Obrabotka metallov (tekhnologiya, oborudovanie, instrumenty) = Metal Working and Material Science. 2024 vol. 26 no. 3 pp. 208–233
ISSN: 1994-6309 (print) / 2541-819X (online)
DOI: 10.17212/1994-6309-2024-26.3-208-233

NSTU NETI

Obrabotka metallov - Metal Working and Material Science

Journal homepage: http://journals.nstu.ru/obrabotka_metallov

Methods of synchrotron radiation monochromatization (research review)

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ARTICLE INFO

Article history:
Received: 24 May 2024
Revised: 10 June 2024
Accepted: 28 June 2024
Available online: 15 September 2024

Keywords:
Synchrotron radiation
X-ray diffraction
Monochromators

Acknowledgements
Thanks to Trofimov Roman Vladimirovich for providing figure number 29 in this paper.

ABSTRACT

The paper presents an analysis of studies related to the monochromatization of X-ray radiation (*XR*) at synchrotron radiation sources. A review of monochromators based on of X-ray diffraction on crystals is given, and the peculiarities of their technical realization are considered. The ideas about monochromators which include multilayer structures are examined. The authors also study technical problems arising during designing devices and its possible solutions. **Introduction.** The possibilities of using X-rays in scientific research are described. The high efficiency of synchrotron radiation sources is noted, and its characterization is given. **Elementary information about diffraction of X-rays.** The paper describes the properties of X-ray radiation and the possibilities of its using while studying various materials. **Degree of monochromaticity.** The degree of monochromaticity is an important characteristic of the synchrotron radiation (*SR*). Depending on the width of the wavelength band, "white", "pink" and monochromatic beams are distinguished. Monochromators based on multilayer structures are used to obtain "pink" beams. Monochromatic radiation is formed using monocrystals. When conducting experiments with "white" beams, the monochromator is not used. The authors also describe the factors that violate the ideal fulfillment of the *Wolf-Bragg* condition and affect the degree of monochromaticity (heat, vibration). The reflectivity values at different beam grazing angles are noted to have different widths. **Monochromators based on multilayer structures.** Periodic structures combining thin layers of two heterogeneous materials make it possible to obtain "pink" beams. The wavelength bandwidth of such devices is one or two orders of magnitude greater than that of monochromators using crystals as optical elements. **Configurations and geometry of optical elements.** There are two types of X-ray diffraction on a crystal: *Bragg* and *Laue* diffraction. *Bragg* diffraction refers to reflective geometry, *Laue* diffraction is based on the passage of beams through the crystal. The section provides examples of monochromators with different configurations of crystals and X-ray mirrors. The arrangement of optical elements in a monochromator plays an important role in the geometry of the beam path. When designing monochromators, it is necessary to take into account the methods of fixation and orientation of the rotation axes of optical elements. Examples of monochromators with different configurations of crystals and X-ray mirrors are given. **Focusing monochromators.** It is possible to provide sagittal and meridional types of deformation by bending the optical element of the monochromator. Due to the curved crystal surface the beam is not only monochromatized but also subjected to focusing. Modern focusing monochromators are equipped with adaptivity elements allowing it to change the radius of curvature of the optical element. Examples of practical realization of such monochromators are presented. **Thermal load of *SR* on optical elements.** The *SR* is characterized by high brightness and a wide spectrum of emitted wavelengths. While operating optical elements of *SR* stations absorb a large amount of thermal power. The problems of heat dissipation have a fundamental influence on the quality of synchrotron radiation monochromatization. **Additional information about monochromators.** Examples of special design solutions for monochromators are given. **Conclusion.** The design of monochromators is relevant to the synchrotron radiation source 4+ "*SKIF*" under construction in Novosibirsk.

For citation: Burdilov A.A., Dovzhenko G.D., Bataev I.A., Bataev A.A. Methods of synchrotron radiation monochromatization (research review). *Obrabotka metallov (tekhnologiya, oborudovanie, instrumenty) = Metal Working and Material Science*, 2024, vol. 26, no. 3, pp. 208–233. DOI: 10.17212/1994-6309-2024-26.3-208-233. (In Russian).

Introduction

Synchrotron radiation (*SR*) has found wide application in various branches of science, including materials science, physics, chemistry, crystallography, medicine, biology, mineralogy, etc. To form ideas about the structure of the object under study, it is necessary to "look inside the material" that can be realized with research methods based on using synchrotron radiation sources (*SRS*). In a way, the experimental setup

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at the *SRS* can be treated as a kind of "powerful microscope". The spectral range of synchrotron radiation energies is very wide (from 10 eV (and less) to 100 keV (and more)). At the same time, different methods of research realized at *SR* sources require using photons of different energies (or wavelengths). Accordingly, researchers need to select from a wide spectrum of radiation that part of it which is the most important for the technique. In most cases, the range of X-ray radiation (*XR*) should be highlighted from the broad spectrum of *SR*. The wavelengths are comparable to the size of atoms, which allows researchers to analyze the atomic crystalline structure of solids, the near-order of liquids and amorphous objects. Radiation corresponding to the X-ray range of electromagnetic waves is characterized by energy values ranging from 1 to 100 keV.

A "white" beam (i.e., radiation with a wide range of wavelengths) emerges from the insertion devices of the storage ring in which a stream of elementary particles, usually electrons or positrons, circulates. However, for most experiments, there should be a beam with a "narrower" range of parameters necessary to solve the problems set by a researcher. In most cases, monochromatic radiation is used at beamlines, and its formation is provided by special devices called monochromators. Those located at beamline together with slits, filters, focusing systems form radiation with the required characteristics. From a technical point of view, monochromators are one of the most complex and high-tech devices for beamline. The production of monochromators belongs to the critical technologies that ensure the effective use of *SR* for studying the structure of materials. As a rule, the main unit of a monochromator is a pair of crystals that allow to extract from the whole *SR* spectrum a diffracted beam corresponding to a narrow band of wavelengths and to direct it to a specimen. Passing through the monochromator, the incoming beam which includes the entire spectrum of radiation generated by the insertion device is converted into monochromatic or "pink" radiation. These types of radiation differ from each other by the degree of monochromaticity which is understood as the ratio $\Delta\lambda/\lambda$, where λ and $\Delta\lambda$ are the peak value of the wavelength and spectral width of the radiation that has passed through the monochromator, respectively. Synchrotron radiation corresponding to the ratio $\Delta\lambda/\lambda = 10^{-4}\div10^{-3}$ is called monochromatic [1]. To solve some problems, "pink" radiation is also used, its degree of monochromaticity is $\Delta\lambda/\lambda = 10^{-2}\div10^{-1}$ [2]. When conducting experiments with a "white" beam, a monochromator is not necessary. Thus, for example, the *Laue* method assumes the exposure of a fixed single crystal to "white" (continuous) radiation. The presence of a wide range of wavelengths in the X-ray spectrum makes it possible to fulfill the Wolf — Bragg condition, i.e., to manifest the effect of diffraction of X-rays. If it is a question of conducting experiments by methods associated with using "pink" and monochromatic radiation, various types of monochromators are used; its features are discussed in this paper.

The principle of monochromators operating is based on diffraction of X-rays. The features of diffraction on crystals were described by *Bragg* and *Wolf* in 1913. Based on the condition now called the *Wolf – Bragg* law, "white" radiation hitting a crystal can be decomposed into beams characterized by a narrow band of wavelengths. Depending on the technique and the objectives, different wavelength ranges may be required for experiments. According to the *Wolf – Bragg* law, extracting a given wavelength (and thus photon energy) requires a certain angle of incidence of radiation on the crystal, which is controlled by the goniometer, one of the most important mechanisms of a monochromator. In addition to the goniometer, which allows a monochromator to be adjusted to different energy levels, the device includes such elements as vacuum pumps, a cooling system and sensors that ensure the operation of all devices.

While designing and subsequent operating a monochromator, it is important to have a quantitative understanding of the intensity and brightness of the beam, which are directly dependent on crystal positioning, technical errors and deviations specific to the instrument. In addition, it is important to understand the properties of the radiation source (in the context of this paper, the source is understood as rotating magnets or insertion devices located on the storage ring of the synchrotron). Fig. 1 shows the schematic diagram of a specialized *SRS* [3]. It consists of such elements as an electron gun based on the thermoelectron emission effect (**1**), a linear accelerator of electron (linac) (**2**), a booster (**3**), bending magnets (**4**), radiofrequency resonators (**5**), insertion devices (undulators, wigglers) (**6**), a beamline (**7**), a front-end (**8**), an optical hutch – the first room with optical devices (**9**), and an experimental hutch (**10**).

The electron gun (**1**) emits electrons and leads it to the linear accelerator (**2**), where the particles are accelerated according to the resonance principle, passing through the gaps of a high-frequency electric

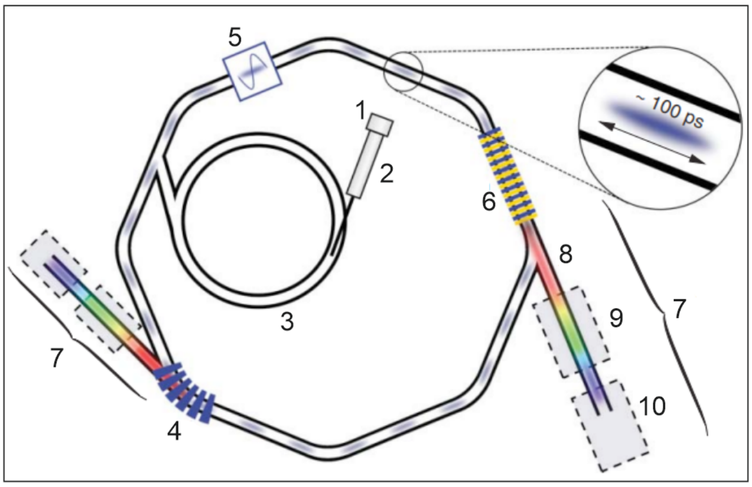


Fig. 1. A conceptual sketch of the *SRS*. Taken from [3]

field. The beam leaving the linear accelerator is directed to the booster (3), where electrons are accelerated to relativistic velocities. Then the beam passes to the storage ring, the main elements of which are bending magnets (**4**), which form a closed trajectory of electron motion, radio-frequency resonators (**5**), which replenish the beam energy that is spent when emitting *SR*, and insertion devices (**6**). The bending magnets and insertion devices are used to generate synchrotron radiation, which is directed to the beamline (**7**) and, after passing through the optical hutch (**9**), enters the experimental hutch of the station (**10**) with the analyzed object inside it.

