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FLUORESCENCE EXAFS AND SAXS STUDIES OF Co/C AND Ni/C MULTILAYERS PREPARED BY PULSED LASER EVAPORATION METHOD

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НОВОСИБИРСК

FLUORESCENCE EXAFS AND SAXS STUDIES OF CO/C AND NI/C MULTILAYERS PREPARED BY PULSED LASER EVAPORATION METHOD

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Abstract

In the present work we have used the EXAFS and SAXS methods for study the inner structure of "classical" Co/C and Ni/C multilayers. This enabled us to study in detail the structure of multilayer as a function of thickness of a metal layer in these multilayers. On the basis of these data the model for the multilayer growth produced with pulsed laser evaporation technology is proposed.

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1. Introduction

For improvement of the multilayer x–ray mirrors production technology the interests of researchers are concentrated on a study of the inner structure of the film materials [1], boundary conditions [2,3,4], the time and temperature stabilities [5,6,7] of multilayer structures. The most studied methods for the synthesis of multilayer structures are sputtering in various versions [8,9] and electron–beam evaporation [10]. The papers [11,12] have informed a successful use of the pulsed laser evaporation (PLE) method for obtaining of multilayers.

The PLE method has a number of unique features. A high kinetic energy of a condensate evaporated on a substrate (hundreds of eV) and a momentary rate of evaporation (10^5-10^6 Å/s) lead to the formation of condensation centers homogeneously over the entire surface of a substrate. On the other hand, high velocity of kinetic energy of a flow can lead to a noticeably mix of layers in a multilayer structure.

So far there is no clear picture of the inner structure of multilayers produced with this technology. In the work presented here we have used the EXAFS and SAXS methods to study of the structure of "classical" Co/C and Ni/C multilayers. This enabled us to study in detail the structure of multilayer as a function of thickness of a metal layer in these multilayers. On the basis of these data the model for the multilayer growth produced with PLE technology is proposed.

2. Multilayer production with PLE technology

The production of multilayers is performed on the UHV device whose schematic diagram is given in Fig.1. The loading of substrate and targets into chamber is provided by the locks pumped out down to a pressure of 10^{-6} Torr. The deposition chamber is pumped out by the magnetic discharge and titanium–sublimation pumps down to a pressure of $10^{-8}-10^{-10}$ Torr.

An optical scheme of the laser evaporator is designed according to the projection scheme proposed in Ref. [13]. The spraying of target materials is performed by a commercially available pulse Nd–YAG laser (λ=1.06 µm, E=0.35 J, t=10 ns, F=30 Hz, energy stability– 1.5 %).

