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Design and Application of X-Ray Lens in the Form of Glass Capillary Filled by a Set of Concave Epoxy Microlenses

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1. Introduction

Glass capillaries are widely used in X- optics. It is a well know fact that a simple glass capillary acts as a waveguide for X-rays because refractive index for X-rays in any medium is less than unity. X-rays transmit glass capillary in the regime of total external reflection instead of visual light that propagates inside glass fiber in the regime of total internal reflection. X-rays also may transmit curved capillaries and, as was proposed by Kumakhov, a bunch of curved capillaries act as a lens that focuses X-rays from a point source into a focal point. This device is known as Kumakhov X-ray lens (Kumakhov & Sharov, 1992). Another well-known X-ray device is a taper or parabolic single capillary that is used to condense or focus synchrotron X-rays into micron-sized spot (Thiel et al., 1992).

Recently a new application of glass capillaries for X-ray optics was proposed: it was demonstrated that capillaries are suitable for designing so named compound refractive X-ray lenses.

Compound refractive X-ray lens at the first time was proposed by A. Snigirev, V. Kohn, I. Snigireva and B. Lengeler (Snigirev et al., 1996) and their idea is based on the following principles. It is a well-known fact that refractive lens for X-rays should be concave instead of convex for visual light. Calculation shown that the focal length F_1 of such biconcave spherical lens is determined by the following ratio:

$$F_1 = R / (2 \delta), \quad (1)$$

where R -radius of the lens and $(1-\delta)$ is real part of refractive index n . The focal length of the lens is rather large (5-10 m for 5-8 keV X-rays) even when the curvature radius of the lens is equal to hundred of micrometers. The large value of the lens focal length was a reason of the conclusion that there is no any practical interest to focus X-rays by refractive lens. Attempts to reduce focal length of the lens have resulted in creation of a compound refractive lens (CRL) for X-rays with energy 5-30 keV (Snigirev et al., 1996). The lens consists of a large number (10-300) of biconcave lenses, made of material with a low-atomic weight (beryllium, carbon, polymers, aluminium). Focal length F of such lens is defined by the following ratio: $F = F_1 / N$, where N - is the number of lenses. The equation for F shows that the focal length

of a compound lens can reach value of 1 m at $R=0.5$ mm. That is quite acceptable for practical applications.

At present compound X-ray lenses are designed by some ways: by using pressing technique for individual lens, by lithographic method, by drilling holes in a plate by a laser (Lengeler et al., 2005). The problem of the lens design is how to produce individual concave lens with a high quality parabolic or spherical shape surface and with curvature radius up to 50 microns or less. Another problem is to stack the lenses coaxially to form compound lens.

The idea of compound X-ray lens was advanced in our work (Dudchik & Kolchevsky, 1999), where it was realised in the form of glass capillary filled by a large number of epoxy drops. The lenses were designed at the Institute of Applied Physics Problems of Belarus State University. The lens was named as microcapillary one and applied at synchrotron SPring-8 for focusing of 18 keV X-rays and as an objective of X-ray microscope (Kohmura et al., 1999). The lens consists of a glass microcapillary, filled by a plenty of biconcave microlenses. The concave microlenses inside the capillary were formed by putting air bubbles into epoxy. The schematic view of the lens is shown in Fig. 1.

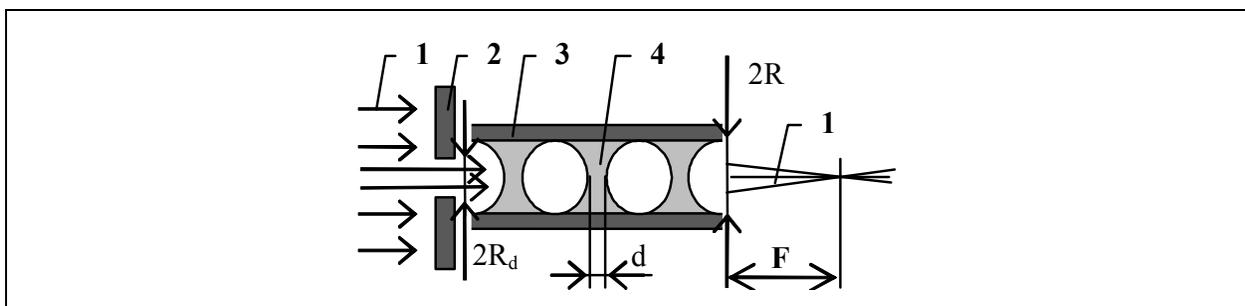


Fig. 1. Schematic view of the microcapillary X-ray lens. 1- X-ray beam; 2- diaphragm; 3- capillary; 4- epoxy lens

