

Polycapillary X-ray Microbeams

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ABSTRACT

As known, Kumakhov optics, or polycapillary optics, is based on the effective passage of X-ray radiation through the bundles of monicapillaries of various configurations; the latter takes place due to the phenomenon of total external reflection of X-rays from the inner capillary walls. In our work the basic characteristics of intense quasi-parallel X-ray polycapillary microbeams from a laboratory source in a scheme of microfocus X-ray tube/polycapillary structure, are investigated theoretically as well as experimentally. Experimental studies are carried out by means of a special automated stand. The received experimental data are compared with the results of computer statistical modeling. Modeling includes of the several theoretical models for various types of deviations of a reflection surface from ideal such as the roughness, waviness and bending. The effect of decrease of full divergence of microbeams near the exit end face of polycapillary system (quasi-decrease of divergence) is found out. But, unlike the observable effects of decrease of local divergence, proposed and discovered in works of Dabagov et al. and described on the base of surface channeling, the given effect can be well enough described within the limits of geometrical optics. Now on the base of Kumakhov optics at the Institute for Roentgen Optics the new generation of devices is developed and created: «laboratory synchrotron», fluorescent spectrometers, reflectometers, diffractometers, X-ray microscopes and combinations of several devices in one.

Keywords: X-ray microbeams, Kumakhov optics, polycapillary optics, X-ray analytical instruments, laboratory synchrotron

1. INTRODUCTION

As known, Kumakhov optics (KO), or polycapillary optics, is based on the effective passage of X-ray radiation (XR) through the bundles of monicapillaries of various configurations; the latter takes place due to the phenomenon of total external reflection (TER) of X-rays from the inner capillary walls [1].

Nowadays on the base of KO at the Institute for Roentgen optics (IRO) the new generation of devices is created: fluorescent spectrometers, reflectometers, diffractometers, X-ray microscopes, «laboratory synchrotron» and combinations of several devices in one [2]. Such significant amount of applications were realized due to the distinctive features of KO such as the large enough angular aperture ($\sim 0,1$ rad) and broad-band energy spectrum (0,1-60 keV [3]).

Application of KO to conventional low-power X-ray tubes allows receiving X-ray microbeams with high density of a flux. So, a microfocus semilens with focal length of millimeter order allow to receive X-ray microbeam with the divergence of about critical angle of TER and the density of the order of 10^{10} photon/(s mm²) for monochromatic characteristic radiation [4,5]. But thus the area of cross-section of resulted beams is defined by the exit diameter of a semilens that finally provides the focal spot size of the order of millimeter.

In [6,7] the authors are received the intense quasi-parallel X-ray microbeams with the density of a radiation flux close to that provided by synchrotron, at the output of system of the microfocus X-ray generator/cylindrical polycapillary system. In [8,9] the application of this method of getting the microbeams to the scheme of scanning X-ray microscopy on the base of a raster X-ray system is considered.

In this work the results of the research and the comparison of traditional monicapillary X-ray microbeam technique (MXM) and new technique [6,7] - polycapillary X-ray microbeams (PXM), are given. Properties of X-ray microbeams,

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received in the scheme of microfocus X-ray source/mono- or polycapillary structure, are considered. Measurements were carried out at the automated stand for investigation of KO [10-12].

2. EXPERIMENTAL PART

2.1 Transmission vs. Length

Monocapillary X-ray microbeams

The glass monocapillaries, created in IRO with the purpose of reception of intensive X-ray microbeams and comparisons MXM and PXM, are experimentally investigated. Dependence of transmission factor of monocapillary on its length for X-ray energy - 8 keV is resulted on fig.1. Following values of parameters of the source are used: the copper anode; the anode voltage - 40 kV; the current - 25 μ A; the focal spot diameter - 10 microns. External diameter of the monocapillary made 5 mm, internal – 10 microns. The distance between the source and the capillary was minimally possible ($\approx 0,2$ mm).

