

https://doi.org/10.1070/QEL16990

Comparison of approaches in the manufacture of broadband mirrors for the EUV range: aperiodic and stack structures

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***Abstract*. We have developed the design and experimentally studied aperiodic and stack broadband Mo/Si mirrors for the purposes of the KORTES project, optimised for uniform reflection in the 17–21 nm wavelength range. It is shown that stack structures with an insignificant loss in the reflection coefficient are much more preferable from the point of view of manufacturing and certification, which, in turn, makes it possible to correct the deposition process and to reach the calculated parameters of a multilayer mirror in a small number of iterations.**

Keywords: *EUV, broadband mirrors, aperiodic structures, block structures, stack structures, magnetron sputtering, inverse problem.*

1. Introduction

Since the 1980s, periodic multilayer mirrors have been a universal element of optical systems designed to work in the entire X-ray and extreme ultraviolet (EUV) wavelength ranges (0.01–60 nm). The development of manufacturing technology and methods for certifying such structures has now allowed a close approach to the theoretical limit of the reflection coefficients [1–5]. At the same time, almost from the very beginning [6, 7], the task was to manufacture mirrors with a period variable in depth, with better characteristics, first of all, an integral reflection coefficient greater than that of periodic mirrors, and an increased spectral or angular reflection band. For the Kirkpatrick–Baez astrophysical telescope [8], multilayer W/Si mirrors were proposed, reflecting radiation with wavelengths up to 0.18 Å at grazing incidence angles of 3 mrad. The period of the mirror monotonically decreased with depth according to the law $d_i = a(b + i)^{-c}$, where d_i is the value of the ith period from the surface; $c \approx 0.26$; and a and b are fitting parameters. This approach is called the ABC model, or super mirror model. In Refs [9, 10], numerical and analytical methods were proposed for calculating the parameters of such mirrors, which provide a given profile of the reflection curve. Later, this approach was used to design mirrors for synchrotron radiation channels and to increase the radiation flux from the X-ray tube on the sample [11].

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Received 6 March 2019
Kvantovaya Elektronika **49** (4) 380–385 (2019)
Translated by V.L. Derbov

A monograph [12] is devoted to the development and application of aperiodic mirrors in the field of soft X-ray and EUV radiation. The special relevance of this topic is due to studies of the extreme state of matter, for which systems are developed to control the spatial, temporal and spectral characteristics of femtosecond and subfemtosecond [13], and more recently attosecond and subattosecond electromagnetic radiation pulses, whose spectrum lies in X-ray or EUV range [14]. For such ultrashort pulses, the spectral width is comparable to the carrier frequency; therefore, it is necessary to use broadband optical elements instead of the classic narrowband periodic X-ray mirrors.

In problems of X-ray microscopy in the water transparency window (λ = 2.3–4 nm), aperiodic mirrors make it possible to increase the recorded signal by two to three times [15]. They are also used in ‘stigmatic’ spectrometers with diffraction gratings for studies of the Sun [16]. The complex of KORTES equipment for the study of the Sun contains a spectroheliograph, which allows recording the emission spectra of solar flares, micro flares and coronal mass ejections. The spectral range of the instrument is determined by its broadband focusing X-ray mirror. In this regard, it is necessary to develop and manufacture a mirror with the maximum uniform reflection in the spectral range of 17–21 nm.

Despite certain successes in the manufacture of aperiodic mirrors, the practical solution of this problem is associated with great difficulties. The main problem in the manufacture of X-ray mirrors, consisting of a large number (often more than a hundred) layers with individual thicknesses and having an optimal preset reflection coefficient, is primarily the complexity and duration of the calibration process. At the initial stage of deposition, the film thickness grows nonlinearly, the particle sticking coefficients vary, and mixing of materials occurs. Noticeable distortions introduce [17] technological errors in the layer thickness of 1%–2%, which do not have a significant effect on the reflection coefficient of the periodic multilayer structure. In fact, in order to deposit N_{AMS} layers of various thicknesses correctly, it is necessary to carry out approximately as many different calibrations, provided that the materials are sufficiently well studied at the stage of fabricating periodic mirrors. The latter implies the knowledge of both film density (often depending on the thickness that determines amorphous or crystalline state of matter [18]) and interlayer roughness. Such a priori knowledge is necessary, since the result of solving the optimisation problem will be substantially determined by the roughness, as shown by the authors in Ref. [17], i.e., the optimal thicknesses of the aperiodic multilayer mirror will be different depending on the characteristics of the interlayer boundaries. After the mirror is manufactured, its certification is carried out. If the reflective characteristics of the fabricated multilayer structure differ from the calculated values, it is necessary to solve the inverse problem of reconstructing the mirror structure in order to correct the technological process. The solution of the inverse problem in the class of aperiodic structures is extremely complex, and it is often impossible to obtain any significant information about individual films from the data of reflectometric measurements.

