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Layered structure analysis of GMR multilayers by X-ray reflectometry using the anomalous dispersion effect

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As a basic layered structure for giant magnetoresistive (GMR) heads, NiFe/Cu/NiFe/Ta/Si substrate was measured by X-ray reflectometry at Cu $K\alpha$, Cu $K\beta$ and Cu K-absorption-edge energies. The accuracy of both the Cu thickness and the interface width between the upper NiFe and the Cu layers was found to improve in the order Cu $K\alpha$ < Cu $K\beta$ < Cu K-edge. The final thickness and interface width values obtained from Cu $K\beta$ reflectivity are in good agreement with those from the Cu K-edge. The anomalous-dispersion effect is useful in the more accurate analysis of the layered structure of transition metal multilayers because it causes a large difference in the refractive indices of specific elements near the absorption edge. The $K\beta$ X-rays, which can be produced from conventional X-ray sources, are also available for the accurate analysis of reflectivity measurements.

Keywords: X-ray reflectivity; anomalous dispersion; layered structure analysis; giant magnetoresistivity.

1. Introduction

Giant magnetoresistive (GMR) spin valve heads have been investigated for high-recording-density rigid disk drives because of their high sensitivity in reading magnetic records (Dieny *et al.*, 1991). The layered structure of the heads consists of two ferromagnetic layers separated by a noble metal spacer of a few nm thickness. Their magnetic properties, such as the magnetoresistance and interlayer coupling between the two ferromagnetic layers, strongly depend on the thickness and the interfacial roughness of each layer. Therefore, a precise structural characterization of the GMR multilayers is important for producing good heads and for improving their magnetic properties.

The X-ray reflectivity technique is a powerful tool for investigating layer thickness, electron density and interface roughness. Huang *et al.* (1992) applied it to GMR multilayers using the Cu $K\alpha$ line from a conventional X-ray source. However, in transition metal multilayers, such as NiFe/Cu/NiFe GMR multilayers, the difference in the refractive index between NiFe and Cu at Cu $K\alpha$ energy is too small to analyse precisely the layered structure, because of the lower intensity of specular X-rays reflected from NiFe/Cu interfaces. The refractive index is a strong energy-dependent variable and rapidly changes near the absorption edge of the material. Using this anomalous-dispersion effect to enhance the X-rays reflected from the interfaces, Bai *et al.* (1996) measured the composition profile on an Fe/Cr superlattice from the reflectivities around the K-edge of Fe and Cr. Usami *et al.* (1997) reported reflectivity measurements of NiFe/Cu/NiFe multilayers using a Cu $K\beta$ line from a conventional X-ray source.

In this report, using a synchrotron radiation facility, the availability of reflectivity measurements using the dispersion effect was studied for the precise layered structure analysis of transition metal multilayers. Reflectivities of NiFe/Cu/NiFe multilayers were measured at Cu $K\alpha$, Cu $K\beta$ and Cu K-edge energies. The accuracy of the layered structure analysis for each X-ray energy was investigated.

2. Experimental

The Fresnel reflection coefficient $F_{i,j}$ between layers i and j is expressed as

$$F_{i,j} = (g_i - g_j)/(g_i + g_j) \simeq (\delta_j - \delta_i)/2\theta^2, \quad (1)$$

where $g = (n^2 - \cos^2\theta)^{1/2}$, $n = 1 - \delta - i\beta$ is the refractive index and θ is the grazing-incidence angle; the last approximation uses $\beta \simeq 0$ and $\theta^2 \gg 2\delta$. Because the smaller value of δ_i near the absorption edge of the i material increases the X-ray intensity from the interface between the layers i and j, one can analyse the layered structure of the multilayers with high accuracy. For example, in NiFe/Cu/NiFe multilayers, $(\delta_{\rm NiFe} - \delta_{\rm Cu})/\delta_{\rm Cu}$ can be calculated as 3, 13 and 30% at Cu $K\alpha$, Cu $K\beta$ and Cu K-edge energies, respectively.

