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**NOVEL HIGH FLUX X-RAY SOURCE:  
A LABORATORY SYNCHROTRON**

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**Abstract**

A novel x-ray laboratory system optimized for diffraction studies of protein crystals and macromolecules as well as for phase contrast analysis and other applications is described. The system is based on a micro-focus X-ray source of 10W power and provides the minimum focal spot size of  $\sim 10 \mu\text{m}$  and the flux of  $\sim 4.5 \times 10^{12}$  ph/sec at the Cu K $\alpha$  line.

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## 1 INTRODUCTION

Achievements in X-ray optics stimulated interest to creation of new radiation sources, which in a combination with optics enables considerably to improve the results of measurements in X-ray optics. In particular, the big progress is observed in the technology of capillary optical elements that, in turn, allows X-ray bunches to be rather effectively operated [1]. These optical elements consist of many thousands hollow tapered tubes (channels) that condense the radiation by multiple reflections from the inner channel surface. The main parameter, in most cases, is the density of a beam of radiation that can be raised on some orders by the use of capillary optical samples [2-4]. Due to this fact the given optics is widely used in various laboratories operating both with traditional sources of radiation and with synchrotron radiation sources [5]. However, it is necessary to note, that the basic advantage of capillary optics, most likely, is the fact that it provides essential increase of the efficiency of desktop X-ray sources. Quite big angular aperture and opportunity of turn of a radiation bunch through large angle with simultaneous focusing in a spot of small diameter allow the density of radiation comparable with that from synchrotron sources to be reached on traditional laboratory sources of X-radiation.

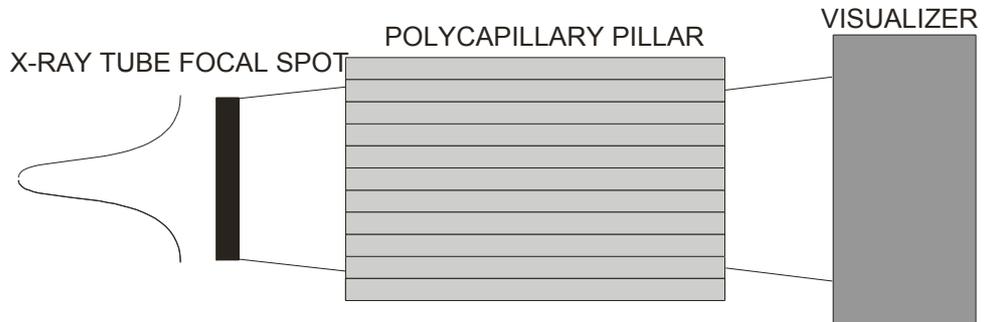
In the given work the study of a new X-ray source representing a micro focus tube in combination with capillary optics is resulted. The first data allow us to conclude, that by correct optimization of parameters of a tube and optics it is possible to create a powerful source of X-radiation. The new source, despite of high density of radiation on an output, is compact and can be quite well attributed to the class of desktop X-ray sources.

## 2 MICRO FOCUS X-RAY SOURCE

It is obvious that the increase of radiation density can be received by means of optics. However, as the analysis shows, a source already defines a certain limit, which can be realized in a specific experiment. Therefore in order to improve radiation characteristics it is necessary to optimize not only parameters of optics, but also of a source. One of the most important characteristics of a source is the size of a focal spot. Its reduction provides essential increase of overall source performance, some problems with a heat-conducting path become simpler and the efficiency of the optics sharply rises.