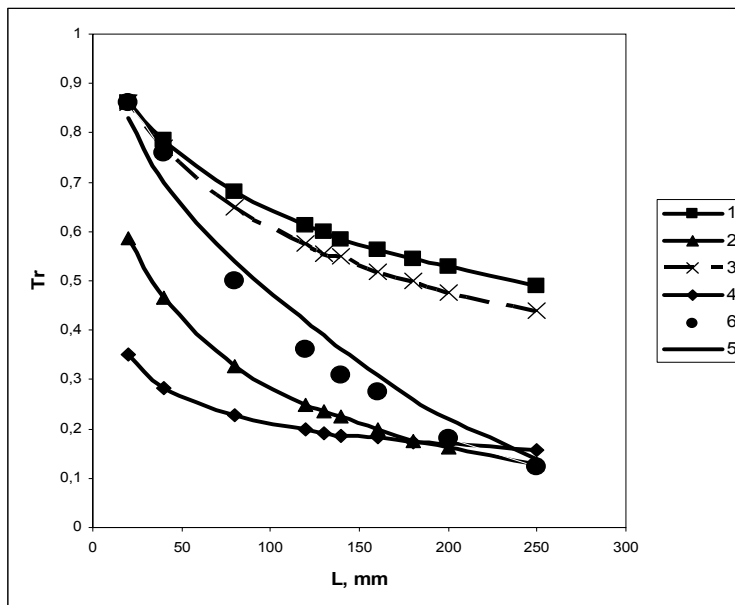


Fig.1 Results of experiment and modelling of dependence of transmission coefficient Tr on monocapillary length L :

- 1 - Modeling of waviness on Wang et al. $\Delta\theta_{\max} = 3 \cdot 10^{-4}$; 2 - Modeling of waviness on Wang et al. $\Delta\theta_{\max} = 2,25 \cdot 10^{-3}$;
- 3 - Modeling of roughness on Vinze et al., $\sigma = 4,5 \text{ \AA}^0$; 4 - Modeling of roughness on Vinze et al., $\sigma = 40 \text{ \AA}^0$;
- 5 - Modeling $\sigma = 2 \text{ \AA}^0$ (waviness); $\Delta\theta_{\max} = 2 \cdot 10^{-5}$ (roughness); $R_{\text{bend}} = 25 \text{ m}$ (bending); 6 - Experimental data.

Polycapillary X-ray microbeams

Characteristics of some the polycapillaries, created in IRO, are investigated. Dependence of transmission of a direct polycapillary on its length, by means of consecutive cycles of shortening of a capillary and measurements, was defined. Values of transmission of the polycapillaries in the given energy range are close to values of transmission of the monocapillaries, received in our researches. Experimental results of transmission dependence from length will well be coordinated with independent researches (for example, [13,14]).

2.2 Divergence

For applications the information about change of the size of a microbeam with distance, that is characterized by divergence, is important. Divergence of polycapillary beams usually is supposed a constant and, within the limits of geometric optics considerations, equal, without taking into account the losses, to two critical angles of TER of X-ray radiation from internal polycapillary walls.

Divergence after polycapillary structures can be subdivided on local and global [5-8]. Local one is defined by divergence owing to TER from walls, global - characterizes a angles between axes of monocapillaries.

It is expedient to enter still concept of the full or effective divergence, describing an aggregate effect and defining change of the effective size of a beam. The effective size of a beam is understood as the size defining the basic area of influence in given section. Value of effective divergence also will give a method of a beam knife scanning in different sections. As the effective size we shall consider FWHM of Gaussian curve, approximating experimental data - the differentiated curve of intensity at a beam knife scanning.

Detailed consideration of characteristics of full divergence of polycapillary microbeams by a method of knife scanning applied by us [10,11] in the literature it is not revealed. We carry out attempt to fill the designated blank [19, 20].