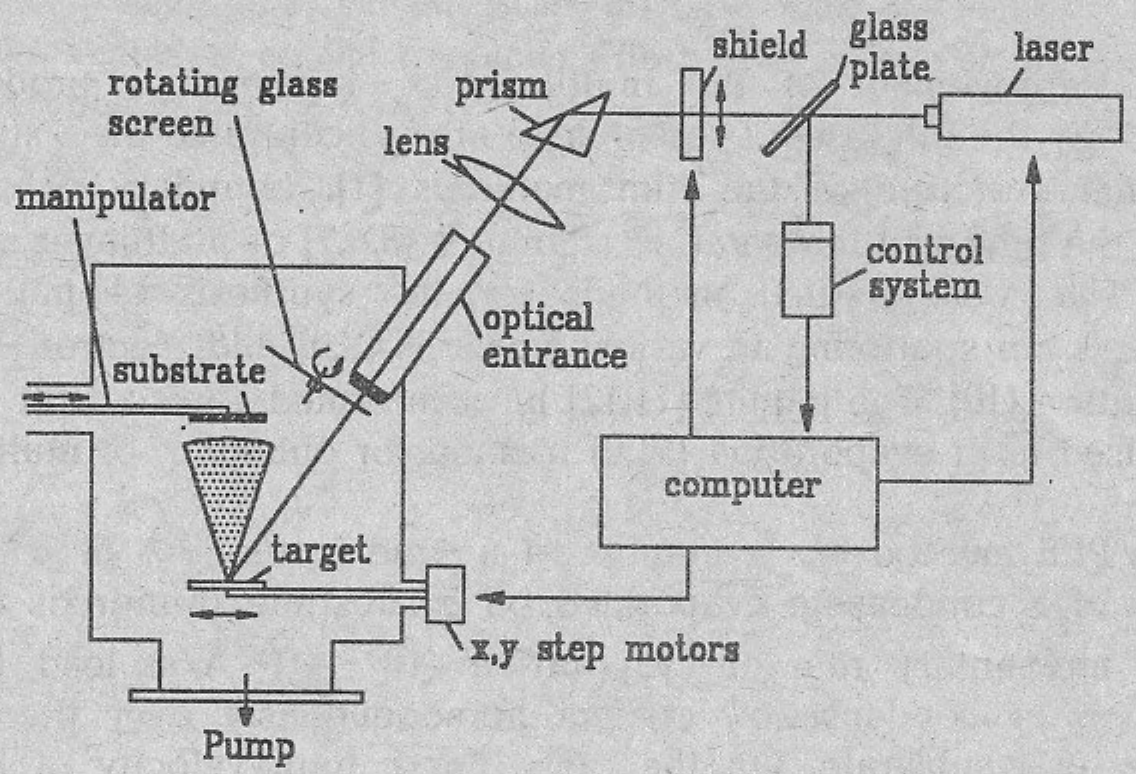

Fig.1 Schematic diagram of a super high vacuum installation for the multilayer X-ray mirrors production by the pulsed laser evaporation method.

The mirrors deposited on "float glass" substrates with a surface roughness of 8 Å. The thickness uniformity of coatings is ±1 % on the 25×25 mm surface .

3. SAXS measurements

Small angle x–ray scattering (SAXS) experiments were performed on a conventional diffractometer. As a monochromator a Si(111) crystal, delivering $Cu_{K\alpha 1}$ monochromatic radiation (λ=1.540562 Å) and an angular divergent beam better 36" was used. The diffractometer can operate automatically in the scanning mode $\theta/2\theta$ with the step 3.6".

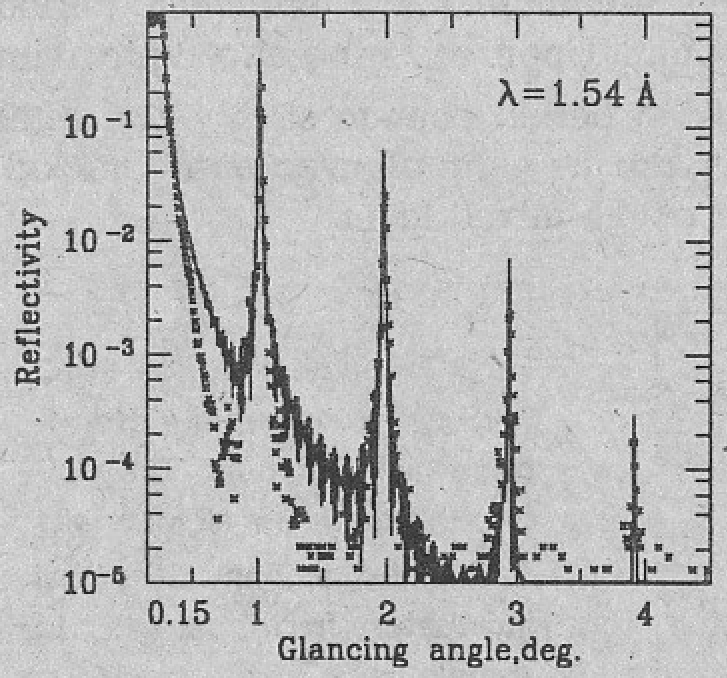

Fig.2 Angular dependencies of the reflection coefficients for radiation with wavelength λ=1.54 Å from the Co/C multilayer: experimental (+), theoretical (continuously line). The parameters used are given in Table 1.