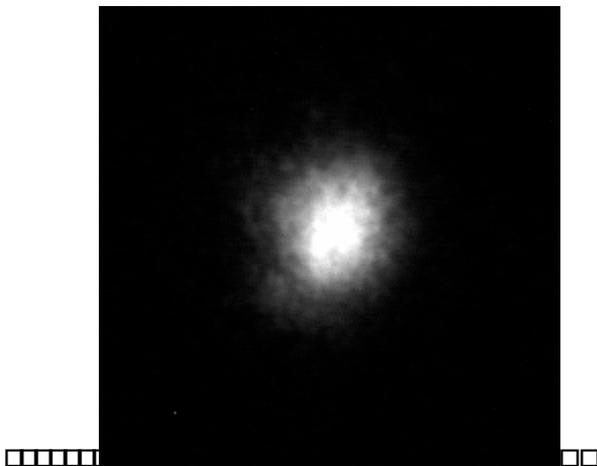
As the first step an X-ray micro focus generator has been developed on the base of transmission anode X-ray tube with a point focus. For the purpose of adjustment the generator provides voltage on the tube within the range of about 40 kV and current of about 500  $\mu$ A, maximum tube power is about 10 W. The source contains an electromagnetic system allowing electron beam formation inside the tube. The system comprises a focusing coil to ensure electron beam compression into a spot with diameter of down to 10  $\mu$ m on the target and also two deflecting windings. One of them is used for electron beam alignment in two mutually perpendicular directions. The results obtained by a Cu anode X-ray tube will be presented below.

To assess the tube focal spot size, a polycapillary pillar (straight collimator system) was used with a channel diameter of 2  $\mu\text{m}$ . An image was registered with the help of a 2D X-ray visualizer with a spatial resolution of 2  $\mu\text{m}$ . The experiment's block scheme is shown in Fig. 1.



*Fig. 1. The block diagram of tube focal spot size assessment experiment.*

For the system calibration the polycapillary pillar was replaced with a thick-walled moncapillary (single capillary) with 18  $\mu\text{m}$  diameter of hole; image was registered at the exit in the same experimental geometry conditions. The images obtained are shown in Figs. 2 and 3.



*Fig.2. Image of X-ray tube anode.*



*Fig.3. Image at the exit of 18  $\mu\text{m}$  moncapillary.*

Analysis of the images has shown that the focal spot size of the tube is much less than 18  $\mu\text{m}$ .

More accurately, the focal spot size was measured with the help of extended collimator, consisting of two polished glass plates, 70x70 mm in size. In this case, the distance between closely jammed plates makes  $\sim 1 \mu\text{m}$ . In order to increase the slit size and, respectively, the flux of quanta passing through the obtained gap, the 5  $\mu\text{m}$ -thick foil strips were placed between the plates. The

central plate part was grinded to prevent X-ray total external reflection and to reduce the inlet angular acceptance of a collimator (Fig. 4).

In our experiment, the collimator was placed on a special holder movable in three orthogonal directions. The step of scanning was 2  $\mu$ m. This ensured precise alignment of the collimator respect to the X-ray focal spot. X-radiation was registered with the help of a semiconductor detector. Measurements were done under the following X-ray source mode of operation: 40 kV voltage, and 250  $\mu$ A current.

Results of the measurements are given on Fig. 5.

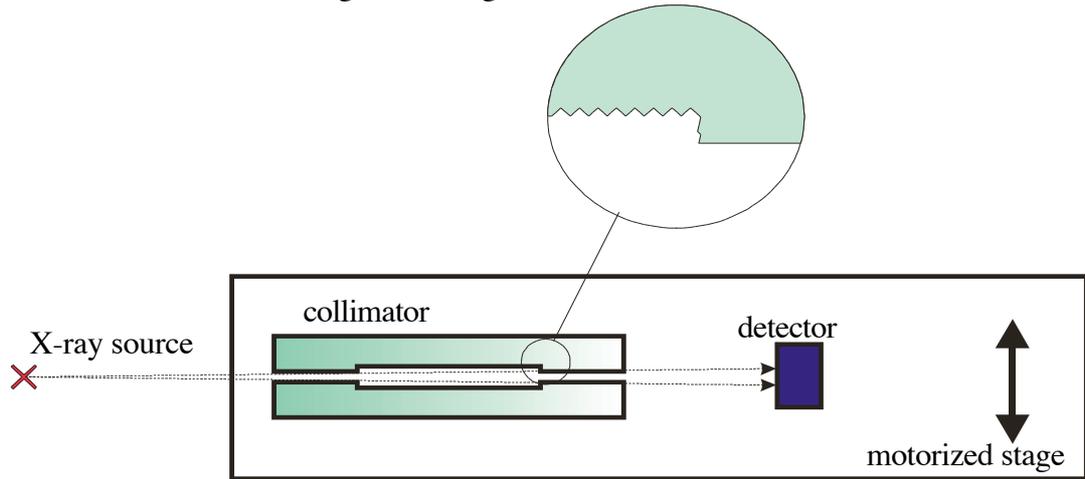


Fig.4. Block-diagram of collimator for measurement of X-ray focal spot size.