Understanding these problems makes us pay attention to the approach, proposed for the first time, as far as we know, in Ref. [19] for the X-ray telescope of the hard X-ray range (radiation with $h\nu$ = 20–40 keV, radiation grazing angle 0.3°). The proposed structure of a multilayer Pt/C mirror consisted of eight periodic multilayer mirrors deposited at each other with periods of 60–30 Å, decreasing as the layers approach the substrate. In total, the structure consisted of 168 layers, and the Pt layer thickness was fixed at 15 Å, except for the upper layer, in which the Pt and C fractions were chosen equal to suppress the second Bragg peak. Because of its simplicity, this method has been successfully applied in the development of grazing-incidence X-ray mirrors for X-ray telescopes InFOCμS and ASTRO-H.

Later in Ref. [20], based on the approach developed in [10], an analytical expression for the reflection coefficient of such a structure was obtained in the kinematic approximation and the basic principles of its design were formulated. These principles imply that the condition $N_i d_i$ = const must be fulfilled for all periodic mirrors incorporated in the structure, the number of N_i periods of neighbouring mirrors differing by one and the period d_i decreasing with depth, which corresponds to the ABC model [8]. As in the case of a supermirror, the broadened reflection band, as compared to a periodic mirror, is formed as a result of the penetration of short-wavelength radiation to a greater depth, where, in accordance with the Bragg condition, it is reflected from a periodic mirror with a smaller period.

Aperiodic mirrors of this type, representing a stack of periodic mirrors deposited on each other with a set of characteristics $\{N_i, d_i, \gamma_i\}$ (respectively, the number of periods, the period value and the proportion of the strongly absorbing layer for each ith mirror), were called block structures in Refs [19, 20] or stack structures in Refs [21–24] devoted to the development and manufacture of broadband mirrors for soft X-ray and EUV ranges. In Refs [21, 22], the aperiodic and stack approaches to the fabrication of broadband Mo/Si mirrors of normal incidence with a constant reflection coefficient in the range of incidence angles of 0–20° for radiation with λ = 13.5 nm were compared for the first time. The calculated reflection coefficient for an aperiodic mirror formed by 101 layer of Mo and Si with thicknesses in the range 2.7–4.5 nm was 45% (deviation is within units of percent). An optimised mirror composed of three periodic structures with parameters (with distance from the substrate) N_1 = 30 (d_1 = 7.22 nm), N_2 = 15 (d_2 = 6.90 nm) and N_3 = 5 (d_3 = 6.10 nm) had a calculated coefficient of reflection in the range of angles of incidence 0–20°, varying within 43%–53%. Broadband mirrors made by magnetron sputtering in both cases provided a reflection coefficient of more than 30% (the difference from the calculated value is explained by the existence of roughness) with a greater smoothness of the measured reflection coefficient for the aperiodic structure. At the same time, the relative simplicity of manufacturing and certification of stack structures is noted, which makes them promising for the EUV range optics.

This paper discusses the application of such an approach to the development and manufacture of broadband mirrors for the KORTES complex, optimised for uniform reflection of radiation in the 17–21 nm wavelength range. At the same time, no a priori restrictions are imposed on the change in the period or the number of layers in the blocks and the layer thickness variation with depth. When numerically solving an optimisation problem, these parameters are considered as free in a wide range of values.