It was shown (Dudchik et al., 2000) that the microlenses inside the capillary are spherical ones and its curvature radius is equal to capillary one. This founded dependence of the lens curvature radius on the capillary one leaves a room to decrease the lens focal length. For example the lens in the form of 200 microns in diameter capillary and filled by 103 microlenses has 13-cm focal length for 8 keV X-rays. It was shown experimentally by Adelphi Technology, Inc. using beamline 2-3 at the Stanford Synchrotron Radiation Laboratory (SSRL) (Dudchik et al., 2004). Such short-focal-length lenses are suitable for imaging not only with synchrotron X-rays, but with X-rays from laboratory sources of radiation (Piestrup et al., 2005).

The purpose of the paper is to consider details of microcapillary lens design as the lens application for focusing and imaging of X-rays.

2. Design and application of microcapillary X-ray lens

2.1 Fabrication technique for the microcapillary refractive X-ray lens

The method of the microcapillary lens preparation consists (Dudchik & Kolchevsky, 1999; Dudchik et al., 2000) in consecutive producing of air bubbles inside of capillary 1, filled by epoxy 2 with using of capillary 4, connected with a cylinder with compressed air 5, as is

shown in Fig.2. The growth of the bubble inside of the capillary 1 is supervised by visual light microscope. When the radius of the bubble is becoming equal to the radius of the capillary 1, the capillary 4 is moving to a distance of few microns from the received bubble and the process is repeated. The liquid between two bubbles has a form of biconcave lens. This technique actually has not restrictions in number of lenses. The photo of epoxy lenses, made by the method, is shown in Fig. 3. The diameter of the capillary is equal to 0.2 mm (a) and 0.8 mm (b). The air bubbles between lenses are observed as black ones. Used epoxy consists of carbon, oxygen, hydrogen and nitrogen which are chemically bonded in proportion $C_{200}H_{100}O_{20}N$. The epoxy density is 1.08 g/cc.

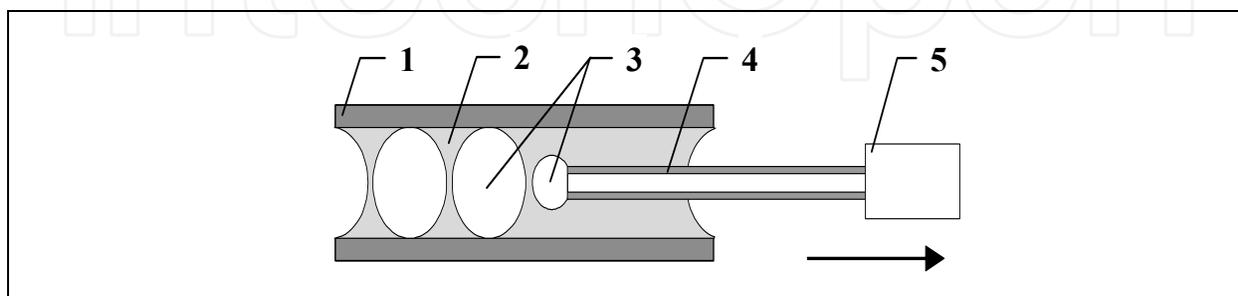


Fig. 2. Schematic view of the setup for microcapillary X-ray lens fabrication. 1- glass capillary tube; 2- glue; 3- air bubbles; 4- injector needle; 5- cylinder with a compressed air