Decreasing of full divergence of PXM [20]

In our work dependence of diameter of section of the polycapillary microbeam on distance between the place of knife edge scan and the exit end face of polycapillary structure is experimentally received. The used polycapillary had diameter of the channel 2,5 μm, the general diameter of the polycapillary structure – 2,5 mm. Length of the polycapillary structure – 30 mm.

The microfocuss X-ray generator on the basis of tube BS-11 with the copper anode was applied, in experiment characteristic radiation $Cu K_{\alpha}$ was used. For scanning a beam the tantalum knife was used.

For definition of the beam size in an interesting point of an axis dependence of signal level of the detector on knife position was obtained at its cross-section of a beam. Then the received dependence was differentiated and approximation by Gauss curve, FWHM of which was considered as the size of a beam in the given point.

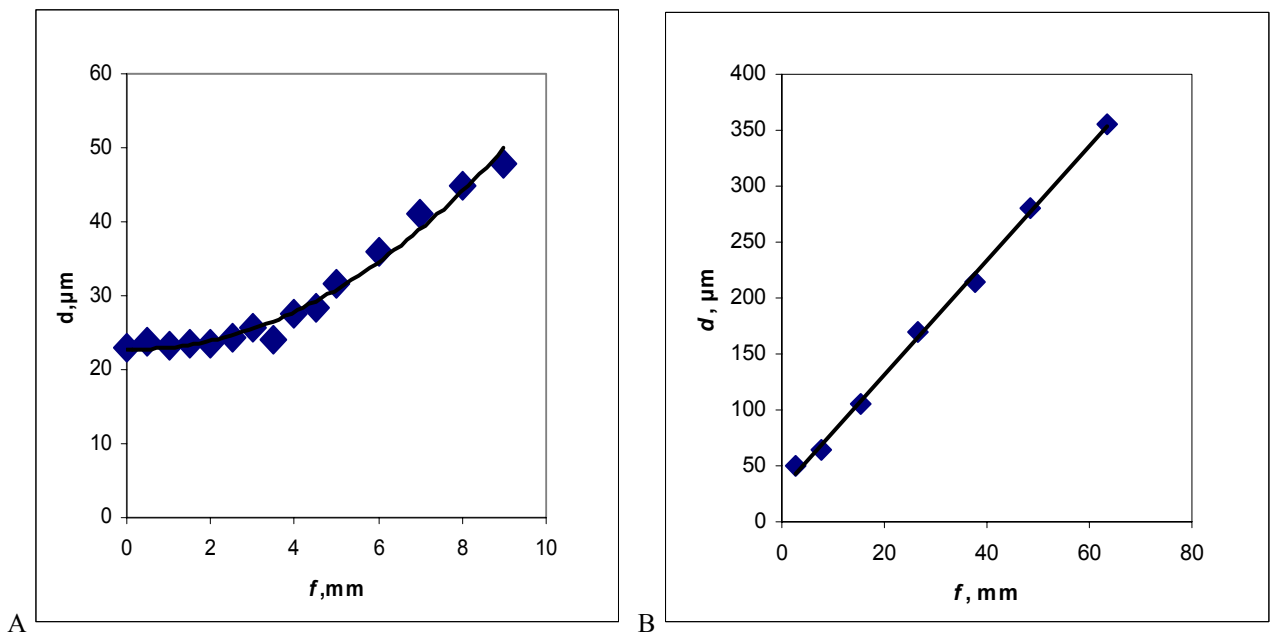


Fig.2 Dependence of diameter of a beam section d on an output of a polycapillary column on distance from a exit end face of a column f , rhombuses – experimental points, a continuous line – the best approach:

A - near the end face, $y = 0.3516x^2 - 0.1476x + 22.787$, $R^2 = 0.9791$;

B - on a site of the maximal divergence, $y = 5.0871x + 30.479$, $R^2 = 0.9982$.