Synchrotron radiation is magnetically induced electromagnetic radiation emitted by relativistic charged particles (traveling at a radiation velocity close to the speed of light), which are forced by a constant magnetic field to move in circular orbits. Devices generating synchrotron radiation can be bending magnets, wigglers, or undulators. The magnetic field of these devices, due to the *Lorentz* force, leads (**1**) to a change in the trajectory of electrons and (**2**) to the formation of synchrotron radiation. The synchrotron radiation directed tangentially to the storage ring enters the beamline, moving along which it is brought to the specimen located in the experimental hutch (**10**).

The optical circuitry of the station includes many devices performing various functions. These include optical elements that change the geometric parameters of the beam (slits, collimating and focusing lenses, X-ray mirrors, etc.), filters, and windows that separate vacuum volumes. The optical scheme should also include beam monitoring elements, which can be divided into two groups. The first group includes detectors that determine the beam position, and the second group includes detectors that record the intensity and spectral composition of the radiation.

The field of applying synchrotron radiation is large and for this reason the methods of experiments may differ significantly. The necessity of solving different kinds of problems leads to the development of beamlines that differ in the set of their elements. Monochromators are one of the key elements of beamlines, which are spectral optical-mechanical devices that allow separating narrow bands of radiation from a wide range of wavelengths.

The monochromators used at *SRS* include units that are identical in purpose. However, the monochromators differ from each other structurally. Technical features of the monochromators are determined not only by the tasks solved at the beamlines, but also by the magnitude of the incoming heat flux, the cooling system of crystals, and the accuracy of their adjustment.

Elementary information on X-ray diffraction

In 1985, *Wilhelm Conrad Röntgen*, conducting experiments with the *Crookes* tube, discovered a previously unknown radiation, which he called X-rays. Fig. 2 shows a scale of wavelengths and frequencies of electromagnetic radiation, and highlights the conditional range corresponding to X-ray radiation, determined at different periods of time based on the works of *K. Röntgen*, *M. Laue*, *C. Barclay*, *D. Thompson*, *G. Moseley*.

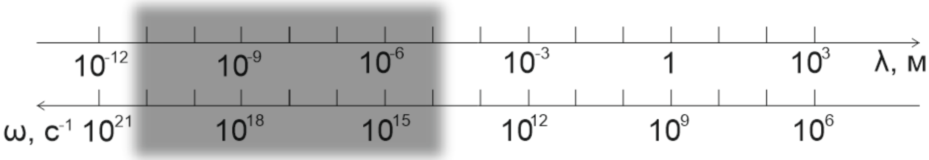


Fig. 2. The scale of electromagnetic radiation. The area of synchrotron radiation is marked in gray

The X-ray region is often divided into ranges of hard (0.1 < λ < 10 Å), soft (10 < λ < 300 Å), and ultrasoft (300 < λ < 1,000 Å) radiation [34]. This division is conventional, but it is important from the point of view of the physics of the monochromator process because these ranges are characterized by different refractive indices, absorption coefficients, and wave polarization features. X-rays are distinguished from visible light by its ability to penetrate deep into substances. The properties of X-ray radiation are straightness of propagation at the speed of light, refraction at interfaces, reflection and scattering at obstacles, interference and diffraction, polarization when scattering or passing through a substance, absorption by substances, and the ability to cause the photoeffect [3, 4].

It has been noted that the principle of operation of monochromators is based on diffraction of X-rays. The first experiments related to this physical phenomenon were performed in 1912 by *Max von Laue* with his young employees *Knipping* and *Friedrich*. A simple condition allowing to determine the angle corresponding to the diffraction maximum was obtained by the English physicist *Bragg* [3] and, independently, by the Russian scientist *Wolf*:

$$2d_{hkl} \sin\theta = n\lambda \,. \qquad (1)$$

Expression (1), referred to as the *Wolf – Bragg* condition in the Russian-language literature, shows how the angles **θ** at which constructive interference of X-ray radiation scattered by a crystal occurs are related to the X-ray wavelength **λ** and the distance between atomic planes $\boldsymbol{d_{hkl}}$ (Fig. 3). The parameter ***n*** in formula (1) represents the order of reflection. The *Wolf – Bragg* condition serves as a theoretical basis for the design of any monochromator for a synchrotron radiation source.

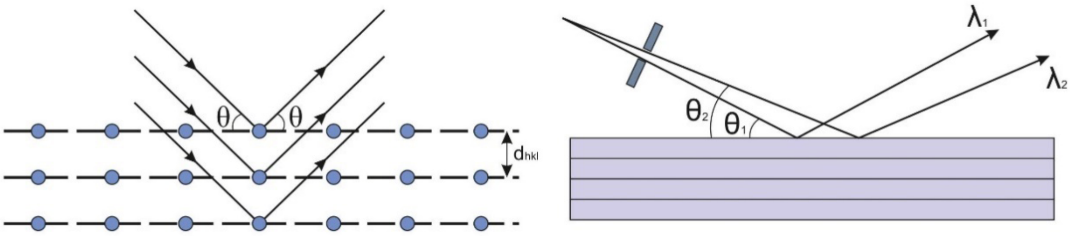
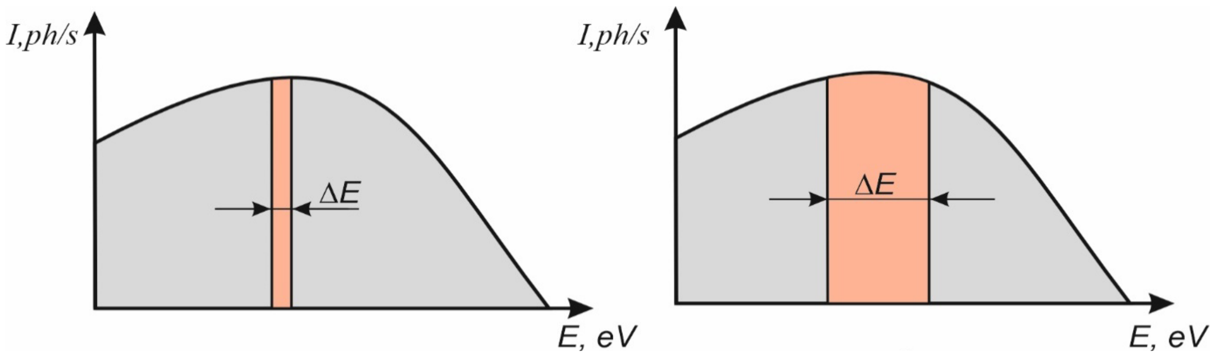


Fig. 3. Schematic of X-ray diffraction on the atomic planes of the crystal and illustration of the angular divergence of the beam

Proceeding from the idea that a crystal could be represented as a set of parallel planes with the same $\boldsymbol{d_{hkl}}$ distance between them, *Bragg* and *Wolf* believed that X-rays falling on a crystal would diffract only if the grazing angle **θ** satisfied the condition (1). It means that if a polychromatic ("white") beam falls on an ideal nonabsorbing crystal of infinite depth, the band of diffracted radiation will be infinitely narrow [3, p. 149]. Such a diffraction mode is ideal, and practically unattainable. In fact, a spectral band of some width **Δλ** is formed by a monochromator. The causes of broadening are related to thermal deformations of the crystal, rigidity of its mounting, imperfection of the crystal structure, vibrations from the coolant flow, vibrations of the Earth's crust, and so on. The mentioned circumstances complicate the description of the X-ray diffraction on the crystal. Thus, the ideal variant of fulfillment of the *Wolf – Bragg* condition is not realized in practice and some scattering of radiation actually occurs (Fig. 3 *b*).

Degree of monochromaticity of radiation

"White" radiation entering the monochromator includes a wide range of wavelengths, which is determined by the radiation source (for example, a bending magnet or wiggler). A monochromator, as noted earlier, should select a narrow band of wavelengths from this range (Fig. 4).



a *b*

Fig. 4. Dependence of *SR* photon flux density on radiation energy (***E***):
a – monochromatic radiation (degree of monochromaticity $\Delta E/E = \Delta\lambda/\lambda = 10^{-4}$–$10^{-3}$); *b* – "pink" radiation ($\Delta E/E = \Delta\lambda/\lambda = 10^{-2}$–$10^{-1}$)

The ratio of the select band width **Δλ** to the peak value of the transmitted radiation wavelength **λ** determines the degree of monochromaticity. By differentiating the *Wolf – Bragg* dependence, we obtain:

$$2d_{hkl} \cdot \cos\theta \cdot d\theta = d\lambda. \quad (2)$$

Substituting $2d_{hkl}$ with λ/sinθ, we have:

$$\frac{\Delta\lambda}{\lambda} = \text{ctg}\theta \cdot \Delta\theta, \quad (3)$$

where θ is the slip angle; Δθ is the deviation of the slip angle caused by thermal deformation and vibration of the crystal.

In some cases, it is convenient for the specialists dealing with the diffraction of X-rays to operate not only with the value of the radiation wavelength **λ**, but also with the value of the photon energy **E** corresponding to a certain wavelength. It is not difficult to show that:

$$\frac{\Delta E}{E} = \frac{\Delta\lambda}{\lambda} = \text{ctg}\theta \cdot \Delta\theta. \quad (4)$$

Thus, the degree of monochromaticity is determined by the grazing angle **θ** and the value of the deviation **Δθ** [4]. In an ideal case, the value of the diffraction angle **θ** on the crystal does not change during the experiment. However, in fact, as we have noted, this assumption is not fulfilled. One of the most important reasons for the deviations observed in the experiment is related to the influence of heat flow, which results in heating of some local zone of the crystal (Fig. 5, highlighted by a circle). The result of such an effect, accompanied by thermal expansion of the material, is a distortion of its crystal structure [5].

