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A Grid-Type Semitransparent X-ray Monochromator

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Abstract—A grid-type semitransparent X-ray monochromator is described. It can be used for splitting polychromatic beams, mixing beams of different spectral compositions, and radiation intensity monitoring. The monochromator is manufactured from a Si single crystal and contains a set of periodically spaced slit openings oriented in parallel to the diffraction plane. The data of reflectivity and transparency measurements obtained by scanning the diaphragm perpendicular to the measurement plane are presented and compared with the results of computer simulation.

The characteristics of an X-ray echelon–monochromator consisting of semitransparent plates of pyrolytic graphite arranged in series were studied in our work [1]. On the basis of those results, a two-wave X-ray reflectometer was constructed, which makes it possible to perform simultaneous measurements in different spectrum regions [2], for example, at K_α- and K_β-lines of the characteristic radiation of the X-ray tube anode. In this case, as was shown in [2, 3], when operating in the relative measurement mode, we managed to eliminate a number of basic errors of X-ray reflectometry associated with uncontrollable conditions of surface illuminance at small glancing angles of the probing beam and with a drift in the parameters of the X-ray equipment.

However, the measurement scheme with thin monochromator plates proposed in [2] can be efficiently applied at wavelengths of <0.5 nm, because, at softer X-rays, the mass attenuation factor of radiation abruptly rises, thus making it impossible to transmit X-rays through plates with a thickness of $\geqslant 10$ μm used in the echelon [1, 2]. Attempts to reduce the thickness of crystalline samples with areas of ~1 cm^2 to ~1 μm lead to their uncontrollable deformations and destruction, as a result of internal stresses and nonuniform chemical and mechanical actions on samples during their treatment.

Another problem is related to the fact that, for precise measurements of periodic-structure parameters and spectral composition, a narrow spectrum region should be selected from the incident radiation, for example, for reliable discrimination of the doublet of frequently utilized K_α characteristic lines. Yet, this remains unaccomplishable in practice with pyrographite because of the imperfections in its crystal structure.

This work describes grid-type semitransparent monochromators that allow to avoid the above drawbacks.

Let us consider a typical measurement scheme (Fig. 1), in which the linear focus of the X-ray tube d_f in size is located at a distance L from the sample's rotation axis O_s parallel to the linear focus ($L >> d_f$). Collimation slits form a ribbon beam with a width near the O_s-axis equal to d_f. Let us draw a measurement z-axis parallel to O_s. In the absence of a monochromator, the expression for the flux density through an area ΔS on the O_s-axis in the spectral band $\Delta\lambda$ created by a focus element Δz can be written in the form

$$\Delta^3 P = J(\lambda, \mathbf{k}, z)|(\mathbf{s}, \mathbf{k})|\Delta\Omega\Delta z\Delta\lambda, \quad (1)$$

where $J(\lambda, \mathbf{k}, z)$ is the spatial spectral–angular density of the X-ray flux, $\Delta\Omega$ is the solid angle subtended by the area ΔS with the vertex on a radiating element of the focus, and $\mathbf{k}$ and $\mathbf{s}$ are the unit vectors directed from the radiating focus element to the center of ΔS and normal to its surface, respectively.

Assume that a thin plate-type crystal–monochromator (CM) is set on the beam path between the focus and sample axis. The CM has several slit openings positioned over the entire cross section of the ribbon beam with a period d and oriented perpendicular to the O_s-axis. It is then obvious that, at small d, by selecting the appropriate dimensions of discretization elements,

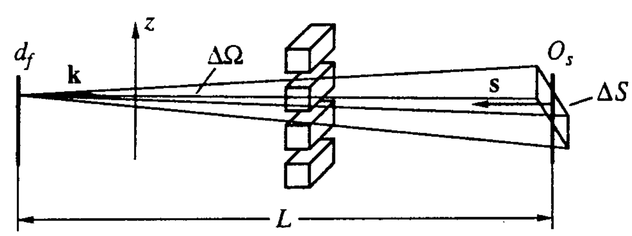

Fig. 1. Geometry of ray paths from the focus to the sample axis through the grid-type structure within a solid angle ΔΩ.

expression (1) for the flux density per area ΔS on the O_s-axis can be written in the form

$$\Delta^3 P = b/dJ(\lambda, \mathbf{k}, z)|(\mathbf{s}, \mathbf{k})|\Delta\Omega\Delta z\Delta\lambda, \qquad (2)$$

where b is the slit width. The ratio b/d is the transparency factor and $b/d\ \Delta\Omega$ can be considered as the effective solid angle of radiation extraction from the surface of the emitting element of the focus. Thus, after a grid-type monochromator with a period of slits $d \longrightarrow 0$ is introduced into the system, the flux density distribution in the zone of the sample axis remains unchanged to within a constant factor. This is valid, if $a\tan\varphi/\sin\theta \ll b$ (a is the CM thickness, φ is the maximum divergence angle in the ribbon beam plane, θ is the Bragg rotation angle), because, in the case of violation of this condition, the CM may substantially modify the angular distribution of radiation.

