Analysis of parameters of multilayer carbon interference structures in the soft xray range

P. E. Kondrashov, I. S. Smirnov, E. G. Novoselova, and A. M. Baranov

Citation: Applied Physics Letters **69**, 305 (1996); doi: 10.1063/1.118041
View online: http://dx.doi.org/10.1063/1.118041
View Table of Contents: http://scitation.aip.org/content/aip/journal/apl/69/3?ver=pdfcov
Published by the AIP Publishing

Analysis of parameters of multilayer carbon interference structures in the soft x-ray range

P. E. Kondrashov,[a] I. S. Smirnov, E. G. Novoselova
Institute Electronics and Mathematics, Bol. Vuzuvsky per., 3/12, Moscow 109028, Russia

A. M. Baranov
Research Institute of Vacuum Technique, Nagorny proezd 7, Moscow 113105, Russia

(Received 19 February 1996; accepted for publication 7 May 1996)

A new type of x-ray interference mirrors using hydrogenated carbon films of various densities is described. The reflection coefficient and resolution of multilayer carbon structures obtained in the soft x-ray range (1.54–44.7 Å) are investigated experimentally and theoretically. As is shown, hydrogenated carbon films can be used to create mirrors with high resolution. © *1996 American Institute of Physics.* [S0003-6951(96)02129-8]

The most important elements of the x-ray optics are grazing incidence mirrors and multilayer interference structures (MIS). At present, the most commonly used structures are MIS in which highly absorbing layers of heavy metals (W, Re, Ni, etc.) alternate with weakly absorbing layers of light atoms (C, Si, etc.).[1] However, in manufacturing x-ray mirrors based on two-component MIS with metallic layers, a number of problems arise which are quite difficult to resolve. For example, it is impossible to obtain both high eflectivity and high resolution.[2] Moreover, in such structures it is difficult to create sharp interfaces because of the mutual diffusion in the regions of large atomic concentration gradients.

These problems can be solved by using periodic multilayer structures consisting of weakly absorbing layers with close dielectric constants as interference mirrors in the x-ray range. We have manifactured and studied new x-ray mirrors consisting of hydrogenated carbon films (a-C:H) of various densities. The choice of carbon for our purpose is motivated by several reasons. On the one hand, carbon is one of the elements with the lowest absorption in the range 1.54–130 Å, being inferior in this respect only to lighter elements: H, He, Li, Be, and B. On the other hand, the presence of several types of bond hybridization (sp^1, sp^2, and sp^3) enables one to obtain various structures (from graphitelike to diamondlike ones) with a great diversity of properties. Furthermore, the variation of the hydrogen content (from 10% to 30% in our case) provides additional possibilities to control the properties of carbon films. The presence of hydrogen can also give rise to a decrease in x-ray absorption compared to carbon films without hydrogen.

Multilayer carbon interference structures (MCIS) were prepared by plasma deposition from the mixture $Ar+C_6H_{12}$.[3] Plates of fused quartz of diameter 30 mm with mean square surface roughness $\sigma=8$ Å were used as substrates. For better adhesion, the substrates were treated by plasma before the layer deposition. The measurements of the structure parameters were performed in the wavelength range (λ) 1.54–44.7 Å. The measurements at 1.54 Å were made using an x-ray reflectometer designed to study total external reflection. Measurements at 7.13 and 8.33 Å were made using a modified fluorescence spectrometer with Si and Al targets. The beam was directed to the sample via the collimator with angular divergence 0.003° at 1.54 Å and 0.15° at 7.33 Å and 8.33 Å. For a comparative analysis, Ni/C mirrors were used. The measurements at 13.3 and 31.4 Å were made by the x-ray reflectometer described elsewhere.[4] The scanning steps were $2\Delta\Theta=0.01°$ at 1.54 Å and $2\Delta\Theta=0.02°$ at the other wavelengths.

The measured dependence on the incidence angle, $R=f(\Theta)$, of the reflection coefficients for MCIS and Ni/C is shown in Fig. 1(a) (curves 1 and 2, respectively). The calculated curves for these structures are plotted in Fig. 1(b) (curves 1 and 2); the calculations were made using the recursion relations.[5] For a MCIS with period $L=94$ Å, mean square substrate roughness $\sigma=8$ Å, and the number of layers

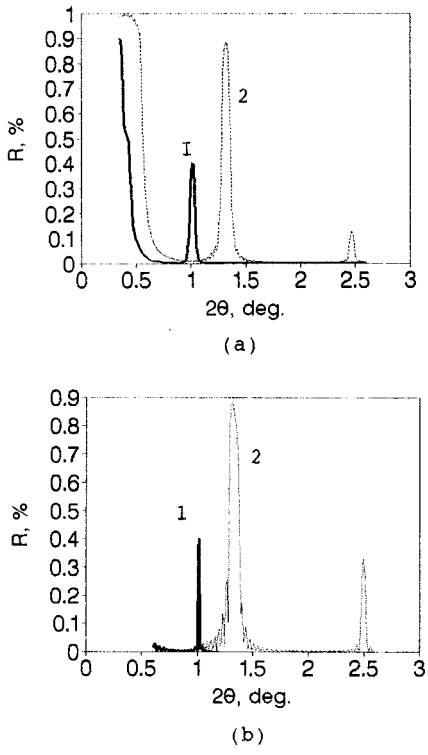

FIG. 1. (a) Experimental and (b) theoretical dependencies $R=f(\Theta)$ for $\lambda=1.54$ Å. (1) Multilayer carbon interference structure ($N=118$, $d=94$ Å); (2) Ni/C mirror ($N=60$, $d=72$ Å).