The thermal effect leads to deformation, first of all, of the first crystal of the monochromator, thus causing a deviation from the *Wolf – Bragg* condition. The result of heating is a local distortion of the crystal lattice of the material (increase in the interplanar distance $\boldsymbol{d_{hkl}}$). The heat accumulated in the crystal is the cause of material expansion, formation of a convex zone and scattering of radiation at different angles. It should be noted, however, that the change in the $\boldsymbol{d_{hkl}}$ parameter can be associated with the quality of manufacturing the crystal, and the presence of defects of various nature in it. Thus:

$$\theta = \theta_d + \delta\theta; \quad (5)$$

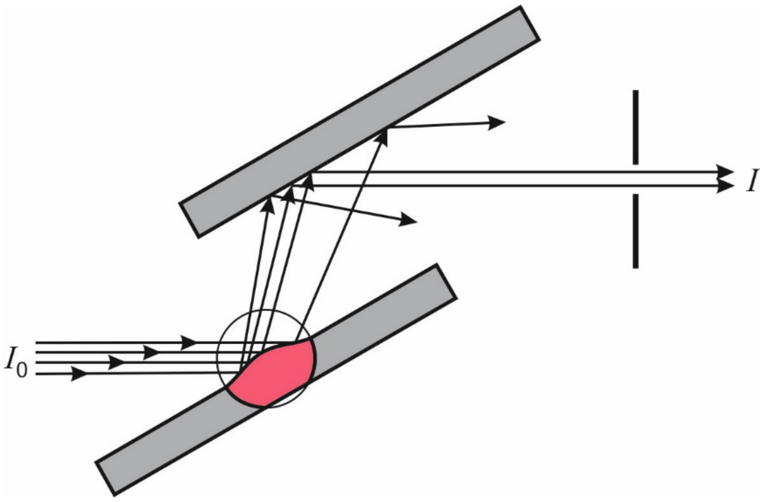


Fig. 5. Demonstration of the effect of thermal expansion of the local crystal zone (highlighted with a circle), caused by the thermal effect of synchrotron radiation, on the diffraction of X-rays. According to [5]

$$d_{hkl} = d_d + \delta d_{hkl}, \quad (6)$$

where θ_d and d_d are the diffraction angle and the interplanar spacing for the case corresponding to the ideal fulfillment of the *Wolf – Bragg* condition; $\delta\theta$ and δd_{hkl} are the possible errors of the corresponding quantities.

Deviations from the ideal operation mode of the monochromator, caused by the expansion of the crystal during its heating, as well as by vibration due to the turbulent flow of the cooling liquid, or transmitted from the foundation, are the reasons for the device to transmit diffracted waves corresponding to a certain range of energy. This means that the radiation passed through the monochromator is a beam characterized by a range of wavelengths $\mathbf{\Delta\lambda}$. The reasons mentioned above affect the grazing angle $\boldsymbol{\theta_d}$ and the interplanar spacing $\boldsymbol{d_d}$, the values of which are related by condition (1) to a specific wavelength corresponding to a specific radiation energy.

Thermal heating and vibrations of various nature are negative factors that are manifested directly during operation of the monochromator. At the same time, there are additional factors that decrease the degree of monochromaticity of radiation caused by imperfections in crystal structure (non-planarity of the outer surface, mosaicity of the material). Mosaicity is one of the possible defects of crystal structure. A monocrystal installed in a monochromator consists of many "blocks" disoriented relative to each other at small angles (Fig. 6). The mosaicity of the crystal, as one of the factors determining the degree of monochromaticity of radiation, was analyzed by *Darwin* as early as 1922 [6]. Nonplanarity is understood as the distance between the real and ideal surface of a crystal plate. Roughness (depressions and protrusions) that has appeared at the last stage of surface processing also has a negative effect on the monochromaticity of radiation.

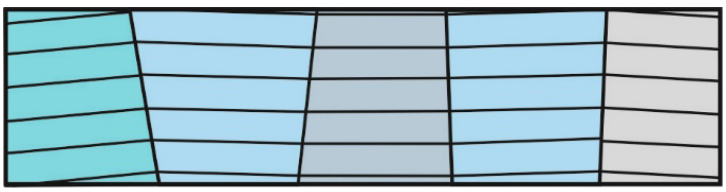


Fig. 6. Demonstration of crystal mosaicity on the example of five blocks disoriented relative to each other at small angles

When solving a certain class of problems, it is sufficient (and sometimes necessary) to provide a degree of monochromaticity corresponding to the so-called "pink" beam (Fig. 4 *b*) [7]. The spectral width of the "pink" radiation is 1÷3 orders of magnitude greater than that of monochromatic radiation. To form a "pink" beam, monochromators equipped with X-ray mirrors are used, which are multilayer structures in the form of alternating pairs of layers made of different materials characterized by different refractive indices (Fig. 9).

An important characteristic of a monochromator is related to its reflectivity (Fig. 7). This parameter is described by the *Darwin* curve or, as it is also called, the rocking curve, which is a function of the angular displacement from the *Bragg* angles for the main X-ray emission [6, 8]. "White" beam of synchrotron radiation falls on an optical element at some angle $\boldsymbol{\theta}_d$. According to condition (1), monochromatic radiation of the desired wavelength is "extracted" from it. The rocking curves illustrate the dependence of the reflectivity $\boldsymbol{R}$ at angle $\boldsymbol{\theta}$ with some angle offset $\boldsymbol{\delta\theta}$, whose magnitudes are comparable to angular seconds [8]. The reflectivity curve shows the percentage of reflected radiation in a certain range of angles, i.e., indicates the "quality" of the interference peak.

In work [9] the results of the experiment, which demonstrates that the reflectivity curves have different widths at different crystal orientation and radiation energy are presented (Fig. 8).

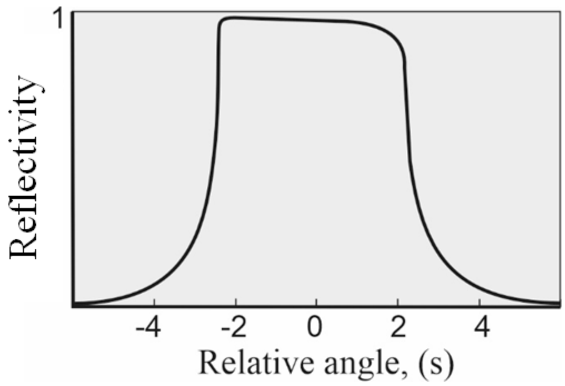


Fig. 7. Reflectivity curve of the monochromator optical element

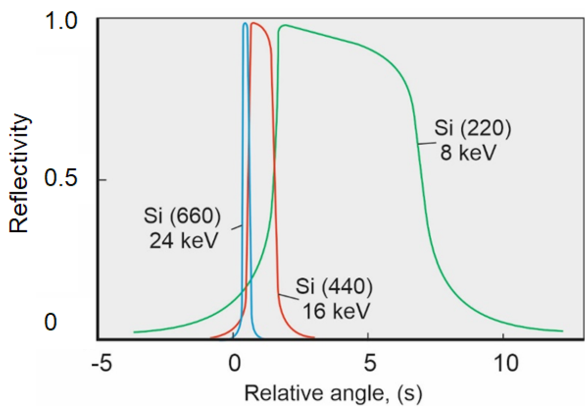


Fig. 8. Reflectivity curve for *Si*(660), *Si*(440) and *Si*(220) at energies of 24, 16 and 8 keV. From [9]

The reflectivity of a crystal depends not only on the grazing angle but also on the orientation of the plane (*Miller* indices). The integral along the rocking curve gives a quantitative estimate of the intensity of monochromatic radiation, and from the values of the angle displacement one can estimate the degree of monochromaticity.

Monochromators based on multilayer structures

One of the widely used practical solutions related to X-ray monochromatization is based on using multilayer structures [10, 11] that are obtained by successive deposition of thin alternating layers of two heterogeneous materials. With this approach, a heterophase structure of the ***A***-***B***-***A***-***B***... (Fig. 9) is formed. Each pair of the multilayer structure is represented by materials that differ from each other by the X-ray

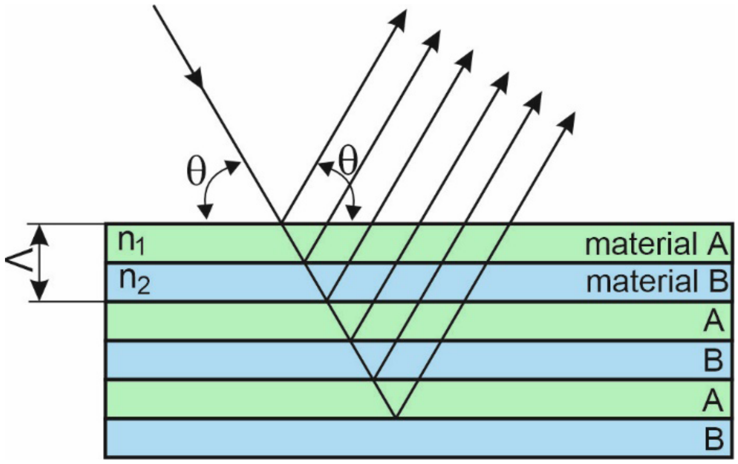


Fig. 9. Schematic of a multilayer X-ray mirror represented by pairs of layers of materials ***A*** and B characterized by refractive indices $\boldsymbol{n_1}$ and $\boldsymbol{n_2}$

refractive index as well as by the atomic mass ***Z***. The thickness of ***A*** ($\boldsymbol{t_A}$) material layers, characterized by low transmissivity (high ***Z*** values), is usually smaller if compared to the thickness of ***B*** ($\boldsymbol{t_B}$) material that has higher transmissivity [3]. The period of a multilayer structure is the value $\boldsymbol{\Lambda = t_A + t_B}$. An example of a heavy element used to produce multilayer X-ray mirrors is tungsten and a light element is carbon. Typically, thin layers are grown using the magnetron sputtering method.

The scattering of X-rays at the interfaces between two sublayers also, as in crystals, leads to forming diffraction maxima. The Wolf – Bragg law for multilayer materials can be written in the following form:

$$2\Lambda \cdot \sin\theta = n\lambda, \tag{7}$$

where the integer $n = 1, 2, ...$ is the order of the reflection maximum.

