

Polarized neutron reflectometry at Dhruva reactor

SURENDRA SINGH and SAIBAL BASU

Solid State Physics Division, Bhabha Atomic Research Centre, Mumbai 400 085, India

Abstract. Polarized neutron reflectometry (PNR) is an ideal non-destructive tool for chemical and magnetic characterization of thin films and multilayers. We have installed a position sensitive detector-based polarized neutron reflectometer at Dhruva reactor, Trombay. In this paper we will discuss the results obtained from this instrument for two multilayered samples. The first sample is a (Ni–Mo alloy)/Ti multilayer sample. We have determined the chemical structure of this multilayer by unpolarized neutron reflectometry (NR). The other sample is a Fe/Ge multilayer sample for which we obtained the chemical structure by NR and magnetic moment per Fe atom by PNR.

Keywords. Neutron reflectometry; polarized neutron; multilayers; interface; roughness; magnetic moment.

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1. Introduction

Neutron reflectometry (NR) is an important non-destructive technique today for determining thin film structures [1]. Unlike X-rays, neutrons are neutral and have large scattering length for low atomic number (Z) materials and possess magnetic moment of $-1.91\mu_n$. Also, for the same element thermal neutron scattering length varies from isotope to isotope. Specifically there is a large difference in scattering lengths of H and D [2]. These properties make neutron reflectometry an attractive tool for studying thin films of polymers, magnetic and biological materials [3–5]. Penetrability of neutron allows one to study deeply buried layers and interfaces by neutron reflectometry. Polarized neutron reflectometry (PNR) gives information on magnetic moment density as a function of depth in the stratified medium. In this paper we present the chemical and magnetic structures of some multilayered samples at microscopic level using NR and PNR techniques.

2. Polarized neutron reflectometry

In NR one measures the specular reflectivity profile of a highly collimated neutron beam (divergence \sim few arc minutes) at the air–film interface of a thin film sample. The specular reflection data is collected at grazing incidence to the sample

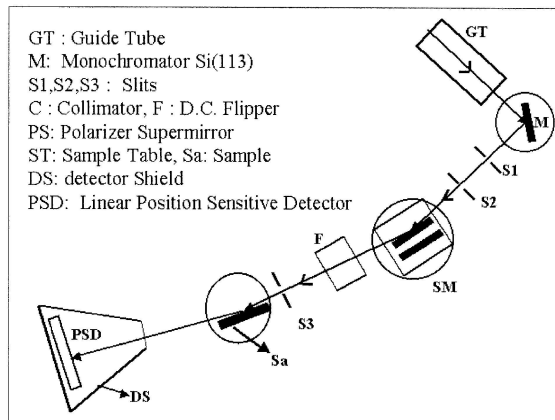


Figure 1. Schematic of polarized neutron reflectometer at Dhruva. GT: Guide tube, M: monochromator Si(113), S1, S2, S3: slits, C: collimator, F: D.C. flipper, PS: polarizer supermirror, ST: sample table, Sa: sample, DS: detector shield, PSD: linear position sensitive detector.

surface (few arc minutes to few degrees). The reflected neutron beam bears signature of the sample thickness, thickness of the constituent layers and roughness at the interfaces. The theory of neutron reflectometry has been discussed, in detail, by Lekner [6] and Zabel [7]. The reflectometry data is collected as a function of $Q (= (4\pi/\lambda) \sin \theta/2$, where θ is the angle of scattering), the momentum transfer. In case of PNR, the sample is magnetized in-plane and incident beam is polarized parallel (+) or anti-parallel (−) with respect to the sample magnetization. The reflectivity data is collected for both the polarizations (R^+ and R^-). One may analyze the polarization of the reflected beam, to obtain R^{++} , R^{+-} , R^{-+} and R^{--} [8]. Parratt's formalism [9] is the most commonly used algorithm for generating theoretical reflectivity profile of a stratified structure. A similar algorithm developed by Blundell *et al* [10] based on transfer matrices has been used by us. We have developed a genetic algorithm-based χ^2 minimization program for obtaining the best possible reflectivity profile and physical parameters, starting from a given set of physical parameters (i.e. thickness, chemical density, roughness, magnetic moment) [11].

The neutron reflectivity data for different samples have been measured at the polarized neutron reflectometer for vertical sample geometry, at Dhruva reactor, Trombay. The description of the spectrometer is given elsewhere [12]. The schematic diagram of this instrument is shown in figure 1.