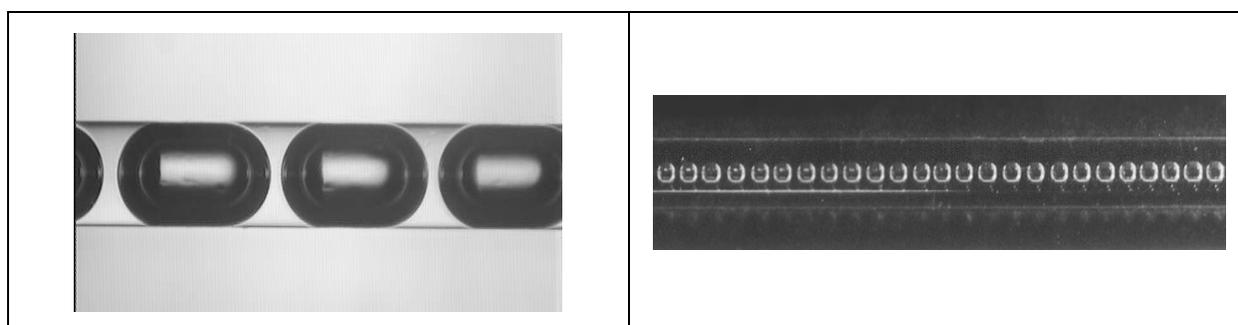


Fig. 3. Visible light microscope image of the microcapillary refractive X-ray lens. The diameter of the capillary is equal to 0.2 mm (a) and 0.8 mm (b).

Important parameter of the lens is thickness d (Fig.1), which for the given material of the lens depends on the diameter of the capillary channel and on the epoxy temperature. We established that for the lens made from epoxy, the lens thickness d might be decreased up to 5-10 microns.

The shape of the lens surface was investigated by an optical method with the help of optical microscope connected with digital camera. The obtained individual lens image was processed by computer. In Fig. 4 (a, b) the images of two lenses are shown. The curve, dividing a light and a dark parts in Fig. 4 was considered as a profile of the lens. At construction of the lens profile we took into account, that the visible lens diameter $2R$ is more than the real diameter of the channel $2R_{real}$ (Fig. 5).

We took into account that the light, that scatters from the inner wall of the lens, does not come directly to the microscope. It is doubly refracted at the (lens-material)-glass and glass-air boundaries. This is a reason why the observed lens profile differs from the real one.

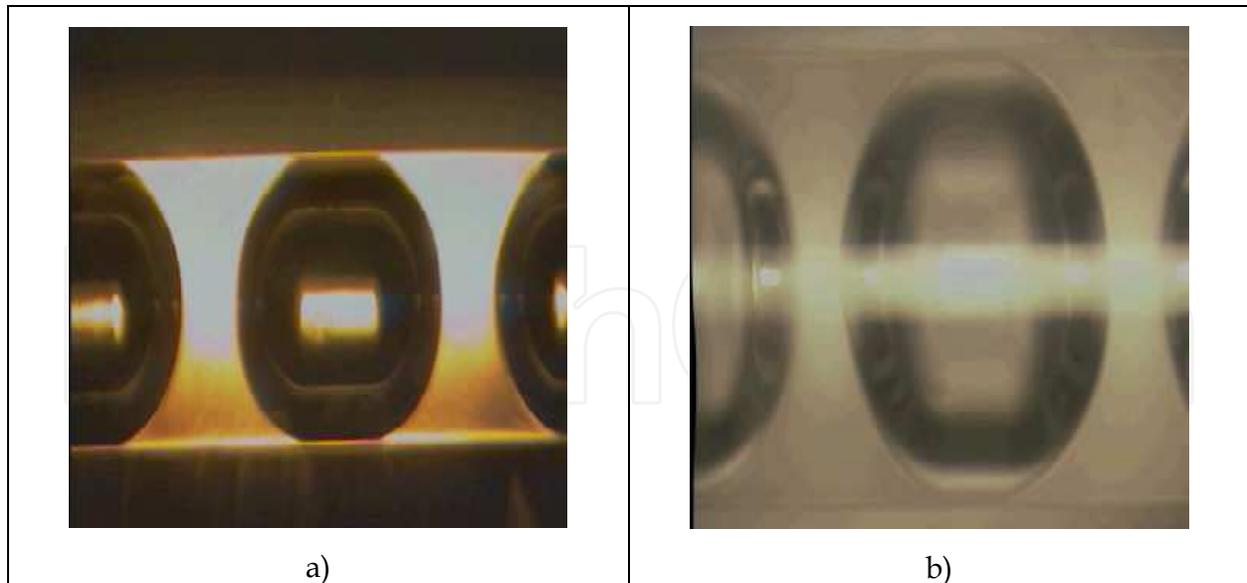


Fig. 4. Visible light microscope images of concave epoxy lenses inside capillary. a) Capillary radius is equal to 0.39 mm; b) capillary radius is equal to 0.21 mm

Formulas for calculating lens profile can be found from a geometrical paths of rays, forming the image of the lens (Fig. 5.). According to the Snell's law:

$$n_{lens} \sin \alpha = n_{glass} \sin \beta; \quad n_{glass} \sin \gamma = \sin \theta, \quad (2)$$

where n_{glass} is the index of refraction of the glass, n_{lens} is the index of refraction of the lens material, $\sin \alpha = R / (n_{lens} R_{chan})$, $\sin \beta = R / (n_{glass} R_{chan})$, $\sin \gamma = R / (n_{glass} R_{cap})$, $\sin \theta = R / R_{cap}$.