[a]Electronic mail: root@onti.miem.msk.su

TABLE I. Parameters of the structures studied.

Structure parameters	Mirror type No. 1 (a-C:H$_I$/a-C:H$_{II}$)	No. 2 (Ni/C)
(1) Structure period (Å)	94	72
(2) Number of period	59	30
(3) Layer thickness (Å)	47/47	24/48
(4) Difference in decrements of refraction of the layers ($\Delta\delta$)	1.2×10^{-6}	18×10^{-6}

$N=118$, the best agreement between the theoretical curve and experimental data was achieved for the difference $\Delta\delta=1.2\times10^{-6}$ in the layer decrements of refraction, which corresponds to the layer density difference $\Delta\rho=0.38$ g/cm^3. Calculations for the Ni/C mirror were made using the tabular values of the decrements of refraction and absorption coefficients for nickel and graphite. The parameters of the mirrors of both types are listed in Table I.

Comparison of the experimental data for different structures demonstrates that for MCIS having maximal reflectivity ($R_{max}=40.7\%$) half as large as for the Ni/C mirror we obtained the resolution that is twice as large (the half-width of the first Bragg reflection peak was $\Delta\Theta=0.02°$). We note that the smaller value of R for MCIS is, to a large extent, due to the large roughness of quartz substrates. For MCIS the curve $R=f(\Theta)$ is below the reflectivity curve for the Ni/C structure. In fact, it is seen from Fig. 1(a) that in the region of total external reflection, R for the Ni/C mirror approaches 98%, whereas for MCIS it is only 90%. It should be noted that the half-width of the Bragg peak of the theoretical $R=f(\Theta)$ curve is about half as large as that of the experimental curve. We believe that this is due to the fact that the experimental curve is the convolution of the true curve and the instrumental function; for this reason the resulting width is greater than the true width. This broadening effect is expected to be especially large for MCIS, as these structures have small peak half-widths.

Since the main parameters that affect reflection at wavelength 1.54 Å are the difference in layer densities and substrate surface roughness, we have also calculated R_{max} which can be achieved by increasing $\Delta\rho$ and decreasing σ. These R_{max} are listed in Table II.

As is seen from Table II, increasing $\Delta\rho$ to 0.5 g/cm^3 at fixed σ leads to an increase in R_{max} up to 61%. Decreasing σ to 4 Å at fixed $\Delta\rho=0.5$ g/cm^3 leads to a further increase in R_{max}, namely, by 11%. A further increase in $\Delta\rho$ to 0.6 g/cm^3 enables one to reach the value of 79% for the coefficient of reflection from MCIS. It is important to note that $\Delta\rho=0.6$ g/cm^3 is practically attainable in the process of carbon film preparation, as the carbon film density usually varies from 1.2 to 3 g/cm^3.[6] Therefore, the goal of preparing

TABLE II. MCIS parameters used in the calculations.

No.	$\Delta\rho$ (g/cm^3)	σ (Å)	R_{max} %
1	0.38	8	40.7
2	0.5	8	61
3	0.5	4	72
4	0.6	4	78

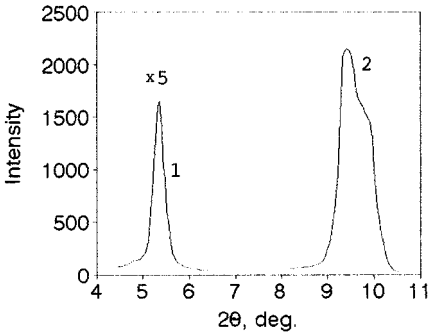

FIG. 2. Experimental dependencies $R=f(\Theta)$ for $\lambda=8.33$ Å. (1) Multilayer carbon interference structure ($N=118$, $d=94$); (2) Ni/C mirror ($N=60$, $d=51$ Å).

films with $\Delta\rho=0.8$–1 g/cm^3 is quite realistic. In this case R_{max} would reach 90% at wavelength 1.54 Å and $d=94$ Å.

We note one more important advantage of MCIS when used in x-ray spectroscopy. There are no even peaks in the reflection spectrum of the structure studied [Fig. 1(a), curve 1]; this indicates that the different layer thicknesses are the same and layer thicknesses are almost constant in any period. One can quench the ''even'' reflection peaks for Ni/C mirrors as well as by the corresponding increase of the Ni layer thickness. However, the calculations show that this decreases the reflectivity of the structure by about 10% and broadens the Bragg peak.