To obtain the main features of the multilayers the experimental small–angle x–ray scattering profiles were simulated by computed spectra. The calculation method we used is based on the recursive optical model [6]. Fig.2 shows the SAXS experimental (+) and simulated (continuous line) profiles for Co/C multilayer with number of periods 70. The parameters used in calculation are given in table 1.

Table 1.The parameters used in calculation. N is the number of periods, d is the period values, β is the fraction of the highly absorbing substance in period and σ is the interfacial roughness.

Materials	N	Dielectric constant of cobalt	Dielectric constant of carbon	d, Å	β	σ, Å
Co/C	70	$1-4.11\cdot10^{-5}$ $+i\cdot6.05\cdot10^{-5}$	$1-1.92\cdot10^{-5}$ $+i\cdot2.88\cdot10^{-8}$	45.2	.185	3.3

To estimate the depth of the mixed layer with the SAXS method we have studied a series of Ni/C MLS. The carbon thickness for all series was 50 Å, the amount of nickel increased from sample to sample. In Fig.3 we have plotted the increase of the MLS period versus the evaporated nickel amount. Upon reaching the equivalent thickness of Ni about a 6 Å the MLS period change slowly and after that begins to grow linearly with the amount of evaporated nickel. This value was taken as the depth of the mixed layer.

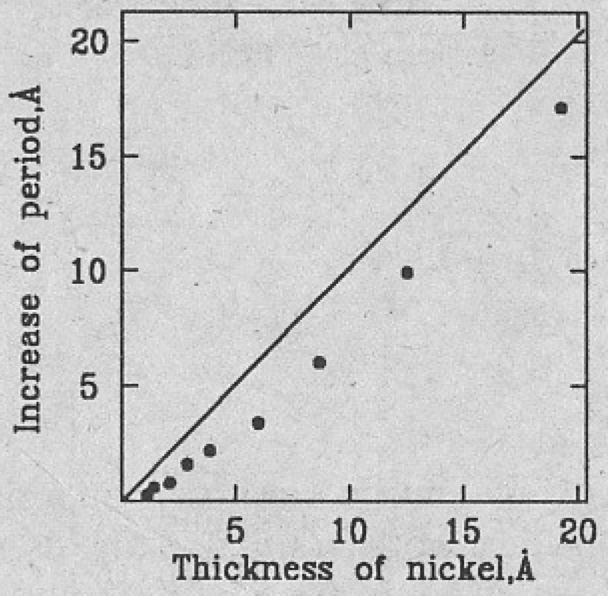

Fig.3 The increase of the Ni/C MLS period versus the evaporated nickel amount. The carbon thickness for all MLS was 50 Å, the amount of nickel increased from sample to sample.

For the study of the influence of the metal layer thickness on the roughness the series of Ni/C and Co/C samples with the same thickness of carbon were made.

The thickness of metal varied from 8 to 70 Å. Fig.4 shows the interfacial roughness as a function of the metal layer thickness for the Co/C MLS. The roughness has a value about 2.5 Å upon reaching of the cobalt thickness 25 Å and after that it increases up to 5.5 Å Similar data were obtained for Ni/C structures.

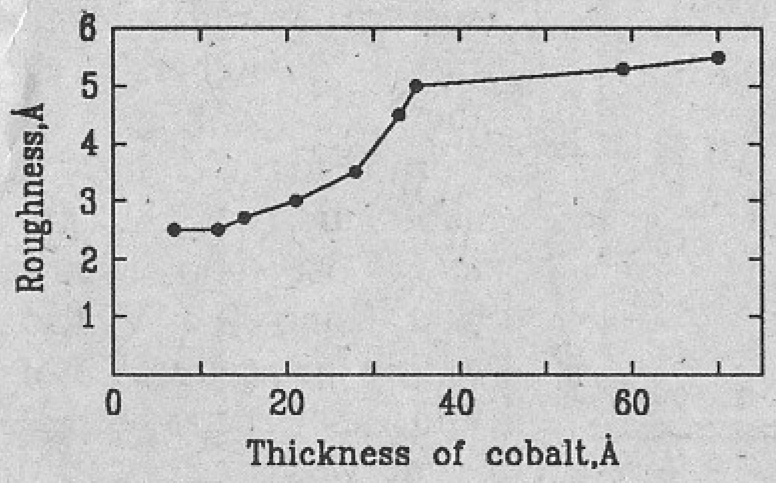

Fig.4 The interfacial roughness as a function of the metal layer thickness for the Co/C MLS.