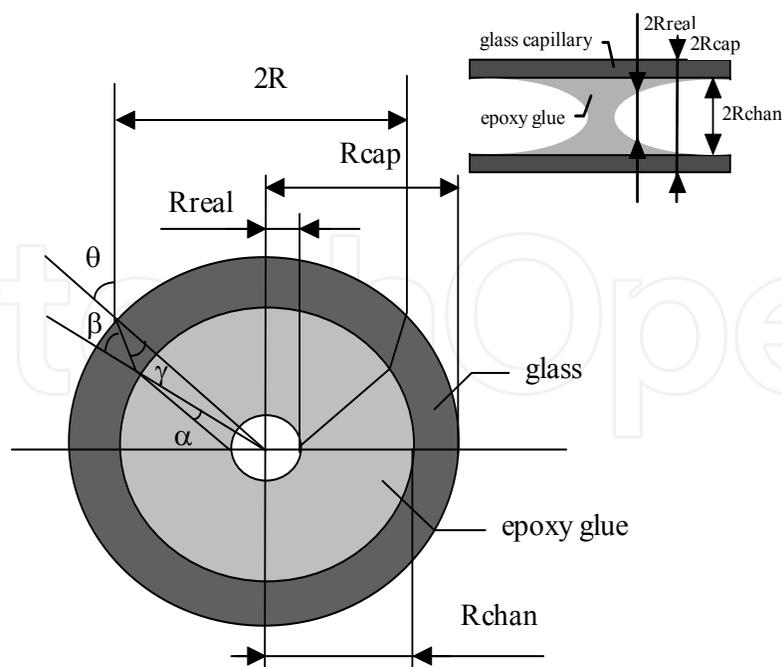


Fig. 5. Schematic view of the transverse section of the microcapillary lens. The rays of visible light forming lens image also are shown. R_{cap} - is the outer radius of the capillary; R_{chan} - is the radius of the capillary channel; R_{real} - is the measured value of the profile; R - visible value of R_{real} .

From eq.(2) the radius of the channel, shown in fig. 4 as R_{real} , is equal to:

$$R_{real} = R / (n_{lens} \cos(\alpha + \gamma - \theta - \beta)). \quad (3)$$

The obtained formula for R_{real} was used for calculation of the lens profile. Fig. 6 shows the profile of lens in comparison to the circle, radius of which is equal to the radius of the capillary. As it can be seen from Fig. 6, the form of the microcapillary lens can be accepted as spherical one, and the radius of lens curvature R is equal to the channel radius R .

This result is in a good agreement with classical molecular theory. The theory states that the form of liquid drop putt into microcapillary can be accepted as biconcave spherical, and the radius of drop curvature is connected by the following ratio to the capillary radius R_{chan} :

$$R = R_{chan} / \cos \varphi, \quad (4)$$

where φ - angle of contact. For epoxy glue located on a glass surface, angle of contact is equal to 0° .