2. Optimisation of stack structure parameters

Consider broadband mirrors for the KORTES Sun Study Facility currently being developed for mounting on board the ISS. It was required to produce a broadband mirror that provides uniform reflection in the spectral range of 17–21 nm with a coefficient $R \geqslant 15\%$ under normal incidence of radiation and at a maximum deviation of the reflection coefficient within a plateau of less than 10%. The traditional pair of materials used for the λ = 12.5–35 nm range is Mo/Si, which is determined by its high optical contrast and high temporal stability along with the smoothness of the spectral dependences of the optical constants and low silicon absorption in this region.

Regardless of the type of the required structure (aperiodic or stack), mathematically, the optimisation problem is the minimisation of the functional

$$F = \int [R(\lambda) - R^{\text{targ}}]^{2m} \mathrm{d}\lambda, \qquad (1)$$

where R^{targ} is the target reflection curve that determines the height of the plateau of the reflection coefficient. The integral is calculated in the R^{targ} domain and is considered as a function of layer thicknesses. The calculation procedure consists of several iterations. The initial height of the plateau is selected at the level corresponding to the coefficient of the reflection of a periodic mirror, and gradually decreases until a satisfactory plateau smoothness is achieved.

In the case of aperiodic multilayer structures (AMS's), the parameters in the optimisation problem are the thicknesses of the Mo and Si layers in pairs h_i(Mo), h_i(Si), $i = 1, ..., N_{\text{AMS}}/2$. The total number of N_{AMS} layers is initially taken to be equal to that for a periodic structure and decreases during the optimisation process, namely, deep layers that do not have a noticeable effect on the calculated reflection coefficient are discarded. Thus, for the aperiodic structure there are several tens of fitting parameters. The roughness and density for the used pair of Mo/Si materials are known [25]; their dependence on the features of the technological process is well studied at the stage of manufacturing periodic multilayer structures. For the stack structure, the additional free parameters are N_i. The number of stacks was chosen to be three, and a protective Si film was deposited on top. Thus, we have only 10 parameters minimising functional (1). As shown below, in our case this is enough to solve the problem, although it is obvious that with an increased number of stacks, it is theoretically possible to obtain more perfect reflection curves, corresponding to an aperiodic mirror (in the limit for the number of stacks equal to $N_{\text{AMS}}/2$, and $N_i = 1$, we obtain an aperiodic structure with the number of layers N_{AMS}).

The optimisation was carried out using the differential evolution algorithm implemented in the Multifitting program developed by M.V. Svechnikov [26]. Unlike its widely used analogue IMD [27] (this work is cited 876 times, three million downloads of the program), Multifitting allows considering the number of periods in stacks N_i as a fitting parameter, which makes it possible to solve the optimisation problem in the class of stack structures. Another important advantage of the Multifitting program is the possibility of simultaneous fitting of several reflection curves, for example, intended for hard and soft X-ray ranges. In addition, the transition layers are represented as a linear combination of the simplest functions, including the error function that best describes the roughness, and the step function describing the stoichiometric layers in the transition region, for example, molybdenum silicide in the case of a multilayer Mo/Si mirror.

In the calculations, the widths of the transition regions (Mo-on-Si (1.2 nm) and Si-on-Mo (0.6 nm) [25]) and the tabulated density of the films were specified. Silicon wafers for the microelectronic industry with effective roughness σ_{eff} = 0.3 nm [28] in the range of spatial frequencies of 0.024–65 μm^{-1} were used as substrates.

In multiparameter problems, the question always arises about the optimality of the solution found. To increase the coverage of the parametric space, a series of automatic fittings were launched with random initial values of the desired parameters, which increases the probability of success, but does not guarantee the achievement of a global minimum. In the case of aperiodic structures, several implementations usually satisfy a given criterion. At the next stage, solutions are screened out from considerations of their resistance to small perturbations. Figure 1a shows the profile of an aperiodic Mo/Si mirror. The layer number is counted from the substrate, there is a Mo layer on the surface, and the total number of layers is 160. For stack Mo/Si structures (17–21 nm wavelength range), running the fitting procedure multiple times allows quick determination of optimal N_i values for the upper stacks (in the bottom stack the number of layers is chosen large enough, so that its thickness exceeds the radiation extinction length). The obtained thickness distribution of the layers of the stack structure over depth is shown in Fig. 1b. Note that, in contrast to the results of Ref. [20], the periods of the mirrors constituting the stack structure increase for layers approaching the substrate, and the condition $N_i d_i$ = const [20] obviously does not hold. Since in the soft and EUV ranges the absorption of radiation in the material plays a large role, a relatively thin mirror should be located on the surface of the stack structure, transmitting radiation to the lower layers, as in Refs. [21, 22].