Results are presented on fig.2A. As we see, character of the divergence, equal to a tangent of angle of a tangent line to a curve, varies. Near the end face of polycapillary structure the effect of decreasing of full divergence is observed. It is caused by characteristic redistribution by polycapillary structure of a radiation field of a source. The effect of reduction of divergence was proposed and discovered also in works of Dabagov et al [21,22]. But, as against observable effect of

reduction of local divergence in these works, described on the base of surface channeling, the observable by us effect can be well enough described within the framework of geometrical optics.

Apparently from graph and as follows from the general reasons, values of diameter near the end face display the size of a source [23] that makes value $\approx 23 \mu\text{m}$. On the maximal divergence defined by local divergence – that is provided by a angle of repeated TER, the microbeam apparently comes out on distance $F_{m.d.}$ defined by value of diameter of a tube focal spot $d_{f.s.}$ and the maximal divergence $Q_{max} \sim \theta_{cr}$ assigned to divergence of a beam of a separate moncapillary, proportional to a critical angle of TER:

$$F_{m.d.} \approx d_{f.s.} / Q_{max} \quad (1)$$

On full divergence the polycapillary beam comes out at an output of all separate moncapillary beams from a polycapillary beam that is when all the internal moncapillary microbeams start to participate in formation of divergence of a polycapillary microbeam.

From here diameter of a polycapillary beam d on distance from an exit end face f will be defined by following expression:

$$d \approx d_{f.s.} + Q_{max} (f - F_{m.d.}) \quad (2)$$

And to estimate the size of a focal spot of a source on the size of section of a beam on an output of polycapillary system accordingly it is possible as

$$d_{f.s.} \approx d - Q_{max} (f - F_{m.d.}) \quad (3)$$

The course of an experimental curve of full divergence is interesting after when the additive of moncapillary beams in general beam ceases to occur, that is on the distance, exceeding $2 d_{f.s.} / Q_{max}$ (fig.2B). Distribution of intensity in section of a beam in this case is received by a photomethod, with use of the detector on the basis of the CCD-sensor, mathematical processing of experimental data was used former. As we see, in the field of further distance $2 d_{f.s.} / Q_{max}$ effective divergence has constant character and is defined approximately 2 critical angles of TER.

Also, the similar effect quasi-decreasing areas of capture, possibly, will be present at polycapillary structure of the limited size compared to a spot of greater diameter that it is necessary to consider by optimization of the size of a tube spot.

It is necessary to pay attention to the given effect at researches of divergence of polycapillary microbeams, at calculation of beam diameter both at application in the microanalysis [6-9], and for diagnostics of X-ray spots [23].

Apparently, so effect in work [13] there were significant difficulties with finding of divergence of Kumakhov polycapillary semilens by methods of reception of an image of a beam section on various distances. For semilens in the scheme of increase the effect of decreasing of full divergence near exit end face of structure will be more expressed, than for a polycapillary column. It is caused as additional reduction of divergence due to curvature semilens and increasing by semilens of a source size. Thus, for these polycapillary systems, transforming a source size, the formula (1) will be transformed in

$$F_{m.d.} = (k d_{f.s.}) / Q_{max} \quad (4)$$

where k - factor of transformation (increase).

The given effect, apparently, has not been found out earlier because of reception of cross-sections mainly on significant distances from exit end face and that polycapillary microbeams were not considered earlier as a separate kind of quasi-parallel microbeams. By means of Kumakhov optics quasi-parallel beams received by means of semilens, where this effect is difficult for finding out.

Absolute values, and also, apparently, the course of curve of dependence of a beam size on distance from an exit end face of structure will depend on a way of definition of the effective size that has been shown in [18]. In this work for a beam after a diaphragm curve dependences of a beam size on distance from a diaphragm are resulted. And a beam size was defined by next ways: FWHM, FWHM of approximating Gauss curve and size of 90 % counts area. Character of curves differs on absolute size and growth rate with distance.

It is expedient to carry out the further researches of the given effect on dependence on a focal spot size, polycapillary length, moncapillary size, to analyze change at various type of an estimation of a beam size that can give various

characteristics of divergence [18], and also try to describe analytically curve of effective beam size depending on distance from an exit end face of a polycapillary structure.

