

# Effects of the process parameters on the microstructure and magnetic properties of nanocrystalline FeTaCN films

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## Abstract

FeTaCN films were prepared by DC-magnetron reactive co-sputtering of Fe target and TaC composite target with Ar + N<sub>2</sub> sputtering gas. Effects of annealing temperature and sputtering power density of the Fe target on the magnetic properties and microstructure of the FeTaCN film were investigated. Transmission electron microscopy analysis indicated that the FeTaCN film was nanocrystalline structure. The in-plane coercivity  $H_{c||}$  is about 1–3 Oe and saturation magnetization  $4\pi M_s$  is about 12–15 kG for the as-deposited film.

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We have reported that the as-deposited FeTaCN film with nanocrystalline structure and good soft magnetic properties (in-plane coercivity  $H_{c||} = 1–2$  Oe and  $4\pi M_s = 12–14$  kG) can be obtained by simultaneous addition of C and N to FeTa alloy film as well as controlling the N<sub>2</sub> flow rate ratio and film thickness [1]. In this work, the effect of the sputtering power density of the Fe target ( $SPD_{Fe}$ ) on the magnetic properties and microstructure of the FeTaCN thin film was investigated.

FeTaCN films were deposited on quartz substrates by DC-magnetron reactive co-sputtering of TaC composite target and Fe target at room temperature. The N<sub>2</sub> flow rate ratio (N<sub>2</sub>/Ar + N<sub>2</sub>) in the sputtering gas was fixed at 5%. The TaC composite target was made by Ta disk overlaid with C chips which covers 18% of the disk surface area. The sputtering power density of the TaC target was fixed at 1.97 W/cm<sup>2</sup>. The  $SPD_{Fe}$  was varied from 3.2 to 4.44 W/cm<sup>2</sup>. The film thickness was fixed at 200 nm. An SiN<sub>x</sub> cap layer of about 20 nm was

deposited on the FeTaCN film to prevent the oxidation of magnetic film. After deposition, the films were post-annealed in vacuum below  $1 \times 10^{-5}$  Torr for 30 min at temperature between 200°C and 500°C, then quenched in ice water. Composition of the film was analyzed by X-ray photoelectron spectroscopy (XPS). The microstructure and crystal structure of the film were investigated by transmission electron microscopy (TEM). Magnetic properties of the films were measured by a vibrating sample magnetometer (VSM) at room temperature.

Fig. 1(a) and (b) shows the TEM bright field images and the corresponding selected area diffraction (SAD) patterns of the annealed Fe<sub>72.06</sub>Ta<sub>6.35</sub>C<sub>6.92</sub>N<sub>14.63</sub> and Fe<sub>77.75</sub>Ta<sub>5.85</sub>C<sub>6.16</sub>N<sub>10.25</sub> films, respectively. The annealing temperature is 500°C. The  $SPD_{Fe}$  is 3.7 W/cm<sup>2</sup> for the Fe<sub>72.06</sub>Ta<sub>6.35</sub>C<sub>6.92</sub>N<sub>14.63</sub> film and 4.44 W/cm<sup>2</sup> for the Fe<sub>77.75</sub>Ta<sub>5.85</sub>C<sub>6.16</sub>N<sub>10.25</sub> film. We can see that both of the films have nanocrystalline structure, which consists of small  $\alpha$ -Fe grains and more smaller Ta(C, N) precipitates. The average grain size of  $\alpha$ -Fe is about 9 nm for the Fe<sub>72.06</sub>Ta<sub>6.35</sub>C<sub>6.92</sub>N<sub>14.63</sub> film, which is smaller than that of the Fe<sub>77.75</sub>Ta<sub>5.85</sub>C<sub>6.16</sub>N<sub>10.25</sub> film. The average grain size of  $\alpha$ -Fe is about 11 nm for the Fe<sub>77.75</sub>Ta<sub>5.85</sub>C<sub>6.16</sub>N<sub>10.25</sub> film. During annealing at 500°C,

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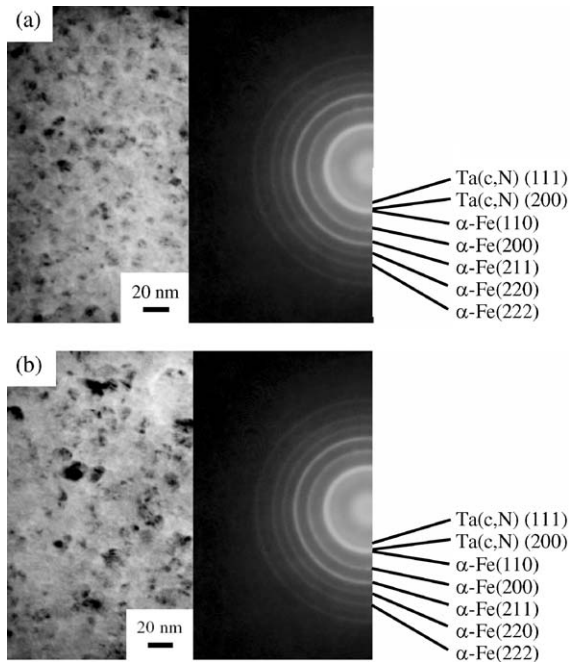