4. EXAFS measurements

The samples were measured on the EXAFS station [14] of the VEPP−3 storage ring of the Siberian Center of Synchrotron Radiation at an electron energy of 2.0 GeV and current 70−120 mA. The data were obtained with the use of a double−crystal Si(111) monochromator. The spectra were taken by the fluorescent method with the use of a gas scintillation detector in the current mode of operation. Following from the shape of the pre−edge 3d−feature of the Cu K−edge (8980 eV) the energy resolution in operating range was found to be 3 eV. In order to avoid the influence of the Bragg's X−ray reflection from the multilayer under study in the process of scanning over energy the samples were set up at an angle of 6° with respect to the monochromatic beam. When checking the "float glass" substrates we have not found any significant traces of Ni and Co. Thus, the FIEXAFS spectra obtained are only due to the metal of multilayer structures.

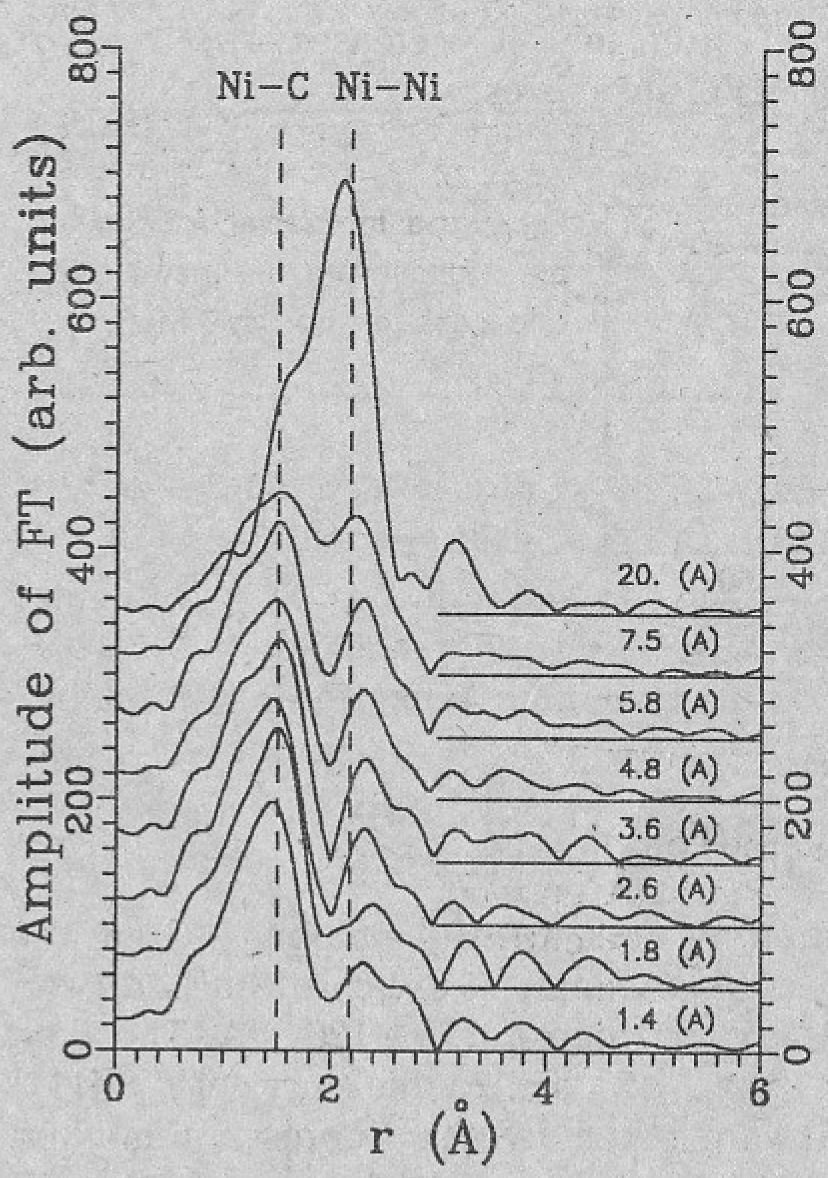

Fig.5 The Fourier transform of the K^3- weighted fine structures (K- range=2÷12 Å^{-1}) at the Ni K-edge for Ni/C multilayers as a function of the layer thickness. Peak positions are uncorrected for phase shifts.

The processing of spectra has been performed according to a standard procedure. The spectra were cleaned from "gleatchers", then their oscillating parts were found by the subtraction of the pre- and post-edge smooth part using the Victorian tuning procedure. Further, the Fourier treatment was performed for obtaining the curve of radial distribution of atoms (RDA).

For finding out the thickness of the intermediate layer and structural changes in Ni films we have studied a series of Ni/C MLS as a function of equivalent thickness of evaporated Ni layers. The series of RDA around Ni without phase correction is given in Fig.5. The first peak corresponds to Ni–C bound (the length is 1.9 Å), and the second peak corresponds to Ni–Ni bound (the length is 2.48 Å).

Fig.6 shows RDA curves corresponding to the Co/C multilayer (number of periods 31, thickness of cobalt 12.4 Å), the Co film (thickness 51 Å) and the Co foil.