Divergence vs. Length

Dependence of maximal divergence of PXM on length of a polycapillary is investigated. The maximal divergence was obtained from dependence of section diameter of a polycapillary microbeam on distance between a place of a knife edge scan and an exit end face of polycapillary structure. The tangent of an angle of a tangent line to curves of the dependences, describing divergence, took on a site of the maximal divergence. Change of beam diameter on a site of the maximal divergence is presented on fig. 2B.

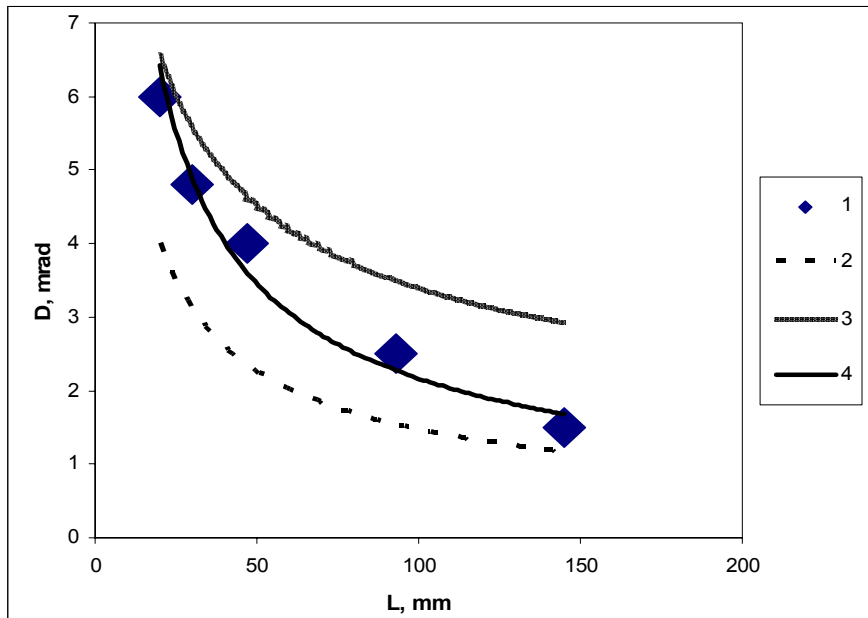


Fig.3 Results of experiment of dependence of the maximal divergence D on polycapillary length L and its modeling in view of waviness on Wang et al.: 1 – Experiment; 2 – Modeling $\Delta\theta_{\max} = 4 \cdot 10^{-4}$; 3 – Modeling $\Delta\theta_{\max} = 1 \cdot 10^{-4}$; 4 – Modeling $\Delta\theta_{\max} = 1.5 \cdot 10^{-4}$; approximating curve – $y = 48.56 x^{-0.68}$.

Investigated polycapillary structures had external diameter of 2,5 mm, internal diameter of a capillary – 2,5 microns. The received dependence is presented on fig.3. The effect of reduction of the maximal divergence with increase in length of polycapillary system is observed.

2.3 Comparison MXM and PXM

Received on the basis of microfocuss X-ray tube the maximal density of a radiation flux as in MXM, so in PXM make value of the order 10^{10} foton / (s mm^2), that is close to density of a flux in the some synchrotron sources (see, for example, [25-27]). But there are following advantages of use of polycapillary structures over monocapillary for the producing of intensive microbeams from a source on base of an X-ray tube.

1. Difficulty of optical system alignment concerning a source sharply decreases.
2. There is an opportunity of reception scanning quasi-parallel microbeam by means of electron beam scanning an X-ray spot of a tube [8,9].
3. The exit field of radiation of a polycapillary differs from those for a monocapillary due to redistribution of radiation by polycapillary structure. There is an effect of decreasing of a full divergence of a polycapillary microbeam near end face of a polycapillary.

