Structure and Magnetic Properties of Fe/Ag Multilayer Films Prepared by DC Sputtering

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Fe/Ag multilayer films were prepared by a double-facing-type dc sputtering system. The multilayer films were investigated by XRD, HRSEM, TEM, X-TEM and HRTEM. From the XRD profiles, we observed stacking of Fe\{110\} and Ag\{111\} planes and satellite peaks around the Ag 111 and 222 reflections clearly. X-TEM diffraction patterns showed the same preferred orientation as XRD results. HRSEM and HREM images showed columnar structure and well-controlled film period. The multilayer films were also characterized by VSM. These films had some magnetic anisotropies in the plane. Some M-H loops showed several steps for their easy axis in the film plane. These magnetic features were discussed on the basis of microstructures of the films.

1. Introduction

Until now, a great number of studies about Fe/Ag multilayer films have been published. However, it seems likely that their features concerning macroscopic magnetic properties and microstructures have not been established. In this study we investigated Fe/Ag multilayer films to characterize microstructures and the magnetic properties.

2. Experimental Procedure

Fe/Ag multilayer films were prepared by a double-facing-target-type dc sputtering system shown in Fig.1. We made these films on Si \{111\} wafers for X-ray diffraction study and for measuring the magnetic properties by VSM, and on evaporated SiO thin films for magnetic structure observation by Lorentz microscopy. In this system substrate temperature was less than 373K because of plasma free arrangement. A magnetic field of about 70 Oe was constantly applied parallel to the substrate surface by means of internal ALNICO magnets that maintain plasma. Base pressure was lower than $6 \times 10^{-7}$Pa and Ar gas pressure was set at 4.5$x10^{-3}$Pa. The deposit rates were 0.02nm/s for Fe and 0.2nm/s for Ag, which were measured by a quartz oscillator monitor and calibrated by Talystep profilometer. Total thicknesses of film were approximately 1000nm and modulation wavelength (film period) was from 3.5nm to 1000nm. The thickness ratio of Fe to Ag was 1:10. One of these specimens was measured under three conditions to compare with each other: as-prepared, after kept in a thermostatic bath at 373K for 24h and after kept in vacuum furnace at 473K for 2h. On the other hand, we made the specimens for the cross-sectional observation by TEM (200kV) and HREM (1000kV) were made by mechanical polishing followed by ion milling and chemical etching. And for monitoring of layer formation, one specimen, whose thickness ratio of Fe and Ag was 1:1, was cut together with Si substrate and observed by cross-sectional high resolution SEM without evaporation of metals to prevent the charging.

3. Results and Discussion

3.1 Structural Features

X-ray diffraction profiles showed that most of these films were composed of an alternating stacking of Fe \{110\} and Ag \{111\} planes. Satellite peaks were seen clearly around Ag 111 and 222 reflections, as partly shown in Fig.2. In connection with this, high resolution cross-sectional SEM observations revealed that the film was formed with many columnar crystals of more than 50nm in diameter and the film had a well-controlled layer period. The cross-sectional TEM observations also showed such columnar structures, in Fig.3, for an as-prepared film. The diffraction pattern of this specimen proved that the film mainly had the stacking of Fe \{110\} and Ag \{111\} planes. Detailed observations proved that a lot of big Ag columnars grew throughout the film. But along their growth direction, there are some Fe layers to separate the Ag layers clearly. In spite of existence of such Fe layers, Ag grain boundaries of columnars seem to continue over several layers. The results indicate that each Ag layer inherits its crystallographic relationship with previous Ag layer in the columnar. Therefore, this suggests that a reasonable crystallographic relationship between neighboring Ag and Fe layers is realized in each layer. On the other hand, we carried out similar
observations for a specimen heated in a furnace. Bright and dark field images showed that Ag layers were connected with the neighboring Ag layers with each other in some columns through Fe layers between them. And the diffraction pattern showed a 110 ring pattern for Fe. The result indicates that Fe grains in each layer already have no preferred crystallographic orientation perpendicular to the film plane unlike the as-prepared films.

3.2 Magnetic Properties

Generally, M-H loops observed for the films prepared by this sputtering system, showed some magnetic anisotropies in the film plane, as shown in Fig.4. Both Ms and Hc evaluated from measured M-H loops tend to decrease with decreasing Fe layer thickness (Fig.5). Especially, they show a sudden decrease for layers less than 2nm in thickness of Fe layer. When Fe layer thickness is thicker than 6nm, several steps are observed in each M-H loop. And the number of steps in the loop appears equal to the number of Fe layers in the Fe/Ag multilayer film. Such steps became clear after a heat treatment at 373K but were not observed clearly after a heat treatment at 473K (Fig.6). These phenomena may be related with microstructure change as stated above for the X-TEM observations. The features of magnetic microstructures in Fe films such as like domain walls, were investigated by Lorentz microscopy. The images showed that domain walls in pure thin Fe films easily move by tilting them against the electron beam or by a magnetic field caused by changing the lens mode. Fe thin films on Ag buffer, whose thickness ratio was equal to that of the specimen for VSM measuring, were also observed.

However, domain walls hardly moved. Such behavior of Fe layer on Ag layer and crystallographic features of Fe layer obtained from X-TEM observations may be related with the appearance of steps in the M-H loops.

4. References


Fig.1 Double-facing-target-type dc sputtering system.

Fig.2 X-ray diffraction profiles.
Fig. 3
Cross-sectional image and diffraction pattern of a Fe/Ag multilayer film by HVEM. (20/200)$_s$

Fig. 4
Typical M-H loops observed in the Fe/Ag multilayer films, prepared by this sputtering system shown in Fig. 1. (14.8/148)$_s$. The applied magnetic field to
a) the hard axis in the film plane.
b) the easy axis in the film plane.

Fig. 5
Ms(a) and Hc(b) as a function of Fe layer thickness.

Fig. 6
Observed M-H loops by VSM. They were measured parallel to the easy axis in the film plane. (20/200)$_s$

a) as-prepared.
b) after a heat treatment at 373K.
c) after a heat treatment at 473K.