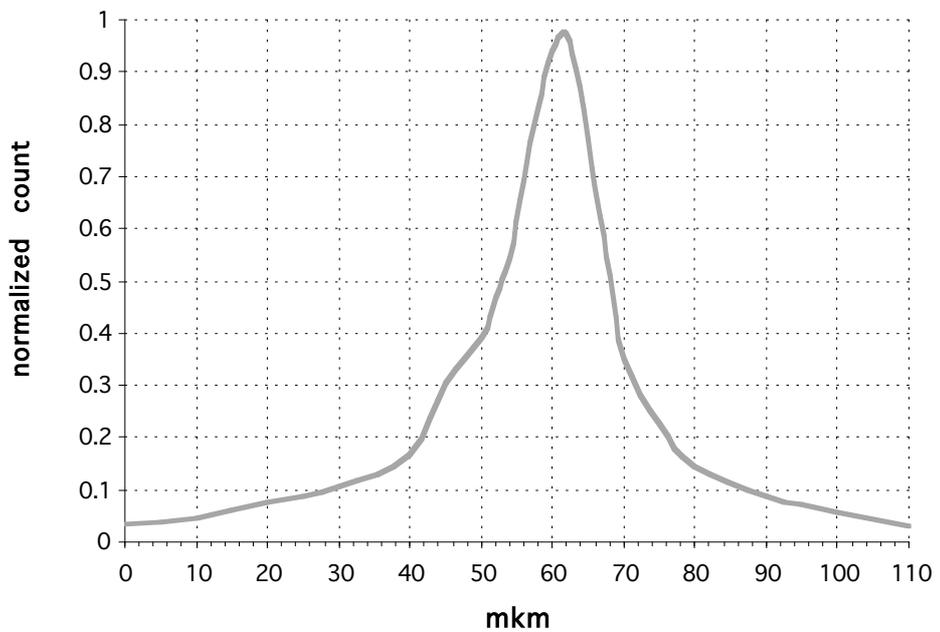
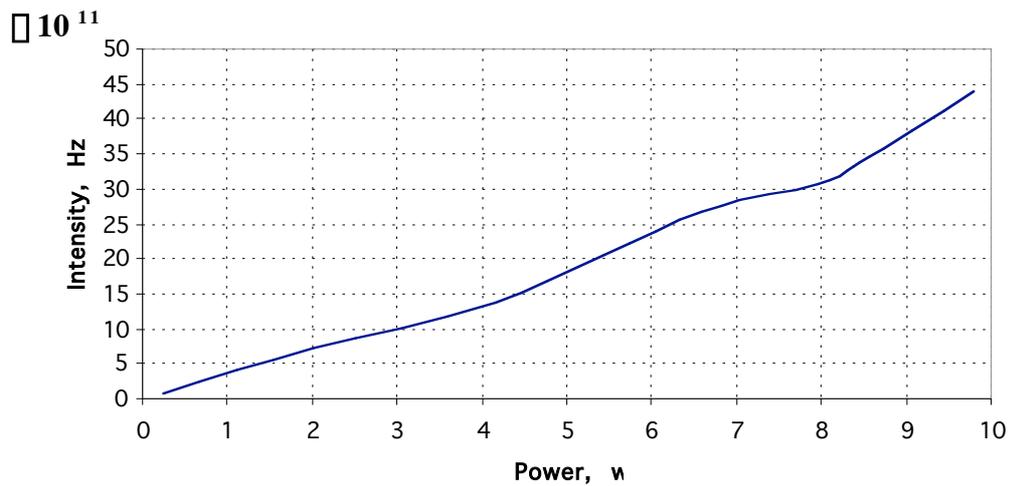


Fig.5. Intensity distribution in X-ray tube focus.

The obtained diagram shows that the focal spot size measured at half maximum of the curve (FWHM) less than effective size of the collimator slit. Asymmetric nature of the curve can be explained by the fact that the flux density of electrons hitting the X-ray tube anode has uneven distribution.