The $Ni_{81}Fe_{19}(10)$/Cu(10)/$Ni_{81}Fe_{19}(10)$/Ta(10)/Si substrate sample was deposited by RF magnetron sputtering. The numbers in parentheses are the nominal thicknesses in nm calculated from the deposit condition. Reflectivities of the sample were measured at BL8C2 at the KEK Photon Factory, Japan. X-rays monochromated through an Si(111) double-crystal monochromator were used to undertake the measurements. The X-ray wavelengths were 0.13805 nm (Cu K-edge), 0.1392 nm (Cu $K\beta$) and 0.1540 nm (Cu $K\alpha$). The incident X-ray intensity was typically 10 Mcounts s^{-1} and the exposure time was 3 s per point. The reflectivity data were collected using the θ–2θ scanning technique and analysed by the least-squares method, which uses the reflectivity formula and includes interfacial effects due to roughness and/or interdiffusion (Parratt, 1954; Névot & Croce, 1980). The values of δ, thickness (t) and interface width (σ) for each layer were refined by minimizing χ^2,

$$\chi^2 = \sum_i \left(\log I^i_{\rm exp} - \log I^i_{\rm cal}\right)^2, \quad (2)$$

where $I_{\rm exp}$ and $I_{\rm cal}$ are the experimental and calculated reflectivity intensities, respectively. To evaluate the reliability of the least-squares refinement analysis, the reliability factor R was calculated from

$$R(\%) = \left[\chi^2 / \sum_i (\log I^i_{\rm exp})^2\right]^{1/2} \times 100. \quad (3)$$

A fitting model was used, containing an oxidized surface of NiFe and an interface layer between the Ta and Si substrates, because the fitting model containing the two layers drastically decreased the value of R.

3. Results and discussion

Fig. 1 shows the experimental and calculated reflectivities of the NiFe/Cu/NiFe/Ta/Si sample obtained with the Cu $K\alpha$, Cu $K\beta$ and

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Table 1
Refined δ, thickness (t) and interface width (σ) of NiFe/Cu/NiFe/Ta/Si multilayers.

Assumption: each δ of the upper and lower NiFe layer was the same.

	$\delta \times 10^{-6}$			t (nm)			σ (nm)		
	Cu $K\alpha$	Cu $K\beta$	Cu K-edge	Cu $K\alpha$	Cu $K\beta$	Cu K-edge	Cu $K\alpha$	Cu $K\beta$	Cu K-edge
Oxide	9.00	12.57	14.08	1.24	1.36	1.43	0.72	0.80	0.83
NiFe	23.67	20.16	19.91	11.03	10.70	10.57	0.98	0.81	0.76
Cu	24.42	17.78	15.38	9.85	10.21	10.15	0.70	0.76	0.77
NiFe	23.67	20.16	19.91	12.03	11.74	11.68	0.40	0.49	0.50
Ta	38.55	30.68	30.18	10.67	10.68	10.69	0.42	0.44	0.47
Interface layer	9.39	8.00	7.80	1.57	1.39	1.39	0.40	0.37	0.41

Cu K-edge X-rays. Each R factor was less than 1% and the refined reflectivity curves closely match the experimental data. The refined δ, t and σ values are listed in Table 1. The refined thickness measured at Cu $K\beta$ was close to that at the Cu K-edge, within 0.1 nm, whereas the thickness at Cu $K\alpha$ was different by about 0.3 nm. Similarly, the refined interface widths at Cu $K\beta$ and at the Cu K-edge were in good agreement with each other, but the interface width at Cu $K\alpha$ differed by up to 0.2 nm. Moreover, note that the interface width of the upper interface of Cu was larger by about 0.3 nm than that of the lower interface, whereas the interface width of NiFe did not change at the upper or lower interface. This is due to the larger size of the Cu grain due to crystal growth.

In order to investigate the accuracy of the layered structure analysis for each X-ray wavelength, the Cu thickness (t_{Cu}) was kept fixed to a value offset from the optimum ($t_{Cu,opt}$), other parameters were refined again and the fitting reliability was examined. Fig. 2(*a*) shows the χ^2 distribution, *i.e.* the difference of χ^2 from the minimum value (χ^2_{min}) *versus* the offset value of the Cu thickness from the optimum. A similar analysis for the interface width between the Cu and upper NiFe layer (σ_{Cu}) and δ_{Cu} was undertaken and the results are shown in Figs. 2(*b*) and 2(*c*). As $|t_{Cu} - t_{Cu,opt}|$ is larger in Fig. 2(*a*), $\chi^2 - \chi^2_{min}$ from the Cu K-edge measurement increases but $\chi^2 - \chi^2_{min}$ from the Cu $K\alpha$ measurement almost never changes. This indicates that t_{Cu} from the Cu K-edge measurement can be refined more accurately than that from the Cu $K\alpha$ measurement. The accuracy of the refined t_{Cu} was found to improve in the following order: Cu $K\alpha$ < Cu $K\beta$ < Cu K-edge X-ray energy. Similarly, for the interface width, σ_{Cu} refined from the Cu K-edge measurement was the most accurate of all the measurements. In contrast, the χ^2 distributions of δ_{Cu} are the same in all the measurements and the accuracy of δ_{Cu} is equivalent in every measurement. These results reveal that the anomalous-dispersion effect is highly accurate in analysing the layered structure