It should be emphasized that in this case the period $\boldsymbol{\Lambda = tA + tB}$ is not the distance between atomic planes in the crystal. Taking into account the circumstances, multilayer materials can be considered as "artificial crystals" [3].

An example of an artificially created heterophase material is a multilayer Ru/B_4C type structure used as the material of an X-ray mirror at the *BM5* beam line at *ESRF* (France, Grenoble) equipped with a bending magnet [12]. Seventy equally thick *Ru* and *B4C* layers were formed on a silicon substrate. The period of the **Λ** multilayer structure was 4.0 nm. The simulation results indicated the roughness of the interfacial surface at 0.3 nm.

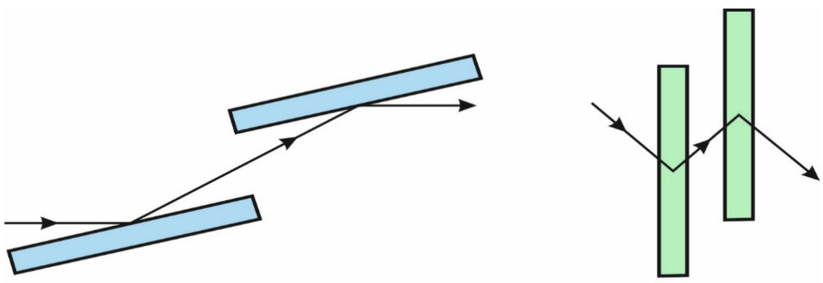
The monochromatic beam increases the image contrast by reducing artifacts and also provides access to more sophisticated contrast techniques. At the same time, the manifestation of contrast is facilitated by an increasing number of photons which can be provided by using mirror monochromators [13]. Compared to crystal monochromators, multilayer monochromators have a number of other advantages. In particular, it is possible to form a periodic structure with the distance between layers much larger than 1 nm, which allows increasing the wavelength range of reflected photons. Thus, the bandwidth of the multilayer material $\boldsymbol{\Delta E/E}$ is one or two orders of magnitude greater than the bandwidth of the monochromator. Consequently, multilayer mirrors reflect a larger part of the spectrum, which leads to an increasing integral intensity of radiation [14]. For this reason, multilayer mirrors and two-mirror monochromators are widely used to form "pink" beams.

Various combinations of materials can be used to obtain multilayer structures. In some cases, such pairs of substances as W/Si, Mo/Si, Pd/B_4C, W/B_4C, Mo/B_4C, Ru/B_4C are preferred. Other systems including Ni/C, Cr/Sc, Cr/Be, La/B_4C, etc. may be used. [9, 14, 15, 16] reflect the examples of studying multilayer structures. When selecting bilayer types for multilayer monochromators, it is important to take into account other factors arising during manufacturing and operating the equipment. One of it is the level of thermal load acting on the material. Multilayer monochromators are characterized by the manifestation of mutual atoms diffusion in heterogeneous materials and the possibility of forming new phases at the layer boundaries. In English-language literature, double mirror monochromators are referred to as *DMM* (Double Multilayer Monochromator).

Configurations and geometry of optical elements

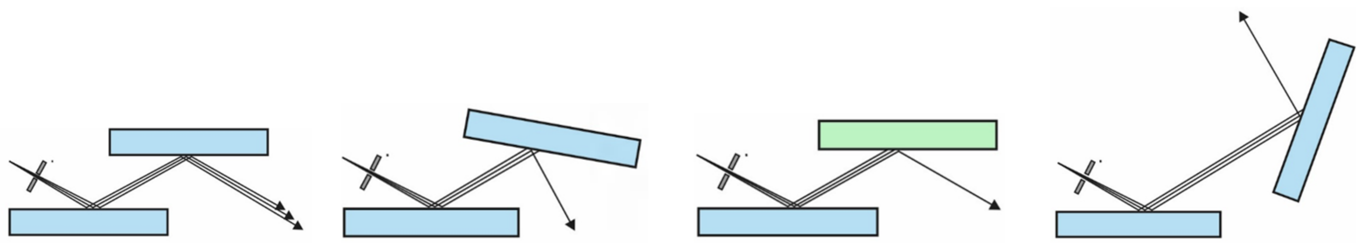
The principle of diffraction providing monochromatization of X-ray radiation can be realized using two schemes. One of them, the *Bragg* scheme (Fig. 10 *a*), assumes reflection of rays by the surface layers of the crystal. The first monochromators based on this scheme were proposed in 1921 by *Davis* and *Stempel* [17]. In contrast to the *Bragg* scheme, the *Laue* scheme presented in [18] is based on the passage of radiation through the crystal (Fig. 10 *b*). In both cases, the *Wolf – Bragg* condition is realized.

Depending on the choice of geometry and orientation of the monochromator crystals, the radiation received from the monochromator is characterized by a different degree of monochromaticity (Fig. 11). Two crystals (corresponding to the variant shown in Fig. 11 *a*) have the same spatial lattice. Its crystallographic surfaces are oriented in the same way in space. In such a "non-dispersive" configuration, the beam monochromaticity when reflected from the second crystal is not improved. The role of the second crystal when realizing this scheme is to restore the original direction of the beam path. In English-language literature, such two-crystal monochromators are referred to by the abbreviation *DCM* (double crystal monochromator).



a *b*

Fig. 10. Diffraction of radiation on crystals of a monochromator:
a – diffraction according to the *Bragg* scheme (reflection of X-rays); *b* – diffraction according to the *Laue* scheme (passage of X-rays through the crystal)



a *b* *c* *d*

Fig. 11. Configuration of non-dispersive (*a*) and dispersive (*b*–*d*) crystals

When improving the degree of monochromaticity is necessary, one of the three solutions schematically presented in Fig. 11 *b*–*d* can be used. The second crystal, unlike the first one, has a different crystallographic orientation (Fig. 11 *b*, *d*), or is made from a different material (Fig. 11 *c*) with other orientation of the surfaces. Dispersion schemes provide an increased monochromaticity of radiation due to additional diffraction on the second crystal, contributing to the isolation of a narrower band of wavelengths. The number of photons transmitted in the required direction is reduced. In [19] the analyzed schemes are described in more detail. In [20] the dispersive crystal arrangement corresponding to Fig. 11 *b* is used. Work [21] presents a configuration similar to the scheme corresponding to Fig. 11 *d*. Asymmetric reflection of rays takes place in both cases.

Devices providing more than two reflections on the crystals can be used to improve the monochromaticity of the radiation. In [21–23] monochromators with two and three pairs of crystals (four and six reflections), each of which emits a certain spectral band, are presented. Devices of this type, having improved monochromaticity indices, make it possible to preserve the beam axis from the entrance to the monochromator to its exit. In some cases, the fulfillment of this condition is important in designing synchrotron radiation stations [24, 25]. Most monochromators installed on *SRS* are equipped with two-crystal nodes [26].

One of the most common designs of crystal monochromators involves using channel-cut crystals. In this case, both plates of the monochromator belong to the same monocrystalline block. In the Russian-language literature, the structures made according to this scheme (Fig. 12) are called "butterfly" [27]; in the English version these are called channel-cut monochromator (*CCM*).

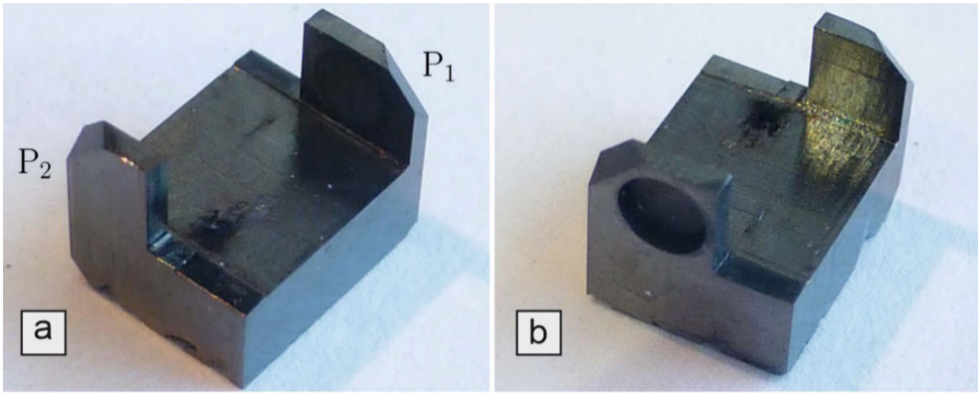
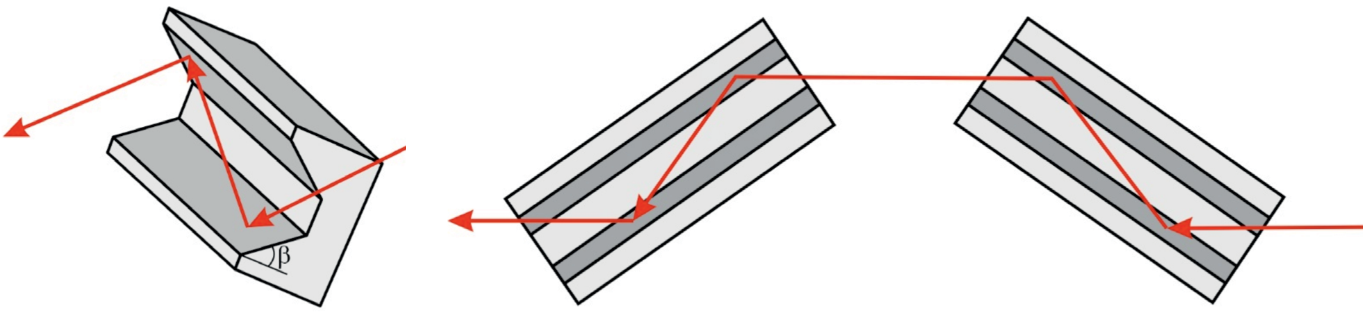


Fig. 12. The working element of the channel-cut monochromator. Two faces of the crystal, on which the beam reflection occurs, belong to a single monocrystal [28]