The calculated reflection coefficients $R(\lambda)$ for the mirrors described are shown in Fig. 2. It can be seen that the aperiodic mirror certainly exceeds the stack reflection coefficient in terms of plateau smoothness, the average reflection coefficient for it in the region of λ = 17–21 nm being also slightly higher, 18% instead of 15.8%. However, turning to the manufacture of multilayer aperiodic structures, we find that this slight superiority is completely levelled by the complexity of their thickness calibration and subsequent synthesis. At the same time, stack-type structures make it possible to manage with just a few preliminary depositing.

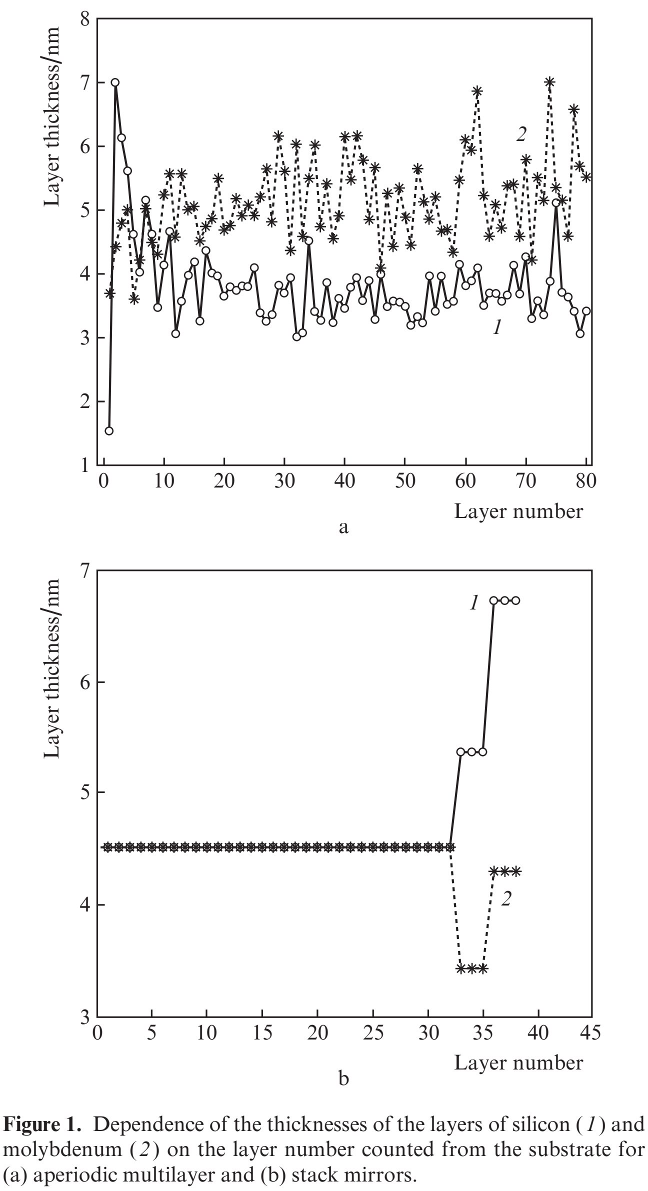

Figure 1. Dependence of the thicknesses of the layers of silicon (*1*) and molybdenum (*2*) on the layer number counted from the substrate for (a) aperiodic multilayer and (b) stack mirrors.