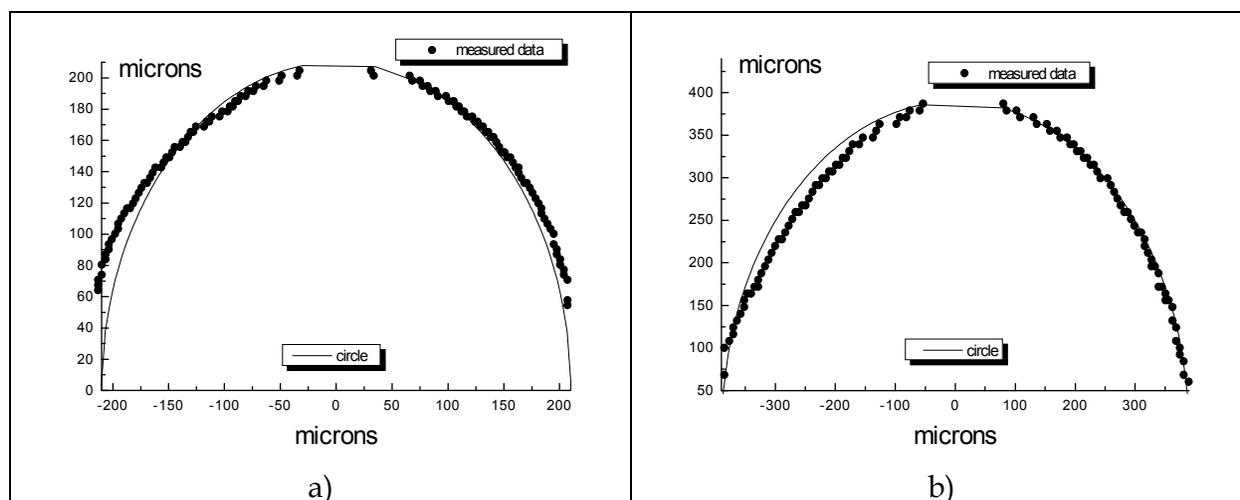


Fig. 6. Measured profiles of the lenses. a) Capillary radius is 210 microns; b- capillary radius is 390 microns

2.2 Parameters of the microcapillary refractive lens

Lens focal length f of compound refractive X-ray lens is calculated as (Snigirev et al., 1996):

$$f = \frac{R}{2N\delta}, \quad (5)$$

where R - curvature radius, N - number of microlenses, $(1-\delta)$ - real part of refractive index for X-rays. Parameter δ for used epoxy may be calculated from the epoxy chemical formula as

$$\delta = 0.5 \left(\frac{22}{E} \right)^2, \quad (6)$$

where E is photon energy measured in eV. Experiments on measuring lens focal length of compound epoxy lenses at Stanford Synchrotron Radiation Laboratory and at Advanced

Photon Source by Adelphi Technology, Inc. shown validity of formula 6 for calculation lens focal length (Dudchik et al., 2004).

Compound X-ray lens consisting of spherical microlenses may be characterized by absorption aperture radius R_a that in a good approximation can be calculated as (Snigirev et al., 1996; Dudchik et al., 2004; Piestrup et al., 2005):

$$R_a = \left(\frac{2R}{\mu N} \right)^{\frac{1}{2}}, \quad (7)$$

where μ is the linear absorption coefficient for the lens material.

The discussed X-ray lens is a linear combination of spherical microlenses and spherical aberrations occur just in the same way as for spherical visual-light lens. To take into account this phenomenon at least two planes around the lens focus may be denoted: they are shown by the lines MS and PP in Fig.7 which shows trajectories of 8-keV X-rays forming focal spot of compound lens consisting of 103-microlenses.

The plane PP represents a focal plane. The plane MS represent the circle of the least confusion (Born @ Wolf, 1975). For spherical lens the size of X-ray beam at MS and PP planes may be decreased by using diaphragm. The beam radius R_{pp} at the PP-plane depends on the radius of used diaphragm R_d and is calculated from the third order aberration theory as (Born @ Wolf, 1975):

$$R_{pp} = fBR_d^3 \quad (8)$$

where f is the lens focal length, B is Seidel coefficient, R_d is radius of the diaphragm placed before the lens. Eq. 8 is valid for the case of the point source located at the infinity. As it was shown in (Dudchik et al., 2000), the equation (8) for compound X-ray lens is rewritten as

$$R_{pp} = \frac{1}{2} \frac{R_d^3}{R^2}. \quad (9)$$

The eq. 9 is valid for the case when $R_d < 0.6 R$ as was estimated in (Dudchik et al., 2000) by numerical calculations. The beam radius R_{ms} at the MS-plane is related to the beam radius R_{pp} at the focal plane as

$$R_{ms} = \frac{1}{4} R_{pp} = \frac{1}{8} \frac{R_d^3}{R^2}. \quad (10)$$

The distance L_{ms} from the lens to MS-plane is calculated as:

$$L_{ms} = f \frac{(R_d + R_{ms})}{(R_d + R_{pp})}. \quad (11)$$

For example $L_{ms} = 0.917 f$ when $R_d = 0.5 R$ and it is illustrated by Fig. 7 where position of MS-plane is shown for the discussed case.