The data $I=f(\Theta)$ for the MCIS and Ni/C mirror in the soft x-ray range at wavelength $\lambda=8.33$ Å (Al anode) are shown in Fig. 2. The Ni/C mirror with period $L=51$ Å having reflectivity of 63% at $\lambda=1.54$ Å was chosen as a reference sample.

The reflection coefficient from the MCIS at the maximum of the first Bragg peak at $\lambda=8.33$ Å was calculated using the formula $R_{max}=(I_{max}/I_0)*g$, where I_{max} is the intensity of reflected beam at the maximum, I_0 is the intensity of the incidence beam, and g is the geometry factor. The latter was introduced to take account of the sample size. For MCIS we have $g=3.4$ but for Ni/C—mirror $g=1.1$. In this case $R_{max}=10\%$, which is half as much as the reflectivity for the Ni/C mirror ($R=22\%$). Just as in the short wavelength range, the half-width of the Bragg peak for MCIS was appreciably smaller ($\Delta\Theta=0.12°$) than for Ni/C mirrors ($\Delta\Theta=0.4°$). The error in the determination of R is mainly due to the small size of experimental structures, the small angle of incidence of x-rays, and the design of the experimental setup.

The resolution ($\Delta\lambda/\lambda$) and the half-width of the peak ($\Delta\Theta$) are related by $\Delta\lambda/\lambda=\Delta/\mathrm{tg}(\Theta)$, where Θ is the angular position of the Bragg peak.[2] Using this relation, we evaluated the resolutions for the MCIS and the Ni/C mirror to be 0.043 and 0.085, respectively.

Measured and calculated R_{max} in the range from 7.33 to 44.7 Å are shown in Table III. The calculations were made for the MCIS, whose parameters are listed in Table II (No. 1).

The measured reflectivity for MCIS is seen to decrease from 12% to 0.2% as the wavelength increases from 7.33 to 44.7 Å. This is the result of the increase in the carbon absorption coefficient as λ approaches the carbon absorption

TABLE III. Experimental ($R_{\max}$) and theoretical (R_{th}) values of reflectivity for MCIS in the soft x-ray range.

λ (Å)	Θ (°)	ΔΘ (°)	$R_{\max}$ (%)	R_{th} (%)
7.13	2.3	0.1	12	19
8.33	2.68	0.12	10	16.5
13.3	4.35	0.17	6.5	7.6
31.4	10.2	0.65	0.95	1.1
44.7	14	⋯	0.2	⋯

edge (λ=44.7 Å). At λ=44.7 Å MCIS reflection practically vanishes, since at the absorption edge the carbon decrement of refraction drops abruptly. Therefore, the difference in decrements of refraction of different carbon layers of the MCIS is also decreased. In this respect the reflection from MCIS measured with the characteristic carbon radiation radically differs from the reflection from Me/C mirrors. In fact, for Me/C mirrors the difference in decrements between metal and carbon goes up near the absorption edge due to the decrease of the carbon decrement. Thus, the reflectivity from Me/C mirrors increases. However, for λ>44.7 Å the reflectivity from MCIS must drastically increase, due both to the increase of $\Delta\delta$ and to the decrease of absorption by carbon atoms.

Thus, we demonstrated experimentally the new method for creating x-ray mirrors with nanometer a-C:H layers using rather simple methods of plasma deposition technology. The absence of concentration gradients at interfaces allows one to minimize the interface diffusion broadening and thus to obtain more perfect structures.

The carbon mirrors studied in this letter can be considered as a fundamentally new type of mirrors being one-component (carbon) structures and not two-component ones as before. Increasing the number of periods of such MCIS enables one to obtain structures with high resolution and, at the same time, sufficient reflectivity in a large spectral range. Since there are no restrictions on relative layer thickness inside the period, the conditions that are necessary for quenching higher-order reflections are easily satisfied.

The authors thank Professor V. V. Kondratenko for supplying the Ni/C mirror used in the comparative analysis and Professor I. F. Mikhailov and Professor N. N. Salashenko for their help in the rendering of measurements in the soft wavelength range.

[1] E. Spiller, *Soft X-Ray Optics* (SPIE Optical Engineering, Washington, DC, 1994).
[2] A. V. Vinogradov and I. V. Kozhevnikow, Trudy Fiz. Inst. Lebedev (FIAN) **196**, 63 (1989).
[3] P. E. Kondrashov, I. S. Smirnov, H. G. Novoselova, A. M. Baranov, and V. V. Sleptsov, Proc. SPIE **2517**, 126 (1995).
[4] N. N. Salashenko, Yu. Ya. Platonov, and S. Yu. Zuev, Poverhnost: Fiz. Chim. Mehanika N **10**, 5 (1995).
[5] L. G. Parrat, Phys. Rev. **95**, 359 (1954).
[6] H. Tsai and B. Body, J. Vac. Sci. Technol. A **5**, 3287 (1987).