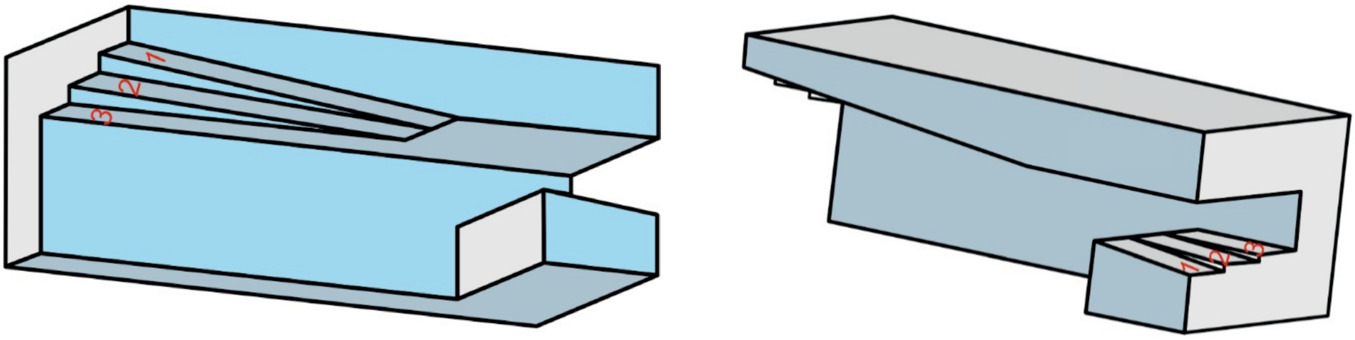
In contrast to two-crystal monochromators, when using the channel-cut monochromator scheme, the crystal is cut out of the monoblock in such a way that its reflecting surfaces are located on the inner sides of the channel (Fig. 12) [28–29]. The main advantage of such a technical solution is the possibility of ensuring perfect parallelism of the reflecting surfaces at the stage of crystal formation. A significant violation of parallelism of the working surfaces of the channel-cut monochromator due to thermal deformation of the material is observed at high radiation energies (over 20 keV [3, p. 152]). When using monochromators with independent crystals that have several degrees of freedom difficulties in parallelism of its working surfaces are possible [28].

Fig. 13 *b* [30] presents one of the varieties of dispersive monochromators with channel-cut, providing fourfold reflection of X-rays [21, 22, 30, 31]. In [32] a scheme of a channel-cut monocrystal with three diffracting faces as working surfaces is demonstrated (Fig. 14).



a *b*

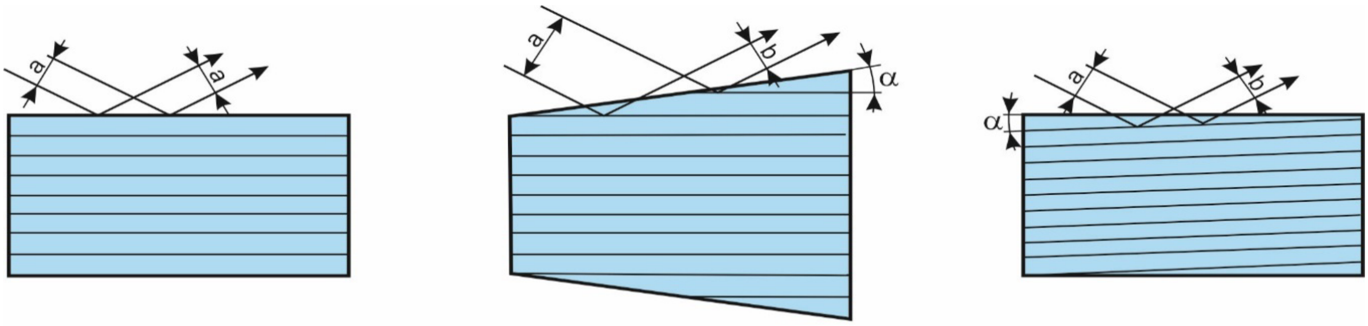
Fig. 13. Monochromator with channel-cut:
β – angle of inclination of the facet [30]



a *b*

Fig. 14. The channel-cut monochromator with three diffracting faces [32]. View of the crystal from two different sides

In [33] symmetric (Fig. 15 *a*) and asymmetric (Fig. 15 *b*, *c*) reflection of X-rays are distinguished when analyzing optical schemes in *Bragg* and *Laue* geometry.

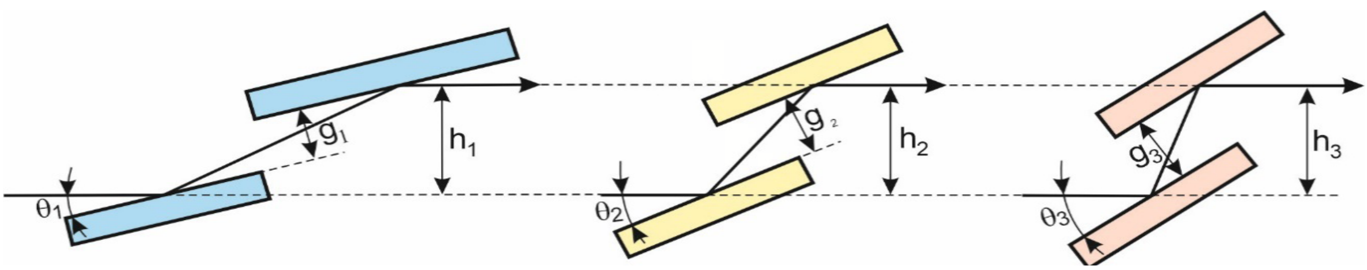


a *b* *c*

Fig. 15. Variants of orientation of atomic planes in monochromator crystals

The schemes with symmetric and asymmetric reflection differ in the length of the ray path in the crystals. In the case of symmetric reflection, shown in Fig. 15 *a*, the atomic planes are parallel to the crystal surface. In the case of asymmetric reflection, the crystal planes are oriented at an angle to the atomic planes, which is usually 5–10° [34, p. 283]. The variants of crystals providing asymmetric reflection are shown in Fig. 15 *b*, *c*. When using the asymmetric reflection scheme, the optical path length of the beam incident on the crystal differs from the path length of the reflected beam [35]. Using such a scheme makes it possible to form a denser monochromatic radiation compared to the scheme with symmetric reflection.

The distance between the *SR* beam entering the monochromator and the beam leaving it is called *beam offset*. In Fig. 16, this parameter is denoted by the symbol ***h***. Depending on the mounting scheme of the



a *b* *c*

Fig. 16. Constant displacement of the beam when changing the angle of incidence **θ** (fixed beam exit from the monochromator: $\boldsymbol{h_1} = \boldsymbol{h_2} = \boldsymbol{h_3}$)

monochromator crystals (mirrors), designs with fixed and non-fixed beam offset can be realized. Fig. 16 shows the positions of crystals corresponding to different grazing angles **θ** of incoming rays [36]. It follows from the figure that the beams exiting the monochromator are at the same height, i.e., its displacement is the same ($h_1 = h_2 = h_3$).

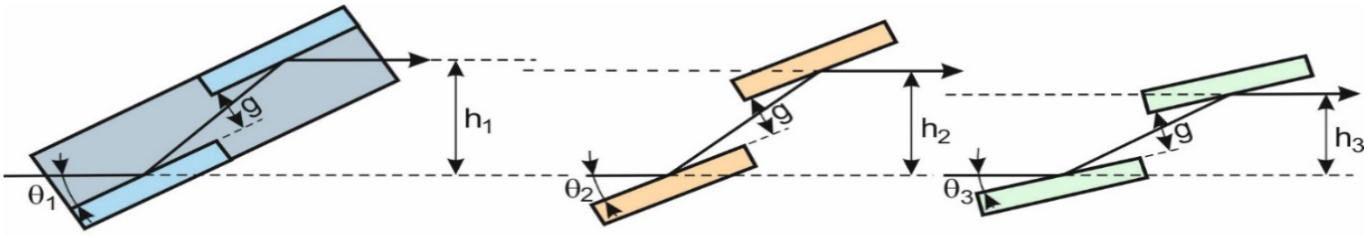
The beam offset depends on the way of attaching the optical elements. It is a question of whether the elements are rigidly connected or fixed independently of each other. A fixed beam offset is achieved by adjusting the gap ***g*** between the crystals. The constant beam displacement ***h*** is determined by the following relationship:

$$h = 2g \cdot \cos\theta, \tag{8}$$

where g is the distance between the optical elements of the monochromator; θ is the beam grazing angle.

For angles corresponding to the range $0 < \theta < 45°$, the second optical element should be extended (Fig. 16 *a*). If the grazing angles are larger than 45°, there is no need to extend the optical element (Fig. 16 *b*, *c*) [37].

Fig. 17 shows the case when the optical elements are rigidly connected to each other and, therefore, the gap ***g*** between them is the same [38]. When this scheme is realized, a change in the beam grazing angle ($h_1 \neq h_2 \neq h_3$) also leads to a change in the displacement value h ($h_1 \neq h_2 \neq h_3$). This is the case of non-fixed beam offset. In [37] a variant of the monochromator corresponding to such an arrangement of elements is described.



a *b* *c*

Fig. 17. Different magnitude of ray displacement ($h_1 \neq h_2 \neq h_3$) when changing the angle of incidence **θ** (monochromator with non-fixed ray output)

While developing a monochromator the choice of the crystal rotation axis is one of the main problems [39]. Three options of its location are possible (Fig. 18). According to one of it, the axis of system rotation of two crystals is located at the first optical element (at the point O_1). It is also possible to locate the axis of rotation at the middle of the beam between the optical elements (at the point O_2). According to the third option, the axis is located at the point where the beam falls on the second crystal of the monochromator (at point O_3). The rotation point plays an important role in the geometry of the beam path in the monochromator.