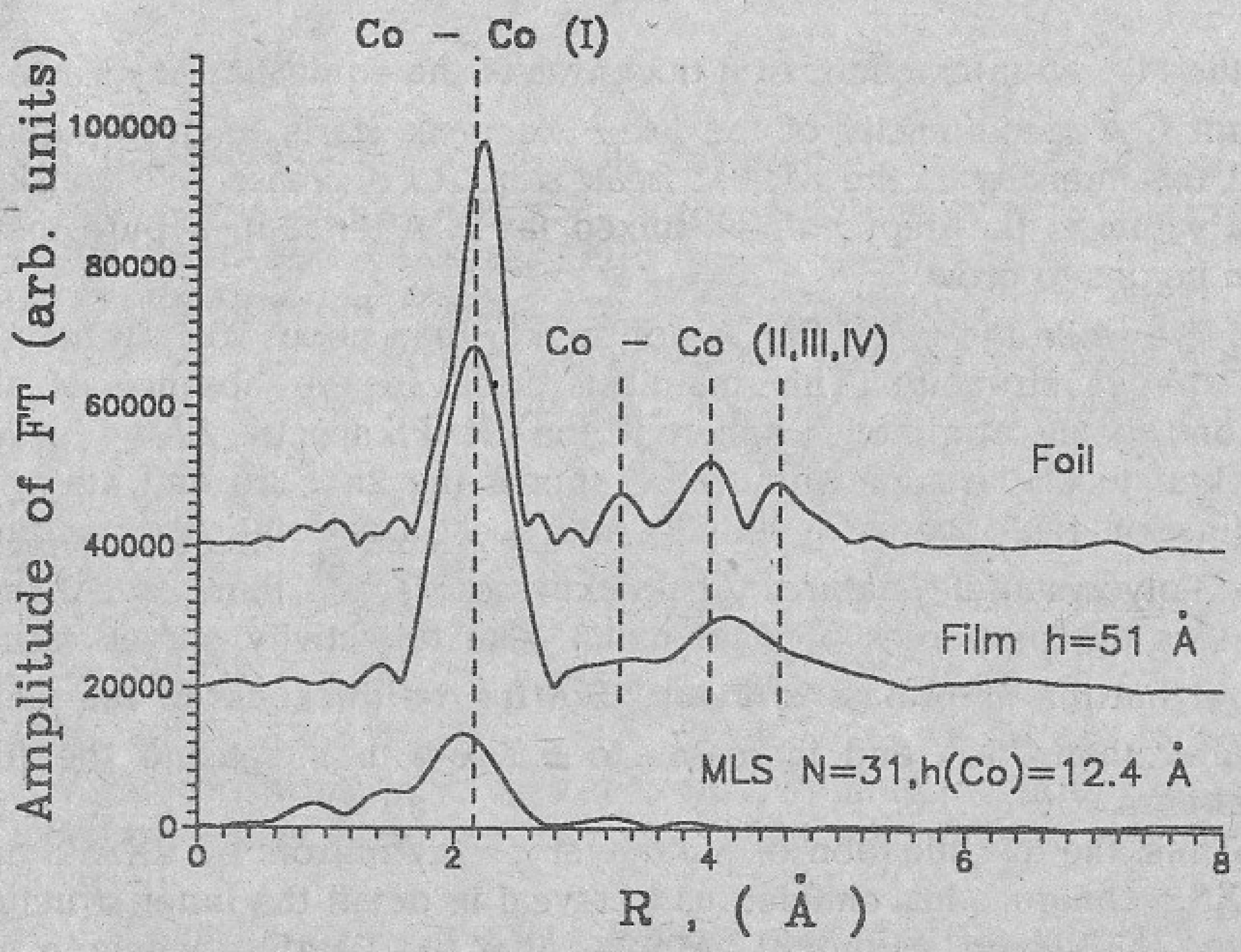

Fig.6 The Fourier transform of the K^3-weighted fine structures (K- range=2÷12 Å^{-1}) at the Co K-edge for a Co/C multilayer, a Co film and a Co foil. Peak positions are uncorrected for phase shifts.

5. Conclusions

Our results allow us to propose the following model of the growth of a multilayer depending on the thickness of the deposited layer of metal (Ni, Co).

At the first stage, as the deposition takes place, as a matter of fact no formation of the nickel film occurs when the metal atoms are introduced into a carbon film. Here the period of the multilayer mirror practically does not increase. The EXAFS measurements indicate the occurring of a Ni–C bounding (the bound length is 1.9 Å) and the absence of the peak corresponding to the Ni–Ni interaction (in this case, the radius is 2.48 Å). The increased amount of metal to be deposited brings about the appearance and the increased intensity of the Ni–Ni interaction. At a thickness of the equivalent layer Me of of the Ni–Ni interaction. At a thickness of the equivalent layer Me of about 6 Å the intensity of the Me–Me peak starts to grow rapidly and the intensity of the Me–C peak starts to decrease. We consider this value as the depth of the mixed layer. After that, a pure metal film begins to grow.

Here while the metal film is not thicker than about 20 Å, it has the amorphous structure. This manifests itself in the absence of the second metal coordination sphere in the EXAFS spectra. In the further thickening of the metal film, there appear the 2nd, 3rd and 4th co-ordination Me spheres on the EXAFS spectra and the films acquire the polycrystalline nature. At thicknesses larger than 50 Å they possess the properties of solid metal. The reflectivity curves results show that the roughness is about 2.5 Å for the thickness of the metal smaller than 25 Å and increases to 5.5 Å with increasing the film thickness.

Thus, the investigation of multilayer x-ray mirrors by EXAFS and SAXS techniques has enabled us to reveal in detail the inner structure of the multilayers. It should be noted that the EXAFS technique has proved to be a very effective tool for the examination of mixed layers.

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Fluorescence EXAFS and SAXS
Studies of Co/C and Ni/C Multilayers
Prepared by Pulsed Laser Evaporation Method

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М.В. Федорченко, В.А. Чернов, Н.И. Чхало

Исследование EXAFS- и SAXS-методами
Ni/C и Co/C МРЗ, изготовленных
методом импульсного лазерного напыления

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