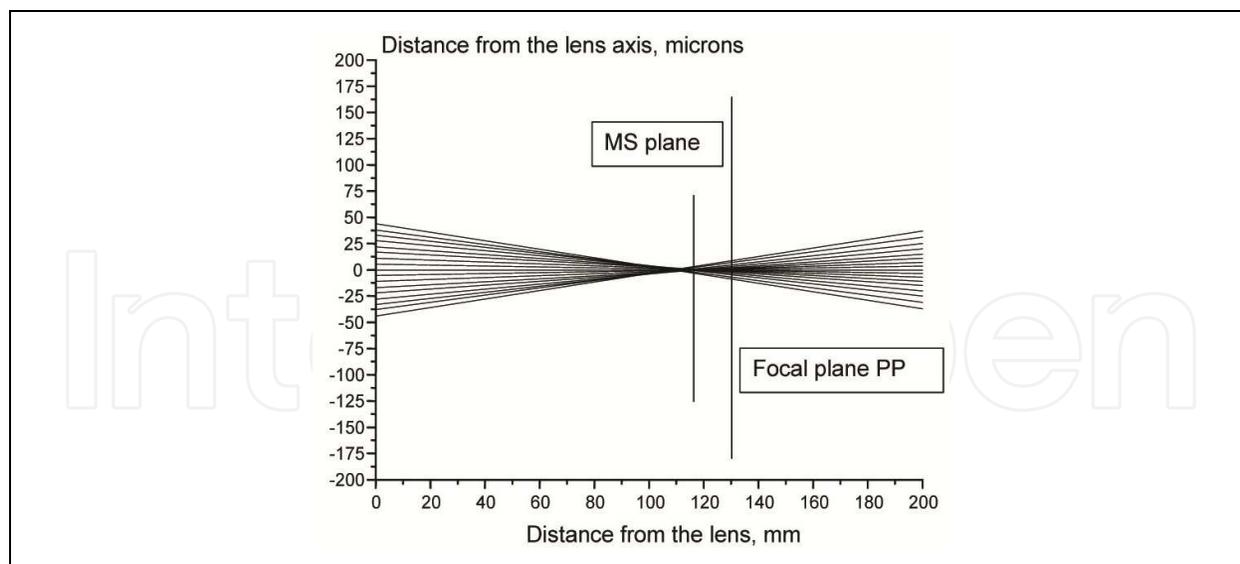


Fig. 7. Paths of 8-keV X-rays forming focal spot of 103- elements lens. Lens radius is 100 microns. Diameter of the diaphragm placed before the lens is equals to 100 microns

Radius R_{ms} of the circle of the least confusion decreases with decreasing of the size of diaphragm and achieves its minimum possible value R_{ms-min} when a diaphragm with an optimal hole radius R_{d-opt} is used. In this case the value R_{ms-min} will be equals to the radius of the first minimum of the Airy diffraction pattern R_{diff} which is $R_{diff} = 0.61 \lambda L_{ms} / R_{d-opt}$. The diaphragm radius R_{d-opt} may be defined by the following equation:

$$\frac{1}{8} \frac{R_{d-opt}^3}{R^2} = \frac{0.61 \lambda L_{ms}}{R_{d-opt}} \quad (12)$$

where λ is the wavelength. The solution of the Eq. (12) for R_{d-opt} and for R_{ms-min} under the assumption $L_{ms}=f$ is:

$$R_{d-opt} = \left(\frac{2.44 R^3 \lambda}{\delta N} \right)^{1/4}, \quad (13)$$

and

$$R_{ms-min} = \frac{0.61 \lambda f}{R_{d-opt}}. \quad (14)$$

It is interesting to compare above result for R_{d-opt} (Eq. 13) with the value of so-named parabolic aperture radius R_p . Parabolic aperture radius R_p is the central portion of the spherical lens that focuses X-rays to the same point. From wave approximation it is known (Snigirev et al., 1996; Piestrup et al., 2005) that the value may be calculated as:

$$R_p = \left(\frac{2 R^3 \lambda}{\delta N} \right)^{1/4}. \quad (15)$$

Comparing Eq.13 and Eq.15 we can see that there is only a small difference in numerical coefficient for these two values. It may be explained that we used f value instead of L_{ms} when solving Eq.12.