4. Due to use of finer capillaries, for reception of demanded divergence the smaller length of structure is required, that also can be advantage to applications.

3. STATISTICAL MODELLING OF X-RAY PASSAGE THROUGH CAPILLARY

The three-dimensional model was applied to calculation of an angle of reflection on the basis of model of Dabagov et. al. [28-30].

2 angles were varied: α and θ , proceeding from which, then counted a angle of falling on a wall:

$$\alpha_{ref} = \arccos \left\{ \frac{\cos \alpha}{\cos[\arctg(\tg \alpha \cdot \sin \theta)]} \right\} \quad (5)$$

and length of run of one reflection:

$$h = 2R \cos \theta \cdot ctg \alpha, \quad (6)$$

where R – radius of a capillary.

Expression for dielectric permittivity was used of the kind:

$$\varepsilon = 1 - \alpha + i\beta, \quad (7)$$

where $0 < \alpha \ll 1$, $\beta \ll 1$.

α - defines an angle of total external reflection:

$$\theta_{\phi} = \sqrt{\alpha}, \quad (8)$$

The factor of reflection of X-rays from substance estimated according to [31]:

$$R_s = \frac{(a - \sin \theta)^2 + b^2}{(a + \sin \theta)^2 + b^2}; \quad (9)$$

$$R_p = R_s \frac{(a - \cos \theta ctg \theta)^2 + b^2}{(a + \cos \theta ctg \theta)^2 + b^2}, \quad (10)$$

where

$$a^2 = 0,5 \cdot [\sqrt{(\sin^2 \theta - \alpha)^2 + \beta^2} + (\sin^2 \theta - \alpha)]$$

$$b^2 = 0,5 \cdot [\sqrt{(\sin^2 \theta - \alpha)^2 + \beta^2} - (\sin^2 \theta - \alpha)].$$

For not polarized radiation:

$$R = (R_s + R_p) / 2. \quad (11)$$

Basic types of deviations of a reflecting surface of a capillary from ideal were considered all: roughness, waviness and bending. In one of the most known models of the account of roughnesses, based on approach Debye by consideration of influence of thermal fluctuations of a crystal lattice on X-ray dispersion, it is applied exponential factor of attenuation to updating Fresnel factor of reflection R_F :

$$R = R_F \cdot \exp[-(4\pi\sigma \cdot \sin \theta / \lambda)^2], \quad (12)$$

where R – factor of reflection of a rough surface, λ -wave length and θ - incident angle of X-ray radiation, σ -the varied parameter describing a roughness. Use of the given model for capillary optics is presented in work Vincze et al. [32].

The account of a waviness of a surface in model Wang et al. [33], applied by us, as a matter of fact, is similar to model of roughness of Arkadiev, based on the random additive to an angle of reflection in a certain interval [14]. In addition restrictions on negative angles are presented:

$$-\Delta\theta_{\max} \leq \Delta\theta \leq \Delta\theta_{\max}, \text{ if } \theta \geq \Delta\theta_{\max} \quad (13)$$

and

$$-\theta \leq \Delta\theta \leq \Delta\theta_{\max}, \text{ if } \theta < \Delta\theta_{\max}, \quad (14)$$

where θ - incidence angle, $\Delta\theta_{\max}$ - limiting size of a deviation of a angle of reflection from specular reflection.

In case of presence of a capillary bend at each subsequent reflection to a reflection angle the value $\Delta\alpha_{ref}$, defined by an inclination of tangents planes to a capillary in points of beam falling in view of a capillary bend, was added:

$$\Delta\alpha_{ref} = (-1)^n h / R_{bend}, \quad (15)$$

where R_{bend} – radius of a capillary curvature, and n – number of reflection. Reduction of length of run of one reflection owing to a bend was considered as negligible value.

Modeling results of dependence of monocabillary transmission on its length are presented on fig.1. Modeling data qualitatively enough describe experimental.

