Contents lists available at ScienceDirect



Journal of Magnetism and Magnetic Materials



journal homepage: www.elsevier.com/locate/jmmm

Degree of chemical reaction of carbon and oxygen to ferromagnetic grains for Co-based alloy-oxide granular structure

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ARTICLE INFO

Available online 6 August 2008

PACS: 75.50.Ss 75.70.Ak 81.15.Cd

Keywords: Co-oxide granular film Saturation magnetization Oxidation Co-carbide

1. Introduction

Granular-type films of Co-based alloy-oxide composites such as CoPtCr-SiO₂ [1] have been commercialized as perpendicular magnetic recording media. To realize media with well-isolated magnetic grain structure, many studies have reported optimized conditions for sputtering onto a rough surface underlayer of Ru, determined from the switching field and loop slope that appears in the magnetic hysteresis loop [2-4]. Sputtering conditions also affect the magnitude of saturation magnetization of granular films, due to the change in the film composition, especially when using metal-insulator targets. However, few reports have systematically discussed this phenomenon. With regard to the reduction of saturation magnetization, a cap layer material is also important when considering reaction with the magnetic grains [5] and oxidation of magnetic grains through the cap layer after fabrication. In this paper, we clarify the saturation magnetization of granular media through the quantitative analysis of film composition and phase formation using a simple system of Co-SiO₂ as the granular layer material.

2. Experimental procedure

Co-SiO₂ and CoPtCr-SiO₂ granular films were prepared by dc magnetron sputtering method at room temperature on glass substrates. Co-(8 mol%)SiO₂ and Co₇₄Pt₁₆Cr₁₀-(8 mol%)SiO₂ composite targets, fabricated by the hot press method, were used. The

ABSTRACT

The saturation magnetization factor, M_s , was investigated for a Co-based alloy-oxide granular film through the quantitative analysis of film composition and phase formation. Using a carbon cap layer deposited directly onto the Co-SiO₂ film, both oxidation and carbonization of cobalt caused a reduction in M_s . The composition of the granular film strongly affects M_s , because the Ar gas pressure and discharge power during Co-SiO₂ sputtering alters the Co and oxygen content in the film. In particular, for a film fabricated under a high Ar gas pressure such as 8.0 Pa, M_s was significantly decreased, due to the formation of the antiferromagnetic CoO phase.

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sputtering conditions for the granular layer were varied using target power (*W*) from 0.4 to 4.0 W/cm^2 in Ar gas pressure (*P*_{Ar}) from 0.6 to 8.0 Pa. The structure of the film stack was Ta (5 nm)/Pt (6 nm)/Ru (20 nm)/granular film (16 nm). On the top of the sample, carbon and/or Ru, and Ta films were used as a capping layer. The film thickness varied by the sputtering time, and the sputtering rate was determined by measuring the thickness of the standard sample with a scanning step height analyzer.

Chemical bonds were analyzed using X-ray photoelectron spectroscopy (XPS). Magnetic properties were measured using a vibrating sample magnetometer (VSM) and a super conducting quantum interference device (SQUID).

The total amount of each atomic element in the granular film was evaluated using the fundamental parameter method by X-ray fluorescence analysis (XRF). Evaluation of the amount of oxygen in the Co-SiO₂ film, required the adsorbed oxygen on the sample surface to be subtracted. Fig. 1 shows a typical determination of the amount of oxygen in a sample. Using the linear portion of the oxygen vs. Co-SiO₂ film thickness (d_{mag}) plot, the amount of adsorbed oxygen can be obtained from the intersection of the extrapolated line with the vertical axis, and the amount of oxygen in the Co-SiO₂ layer is determined from the gradient.

3. Results and discussion

3.1. Chemical reaction of the carbon cap layer

The effect of the carbon cap layer on M_s is first examined. Fig. 2 shows the dependence of M_s on the thickness of the carbon

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^{0304-8853/\$ -} see front matter \circledcirc 2008 Elsevier B.V. All rights reserved. doi:10.1016/j.jmmm.2008.08.013



Fig. 1. Change in the amount of oxygen measured by XRF against the thickness of the Co-SiO₂ film for Al/NiP sub./Co-SiO₂/Ta (10 nm)/carbon (7 nm) film.



Fig. 2. M_s dependence on the thickness of the cap layer (d_{CAP}) for Co-SiO₂ films (*W*: 2.0 W/cm²; P_{Ar} : 4.0 Pa). Cap layer materials are Ta (\odot), Ru (\blacktriangle), and C (\Box). Estimated M_s from the amount of Co in the film (938 emu/cm³) is shown by a dotted line.

cap layer for the Co-SiO₂ films. M_s for the films with Ru and Ta cap layers are also shown for reference. The estimated M_s for the Co-SiO₂ film, M_s^{XRF} (938 emu/cm³), is also shown. M_s^{XRF} is calculated from the amount of Co measured by XRF, assuming that all Co atoms form a ferromagnetic phase and have the same magnitude of saturation magnetization as that of the bulk specimen (1422 emu/cm³ [6]). For the film without a cap layer ($d_{CAP} = 0$ nm), the M_s is 750 emu/cm³. In the case of Ru and Ta caps, M_s for



Fig. 3. M_s dependence on the thickness of the carbon cap layer, d_c , for a Co-SiO₂ film with a carbon/Ru cap layer (•). M_s for the Co-SiO₂ film with only a carbon cap layer is shown by open squares. M_s^{XRF} (938 emu/cm³) is shown by a dotted line.

the film with $d_{CAP} \ge 3 \text{ nm}$ is equal to the estimated value of 938 emu/cm³. This indicates that Ru and Ta cap layers with $d_{CAP} \ge 3 \text{ nm}$ sufficiently protect against oxidation from the surface. For the carbon cap layer, M_s is reduced to 830 emu/cm³ from the estimated value, even with a thick d_{CAP} of 20 nm.

In order to separate another factor of M_s reduction from oxidation, a C/Ru stacked cap layer was investigated. Fig. 3 shows the dependence of M_s on the thickness of the carbon cap layer (d_c) for a Co-SiO₂ film with a C/Ru (10 nm) cap layer. M_s for a Co-SiO₂ film with only a carbon cap layer and M_s^{XRF} (938 emu/cm³) are also shown. Note that the 10 nm Ru cap layer sufficiently prevents oxidation. M_s is decreased from the estimated value from M_s^{XRF} and shows a constant value of 850 emu/cm³ with increasing d_c . This indicates that capping of the carbon layer reduces the M_s of the film by about 10%.

Fig. 4 shows XPS spectra for a Co-SiO₂/C film with various ionetching times, focusing on the binding energies around C 1s (289–279 eV), and Co 2p3/2 (784–774 eV). At the surface, only the peak from C 1s is observed at 284 eV, which corresponds to covalent bonding. As the etching proceeds, the peak shifts toward 283 eV together with the appearance of another peak at 779 eV from Co 2p3/2. According to the binding energies reported in Ref. [7], the peak shift toward lower energy indicates a change in bond character from covalent to metallic bonding. In the present study, metallic bonds are limited to the bond between carbon and Co; therefore, the peak at 283 eV suggests the formation of Co-carbide. This interpretation is also supported by other groups [5,8]. Therefore, for Co-SiO₂ with a carbon cap layer, the half of the 10% reduction in M_s is caused by Co-carbide, and the rest by oxidation.

3.2. Influence of phase formation under various sputtering conditions

The effect of the granular layer sputtering conditions on $M_{\rm s}$ was investigated using an oxidation- and reaction-free cap layer.

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