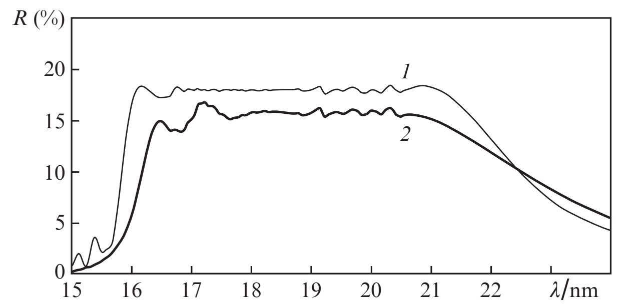

Figure 2. Calculated reflection curves of optimised multilayer Mo/Si mirrors of (*1*) aperiodic and (*2*) stack types. The widths of the Mo-on-Si and Si-on-Mo transition regions are 1.2 and 0.6 nm, respectively.

3. Experiment

Samples were prepared by the method of magnetron sputtering in an argon atmosphere at a pressure of 10^{-3} Torr. Details of the deposition of multilayer structures can be found in [29], but here we note that the thickness of the deposited film of a material is determined by two main parameters: the time the substrate passes over the target and the magnetron current (the higher the current, the stronger the material is deposited). In addition, there is also a weaker dependence of the thickness on the state of the walls inside the sputtering installation, the gas pressure in it and the degree of production of the target. With a significant difference in the thickness of the deposited layers, it is necessary to adjust both the velocity of passage of the substrate over the target and the current of the magnetron together. In the case of a conventional aperiodic mirror, this is not the case: if the current of the magnetron changes from layer to layer, this will lead to a noticeable instability of the process. Thus, there remains only one possibility of changing the layer thickness, namely, changing the velocity of passage of the substrate over the target. However, this speed can vary greatly for layers of different thickness, e.g., for depositing thick layers, it is necessary to make it very small. At the same time, the total sputtering time of the structure will also increase, which means that systematic deviations of the thickness of the deposited layers from the calculated ones will have a stronger effect. To overcome this problem, in Ref. [30] functional (1) is modified to minimise the difference in layer thicknesses in neighbouring 'periods' of aperiodic structure.

At the same time, first, the stack structure itself contains fewer layers. Second, after each stack is deposited, both the magnetron current and the substrate passage velocity can be changed, after which the next periodic structure is deposited. The total time of its deposition will be reduced, which means that the influence of negative effects will decrease.

The process of manufacturing any multilayer mirror is preceded by a calibration procedure, the purpose of which is to determine the growth rate of the film and the time it takes the substrate to pass over the target to achieve a specific film thickness. The fabrication of a quality aperiodic structure ideally requires a separate calibration for each individual thickness. If their number is large, the problem will be technologically solved over the course of several weeks, during which the quality of the calibration will significantly decrease due to the drift of the deposition parameters. In such a situation, it is usually done in the following way: they calibrate for several thicknesses, and for the rest, the depositing parameters are set from considerations of proportionality. Thus, in the case of aperiodic structure, an a priori error is introduced in the thickness of the layers in addition to their inevitable random variation due to microfluctuations of current, insignificant changes in the pressure of the working gas, etc.

The stack structure requires only three to six calibration deposits for periodic mirrors within its structure, and the calibration will be 'fair' and unpredictable, which will increase the accuracy of the synthesis of the final broadband mirror. If it is necessary to re-fabricate a multilayer mirror with the same parameters, the calibration procedure must be repeated, and therefore an adequate quick calibration also means good reproducibility of the result.

The fabricated broadband mirrors were certified for reflection on the reflectometer developed by the authors [31], in which the high-resolution Czerny–Turner spectrometer with a flat diffraction grating and two spherical collimating mirrors was used for monochromatisation of radiation. The source of X-ray radiation was a highly ionised plasma generated by the action of high-power laser radiation with an intensity of 10^{11}–10^{12} W cm^{-2} on a solid-state target. Scanning over spectrum was performed by rotating the diffraction grating, and calibration of the laboratory reflectometer was carried out by comparing the results with the measurement data of reflection coefficients of periodic mirrors using the synchrotron radiation source BESSY-II [32, 33].