### 3 INTEGRAL TUBE FLUX STUDY

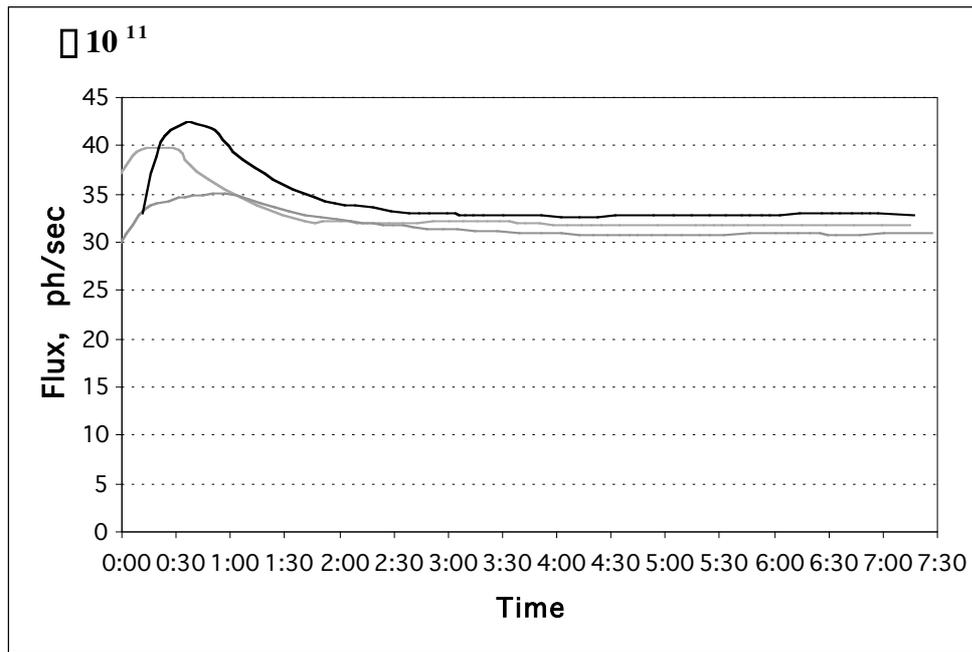
Integral X-ray flux measurements were done using a semiconductor Amptek XR-100T detector installed at 1 m distance from the source of radiation. To reduce the X-ray flux at the detector's inlet, a diaphragm was installed having a hole of 100  $\mu\text{m}$  in diameter. In addition, in order to suppress Cu  $K_{\alpha}$  rays, a 10 $\mu\text{m}$ -thick nickel filter was placed behind the diaphragm. Experiments were performed at 40 kV voltage of the X-ray source. The results of measurements are presented in Fig. 6. It is seen from the diagram, that at 10 W in power, the source emits  $4.5 \times 10^{12}$  monochromatic photons per second.



*Fig.6. Integral intensity of X-ray source.*

For any type of radiation the most important characteristics is temporal flux stability. Study of this parameter was done in the same way as in the previous experiment. The results of measurements are given in Fig. 7.

These experiments have proven that after the system warming-up, deviations from the mean value do not exceed 0.8 % for 5 hours of operation. Maximum temperature on the housing surface is about 40° C.



*Fig. 7. Investigation of source's long-term stability.*

#### **4 PHASE CONTRAST ANALYSIS BY MICRO FOCUS TUBE**

The above described X-ray tube will have a wide range of applications. We used it in a set of applications, but the most attractive result was obtained in phase-contrast analysis experiment.

Obviously, the first very simple idea is to use it for microscopic studies. Herein, the contrast resolution can be significantly improved due to the small source size. Moreover, it is particularly convenient, because in such a system with transmission anode configuration any object can be placed very close to the anode, thus ensuring the maximum magnification of the object under the study.