Eq. (13) for R_{d-opt} and Eq. (12) for R_{ms-min} are useful to calculate expected X-ray beam size in the MS plane for compound X-ray lenses. In Table 1 and Table 2 the diameter $2R_{ms}$ of the circle of the least confusion is calculated for epoxy spherical lenses with different values of number of microlenses N and lens radius R . Table 2 shows result of the same calculations for the case when additional diaphragm is used to decrease size of the beam at MS plane.

From the Table 2 it is seen that spherical compound refractive lens being combined with diaphragm ensures resolution at submicron level.

| Lens parameters | Lens focal length f for 8 keV X-rays, mm | Lens absorption aperture $2Ra$, μm | L_{ms} value for 8 keV X-rays beam, mm | Diameter ($2R_{ms}$) of the circle of the least confusion, μm |
|----------------------------|--|--|--|--|
| N=100, R=100 μm | 132 | 117 | 117.5 | 5 |
| N=200, R=100 μm | 66 | 82 | 62 | 1.72 |
| N=100, R=50 μm | 66 | 82 | 53.5 | 6.89 |
| N=200, R=50 μm | 33 | 58 | 29.4 | 2.44 |

Table 1. Parameters of X-ray beam formed by spherical compound epoxy X-ray lens.

| Lens parameters | Lens focal length f for 8 keV X-rays, mm | Diameter of the optimal diaphragm aperture $2R_{opt}$, μm | L_{ms} value for 8 keV X-ray beam, mm | Diameter $2R_{ms}$ of the circle of the least confusion, μm |
|----------------------------|--|---|---|--|
| N=100, R=100 μm | 132 | 62 | 127.4 | 0.78 |
| N=200, R=100 μm | 66 | 53.2 | 64.3 | 0.46 |
| N=100, R=50 μm | 66 | 37.6 | 62.7 | 0.66 |
| N=200, R=50 μm | 33 | 31.6 | 31.8 | 0.39 |

Table 2. Parameters of X-ray beam formed by spherical compound X-ray lens combined with diaphragm.

2.3 Measurement of the microcapillary refractive lens at Stanford Synchrotron Radiation Laboratory

Refractive X-ray lens work as ordinary lens for visual light and lens formula is also valid to describe its operation. The formula is written as:

$$\frac{1}{a} + \frac{1}{b} = \frac{1}{f}, \quad (16)$$

where a is distance from the source to lens, b is distance from the lens to source image, f - lens focal length. The size of the source image S_1 , as in the case of visual optics, is related to the source size S by the equation:

$$S_1 = S \frac{f}{a-f}. \quad (17)$$

In the case of synchrotron radiation the distance between the source and the lens is high enough and equals, as a rule, to 10-50 m; the size of the source is also, as a rule, less than 1000 microns. When refractive lens with a focal length equal to approximately 10 cm is used, expected size of source image may be equal to some microns in according to formula 17. This is a way for obtaining micro and nano-sized X-ray beams.

We fabricated and tested some microcapillary refractive X-ray lenses for focusing synchrotron X-rays. The lenses were arbitrarily designated as 1-1, 3-1, 3-4, 3-3, 4-1 and 5-1. The calculated and measured characteristics of these CRLs are given in Table 3 below.

| | | | | | | |
|---|------|------|------|------|------|------|
| X-ray lens designation | 1-1 | 3-1 | 3-4 | 3-3 | 4-1 | 4-1 |
| Photon energy, keV | 12 | 12 | 9 | 8 | 8 | 7 |
| Number of individual lenses | 90 | 196 | 103 | 93 | 102 | 102 |
| Lens curvature radius, μm | 165 | 250 | 100 | 100 | 100 | 100 |
| Calculated focal length, cm | 52.8 | 37 | 15.7 | 13.8 | 12.6 | 9.6 |
| Calculated image distance, cm | 54.5 | 37.8 | 15.8 | 13.9 | 12.6 | 9.7 |
| Measured image distance, cm | 32 | 36 | 17.5 | 13 | 14 | 10 |
| Calculated vertical minimum waist diameter, μm | 15.1 | 10.4 | 4.4 | 3.9 | 3.2 | 2.7 |
| Measured vertical minimum waist diameter, μm | 12.8 | 12 | 3.9 | 4.8 | 2.7 | 4 |
| Calculated horizontal minimum waist diameter, μm | 64.1 | 44.0 | 18.8 | 16.6 | 13.5 | 11.5 |
| Measured peak transmission, % | 36 | 30 | 24 | 16 | 27 | 5 |
| Calculated attenuation aperture diameter, μm | 314 | 262 | 143 | 125 | 119 | 96.7 |
| Measured attenuation aperture diameter, μm | 321 | 245 | 147 | 150 | 149 | 149 |
| Calculated 2D-gain | 16.6 | 20.0 | 25.6 | 16.9 | 28.9 | 6.0 |
| Measured 2D-gain | 8.9 | 3.5 | 13.4 | ** | 25.5 | ** |

