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ELSEVIER

The application of the X-ray standing wave method to study Ni/C layered structures obtained by laser-assisted deposition

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Abstract

The X-ray standing wave method using fluorescence yield mode was applied to study the profile of the component-by-component distribution between Ni and C layers in Ni/C layered structures produced by laser-assisted deposition. The method of recurrent relationships was used to calculate the standing wave electric field profile, and to reconstruct the Ni deep distribution. Best fitting was obtained if the intermixing thickness was equal to 3–4 Å.

1. Introduction

X-ray optics on the basis of multilayers (multilayer mirrors, Bragg–Fresnel components on their basis, multilayer gratings, etc.) is progressing rapidly. Meanwhile further improvements of their X-ray characteristics and the thermal and radiation resistance are directly connected with the knowledge of the internal structure of the multilayer and the influence of the fabrication technology on it, as well as with the chemical interaction between the layers. The diversity of the pairs of chemical elements and the compounds used in low- and strong-reflecting layers of multilayers greatly complicates this problem.

The small thicknesses and, as a rule, the amorphous nature of the deposited layers of multilayer mirrors make it difficult, and sometimes impossible, to apply X-ray diffraction techniques. The EXAFS spectroscopy technique has proven to be very informative in the analysis of the local atomic structure. In our previous work, it is the technique that allowed us to reveal the mixing of the metal and carbon layers in Co/C and Ni/C multilayered structures, and also its effect on the structure of metallic layers depending on their thickness [1]. In the process of thermal annealing, the strong structural changes in the metallic layers of these structures were made evident [2]. Nevertheless, this technique gives no direct information on the profile of the component-by-component distribution between layers, which is necessary to understand the X-ray optical characteristics.

In the present work, we used a standing wave method in order to examine in more detail the structure of multilayer mirrors fabricated by the technology of laser-assisted deposition. In this case, we get direct data on the concentration profile of the element under study. Moreover, the application of this technique coupled with EXAFS measurements seems extremely promising because it will make it possible to obtain information on the distribution profile of interlayer chemical compounds formed both in the process of fabrication and under thermal loads.

2. Experimental

For the concentration profile of a Ni layer between carbon layers to be analysed, we fabricated complex multilayered structures of two types using pulsed laser evaporation [3]. In both cases, identical W/C multilayered structures (22 bilayers, period 45.5 Å) were used to form the standing wave. In the first case (sample 1), the Ni(13 Å)/C(50 Å) bilayer was deposited directly on the upper W/C bilayer, and in the second one (sample 2) it was deposited through an intermediate carbon layer (26 Å). Thus, the standing wave phases coincided in the W and Ni layers for the first case and they differed by $\pi/2$ for the second one. It may be noted that the Ni and C layers were deposited immediately after the formation of the W/C multilayered structure, in the same technological cycle.

The standing wave measurements were performed at the X-ray microscopy experimental station of the VEPP-3 storage ring (Siberian SR Centre at the Budker Institute of Nuclear Physics, Novosibirsk) [4]. A schematical sketch of the experiment is depicted in Fig. 1. The synchrotron radiation from a 2 T wiggler-magnet was monochromatized and focused by means of a sagittally focusing double-crystal Si(111) monochromator. Having passed through

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the collimating slit and then through the ionization I_0 chamber, the monochromatized beam (8.98 keV) was incident upon a sample. The illuminated area of the sample was about 3×3 mm^2. The sample was fixed on a Microcontrole goniometer (minimal step 0.001°). Having reflected from the sample, the beam was detected by a NaI(Tl) scintillation detector. The fluorescence X-ray intensity from the sample was measured by a Si(Li) detector placed horizontally and normal to the incident beam.