*Fig.8. X-ray image of a fish body.*

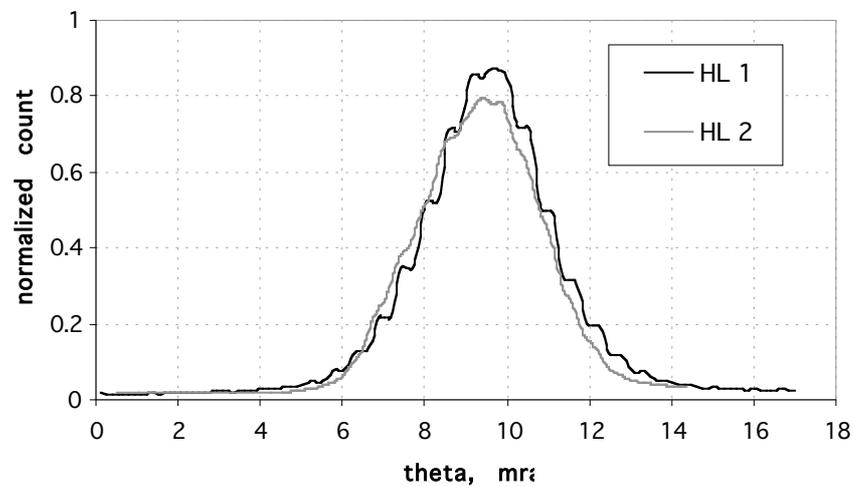
In addition, radiation goes to a semi-sphere permitting a concurrent investigation of several objects.

The system was used for phase-contrast imaging in weak absorption objects []. In this instance, an improvement in the clarity of X-ray image, providing greater definition of weakly absorbing features in a fish body, was obtained (Fig. 8).

## 5 HALF-LENS PARAMETERS

To achieve maximum density of quasi-parallel radiation for a narrow beam, we have used a half-lens (polycapillary half lens), which was optimized to obtain the best correlation between the capture angle (acceptance) and the exit diameter. To satisfy this requirement, it is necessary to have a half-lens with minimum focal length. Thus, the half-lenses with the focal lengths of about 3 mm and less were fabricated. Transmission of the central part (300  $\mu\text{m}$ ) of the developed half-lenses approaches 35-40%.

In order to measure the beam divergence, a diaphragm with 0.3 mm diameter was installed at the half-lens exit and aligned to obtain the maximum X-ray flux. Then the X-ray beam passed a Si monochromator, plane (400), mounted on a motorized  $\square$ -2 $\square$  goniometer that allowed a rocking curve to be taken. The obtained curves are given in Fig. 8.



*Fig.9. Angular divergence of radiation behind polycapillary half-lenses.*

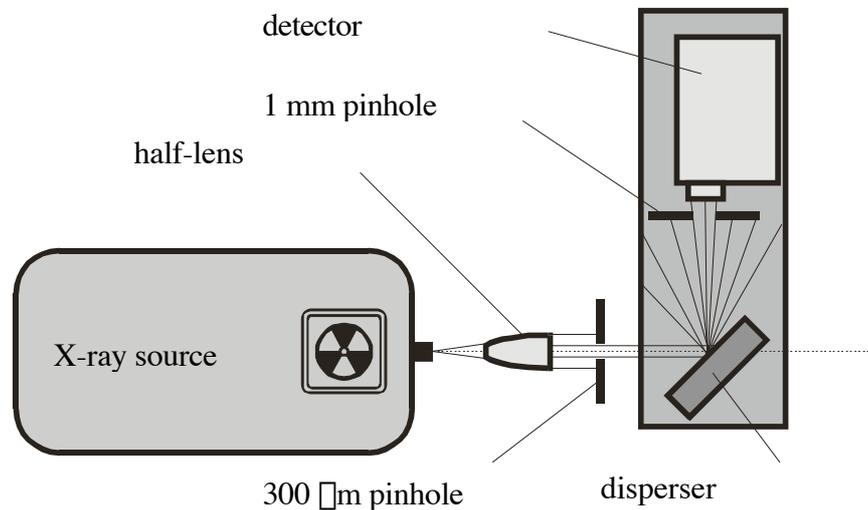
Analysis of these curves allows us to define the divergence of quasi-parallel beam to be of the order of 3.5 mrad.

## 6 FLUX MEASUREMENTS FOR MICRO SOURCE & OPTICS ASSEMBLY

The first tests of a new micro focus source have shown perspectives of research continuation. However, as follows from the simple analysis, irrespective of absolutely new solutions there is a certain limit of an ability of any source that is impossible to overcome by only physical principles. Obviously thus, that for improvement of source characteristics, application of a certain optics is necessary. In our case the combination of the offered new source (microfocus X-ray source) with the capillary lens (the half-lens) optimized for a concrete source was investigated.