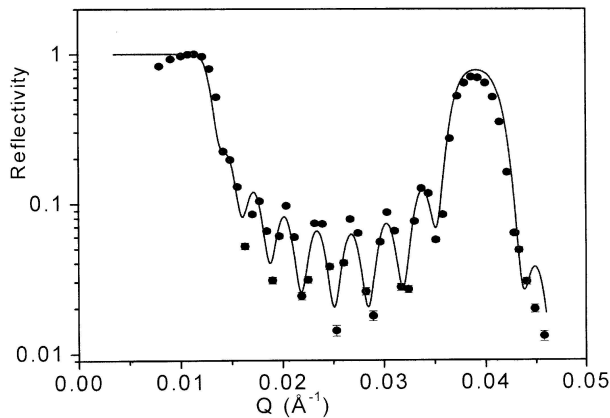


Figure 2. Unpolarized neutron reflectometry data for (Ni-Mo alloy)/Ti multilayer.

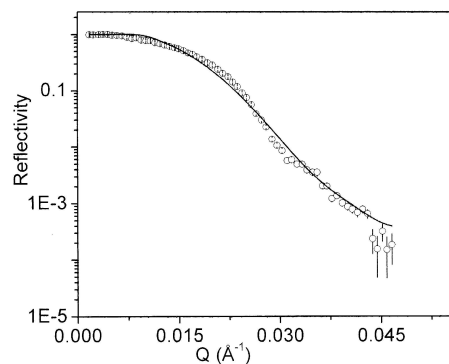


Figure 3. Unpolarized neutron reflectivity plot for (Fe/Ge) multilayer sample.

3. Results and discussion

3.1 (Ni-Mo alloy/Ti) multilayer sample

A multilayer film of (Ni-Mo alloy/Ti)₁₀ (i.e. ten bilayers) was deposited on a glass substrate by sputtering technique by the neutron optics group at PNPI, Gatchina. This combination of materials is suitable to make monochromator and supermirrors in neutron optics (see [13] and the references therein). Unpolarized neutron reflectivity profile of this sample along with the fitted reflectivity patterns are shown in figure 2. The Kiessig oscillations and the Bragg peak are seen clearly. The solid circles in figure 2 represent the background corrected reflectivity data and the continuous line is the profile corresponding to physical parameters that gives best fit to the observed reflectivity profile. The thickness of Ni-Mo alloy layer and Ti layer are 88.6 Å and 78.5 Å respectively as obtained from the fitted profile (designed: 86 Å and 76 Å respectively). The bilayer thickness is 167 Å, which results to a Bragg

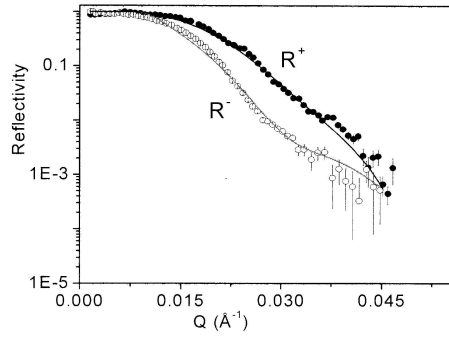


Figure 4. Polarized neutron reflectivity plot for (Fe/Ge) multilayer sample.

Table 1. Physical parameters of sample extracted from best fit of unpolarized reflectivity data.

Layer material	Designed parameters	Unpolarized neutron reflectivity data		
	Thickness (Å)	Thickness (Å)	Density (g/cc)	Average roughness (Å)
Au	30	26 ± 3.0	18.3	4.5
Ge	20	12 ± 1.5	5.11	4.5
Fe	20	16 ± 1.6	7.83	4.5
Buffer Ge	100	89 ± 6.0	5.20	4.5
Substrate (Si)	–	–	2.33	5.0

peak at $Q = 0.039 \text{ \AA}^{-1}$. The best fit gives the average interface roughness of 12 \AA , averaged over the total number of interfaces. In the present multilayer monochromators, the intensity at the Bragg peak is about 70% of the total reflectivity plateau and the multilayer is an ideal neutron mirror.

3.2 (Fe/Ge) multilayer sample

We have used an r.f. sputtered Fe/Ge sample. This sample has Si(1 0 0) as substrate with a Ge buffer layer, on top of which 5 bilayers of Fe/Ge, each 20 \AA thick, were deposited. On top of the bilayers an Au capping layer was deposited. The complete structure of the sample is: Si[1 0 0] substrate/ $\text{Ge}_{100\text{\AA}}$ / $[\text{Fe}_{20\text{\AA}}/\text{Ge}_{20\text{\AA}}]_5/\text{Au}_{30\text{\AA}}$. The NR profile of this sample (circles) and the fitted profile (continuous line) are shown in figure 3. Table 1 gives parameters of this film as obtained from the best fit to the NR. Figure 4 shows the PNR data of the same sample for R^+ (solid circles) and R^- (open circles) neutrons along with the fitted patterns (continuous lines).

We obtained a magnetic moment of $1.43 \mu_B$ per Fe atom in the Fe layers from the best fit. The value of the magnetic moment was obtained by freezing all other physical parameters e.g. thickness and density of layers, as obtained from NR,

while minimizing χ^2 for the PNR data. The magnetic moment of Fe is drastically low compared to the bulk moment of Fe ($2.2 \mu_B$).

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