Fig. 1. TEM bright field image and electron diffraction pattern of the annealed (a) Fe<sub>72.06</sub>Ta<sub>6.35</sub>C<sub>6.92</sub>N<sub>14.63</sub> and (b) Fe<sub>77.75</sub>Ta<sub>5.85</sub>C<sub>6.16</sub>N<sub>10.25</sub> films.

the impeding of the  $\alpha$ -Fe grain growth by high melting point TaC(N) precipitates was decreased as the  $SPD_{Fe}$  was increased from 3.7 to 4.44 W/cm<sup>2</sup>. The decrease of the pinning effect of the TaC(N) precipitates results in the increase of  $\alpha$ -Fe grain size. Since the magnetocrystalline anisotropy is known to be averaged out by the refinement of grains, the coercivity of the film is decreased according to the random anisotropy model [2]. Therefore, the randomly oriented fine  $\alpha$ -Fe nanograins together with TaC or TaN precipitates in the film will result to the low coercivity.

Fig. 2(a) and (b) shows the variations of the saturation magnetization  $4\pi M_s$  and in-plane coercivity  $H_{c||}$  with annealing temperature, respectively. In which the FeTaCN films were deposited at different  $SPD_{Fe}$ . We can see that the  $4\pi M_s$  value of the films was increased with the  $SPD_{Fe}$  as shown in Fig. 2(a). This is due to the increase Fe content in the film or the increase of crystallinity of the film [3]. After annealing at 500°C, the  $4\pi M_s$  value is about 14 kG when  $SPD_{Fe}$  is 3.2 W/cm<sup>2</sup> (The film composition is Fe<sub>69.22</sub>Ta<sub>7.07</sub>C<sub>8.53</sub>N<sub>15.18</sub>) and it will increase to 16 kG as the  $SPD_{Fe}$  is increased to 4.44 W/cm<sup>2</sup> (The film composition is Fe<sub>77.75</sub>Ta<sub>5.85</sub>C<sub>6.16</sub>N<sub>10.25</sub>). From Fig. 2(b), we can see that the  $H_{c||}$  value could be decreased to 0.5–0.8 Oe if the film was annealed at 200–300°C. This is due to the stress relief resulting from the diffusing of C and N atoms out from  $\alpha$ -Fe grains that will reduce the

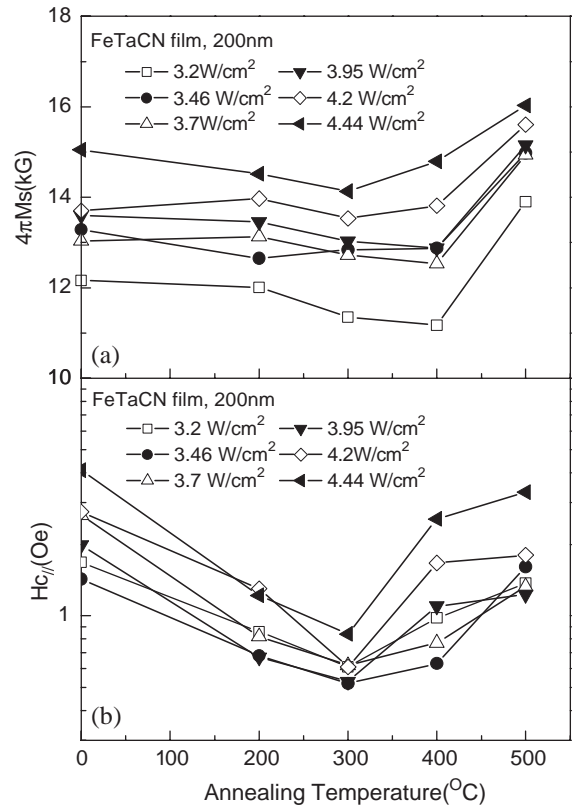


Fig. 2. Variations of (a) the saturation magnetization  $4\pi M_s$  and (b) the in-plane coercivity  $H_{c||}$  with annealing temperature for the FeTaCN films with different  $SPD_{Fe}$ .

Gibbs free energy [1]. After annealing at 500°C,  $H_{c||}$  value of the film was increased slightly. The increase of  $H_{c||}$  value was due to the large residual stress resulting from quenching the film in ice water and the grain growth at high temperature annealing. As the grain grows, the larger value of the magnetocrystalline anisotropy results in higher value of the  $H_{c||}$  [2].

In conclusion,  $4\pi M_s$  of the FeTaCN films is increased with the  $SPD_{Fe}$ . The randomly oriented fine  $\alpha$ -Fe nanograins together with TaC or TaN precipitates in the film will result to the good soft magnetic properties.

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