In addition, the method of small-angle X-ray diffraction was used to study the structure of multilayer mirrors. The measurements were carried out in the range of 0–5° grazing angles θ of radiation with $\lambda = 0.154$ nm using a four-crystal high resolution PANalitycal X'Pert Pro diffractometer.

When solving the inverse problem of restoring the structure of a multilayer mirror in order to further adjust the sputtering parameters, an important advantage of the stack structures over the 'classical' aperiodic mirrors appears. From a mathematical point of view, this problem does not differ from the problem of optimisation of parameters of multilayer structures described above and reduces to minimising the same functional (1), in which experimentally obtained reflection curves $R_{\rm exp}(\lambda,\theta)$ are substituted for $R^{\rm targ}$. However, it has a greater number of optimisation parameters: in addition to the layer thicknesses, the length of the transition regions and the density of the films may differ from those specified and lead to a difference in the reflection curve from the calculated one. In the case of stack structures for solving the inverse problem, we have a well-developed methodology for determining the parameters of periodic mirrors.

4. Results and discussion

Figure 3a shows the reflection curves of the fabricated Mo/Si structure, obtained experimentally and reconstructed using the Multifitting program [26]. The thickness $h_{\rm Mo}$ of molybdenum and $h_{\rm Si}$ of silicon films deviated from the calculated (see Table 1) and corrections were made during the deposition times, which allowed for three iterations (it took about a week of work) to achieve a plateau of reflection coefficient in the region of 17–21 nm. The corresponding reflection curves can be seen in Fig 3b. Noteworthy is the sensitivity of the reflection curve to film thickness changes by units of angstroms.

In the case of aperiodic structures formed by tens and hundreds of films of different thickness, it is almost impossible to solve the inverse problem [10, 30]. The solution obtained (a set of film parameters as part of a multilayer mirror) is certainly not the only one; accordingly, it is impossible to implement a truly iterative procedure for synthesising multilayer mirrors, resulting in a finite number of corrections leading to a change in the reflection curve for the better.

Table 1. Reconstructed parameters (nm) of the stack Mo/Si structure.

Stack number i beginning from the substrate	Number of periods N_i	Calculation		Initial structure		First correction		Final correction	
		$h_{\rm Mo}$	$h_{\rm Si}$	$h_{\rm Mo}$	$h_{\rm Si}$	$h_{\rm Mo}$	$h_{\rm Si}$	$h_{\rm Mo}$	$h_{\rm Si}$
Si film	1		1.40		1.40		1.40		1.40
3	3	4.30	6.72	4.34	6.66	4.33	6.66	4.34	6.72
2	3	3.43	5.37	3.35	5.21	3.35	5.21	3.44	5.39
1	32	4.50	4.50	4.30	4.30	4.40	4.40	4.52	4.52

Figure 4 shows the measured reflection spectra of aperiodic and stack Mo/Si mirrors, the design of which is shown in Fig. 1. It can be seen that the real stack mirror has an even smoother profile of the reflection coefficient, which is associated with the adjustment of the film thickness in the sample manufacturing process. The average reflection coefficient of the stack structure is somewhat smaller; nevertheless, given the ease of fabrication, this type of broadband mirrors seems to be extremely efficient for practical use.