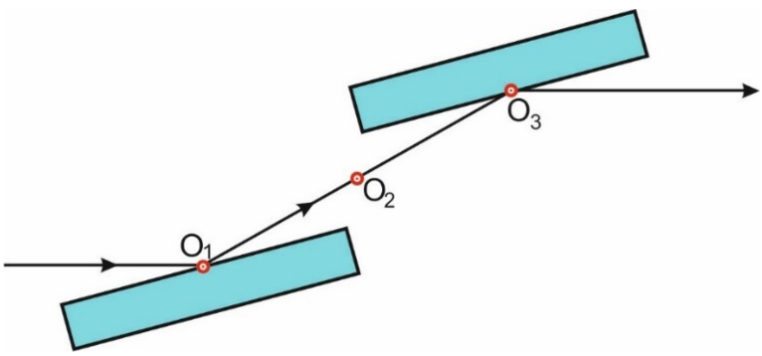


Fig. 18. Three possible arrangements of rotation axes of optical elements of monochromators

Focusing monochromators

When conducting research using synchrotron radiation, there are problems that require either a focused or a divergent beam. One of the approaches allowing to form focused beams is based on using bent crystals. The basic configurations of monochromators with bent crystals are described below. Fig. 10 *b* shows the *Laue* scheme based on the passage of a "white" beam of synchrotron radiation through a monochromator crystal. According to it, the polychromatic *SR* beam enters the crystal, where it experiences reflection at an angle to the atomic planes satisfying the *Wolf – Bragg* condition. Monochromator crystals realizing diffraction according to the *Laue* scheme have been widely applied in solving problems related to the necessity of focusing synchrotron radiation [35]. The background was the development of the *Yvette Cauchois* spectrograph in the early 1930s, which showed that curved crystals allowed focusing X-ray radiation [40]. The course of the rays corresponding to this work (Fig. 19 *a*) can be considered as a modified *Laue* scheme, which is called the *Cauchois – Johann* scheme [40]. Fig. 19 *b* shows the scheme which is called the *Cauchois – Johansson* scheme [40]. The continuations of the atomic planes of the curved crystal converge at point ***N***, and the continuations of the incident rays converge at point ***F'***. In the case of radiation falling from the convex side, as shown in Fig. 19 *a*, *b* X-ray radiation is focused on spot ***F***. The points ***F'***, ***N***, as well as the set of points lying in the spot ***F***, are located on the same circle with radius ***R***, called the focal one. The scheme shown in Fig. 19 *c* is called the *Johann* scheme [41]. Fig. 19 *d* shows the *Johanson* scheme [42]. For cases in Fig. 19 *c*, *d*, the radiation source ***I*** and the focus spot should be located on the focal circle.

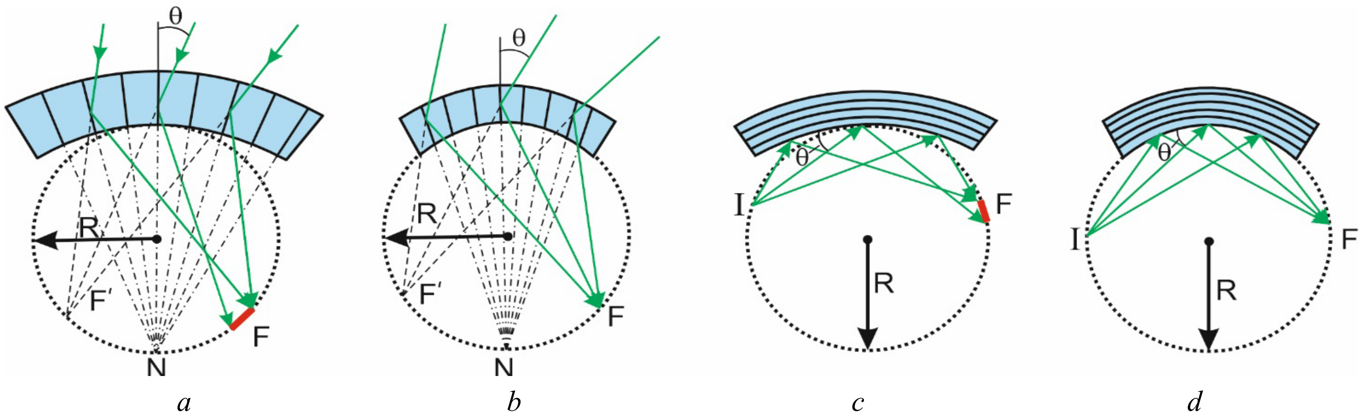


Fig. 19. Schemes of ray path in focusing monochromators with curved crystals:
a – *Cauchois – Johann* scheme (modified Laue scheme); *b* – *Cauchois – Johansson* scheme; *c* – Johann scheme (atomic planes and focal circle have different radii of curvature); *d* – Johansson scheme (atomic planes and focal circle have the same radii)

By directing the radiation from the inside of the crystal, the beam can be broadened as shown in Fig. 20 [43].

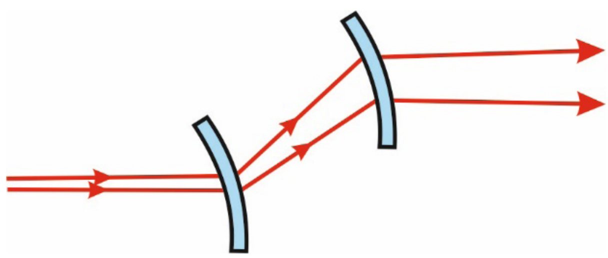
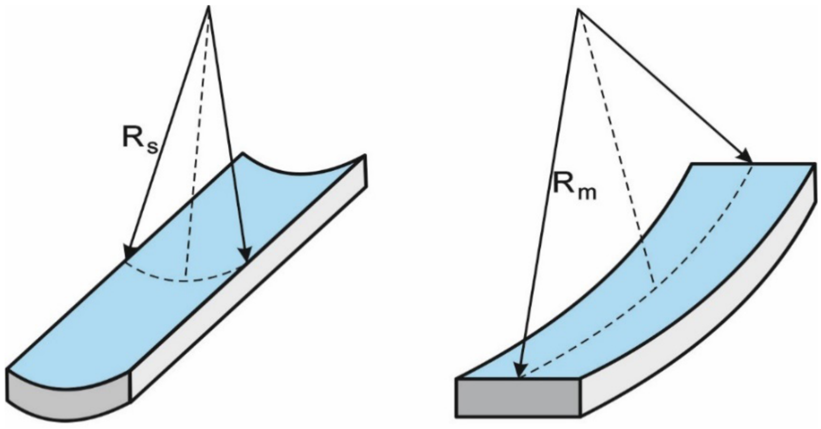


Fig. 20. *Laue* monochromator with bent crystals expanding the beam

In addition to the *Cauchois* scheme, which assumes that the radiation falls on the outer side of the crystal, the focusing effect can also be achieved using the *Johann* (Fig. 19 *c*) and *Johansson* (Fig. 19 *d*) schemes, in which the radiation falls on the crystal from its inner side. Both schemes are based on *Bragg* diffraction of radiation. The difference between them is related to the radius of curvature of the crystals.

In the *Johansson* scheme, the radius of the focal circle and the radius of curvature of the crystal coincide. In the *Johann* scheme, the radius of the focal circle is smaller.

Crystals installed in focusing monochromators can be deformed in various ways. A distinction is made between sagittal [44] and meridional [45] bending (Fig. 21).



a *b*

Fig. 21. Curved optical elements. Sagittal bending of a crystal with radius $\boldsymbol{R_s}$ (*a*) and meridional bending with radius $\boldsymbol{R_m}$ (*b*)

Depending on the type of crystal bending, the focal length of the monochromator is determined according to the formulas:

$$R_s = 2\sin\theta \cdot \frac{pq}{p+q} = 2f\sin\theta; \qquad (9)$$

$$R_m = \frac{2}{\sin\theta} \cdot \frac{pq}{p+q} = \frac{2f}{\sin\theta}, \qquad (10)$$

where p is the distance from the source to the crystal; q is the distance from the crystal to the sample; f is the focal length.

To adjust the focal length of the monochromator, it is necessary to provide a change in the radius of curvature of the crystal. Monochromators are equipped with mechanical devices that make it possible to adjust the necessary curvature of the crystal.

Fig. 22 shows the device of the monochromator described in [46]. The device is equipped with two crystal mounting nodes using a scheme that provides a fixed beam output. This is realized by moving the

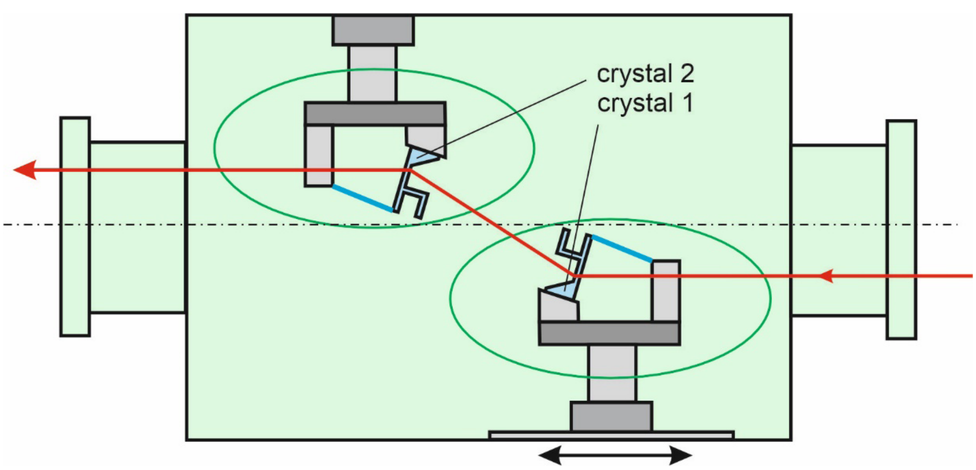


Fig. 22. Curved optical elements of the fixed beam offset monochromator

first (lower) crystal node along the beam propagation axis. And focusing is provided by rods fixed on the crystals. The enlarged image of one of the crystals with a tie rod and deformation mechanism attached to it is shown in Fig. 23.

One of the technical solutions to the problem associated with crystal bending is described in [47] (Fig. 24). The scheme depicted in the figure provides for deformation of the crystal by four levers.