3. Results and discussion

Figs. 2 and 3 show the experimental curves concerning the reflection and yield of Ni K_α fluorescence radiation, which were derived for samples 1 and 2, respectively. The fitting curves for the fluorescent signals may also be found in these figures. To calculate the strength distribution of the electric field of the standing wave in space, a method of recurrent relations was used [5] (see Appendix A). The distribution of the Ni concentration was simulated by a trapezium-shaped profile consisting of several sublayers. For a transient layer, the stepped profile was used. The fitting data have shown that the best agreement between experiment and theory is observed for a 3 Å transient layer. These results are consistent with the EXAFS and SAXS data.

The results, which were obtained by the standing wave method on these samples, demonstrate the sensitivity of

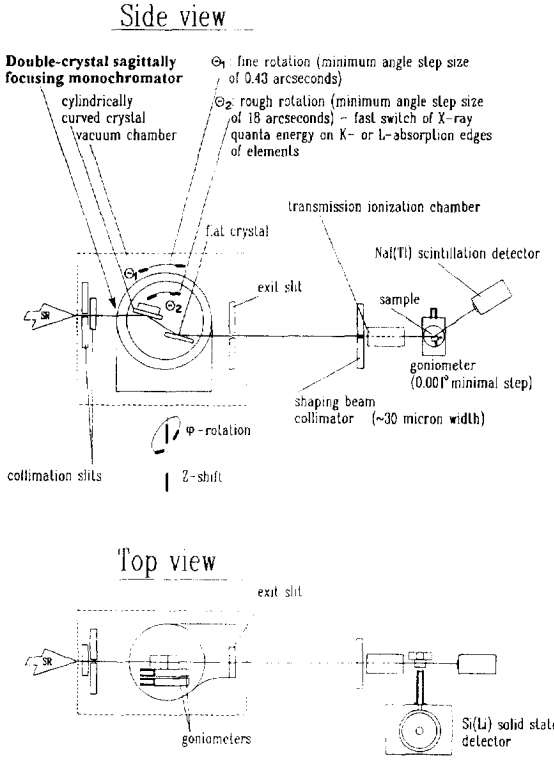

Fig. 1. The layout of the experimental setup.

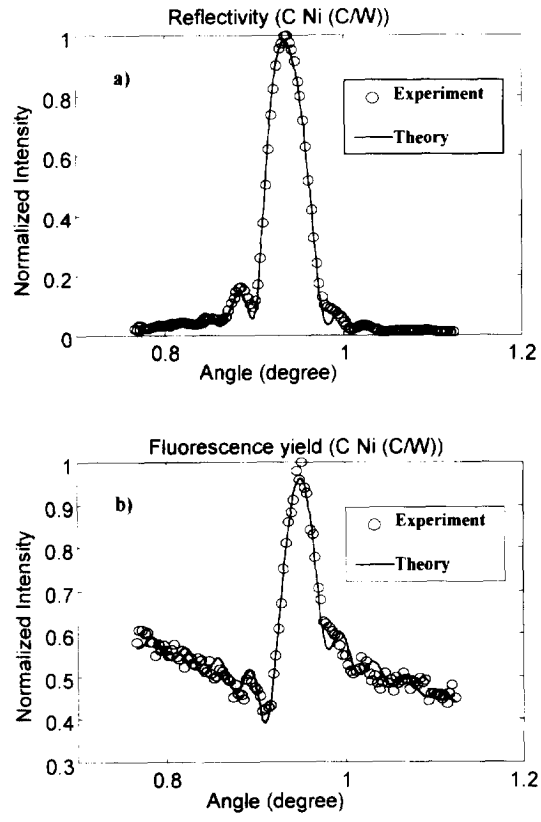

Fig. 2. The reflection (a) and Ni K_α fluorescence yield (b) curves for sample 1 (C Ni (C/W)) (see the text).

the technique used at the ångström level. This will allow one to study fine processes of structure rearrangement, chemical reactions and interlayer diffusion under the conditions of thermal and radiation loads at multilayer structures.

