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Influence of Additions and Stratification on the Magnetic Properties and Out-of-Plane Texture of Nd-Fe-B Films

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Nd-Fe-B films with Ta and Mo additions used as buffer and capping layers and as interlayers have been deposited by r.f. sputtering on heated Si(111) substrates at 370 °C and 470 °C, and subsequently annealed at temperatures between 550 °C and 650 °C. In comparison with the Ta/NdFeB/Ta film, the Mo/NdFeB/Mo film deposited at 470 °C presents an obvious anisotropic character. Very good anisotropic magnetic properties were obtained for Mo/[NdFeB(180)/Mo(5)]×3/Mo multilayer film deposited at 470 °C and then annealed at 550 °C. This sample exhibits an increase in coercivity from 1225 kA/m up to 1651.8 kA/m, increase in M_r/M_s ratio from 0.90 up to 0.99, and increase in the maximum energy product from 60.4 to 121.6 kJ/m^3 as compared to the as-deposited Mo/NdFeB/Mo film. The results demonstrate that after annealing at 550 °C the Mo film is more effective for nucleation of $Nd_2Fe_{14}B$ hard magnetic grains with c-axis perpendicular to the film plane, especially in multilayer variant.

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1. Introduction

The out-of-plane-oriented Nd-Fe-B films, for which high M_r/M_s ratio (M_r-remanent magnetization, M_s-saturation magnetization), high coercivity H_c and large maximum energy product $(BH)_{max}$ can be obtained, have attracted increasing attention due to their potential applications in magnetic micro-electro-mechanical systems (MEMS), magnetic sensors, and ultra-high-density magnetic data storage [1]. The perpendicular texture of the Nd-Fe-B films depends on the substrate temperature, the nature of the material used as buffer/capping layers and interlayers, as well on the annealing temperature. Previous studies have shown that out-of-plane texture of Nd-Fe-B films can be induced by crystallization in two-steps, during deposition onto heated substrates at medium temperatures, followed by annealing at high temperatures [2]. In order to obtain Nd-Fe-B anisotropic films, Ta, Mo, or Ti are used as buffer and capping layers [3, 4]. Ta film was also used as interlayer for multilayer films [3]. A reduced crystallization temperature for the $Nd_2Fe_{14}B$ grains is an important target. In this paper results on the influence of Ta and Mo films used as buffer, capping layers, and interlayers on the hard magnetic properties and out-of-plane texture of Nd-Fe-B films are presented. A comparison between Mo and Ta influence on the nucleation at reduced temperatures of the $Nd_2Fe_{14}B$ grains with out-of-plane orientation is presented.

2. Experimental

Three series of samples have been prepared for experiments: Ta(40)/NdFeB(540)/Ta(20) and Mo(40)/ NdFeB(540)/Mo(20) single layer films, and Mo(40)/[NdFeB (180)/Mo(t)]x3/Mo(20) multilayer films (the thickness of each layer is in nm). The samples have been deposited using r.f. sputtering technique on Si(111) substrates, heated to temperatures T_s of 370 °C and 470 °C. The as-deposited samples have been thermally treated for 20 minutes at temperatures ranging between 550 °C and 650 °C.

The crystallographic structure was investigated using X-ray diffraction (XRD) analysis.

The anisotropic hard magnetic characteristics were measured perpendicular to the film plane using a vibrating sample magnetometer (VSM) and a Physical Property Measurement System (PPMS) with a maximum magnetic field of 2460 kA/m and 3500 kA/m, respectively. The demagnetization factor is not taken into consideration in the magnetic measurements.

3. Results and discussion

The hard magnetic properties and crystallographic structure of anisotropic Nd-Fe-B films are very sensitive to composition and substrate temperature. Figure 1 shows the hysteresis loops for Nd-Fe-B thin films with Ta and Mo films as buffer and capping layers, deposited on substrates heated to 370 °C and 450 ° C. For Ta/NdFeB/Ta thin film deposited at 370 °C the hysteresis loops in the as-deposited and annealed states indicate an isotropic behavior. The 470 °C substrate temperature is favourable for the formation of the out-of-plane texture in as-deposited state, especially for Mo/NdFeB/Mo film. However, this out-of-plane texture is weak. After annealing at temperatures ≥550 °C the anisotropic magnetic characteristics of Ta/NdFeB/Ta film deposited at 470 °C are slightly improved, while a decrease in the anisotropic magnetic characteristics of Mo/NdFeB/Mo thin films was observed.

In order to enhance the out-of-plane orientation of $Nd_2Fe_{14}B$ grains, the Nd-Fe-B magnetic layer was stratified in three NdFeB(180)/Mo(t) bilayers.

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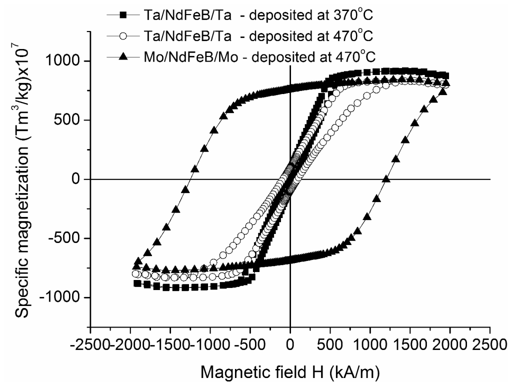

Fig. 1. Hysteresis loops of Nd-Fe-B with Ta and Mo as buffer and capping layers, deposited at 370 °C and 470 °C.

