Nuclear Instruments and Methods in Physics Research A 710 (2013) 106–113

Contents lists available at SciVerse ScienceDirect

Nuclear Instruments and Methods in Physics Research A

journal homepage: www.elsevier.com/locate/nima

X-ray scattering of periodic and graded multilayers: Comparison of experiments to simulations from surface microroughness characterization

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ARTICLE INFO

Available online 8 November 2012

Keywords:
Multilayers
X-rays
Scattering
Roughness

ABSTRACT

To enhance the reflectivity of X-ray mirrors beyond the critical angle, multilayer coatings are required. Interface imperfections in the multilayer growth process are known to cause non-specular scattering and degrade the mirror optical performance; therefore, it is important to predict the amount of X-ray scattering from the rough topography of the outer surface of the coating, which can be directly measured, e.g., with an Atomic Force Microscope (AFM). This kind of characterization, combined with X-ray reflectivity measurements to assess the deep multilayer stack structure, can be used to model the layer roughening during the growth process via a well-known roughness evolution model. In this work, X-ray scattering measurements are performed and compared with simulations obtained from the modeled interfacial Power Spectral Densities (PSDs) and the modeled Crossed Spectral Densities for all the couples of interfaces. We already used this approach in a previous work for periodic multilayers; we now show how this method can be extended to graded multilayers. The upgraded code is validated for both periodic and graded multilayers, with a good accord between experimental data and model findings. Doing this, different kinds of defects observed in AFM scans are included in the PSD analysis. The subsequent data-model comparison enables us to recognize them as surface contamination or interfacial defects that contribute to the X-ray scattering of the multilayer.

1. Introduction

Multilayer coatings are known to enhance the reflectivity of extreme ultraviolet (EUV), neutron, and X-ray mirrors at incidence angles larger than the critical one. As the energy of the incident beam increases, the smoothness of the surface becomes more and more important, because the roughness reduces the specular reflectivity and increases the X-ray scattering (XRS) in non-specular directions, leading to a degradation of the angular resolution. Depending on the specific application, periodic or graded multilayers are deposited using different techniques: anyway, to a variable extent, the deposition process triggers an evolution of the roughness from the substrate to the outermost layer. The interference of waves scattered at layer interfaces [1] result in the final XRS pattern (Fig. 1); therefore, in order to estimate the roughness impact on the Point Spread Function (PSF), a roughness measurement of all the multilayer interfaces would be needed.

However, only the outer surface of the multilayer is accessible to direct topography measurements using, e.g., an Atomic Force Microscope (AFM). X-ray reflectivity (XRR) measurements as a function of the incidence angle, combined with a detailed fit routine to interpret the reflectivity scans [2,3], allows a non-destructive, in-depth analysis of the multilayer stack structure (layer thickness in the stack, uniformity, smoothness), but does not enable the reconstruction of the PSD (*Power Spectral Density*) evolving throughout the stack. Nevertheless, an XRS computation based upon the sole thickness description and the outer surface PSD, assuming the rough topography to be exactly replicated in the stack, would in general return a diagram mismatching experimental data (Fig. 4a).

In this paper, a modeling of the PSD evolution in the stack is used to compute the XRS diagram. A known multilayer growth model [4] provides the PSD growth across the stack, modeled from the measured PSD of the substrate and of the multilayer surface. The model physically describes the roughness of each interface as stemming from two effects in mutual competition: the replication of the roughness of the underlying interface and the roughness introduced by the growth of the layer itself Eq. (1). As a result, the PSD increase from the substrate to the outer surface can be modeled by tuning the values of a few growth parameters [5] that can be tuned to fit the measured external PSD. Once the best-fit parameter values are set, the internal PSDs for all the interfaces and the *Crossed Spectral Densities* (CSD) for all

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http://dx.doi.org/10.1016/j.nima.2012.10.104

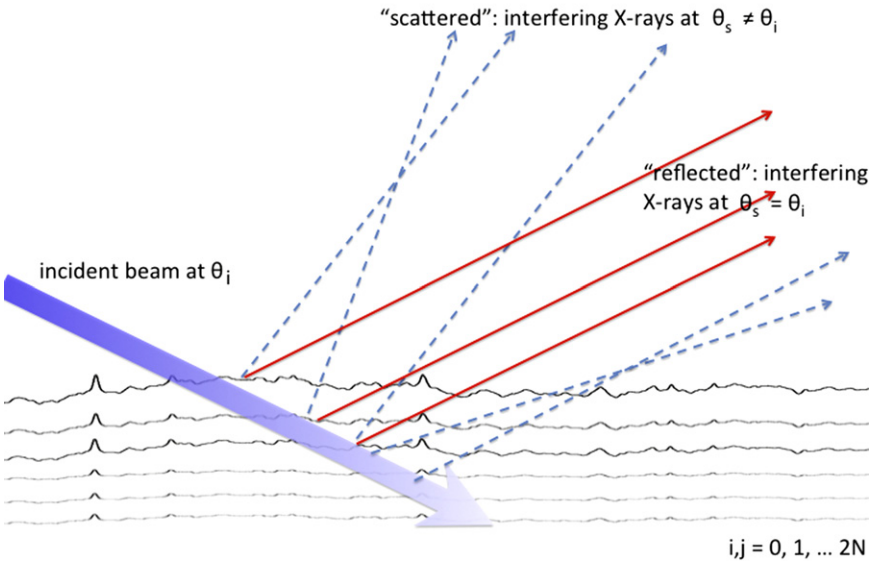

Fig. 1. Scheme of X-ray scattering in a multilayer stack. Both "reflected" (i.e., in the direction specular to that of incidence) angle and "scattered" (i.e., in non-specular directions) rays result from the interference of elementary waves scattered at each boundary in the multilayer.