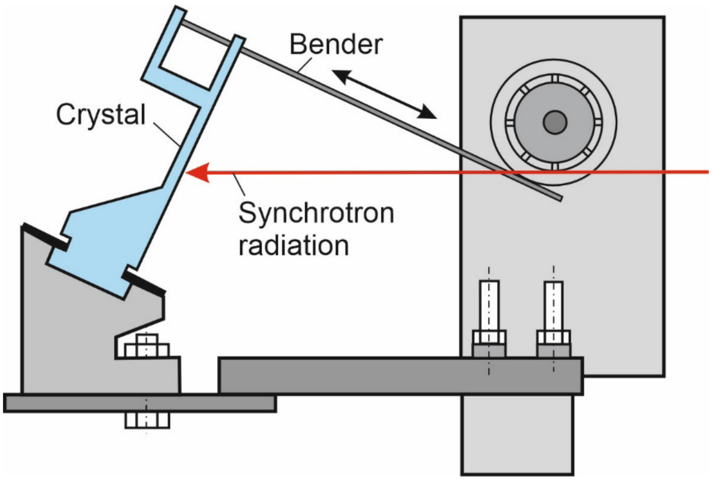


Fig. 23. Node with the first crystal of the monochromator (according to [46])

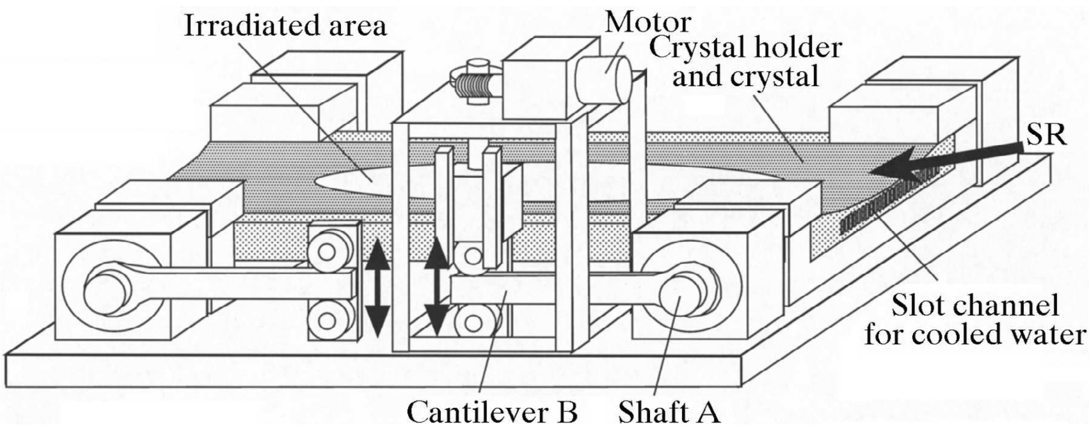


Fig. 24. Schematic of the device for bending the monochromator crystal (according to [47])

The crystal shown in Fig. 24 is characterized by sagittal bending in the initial state. A mechanical device makes it possible to bend it also in the meridional direction (Fig. 25 *a*). Thus, the monochromator crystal can be simultaneously bent in two directions.

Fig. 25 shows one of the variants of the combination of elements providing focusing of synchrotron radiation. The focusing device includes two multilayer X-ray mirrors [48, 49]. One of it is flat and the other is curved. By applying a mechanical load, it is possible to change the radius of curvature of the second mirror.

The design features of the developed monochromators are determined, first of all, by the type of tasks to be solved. In many cases, devices unique in terms of technical parameters are designed.

Thermal load of synchrotron radiation on optical elements

When selecting materials for synchrotron radiation monochromatization, three aspects are analyzed: diffraction, thermal, and mechanical. The most important requirement for materials used for manufacturing

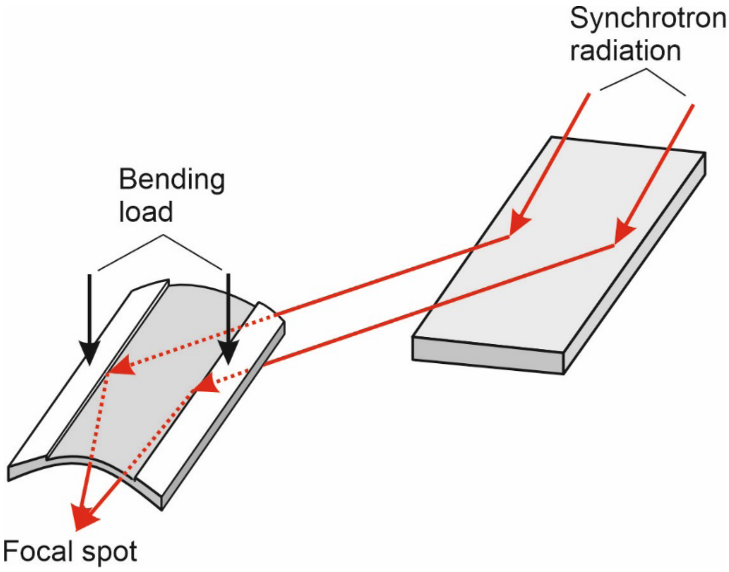


Fig. 25. Configuration of mirrors of the focusing monochromator. According to [48]

optical elements is the ability to dissipate the thermal load caused by the local impact of the "white" *SR* beam on the surface of the product (Fig. 5). In addition, the material should be resistant to radiation damage [3, 5] and meet radiation bandwidth requirements. Section "Monochromators based on multilayer structures" describes the materials used for producing X-ray mirrors of multilayer monochromators.

When manufacturing crystalline monochromators, silicon and germanium crystals with the necessary thermal conductivity are most often used as elements providing diffraction of X-ray radiation [50]. This function can also be performed by diamonds. Diamond crystals are used at sources with high radiation flux characterized by increased thermal load. Nevertheless, in comparison with silicon and germanium, diamond, having excellent thermal conductivity, is characterized by lower values of the reflection coefficient [51], which limits the practical application of this material.

The optical element (monochromator crystal, X-ray mirror) that perceives the main thermal load is subject to deformation accompanied by changes in its shape. To maintain parallelism of working surfaces of crystal pairs (and X-ray mirrors), and, consequently, to ensure the functionality of the device, forced cooling of heat-loaded elements is used. Depending on the amount of heat released per unit volume of material, the type of cooling is selected (water or cryogenic), as well as the method of cooling (direct or indirect).

Problems related to crystal cooling are among the most important when designing monochromators [5]. The peculiarities of the technical solutions used by the developers determine the accuracy of the devices. Several variants of monochromators with different approaches to the problem of cooling of optical elements are given below. One of them consists of using cooling channels (Fig. 26) laid directly in the monochromator crystals [52].

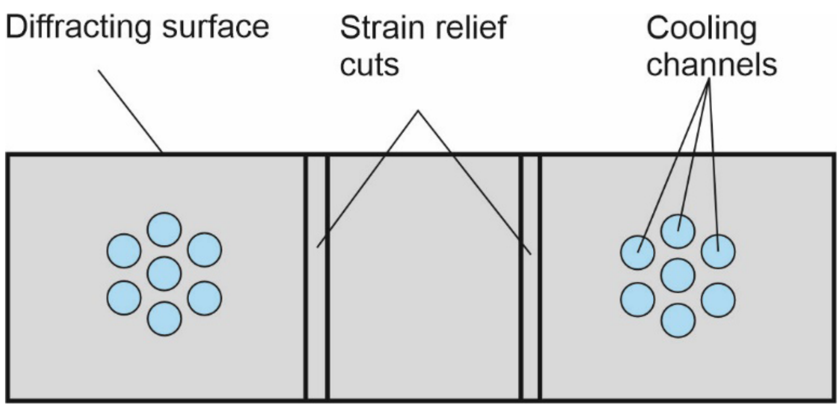


Fig. 26. Schematic of the crystal with direct cooling. According to [52]

The works [53–54] describe monochromators in which the principle of indirect cooling of optical elements is realized (Fig. 27). Its essence consists of heat transfer from the heated crystal to a tightly pressed cooler, and the cooling liquid is pumped through its internal channels.

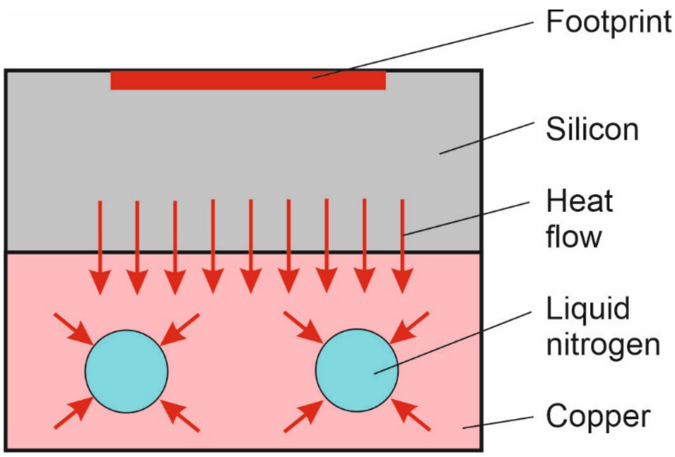


Fig. 27. Scheme of indirect cooling of the monochromator crystal. Red arrows show the heat flow from the crystal to the cooling channels with liquid nitrogen (according to [54])

When designing monochromators, it is also necessary to take into account the aspects related to the effects of vibration "pollution". This refers to vibrations of the ground, electrical appliances and other equipment. The consequence of this effect is the fluctuations of the beam position on the optical elements and the specimen. The vibration of optical elements leads to a violation of the alignment of the device, which prevents the achievement of its optimal parameters [56]. Factors contributing to the manifestation of deformation and vibrations of monochromator elements are related to the influence of thermal load, the impact of clamps on crystals or X-ray mirrors, and vibrations caused by the cooling system. In [57] the peculiarities of vibrations arising during the operation of the monochromator due to the thermal effect of the beam on the crystal are analyzed.

When the thermal effect on crystals and X-ray mirrors is not critical, cooling of optical elements is not required.