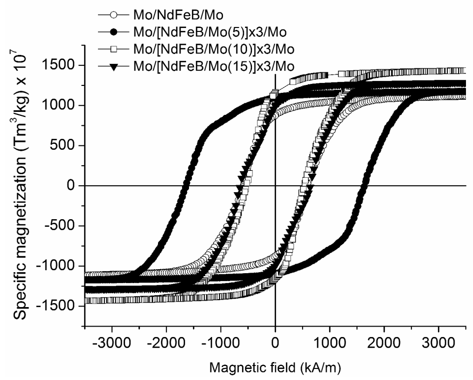

Fig. 2. Hysteresis loops of Mo/NdFeB/Mo and Mo/[NdFeB(180)/ Mo(t)]×3/Mo films, deposited at 470 °C and annealed at 550 °C.

Very good anisotropic magnetic properties were obtained for Mo/[NdFeB(180)/Mo(5)]×3/Mo multilayer film annealed at 550 °C, i.e. the H_c increases from 1225 kA/m up to 1651.8 kA/m, the M_r/M_s ratio from 0.90 up to 0.99, and the $(BH)_{max.}$ from 60.4 up to 121.8 kJ/m^3, in comparison with as-deposited Mo/NdFeB/Mo film (Fig. 2). This increase is due to the enhancement of the perpendicular anisotropy.

The Ta/NdFeB/Ta films deposited at 370 °C are amorphous. Figure 3 (a, b, c, and d) shows the XRD patterns of Ta/NdFeB/Ta (a), and Mo/NdFeB/Mo (b) films deposited at 470 °C, and Mo/[NdFeB(180)/Mo(5)]×3/Mo multilayer film deposited at 470 °C (c) and annealed at 550 °C (d). Ta and Mo films grow during deposition having (110) orientation. The XRD patterns for as-deposited samples reveal the formation of out-of-plane oriented $Nd_2Fe_{14}B$ phases evidenced by (004), (006), (008), (105), and (214) peaks. For samples with Mo addition these phases coexist with a residual amorphous phase which vanishes after annealing at 550 °C, while the degree of orientation of the c-axis improves, especially for Mo/[NdFeB(180)/Mo(5)]×3/Mo film. After annealing at temperatures ≥550 °C, some XRD peaks specific for Mo oxides also were evidentiated, and this is a possible reason for the decrease of the anisotropic characteristics. The oxidation effect is reduced for the multilayer sample due to protection of Mo interlayers.

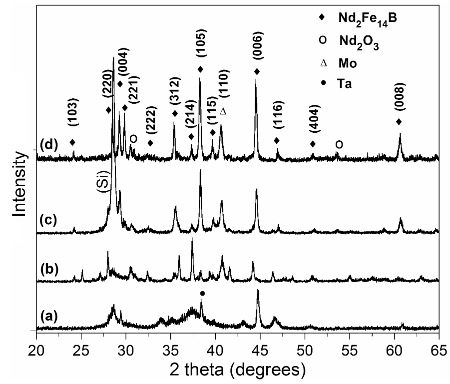

Fig. 3. XRD patterns of Ta/NdFeB/Ta (a), and Mo/NdFeB/Mo (b) films deposited at 470 °C, and Mo/[NdFeB(180)/Mo(5)]×3/Mo film deposited at 470 °C (c) and annealed at 550 °C (d).

In order to further reduce the oxidation effect, in our future investigations, Ta films will be used as buffer and capping layers for Mo/NdFeB/Mo film both in single layer and multilayer variants. Thus, these systems will benefit of the advantages offered by the presence of Mo and Ta.

4. Conclusions

Investigations on the influence of Mo and Ta additions and stratification of magnetic layers on the magnetic properties and out-of-plane texture of Nd-Fe-B films have demonstrated that, in comparison to Ta film, the Mo film is more effective for the nucleation at reduced temperatures (550 °C instead of 650 °C) of $Nd_2Fe_{14}B$ grains with c-axis perpendicular to the film plane. This tendency is stimulated by the multilayered configuration. Thus, very good anisotropic hard magnetic properties were obtained for Mo/[NdFeB(180)/Mo(5)]×3/Mo multilayer films annealed at 550 °C in comparison with Mo/NdFeB/Mo films.

Acknowledgments

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References

[1] V. Neu, S. Melcher, U. Hannemann, S. Fähler, L. Schultz, *Phys. Rev. B* **70**, 144418 (2004).

[2] N.M. Dempsey, A. Walther, F. May, D. Givord, K. Khlopkov, O. Gutfleisch, *Appl. Phys. Lett.* **90**, 092509 (2007).

[3] M. Uehara, *J. Magn. Magn. Mater.* **284**, 281 (2004).

[4] L.K.E.B. Serrona, A. Sugimura, N. Adachi, T. Okuda, H. Ohsato, I. Sakamoto, A. Nakanishi, M. Motokawa, D.H. Ping, K. Hono, *Appl. Phys. Lett. 82* **82**, 1751 (2003).