the couples of interfaces can be reconstructed. These quantities, in turn, determine the XRS expected from the multilayer. In particular, it is the cross-correlation between nearby or distant interfaces to affect the coherence between scattered waves, and, consequently, the amplitude of interferential features in the XRS diagram. The first-order perturbation theory (see Refs. [6,7]) is used to compute the XRS diagram from the roughness PSD evolution in the stack [8,9].

Previous works [5,10] have already implemented this model for periodic multilayers. However, broadband multilayers, like the ones in use in X-ray telescopes, have a graded structure [11]. In this work, we extend the formalism to graded multilayers and we apply it to two multilayer samples, a W/Si periodic sample and a Pt/C graded sample. Roughness analysis of the substrate and the outermost surface of the samples is performed with the Atomic Force Microscope (AFM) operated at INAF/OAB. The layer thickness measurement is obtained from the accurate fit [2] of the XRR measurements performed with a BEDE-D1 diffractometer, also operated at INAF/OAB, at the X-ray energy of 8.045 keV (the CuKα_1 fluorescence line). Eventually, aiming at checking the correctness of the growth description, we have compared the expected XRS diagram to the one measured with the BEDE-D1 at selected incidence angles, finding a very good agreement between the modeling and the experiment. Some preliminary results were already exposed in a previous paper [12].

In Section 2 we retrieve the adopted growth model [4] and the XRS formalism applied to multilayers [9]. In Section 3 we describe the samples and the experimental setup. In Section 4 we show the modeling of the PSDs growth, as well as the predicted XRS vs. the experiments. For the periodic case, we have reanalyzed the data already treated [5], showing that including the modeled PSD growth and adopting a more general electric field modeling leads to the best data/model matching. For the graded case, we show that the thickness trend that describes the stack and fits the XRS peak positions is univocally determined, and also matches the XRR measurement. Finally, we show how XRS can be a powerful tool to discriminate between surface and embedded defects, by including them in the PSD and checking if a proper XRS fit is obtained. The results are briefly summarized in Section 5. A possible derivation of the formula used to model the scattering diagram is sketched in Appendix A, or with more details in Ref. [9].

2. Modeling microroughness growth and X-ray scattering in multilayers

2.1. Microroughness growth model

The roughness growth model [4] solves a kinetic equation to describe the evolution of the rough profile $z(x)$ with the thickness τ of the film. For a single layer deposited onto a substrate, this equation reads

$$\frac{\partial z(x)}{\partial \tau} = -v\left|\nabla^n z(x)\right| + \frac{\partial \eta}{\partial \tau}. \tag{1}$$

The model describes the roughening of the surface as a competition between a surface relaxation process and the increase in roughness due to the random nature of the deposition process. The relaxation process is parametrized with v and the positive integer n that varies with the kinetic mechanism that dominates the smoothing process [4]. The increase in roughness results from the deposition process and is described by a random shot noise term η. The solution of Eq. (1) in terms of surface PSD [4] is

$$P^{\mathrm{int}}(f) = \Omega \frac{1-\exp(-2v\left|2\pi f\right|^n \tau)}{2v\left|2\pi f\right|^n} \tag{2}$$

where $P^{\mathrm{int}}(f)$ is the intrinsic bi-dimensional PSD of the layer surface, i.e., the PSD that the surface layer would have if the substrate were ideally smooth. This PSD is characterized by a plateau up to the maximum frequency corresponding to the cutoff wavelength $l^* = (v\tau)^{1/n}$, then decreases as a power-law of spectral index n. Ω represents the volume of the deposited atom, molecule, or nanocrystal.

When a stack of N alternated layers is considered, the situation is complicated by the presence of two elements with different properties, i.e., different values of the parameters Ω, v, and n. However, the formalism can be extended by considering each single layer (whose upper surface is labelled with $j=0, 1, \ldots, N$ moving from the substrate towards the surface) as growing upon its underlying layer, which acts as its substrate. In this way, one can write [4] $P_0 = P_{\mathrm{subs}}$ and the PSD of the jth interface as a sum of the intrinsic contribution of the layer itself and of a term representing the rough profile partially inherited from the previous layer

$$P_j(f) = P_j^{\mathrm{int}}(f) + P_j^{\mathrm{ext}}(f) = P_j^{\mathrm{int}}(f) + a_j(f)P_{j-1}(f). \tag{3}$$