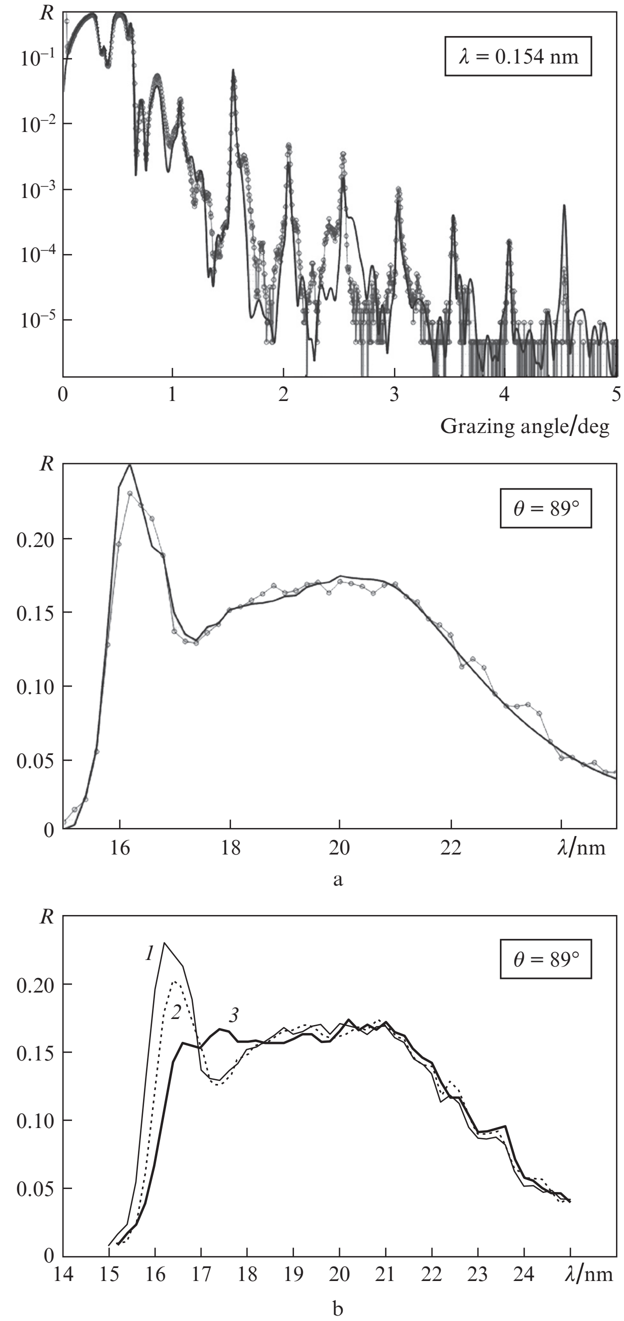

Figure 3. Reflection curves of stack Mo/Si structures obtained experimentally and reconstructed using the Multifitting program: (a) dependences $R(\theta)$ and $R(\lambda)$ for $\theta = 0.154$ nm and $\theta = 89°$, respectively (dots are experimental data, the solid curves are reconstruction results); (b) reflection spectra of synthesised structures (the figures on the curves correspond to the number of iteration.

5. Conclusions

We studied aperiodic broadband Mo/Si structures for the KORTES project, optimised for uniform reflection in the 17–21 nm wavelength range. It is shown that for stack structures consisting of three periodic mirrors deposited on each other with different parameters, it is possible to achieve uniform reflection at the level of 16% with good reproducibility of the results. Slightly yielding to aperiodic structure in the theoretical value of the reflection coefficient, the stack mirror is much more preferable from the point of view of its production rate

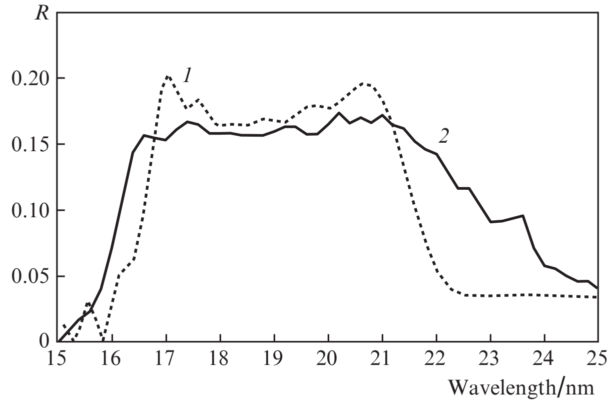

Figure 4. Measured reflection spectra of (*1*) aperiodic and (*2*) stack Me/Se mirrors.

and certification, which ultimately allows for correct adjustment of the deposition process and for a small number of iterations to reach the design parameters of the structure.

Acknowledgements. This work was supported by the Presidium of the Russian Academy of Sciences (Programme I.1 'Extreme Light Fields and Their Interaction with Matter'). The upgrade of the Multifitting software package for solving the optimisation problem in the class of stack structures was supported by the Russian Foundation for Basic Research (Grant for young scientists No. 18-32-00173).

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