X-ray Reflectivity of Polystyrene-Gadolinium Layered Thin Films

Carlos Quioshi Hiramatsu[1,2], Mari Mizusawa[2] and Kenji Sakurai[1,2]

[1]*University of Tsukuba, 1-1-1 Tennodai, Tsukuba, Ibaraki, 305-8577, Japan*
[2]*National Institute for Materials Science 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan*

Abstract. Spin-coated polystyrene layers have been formed on thin metallic gadolinium thin films deposited on silicon substrates. The polystyrene/gadolinium interface has been studied using the X-ray reflectivity technique.

INTRODUCTION

There have been many scientific discussions on the properties of interfaces between extremely different materials, such as the polymer-metal interface, where quite a big gap in density is created, leading to some interesting electronic properties [1]. Metallic coatings on polymer films have been used in a variety of industrial products, and one of the most important issues here is controlling the layered structure, in particular the interface. The coating of metallic layers with polymers constitutes another interesting topic, and we have therefore studied spin-coated polystyrene (PS) films formed on thin metallic gadolinium layers. The X-ray reflectivity (XR) technique [2,3] was employed to investigate the structure along the depth non-destructively. Electron density profiles (EDP) for several samples are discussed.

EXPERIMENTAL

Metallic gadolinium thin film was grown on a silicon(100) substrate (10mm× 20mm×0.6(t)mm), which was cleaned in ethanol for 15 minutes in an ultrasonic bath. The coating was performed by a normal evaporation method in a vacuum chamber, maintained at around 10^{-6} Pa before the deposition. The temperature of the K-cell was set at ~1200°C during the deposition, and the typical evaporation rate is ~95 Å/h [3,4]. Polystyrene (PS) was subsequently spin-coated. A PS solution in toluene was dropped onto the Gd/Si sample, and then rotated at 4000 rpm. After coating, the sample was heated in a vacuum and kept at 120°C for 4 hours to remove the remaining toluene. The XR measurements were performed with CuKα_1 monochromatic X-rays (8.04 keV).

RESULTS AND DISCUSSION

Figure 1 shows the experimental XR data for a Gd/Si sample (without PS layer), which was measured just for comparison, because of its simpler structure. As shown in Fig.1, it would be unsuccessful to attempt a fit with Model 1, which assumes a single Gd layer on a Si substrate. However, Model 2, which takes into consideration the existence of a thin intermediate layer between the gadolinium layer and the silicon substrate, can explain the experimental data well. The values obtained for each parameter are listed in Table 1. The density of the gadolinium layer (6.79 g/cm^3) is slightly smaller than that found in the literature (7.9 g/cm^3 for the bulk) [5].

Figure 2 shows the experimental XR data for a PS/Gd/Si sample. One can see many complicated interference fringes. The critical angle is found at a very low angle (~ 2.6 mrad) because of the extremely low density of the polystyrene layer. From the data analysis, we found it necessary to consider several transition layers in the vicinity of both the PS-Gd and Gd-Si interfaces. The EDP can be expressed as

$$\sum \Delta\rho_i \cdot \{Erf((z - z_i)/\sqrt{2\pi}\sigma) + 1\}/2. \qquad (1)$$

where $\Delta\rho_i$, z_i and σ_i are the electron density gap, the depth position of the interface and the roughness for each interface, respectively. As shown in the inset of the figure, the polymer-metal interface is not very sharp. This gradation is possibly caused during the drying process. One might notice that the fitting shown in Fig.2 is still not perfect. This could be due to asymmetric (non-Gaussian type) grading. Another interesting point is that the surface roughness is quite large. Both the lateral morphology and the density gradation along the depth are contributory factors.

In summary, it was found that there exists some gradation in the PS-Gd interface prepared by combining the spin-coating and vacuum evaporation methods.