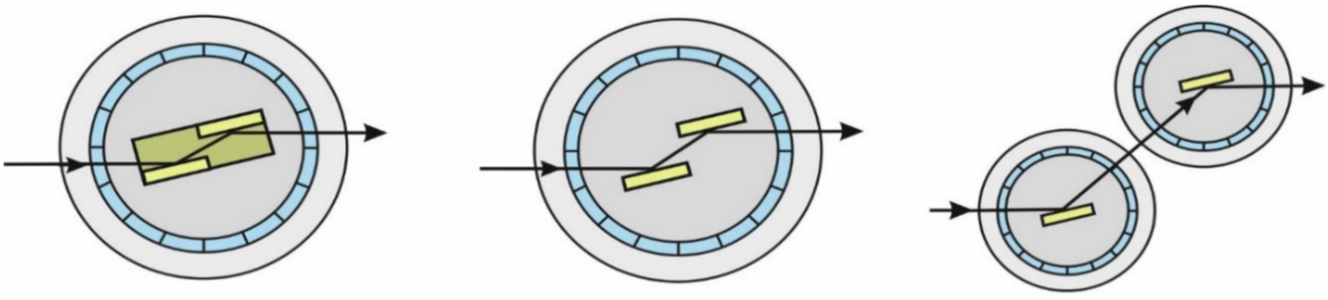
Additional information about monochromators

Monochromator is an optical-mechanical device with a wide range of requirements. Equipment designer determine technical solutions that ensure reliable operation of the monochromator with observing accuracy indicators. At the same time, standard solutions are used in designing most devices. For example, goniometric devices are used for crystal rotation, shown schematically in Fig. 28.

In practice, different variants of goniometer installation are possible. Fig. 29 shows the schemes of devices with horizontally and vertically oriented axes of rotation.

Fig. 22 shows a monochromator illustrating another technical solution related to crystal control. In this device, one of the crystals (the second one) is fixed stationary, and the other is connected with a moving mechanism, which allows fixing the beam offset. In practice, other variants of providing movements on monochromators are also realized. Both optical elements or just one of it can be movable. The schemes of beam reflection in both vertical and horizontal planes are proposed. The final solution is determined by the device designers and depends on the set of tasks to be solved at the beamline.

Monochromators containing several pairs of crystals [58, 59] of different geometric shapes have been proposed for using as a part of stations. Fig. 30 shows the scheme of the monochromator described in [58]. The red arrow highlights the incoming beam of synchrotron radiation. In order to adjust to the



a *b* *c*

Fig. 28. Schemes of goniometers:
a – goniometer of “butterfly” type; *b* – goniometer of quasi channel-cut monochromator; *c* – goniometer realizing the scheme of conjugation of independent crystals

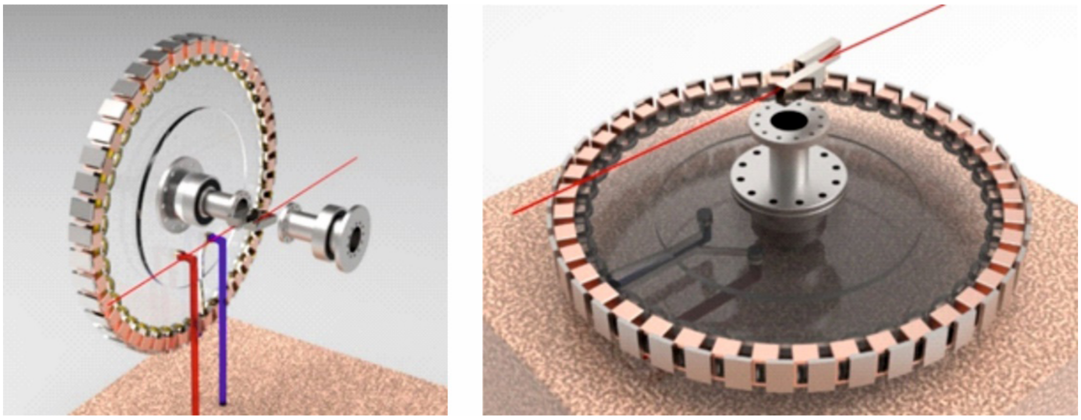


Fig. 29. Schemes of installations with horizontally and vertically oriented goniometer axes

incident beam, pairs of crystals move along rails fixed on the frame that is shown in Fig. 30 in blue color. This technical solution makes it possible to assemble two pairs of optical elements monochromatizing synchrotron radiation in one device.

Several pairs of crystals can be used in the design of monochromators. There are devices with an odd number of optical surfaces (not parallel to each other). In [60] one example of realizing such a scheme is presented (Fig. 31).

In [61] a monochromator with operation involving reflection on four crystals is presented (Fig. 32).

Conclusion

Analysis of the structural features of materials of different nature is one of the most important tasks currently being solved by the scientific community. A large number of such tasks arise in modern materials science, physics, chemistry, geology, biology, medicine, pharmacology, archeology. The tasks related to the study of fast structural transformations in materials at different stages of its processing are among the most difficult ones. The success of its solution depends to a great extent on the technical characteristics of the scientific equipment used by specialists. The research methods developed during the last century allow obtaining unique information about the structure of materials at different scales.

Among the most informative methods of studying materials, it is necessary to emphasize the methods of X-ray structural analysis, which originated from the works of *Röntgen*, von *Laue*, father and son *Bragg*. In recent decades, these methods have been effectively developed in solving problems at synchrotron radiation sources. In accordance with the national project “*Science and Universities*”, modern synchrotron radiation sources “*SILA*” (Moscow region, Protvino) and “*RIF*” (Vladivostok, Russky Island) will be created in

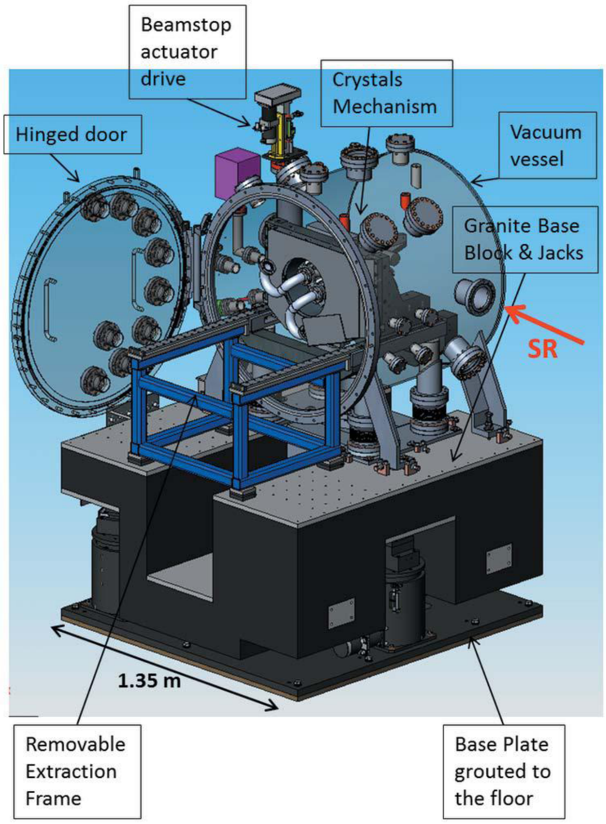


Fig. 30. Principle scheme of a monochromator with two pairs of crystals. (Taken from [58])

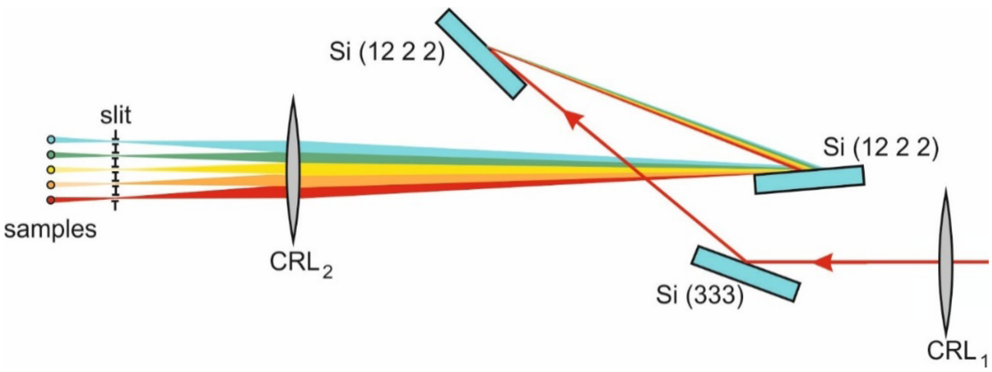


Fig. 31. Principle scheme of the monochromator-spectrograph. Taken from [60]

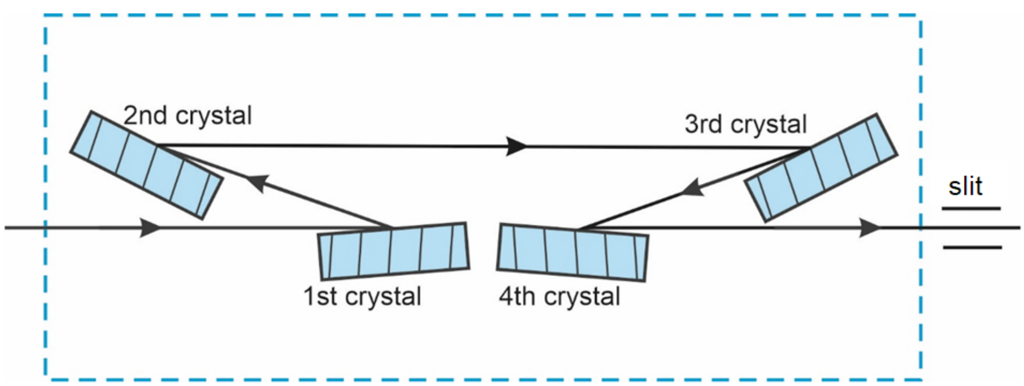


Fig. 32. Schematic of a high-resolution monochromator. (Based on [61])

Russia. At the end of 2024, a specialized synchrotron radiation source of generation 4+ "*SKIF*" is to be put into pilot operation in Novosibirsk (Koltsovo).

The design of synchrotron radiation sources implies the presence of a storage ring of elementary particles (usually electrons) and beamlines, which equipment is used to study materials. The total number of planned beamlines at the *SKIF* source alone is thirty.

Monochromators are one of the types of equipment included in the beamlines. The purpose of these optical and mechanical devices is related to the separation of diffracted beams characterized by narrow wavelength bands from a wide spectrum of electromagnetic radiation. Including Russia in the number of countries focused on creating modern sources of synchrotron radiation means that there is a need to develop its own various types of monochromators. Russian specialists will have to solve this problem in the coming years.

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Conflicts of Interest

The authors declare no conflict of interest.