Manufacturing, by modern photolithography methods, a periodic slit structure with a spacing $d \ll a$ in commercial disk single-crystal wafers with a thickness a = 0.3–0.4 mm is difficult in practice, because of the inevitable and uncontrollable etching of side walls of openings during chemical treatment. In order to solve this problem, we chose the mechanical cutting technique with a diamond disk. Two specimens of a grid-type monochromator in the form of a comb were manufactured (Fig. 2). The first one with the dimensions 24 × 12 × 4 mm was cut directly from a single-crystal Si boule oriented along the [111]-direction. The plate was oriented by using an X-ray diffractometer, which ensured an angular deviation of the (111) crystallographic plane from the crystal surface of ~10′. In order to remove the disturbed layer, the initial plate was chemically etched after cutting. The second sample was manufactured by bonding a Si(111) wafer obtained by chemicomechanical treatment to a graphite substrate by using an epoxy resin. The initial thicknesses of the substrate and Si wafer were 5 and 0.35 mm, respectively. The slits were cut by the inner edge of the diamond disk from the side of the lateral surface of the wafer with a constant spacing d = 500 μm equal to the double disk thickness. The area of the grid zone on the reflecting surfaces of both samples was 6 × 12 mm (Fig. 2). In order to reduce the effective scattering area, the thickness of the graphite substrate of the second sample was reduced to 2 mm; no repeated chemical etching of Si was carried out after cutting.

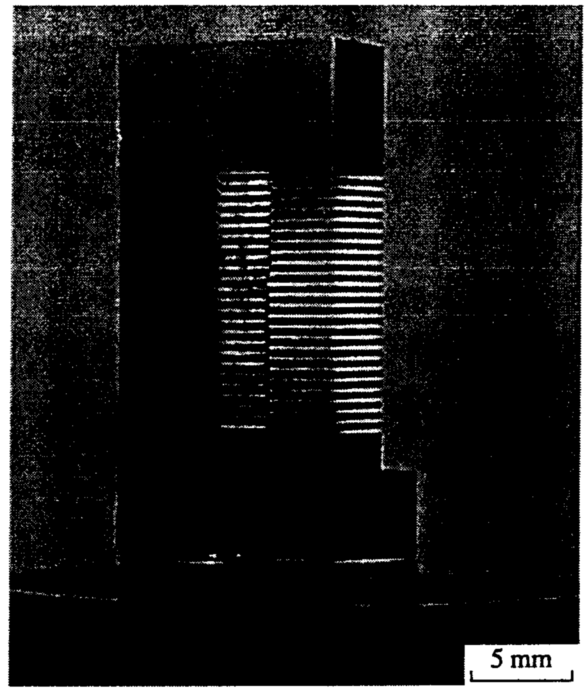

Fig. 2. General view of the grid-type monochromator manufactured from a silicon single crystal.

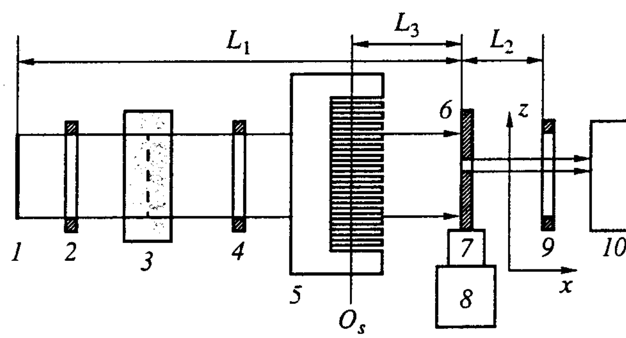

Fig. 3. Arrangement of the elements of the experimental setup in the axial plane: (*1*) focus of the X-ray tube; (*2*, *4*, *9*) vertical slits; (*3*) primary monochromator; (*5*) grid-type monochromator; (*6*) horizontal slit diaphragm; (*7*) movable rod; (*8*) linear scanning unit; and (*10*) detector.