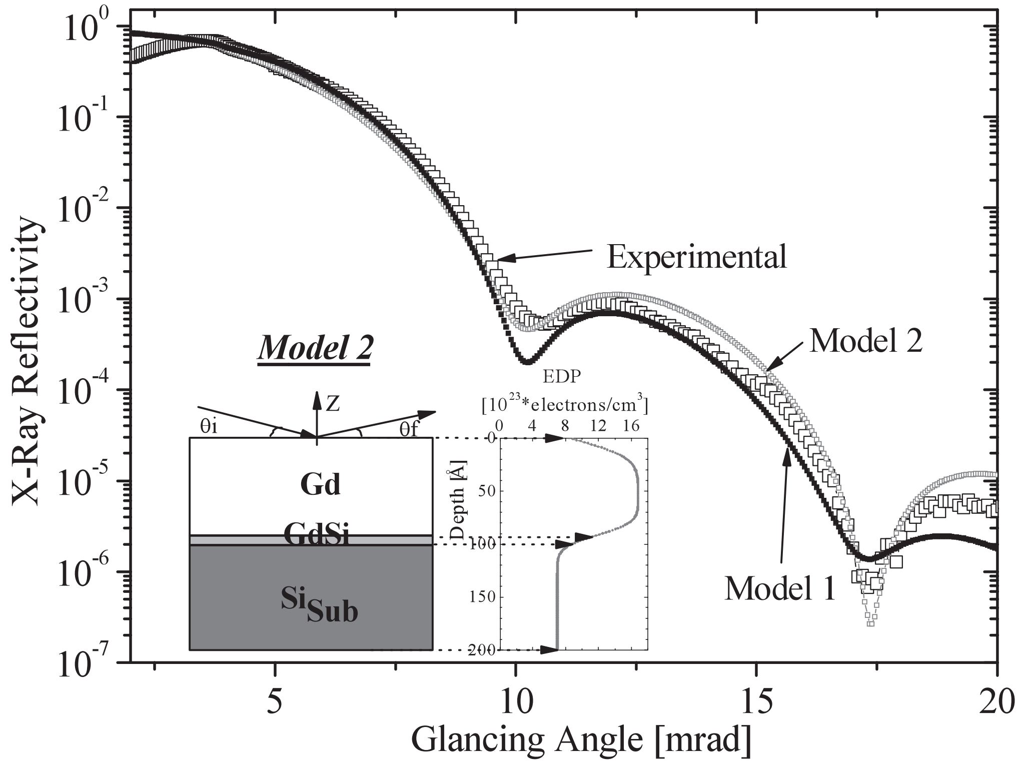

FIGURE 1. The XR from the gadolinium on silicon substrate. Model 1 assumes a single gadolinium layer on a silicon substrate, while Model 2 takes into consideration the existence of the intermediate layer. The corresponding EDP obtained from Model 2 is shown as an inset.

TABLE 1 – Parameters of the Gd/Si (Model 2)

	Density [g/cm³]	***Thickness [Å]***	***Roughness [Å]***
Gd	6.79	91.5	14.3
Gd-Si	5.11	5.3	12.2
Si $_{(sub)}$	2.33	0	9.5

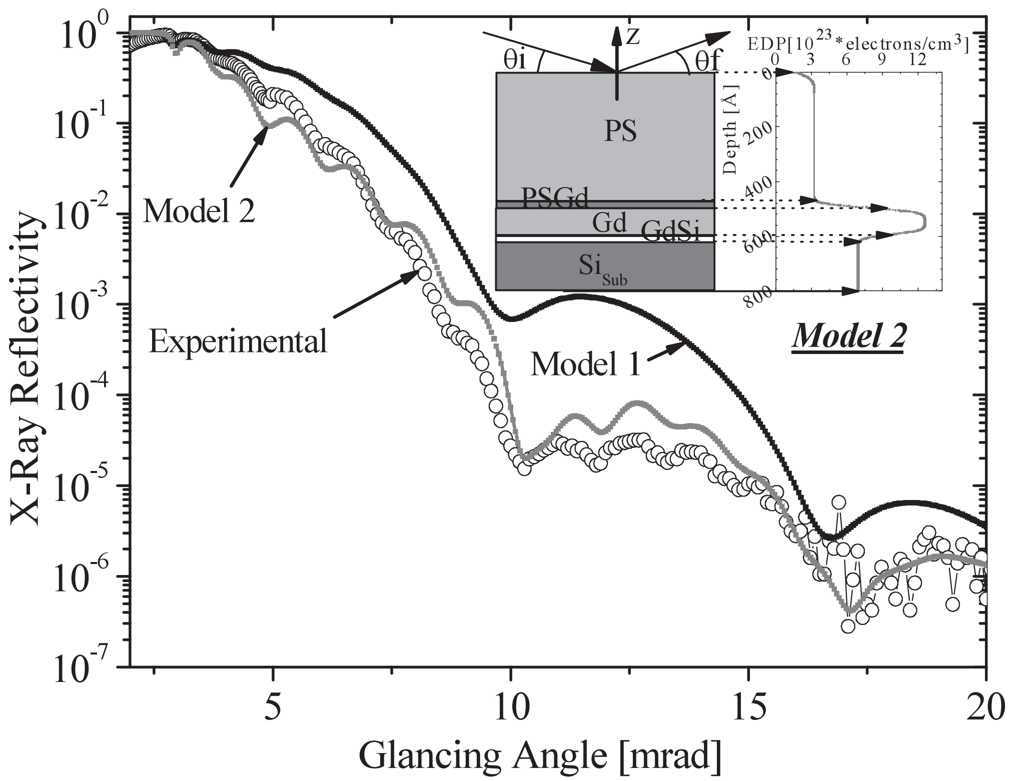

FIGURE 2. The XR from the PS-Gd thin films. Model 1 assuming a simple layered structure cannot explain the experimental results. By considering several additional layers with different densities (Model 2), the fitting has been improved to a fair extent. The corresponding EDP obtained from the Model 2 is shown as an inset.

TABLE 2 - Parameter of the PS/Gd/Si (Model 2)

	Density [g/cm^3]	***Thickness*** [Å]	***Roughness*** [Å]
PS	1.01	498	21.8
PS-Gd	3.59	23.9	12.8
Gd	5.14	55.3	12.7
Gd-Si	4.11	17.2	12.5
Si $_{(sub)}$	2.33	0	10.5

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