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Mossbauer and XPS analysis of Fe-SiO_2 and Fe-SiO_2/SiO_2 granular films

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Nanocomposite materials consisting of ultrafine magnetic particles embedded in a matrix which is metallic or insulating have recently attracted great attentions. We have been investigating magnetic and transport properties in Fe-SiO_2 granular films prepared on glass substrates by rf magnetron sputtering. The superparamagnetic nature and the tunneling giant magnetoresistance are observed in the films having Fe volume fractions smaller than 45% [1,2]. In this paper, we report the chemical structures as a function of film thickness for the single layered Fe-SiO_2 films with 44 vol.% Fe and of the thickness of SiO_2 layer for the Fe-SiO_2/SiO_2 multilayers.

Thicker films show the ferromagnetic hysteresis loops with high magnetization M_s. However, the magnetization decreases with becoming thinner as shown in Fig.1, and the hysteresis characteristics changes to the superparamagnetism. This behavior is confirmed by the conversion electron Mossbauer spectroscopy (CEMS); in thicker films the ferromagnetic sextet peaks appear, but in thinner films the paramagnetic singlet peak appears overlapping two small peaks (Fig.2a) arising from Fe-O or Fe-Si-O forming the shell of the nanogranule of Fe. In the multilayers of Fe-SiO_2 (10nm)/SiO_2 (x), the magnetization curve shows the superparamagnetic nature, and M_s decreases with increasing x. In CEMS spectrum, two peaks arising from Fe-O or Fe-Si-O increases also with x as shown in Fig.2, corresponding to the decrease of M_s.

The XPS spectra indicate also the existence of Fe-O or Fe-Si-O at 711 eV as indicated in Fig.3(a). This peak is very weak in the single layered films, but slightly larger in thinner films than thicker films. In thicker films, the peak intensity increases slightly with approaching the substrates. As shown in Fig.3, the peak of Fe-O or Fe-Si-O becomes remarkable with increasing x in the multilayers. The solid and broken lines indicate the spectra at the Fe-SiO_2 and SiO_2 layers. In (b), the peak of Si shifts to lower energy for the Fe-SiO_2 layer (solid line) comparing to the SiO_2 layer (broken line). Similar energy shift can be observed in the peak of O. These indicate that the oxidation or silicicacidation of Fe occurs by consuming the Si and O of SiO_2. The intensity ratio of Fe-O or Fe-Si-O and Fe is plotted with the peak intensity of Fe in Fig.4 as a function for the multilayers. In the film of x = 3 nm, the composition modulation can be observed clearly, and the oxidization or silicicacidation occurs selectively at the layer boundaries.

These data indicate that oxygen and silicon are very reactive immediately after the deposition, and so the deposited Fe is partially oxidized or siliconized at near substrate or at surface of SiO_2 layer. Thus, M_s decreases with decreasing thickness or increasing SiO_2 layer thickness.

[1] S. Honda, T. Okada, M. Nawate, and M. Tokumoto, Phys. Rev. B 56 (1997) 14566.
[2] S. Honda, M. Nawate, T. Umemoto, S. Mitsudo, S. Mitani, H. Fujimori and M. Motokawa, IEEE Trans. Magn. 35 (1999) 2955.

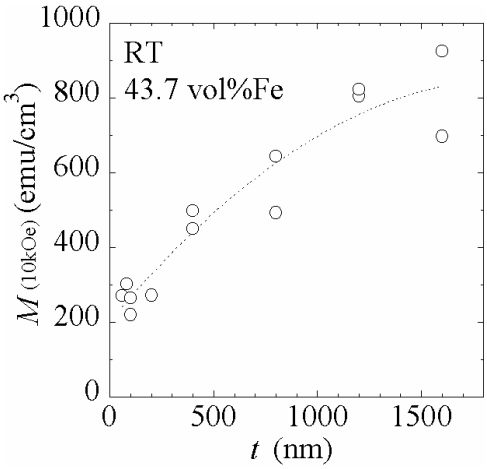

Fig.1 film thickness dependence of magnetization in the Fe-SiO_2 singlelayer films.

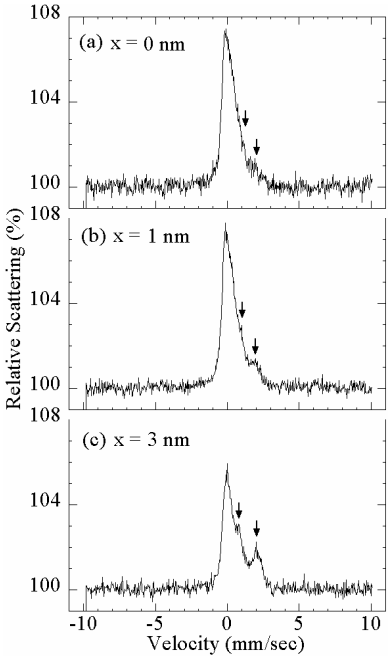

Fig.2 CEMS spectrum of the Fe-SiO_2 (10 nm)/ SiO_2 (x) multilayers.

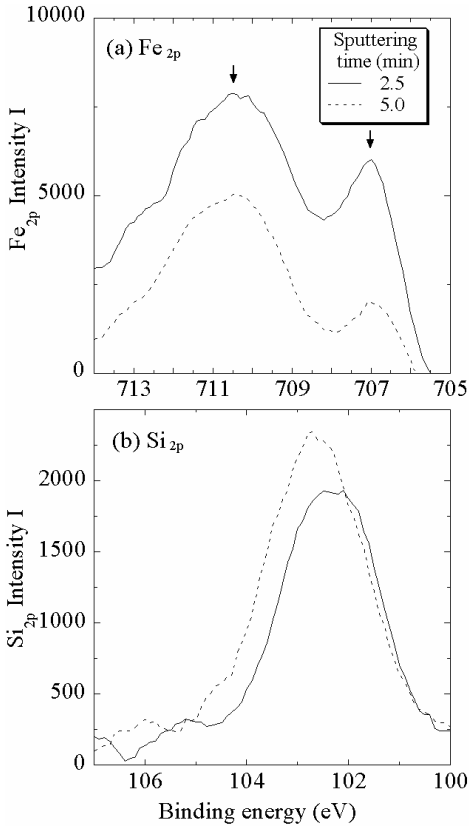

Fig.3 XPS spectrum of Fe_{2p}, Si_{2p} in the Fe-SiO_2 (10 nm)/ SiO_2 (3 nm) multilayer.

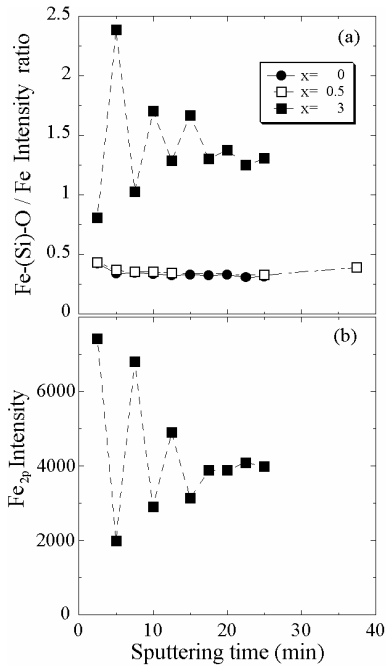

Fig.4 Depth profiles of (a) intensity ratio and (b) intensity for the Fe-SiO_2 (10 nm)/ SiO_2 (x) multilayers.

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