The system flux was measured using a semiconductor Amptek XR-100T detector. As above mentioned, at the exit of half-lens a diaphragm of 0.3 mm in diameter was installed as well as a 9  $\mu\text{m}$ -thick Ni filter to obtain monochromatic radiation. Further along the beam path, at a distance of 45 mm from the lens exit, an acrylic resin disperser was installed. This method allowed us to avoid saturation on the detector due to extremely high flux of radiation. The detector with 1 mm diaphragm at the inlet was placed at 35 mm distance from the point where X-rays hit the disperser. The disperser plus detector system with the same geometry was calibrated to determine a conversion rate. Measurements were done with the source of 10 W power. It was shown that under the given parameters, the half-lens forms a flux with intensity of  $2 \times 10^8$  ph/sec at the exit of  $\varnothing 300$   $\mu\text{m}$  diaphragm, and makes  $2.8 \times 10^9$  ph/sec/mm<sup>2</sup> for 1 mm<sup>2</sup> area.

As seen this is a record flux for desktop (laboratory) sources comparable with those obtained at synchrotron facilities.



*Fig.10. Scheme of experiment for the flux measurement of the assembly of point source and half-lens.*

## 7 CONCLUSION

A micro focus generator has been created featuring a transmission anode with about 10  $\mu\text{m}$  focal spot and 10 W power, i.e. the electron beam density on the anode makes  $10^7 \text{ W/cm}^2$ . This is an unprecedented density for a transmission type of anode by order higher than that at the rotation anodes [7].

The micro focus source has a wide range of applications. In particular, it was used for phase-contrast imaging of biological objects. In this instance very good results were obtained. The development of this new technology has the potential to provide new applications in medical, industrial and biological environments.□

Moreover, the importance of this source of X-radiation will be increased by its combination with definite optics. For instance, we have used a micro focus source-polycapillary half-lens system for protein crystal studies that generally require high-intensity beams from tens to several hundreds microns in diameter [8]. A quasi-parallel beam of about 20  $\mu\text{m}$  in diameter and  $3 \times 10^{10} \text{ ph/sec/mm}^2$  flux density was obtained by means of the half-lens. It is important that the system permits using tubes with various anode materials that is convenient for express protein crystal investigations.

Thus, on the basis of micro focus source and polycapillary optics, a unique source has been created that has no analogues in the world. With its help, an unprecedented quasi-parallel flux of monochromatic X-radiation with density varying from  $3 \times 10^9 \text{ ph/sec/mm}^2$  to  $3 \times 10^{10} \text{ ph/sec/mm}^2$  in dependence on the beam size was detected. The obtained fluxes largely exceed the fluxes obtained at rotation anodes and are comparable with the fluxes usually obtained at synchrotron workstations. This source was named a "Laboratory Synchrotron" [9]. There is also another point to be mentioned, namely, research carried out using the synchrotron workstations has limitations in terms of time and convenience. A Laboratory Synchrotron represents a breakthrough in the production of high flux density X-ray beams, and, on the contrary, offers freedom of creativity to scientists for use at their own laboratories.

## 8 REFERENCES

- [1] M.A. Kumakhov, *Radiation of Channeling Particles in Crystals* (Energoatomizdat, Moscow: 1986).
- [2] M.A. Kumakhov, and F.F. Komarov, *Phys. Rep.* **191** (1990) 290.
- [3] P. Engström, S. Larsson, and A. Rindby, *Nucl. Instr. Meth.* A302 (1991) 547.
- [4] D.J. Thiel, D.H. Bilderback, A. Lewis, and E.A. Stern, *Nucl. Instr. Meth.* A317 (1992) 597.
- [5] S.B. Dabagov, *Physics-Uspokhi* **173** (No. 10) (2003).
- [6] T. J. Davis, D. Gao, T. E. Gureyev, A. W. Stevenson, and S. W. Wilkins, *Nature* **373** (1995) 595.
- [7] <http://www.msc.com/protein/brochures/ruh-brochure.pdf>
- [8] D.E. McRee, *Practical Protein Crystallography*, Sec. Edit. (San Diego, London, New York, etc., Academic Press: 1999).
- [9] <http://www.unisantis.com>