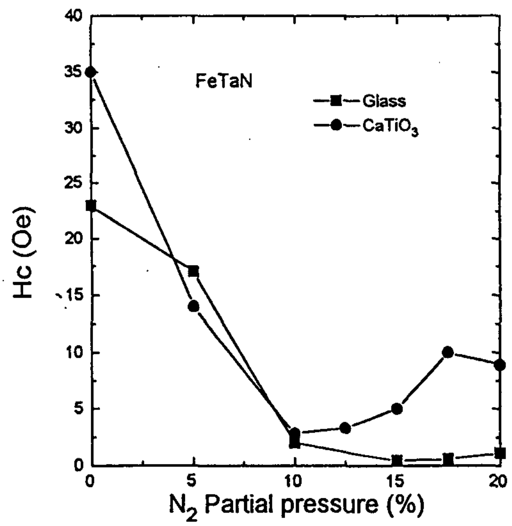

Fig. 9 Substrate effect on the coercivity characteristics in the FeTaN films for various N_2 partial pressure.

have been reported to have significant effects on the magnetic properties. Fig. 9 shows the substrate effects on the H_c characteristics in the FeTaN films for various N_2 partial pressure. As previously mentioned, FeTaN shows low H_c of 0.23 Oe at 15% partial pressure on glass substrate. On the other hand, substantially higher H_c values are observed on the $CaTiO_3$ substrate. From TEM studies, as shown in Fig. 10, we could not recognize any microstructural doifference between two substrates. Even though thermal stress developed to the films is speculated to be the cause of this difference, further investigation is needed to verify the substrate effects.

The effect of substrate of FeTaNC is distinctively different from those of FeTaN or FeTaC. In Fig. 11, highest μ' values are compared between glass and $CaTiO_3$ substrates. Whereas μ' is significantly degrad ed on the $CaTiO_3$ substrate in FeTaN and FeTaC, FeTaNC keeps high value in both substrates. This result indic-ates that the FeTaNC shows distinctive advantage over FeTaN or FeTaC in the minimization of substrate effects.

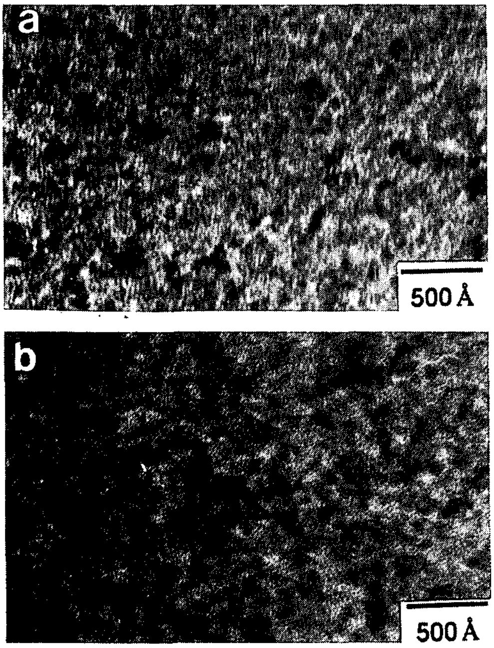

Fig. 10 TEM micrograohs in the FeTaN films for (a) glass and (b) $CaTiO_3$, substrates.

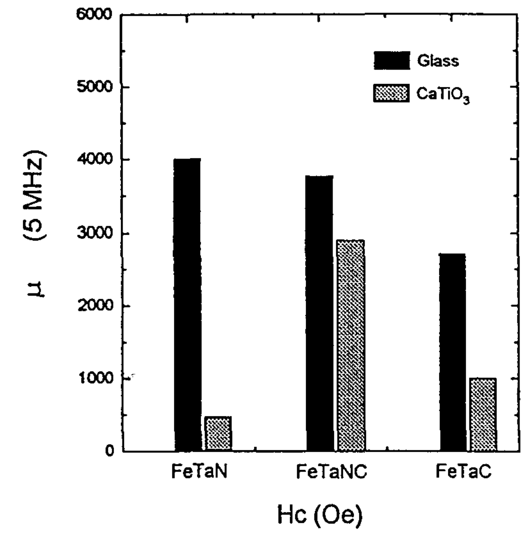

Fig. 11 Permeability values measured for glass and $CaTiO_3$ substrates.

CONCLUSION

FeTaNC quaternary films were investigated and compared with FeTaN and FeTaC films. Addition of N and C to FeTa metals significantly enhance efficiency of grain refinement and soft magnetic properties, compared to N addition (FeTaN) or C addition (FeTaC) only. Good soft magnetic properties of $H_c = 0.17$ Oe, $\mu' = 4000$, and $B_s = 17$ kG can be obtained in the wide process windows. FeTaNC films show good soft magnetic properties on both glass and $CaTiO_3$ substrates, whereas FeTaN and FeTaC films shows inferior properties on the $CaTiO_3$ substrate.

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