On fig.3 results of modeling of dependence of divergence on length are presented. It is established, that experimental data are well enough described by model Wang et al. Value of factor close to received in [14] value - 10^{-4} .

4. DEVICES WITH POLYCAPILLARY X-RAY MICROBEAMS

The applications of above-stated researches of polycapillary microbeams are the opportunity of additional optimization of devices on the base of Kumakhov optics. Nowadays on base of polycapillary optics at the IRO the new generation of devices is developed, created and commercially accessible: «laboratory synchrotron» [5], fluorescent spectrometers, reflectometer, diffractometer, X-ray microscopes [34,35] and combinations of several devices in one [2,27].

4.1 Microfocus high flux X-ray source: «Laboratory Synchrotron»

The microfocus X-ray source «SBXRS 1.0» is developed for creation of beams of the maximal flux density (diverging, quasi-parallel or converging).

Application of specially developed X-ray tube and system of electromagnetic focusing of an electronic beam has allowed to produce the size of an anode spot of a tube no more than 20 microns in diameter in which the record density of radiation is realized owing to the small sizes. In a source are used microcapillary X-ray optic systems of Kumakhov optics that allows using radiation of an X-ray tube most effectively.

For producing of quasi-parallel beam in diameter 0,1-1 mm the source is equipped X-ray semilens, which will transform diverging beam from an X-ray tube in quasi-parallel. It allows at capacity of a source 20 W to produce a beam of XR on a number of parameters compared with the beams received on workstations of synchrotron sources with bending magnets. For producing of converging beams the full lens is used. Use of a full lens allows to obtain intensity in a focal spot of the order 10^8 photon/s in a spot in diameter ~10 microns.

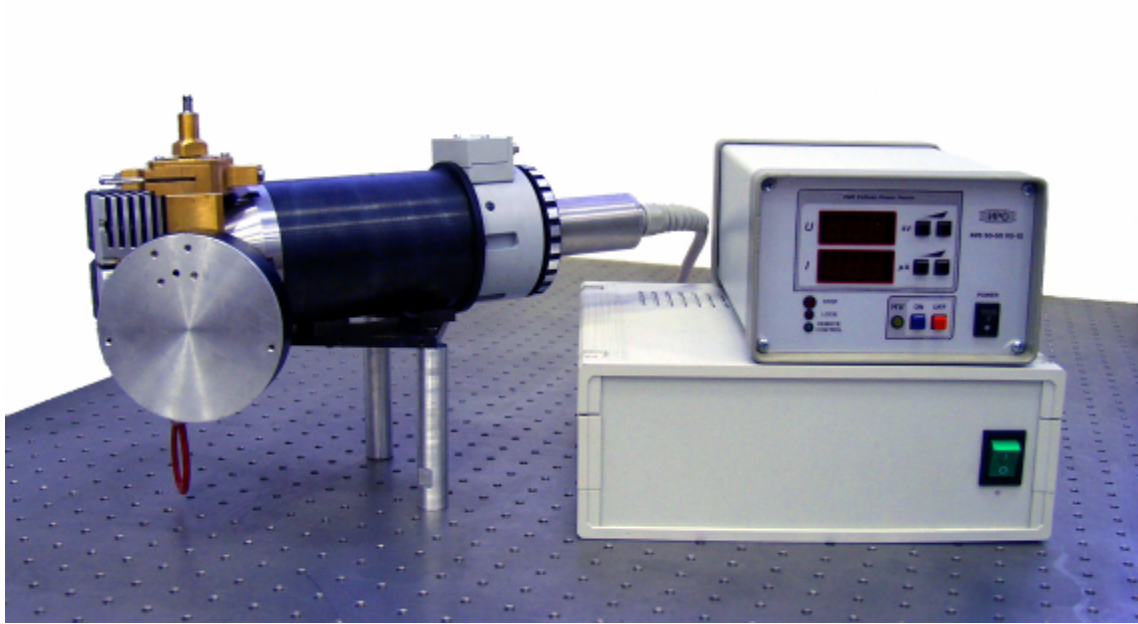


Fig.4. «Laboratory synchrotron» – sources of X-ray radiation of high flux density on a base of microfocus X-ray tube and Kumakhov polycapillary optics: «SBXRS 1.0».