Figure 3 shows a diagram of the ДРОН-3М-diffractometer-based experimental system for measuring the parameters of a diffraction-reflected beam and a beam transmitted through the grid-type monochromator. A БСВ-22 copper-anode tube was used as an X-ray source. Measurements were executed with an initial Si(111) CM *3* in polychromatic radiation. The total path length from the focus to the grid-type monochromator *5* was 330 mm. The linear scanning unit *8*, which was located in front of or behind the monochromator *5*, provided the motion of a 50-μm-wide slit diaphragm *6* with a velocity of 400 or 100 μm/min in the direction of the z-axis perpendicular to the measurement plane. Data acquisition was controlled by a computer.

Figure 4 shows $\theta - 2\theta$ diffraction rocking curves for a monochromator from a monolithic Si plate. The curves were measured in the two-crystal spectrometer mode with grid and continuous CM areas. Their comparison shows that the half-widths of the diffraction curves from these areas are almost identical, which confirms the possibility of obtaining a grid-type CM (ensuring the high spectral resolution) by using the

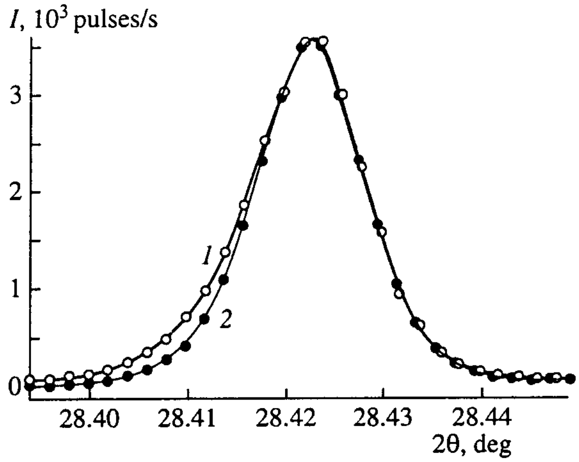

Fig. 4. Diffraction rocking curves (θ – 2θ) of the grid-type monochromator for the Cu$K_{\alpha 1}$-line: (*1*) grid area (scale factor of 2.2); and (2) continuous area.

mechanical cutting technique. A slight increase in the intensity of the smaller angle wing of the diffraction curve is associated apparently with partial penetration of radiation into the side walls of the slit openings, which is equivalent to a shift of a part of the reflecting crystal surface along the normal with respect to the focusing circle.

Figure 5 shows the dependences of the transmitted $I_T(z)$ and reflected $I_R(z)$ radiation intensities on the position of diaphragm *6* moving along the *z*-axis for a monolithic CM and a CM on a graphite substrate. The diaphragm was placed at a distance of 20 mm in front of the grid-type monochromator; no Soller collimator was used. An abrupt decrease in $I_R(z)$ at $z > 5$ mm for the substrate-based monochromator is explained by an angular departure of some comb's elements as a result of uncontrollable deformations arising during cementing and mechanical treatment. This is confirmed by the possibility of setting to the diffraction position various components of the substrate-based CM by rocking it along the sample axis. All elements of the grid structure of the monolithic-Si CM keep the orientation of the initial wafer.

An increase in the recorded $I_T(z)$ and $I_R(z)$ intensities and decrease in the degree of intensity modulation observed during motion of the horizontal diaphragm from the beam edge to its center are explained as follows. In the axial plane, which runs through the sample rotation axis and entrance slit, the scanning diaphragm can be considered as a pinhole camera, which creates an inverted image of the X-ray-tube focus in the plane perpendicular to the forward beam. If this plane coincides with the entrance slit, then the magnification factor is $M = L_2/L_1$, where L_1 and L_2 are the distances from the focus to the diaphragm and from the diaphragm to the entrance slit, respectively (Fig. 3). When the diaphragm scans along the *z*-axis parallel to the sample rotation axis, the focus image moves over the area of the entrance slit of the detector and, at a certain *z* value, leaves the area of the slit opening. This fact accounts for a monotonic fall in the average intensity at the slit edges. In this case, a shift in the focus image outside of the slit means a decrease in the focus d_{fv} size visible within the detector area. This formally corresponds to a decrease in the effective angular beam divergence in the vertical plane. If the distance from the scanning diaphragm to the monochromator is $L_3 << L_2$, then it is necessary to take into account the finite size of the diaphragm aperture d_s; therefore, the effective size of the focus image in the image plane coinciding with the monochromator rotation axis is $d_i = d_s + d_{fv}L_3/L_2$. It is obvious that, at $d_i < b$ ($b = d/2$), the signal intensity modulation depth in the transmitted and reflected radiation reaches a maximum, as the slit moves from the beam axis to its periphery. These considerations are confirmed well by the results of mathematical simulation of reflection and transmission of X-rays by the grid-type CM (Fig. 6). These calculations take into account the blurring of the focus image boundaries resulting from scattering at edges by introducing a smoothing function of the $\exp[-(\alpha\xi)^2]$ type, where ξ is the current coordinate on the *z*-axis and $\alpha \approx d_i/2$ is a fitting parameter.