Appendix A: Calculation

Reflectivity

To estimate the MLS reflectivity the technique of recurrent relationships was used [5]. In this case, the coefficient of reflection r_j from the boundary of layers with numbers j and $j+1$ is depending on r_{j+1}:

$$r_j = \frac{r_j^{\mathrm{F}} + r_{j+1} e^{2i\tau_{j+1} l_{j+1}}}{1 + r_j^{\mathrm{F}} r_{j+1} e^{2i\tau_{j+1} l_{j+1}}}; \quad j = 0,1,\ldots,n,$$

$$r_{n+1} = 0,$$

where $r_j^{\mathrm{F}} = (\tau_j - \tau_{j+1})/(\tau_j + \tau_{j+1})$ is the Fresnel reflection coefficient for σ-polarization, $\tau_j = k(\varepsilon_j - \cos^2\theta)^{1/2}$, $j = 0,1,\ldots,n+1$, $k = 2\pi/\lambda$, $\varepsilon_j = 1 - \delta_j + i\gamma_j$ is the complex susceptibility of material of jth layers. l_j is the

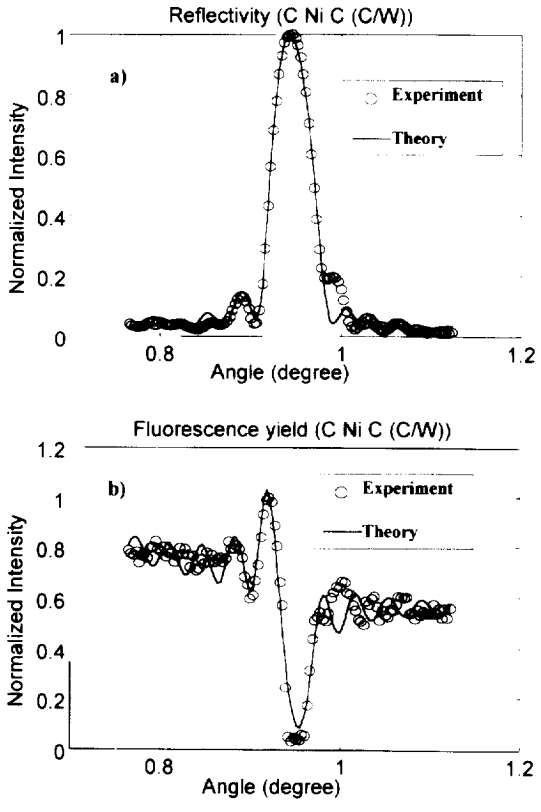

Fig. 3. The reflection (a) and Ni K_α fluorescence yield (b) curves for sample 2 (C Ni C (C/W)) (see the text).

thickness of the jth layer, and θ is the incident angle. The total reflectivity of whole MLS can be found using $R = |r_0|^2$, and the phase jump of the reflected wave may be expressed as $\varphi = \arg r_0$.

Fluorescence yield versus angle

If the MLS is covered by an additional layer with thickness h then the fluorescence yield from this layer versus angle can be estimated as:

$$F \sim \int_{-h}^{0} |E^{I}(z) + E^{R}(z)|^2 \mathrm{d}z$$

$$= E_0^2 \int_{-h}^{0} |e^{i\tau z} + r_0 e^{-i\tau z}|^2 \mathrm{d}z,$$

where $E^{I}(z)$ and $E^{R}(z)$ are distributions of the electric field in the incident and reflected wave on the MLS, $r_0 = r_0(\theta)$ is the complex amplitude reflection coefficient of the MLS, and $\tau = k(\varepsilon - \cos^2\theta)^{1/2} = q + i\mu$ is the wave vector in the layer. After the simplifying it is possible to obtain the next equation:

$$F \sim \frac{\left(1 + |r_0|^2 e^{-2\mu h}\right)\left(e^{2\mu h} - 1\right)}{2\mu}$$

$$+ \frac{|r_0|}{q}\left(\sin(\varphi + 2qh) - \sin\varphi\right).$$

If there are several neighbouring layers which produce fluorescence radiation, the total fluorescence yield is defined as: $F_{\mathrm{tot}} = \Sigma_m F_m w_m$, where w_m is the concentration of the fluorescence element in the layer with number m.

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