Table. 1. «Laboratory synchrotron» parameters.

Key parameters

Maximum power - 50 W.

Maximum anode voltage - 50 kV.

Material of the anode of an X-ray tube - Cu, Mo.

The size of a tube focal spot - no more than 20 microns.

X-ray tubes – sealed off with the mirror anode.

Operating mode - long-term.

Gross weight of system - no more than 25 kg.

Beams parameters at using semilens

Diameter of a beam - 0,1-1 mm.

Radiation flux density of a beam - 10^{10} photon/s/mm².

Divergence of a beam - 4 mrad for Cu; 2 mrad for Mo.

Beams parameters at using full lens

Diameter of a focal spot of a lens - 10-50 microns.

Radiation flux density of a beam - the order 10^{12} photon/s/mm²

Focus length of a lens - 2-10 mm.

The source consists of a generator and two power units. In a source realized auto-focusing of X-ray optics, concerning a source, that as much as possible simplifies adjustment of system at change of optics and X-ray tube.

«Laboratory Synchrotron» can be used for microdiffractometry, including for protein crystals; diffractometry of crystal materials under a high pressure; phase contrast investigations; projective microscopy; small-angle X-ray scattering method etc.

Now IRO has finished the experiments which have shown an opportunity of manufacturing type of a source with highly polarized radiation for scientific researches, including for research of magnetic properties for various materials.

4.2 New generation of analytical instruments

The portable X-ray microanalyzer for element analysis

In structure of the portable X-ray microanalyzer for element analysis [36] is used Kumakhov lens with a focal spot 50-250 microns. Due to use of Kumakhov polycapillary optics the highest efficiency of the device (sensitivity up to 10 ppm) together with increase of compactness (only 15 kg) and reduction of power consumption (an X-ray source only 50 W) is achieved.

Series multifunctional diffractometers for phase, stress and element analysis

Multifunctional diffractometers for phase, stress and element analysis are combination diffractometry and the fluorescent analysis in one device [37, 38]. By means of application of Kumakhov optics no necessity for use monochromators and collimators that has allowed to reduce the sizes of the device at preservation of an opportunity of research of the big samples.

Portable X-ray diffractometers

Portable X-ray diffractometers with Kumakhov polycapillary semilense are used for stress analysis of a responsible details, units and designs [27]. Due to use of Kumakhov polycapillary optics the highest efficiency of the device together with increase of compactness (only 14 kg) and reduction of power consumption (a X-ray source only 10 (5) W) is achieved.

«X-ray MiniLab»

«X-ray MiniLab» [39] offers to material research labs in science and industry a new versatile X-ray instrument based on latest developments in focusing polycapillary optics, semitransparent monochromators, and new conceptual design. This compact facility provides high precision, friendly interface, and unique combination of analytical opportunities: diffractometry; reflectometry; refractometry; small-angle scattering; local x-ray fluorescence. Controlled parameters: surface and interface roughness (down to 0,1 nm); thin layer thickness (1-400 nm); surface layer density; structure period; composition of layers; radius and concentration of nanoparticles; surface and interface roughness (down to 0,1 nm); radius of curvature (up to 300 m).

X-ray Optical Microscope

X-ray Optical Microscope with local composition analyzer [35] - an advanced X-Ray facility providing versatile imaging and varieties of analytical opportunities. There are such operation modes: X-ray microscopy; optical microscopy; local X-ray fluorescence; sterilization by focused radiation; microdiffraction. The design is based on innovative research in polycapillary optics, spectrum modulation, and digital data procession: simultaneous X-Ray and optical imaging; fully automated data acquisition; fast exposures: down to 0,1 s; local composition and structure analysis; complete radiation safety.

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