The above considerations can be utilized for analyzing the radiation intensity distribution behind the monochromator, because, with respect to the incident and reflected beams, the working monochromator zone is a set of periodically spaced slit diaphragms and a set of periodically spaced reflectors with the reflectivity $R(z)$. Then, for a grid-type CM installed in front of a sample and used as a monochromator in a two-crystal spectrometer in order to provide a uniform radiation-intensity distribution on the sample surface, the condition

$$Md_f >> d \quad (3)$$

should be satisfied.

In the measurement schemes of commercial instruments, the ratio between the sample–monochromator and monochromator–focus distances is usually 1.2–2.5. Since a typical focus size is 10 mm, condition (3) is satisfied well at a selected period $d = 0.5$ mm. Note that a nonuniform distribution of the detected radiation on the *z*-axis does not lead to an additional measurement error. Such an edge effect actually occurs upon introduction of the Soller collimator intended for limiting the angular divergence in the direction perpendicular to the measurement plane. When the grid-type CM and Soller collimator are used jointly, in order to minimize the intensity loss, it is necessary to have equal spatial periods for the CM slits and collimator plates. Note that the collimator plates for the beams transmitted and reflected by the CM must be shifted by $d/2$.

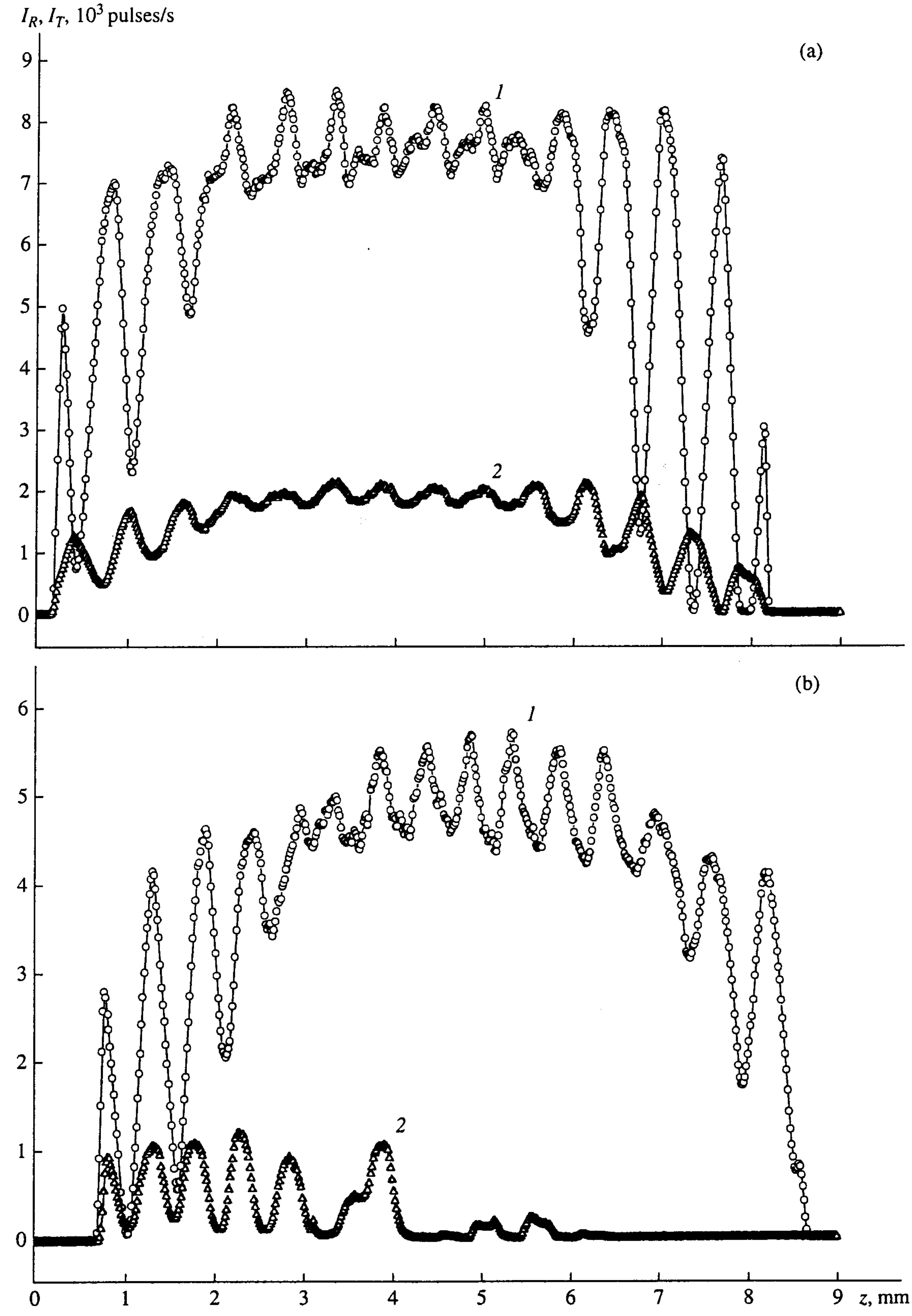