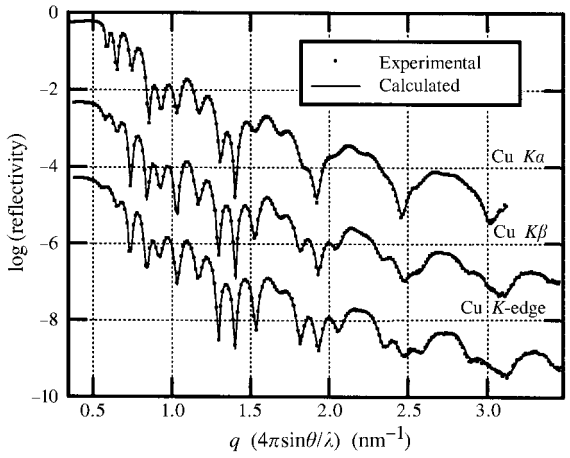

Figure 1
Experimental and calculated reflectivities of the NiFe(10 nm)/Cu(10)/NiFe(10)/Ta(10)/Si multilayers measured for Cu $K\alpha$, Cu $K\beta$ and Cu K-edge X-rays.

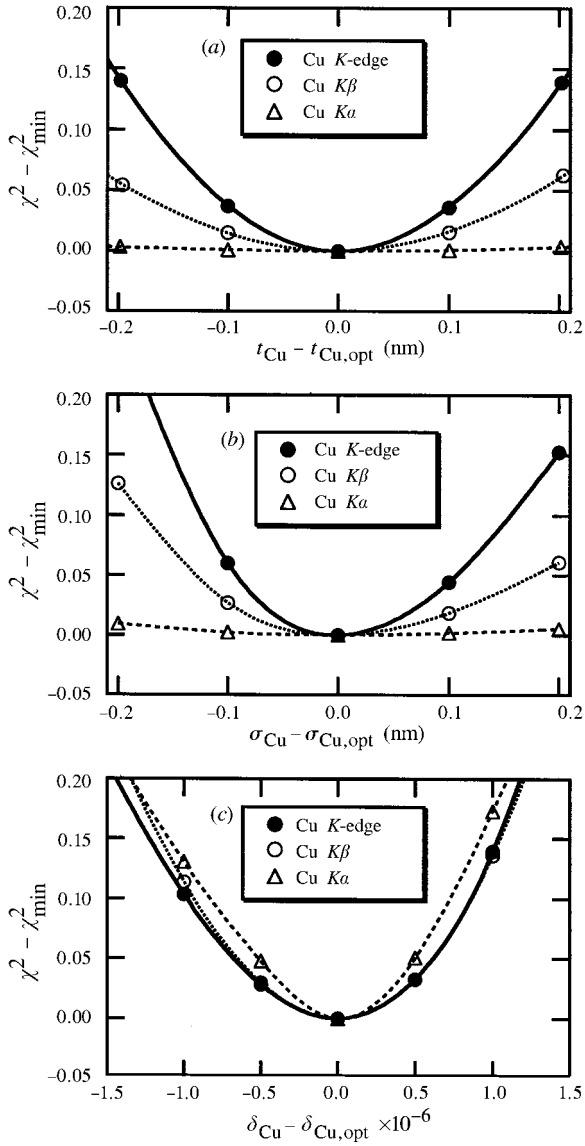

Figure 2
χ^2 distribution *versus* parameters of the Cu layer obtained by least-squares methods for the reflectivities shown in Fig. 1; (*a*) *versus* Cu thickness (t_{Cu}) offset from optimum, (*b*) *versus* interface width (σ_{Cu}) between upper NiFe and Cu layer, (*c*) *versus* δ of Cu.

of the transition metal multilayers. Because the refined parameters obtained from the Cu $K\beta$ reflectivity were in good agreement with those from the Cu K-edge, the $K\beta$ X-rays can also be harnessed for reflectivity measurements for more accurate analysis. This is important for controlling the deposit of the GMR multilayers based on reflectivity measurements, because the $K\beta$ X-rays can easily be produced by conventional X-ray sources.

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