Fig. 5. Experimental dependences of the (*1*) transmitted and (*2*) diffraction-reflected radiations on the position of the scanning diaphragm for the grid-type CM: (a) monolithic Si wafer; and (b) Si wafer on a graphite substrate. The distance between the scanning diaphragm and sample axis is $L_3 = 20$ mm.

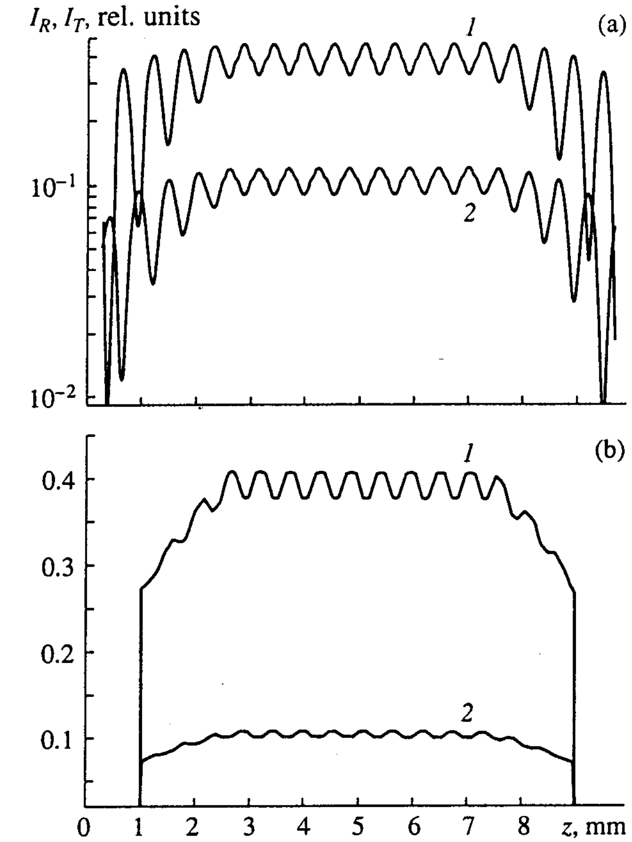

Fig. 6. Calculated dependences of the (*1*) transmitted and (*2*) diffraction-reflected radiations on the position of the scanning diaphragm for the grid-type CM with a local reflectivity of 0.2: (a) L_3 = 14 mm; and (b) L_3 = 47 mm.

The replacement of a single crystal by an artificial multilayer structure, in which the medium permittivity changes periodically, allows one to obtain a peak reflectivity of >0.5 for soft X-rays and simultaneously to select a narrow spectral band [4, 5]. The use of such a structure requires no basic changes in the shape of the grid-type CM. Only the general requirements imposed on multilayer X-ray mirrors need be fulfilled. These stipulate that the departures of the reflecting surface and the law of permittivity as a function of depth from the specified shapes, as well as the interface roughness parameters, should be within acceptable bounds.

The type of semitransparent monochromators proposed can be used in measuring X-ray devices, in which it is expedient to utilize several operating wavelengths. They can also be used to monitor the X-ray intensity and, especially, beams of pulsed X-ray lasers [6, 7], because a standard scheme for a quasi-isotropic radiation source with a part of the beam diverted to a side channel is inapplicable.

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