Table 3. Measured and calculated parameters of microcapillary refractive X-ray lens for SSRL BL 2-3 source

Used was the beamline 2-3 on the Stanford Synchrotron Radiation Laboratory's (SSRL's) synchrotron (Dudchik et al., 2004). This beamline possesses a double-crystal monochromator that was capable of giving x-rays from 2400 to 30000 eV with a 5×10^{-4} resolution. The approximate source size (full width half maximum, FWHM) was $0.44 \times 1.7 \text{ mm}^2$, as specified by SSRL. The experimental apparatus is shown in Fig. 8.

The distance from the source to lens was 16.81 meters. The X-ray beam size from this source was approximately $2 \times 20 \text{ mm}^2$ at the entrance to the experimental station; however, this size

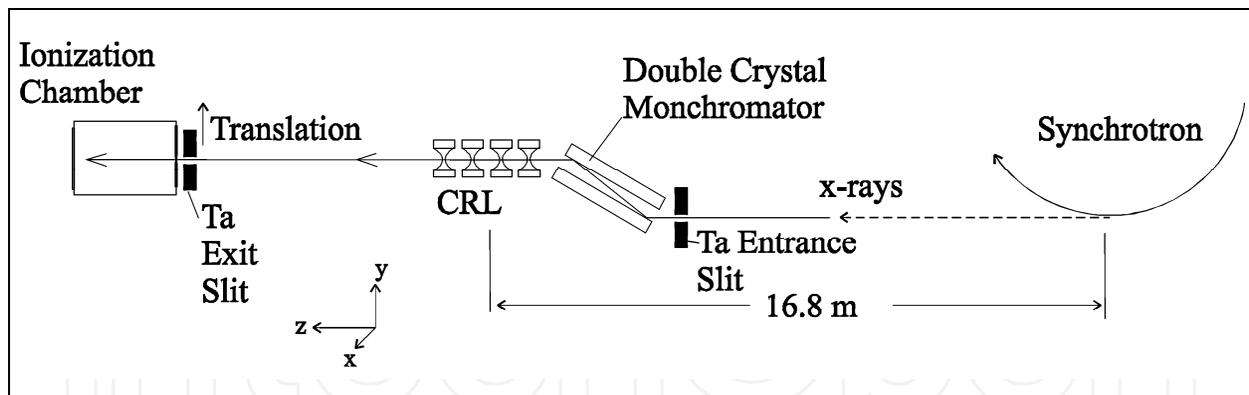


Fig. 8. The experimental apparatus at SSRL for measuring microcapillary X-ray lens

was reduced to approximately $0.4 \times 0.4 \text{ mm}^2$ by Ta slits upstream of the monochromator. The CRL was placed in a goniometer head that could be manually tilted in three axes. The lens could also be remotely translated orthogonally (x and y) to the direction of the x -ray beam to maximize the x -ray transmission through the lens. An x -ray gas ionization detector was placed after a translatable slit for measuring the x -ray beam profile. This Ta slit was adjusted to below $25 \text{ }\mu\text{m}$ by using a thin stainless steel shim. It is likely that, as good as the Ta slit was, the jaws are not ideally parallel at these small dimensions, and the slit width was minimally $> 3 \text{ }\mu\text{m}$ when jaws appeared to be entirely closed.

After the slit width was adjusted, the Ta slit was then translated in the x direction across the focused x -ray beam, and its profile obtained. We then manually moved the slits along the z -axis of the x -ray beam, measuring its vertical widths by scanning the slits in the x direction across the beam at each location.

Using these measured widths, we profiled the beam waist as a function of distance from the lens. Results are shown in Table 3. For example, the source of diameter 0.44 mm was focused by the lens 4-1 to a minimum spot FWHM of $5 \text{ }\mu\text{m}$, at a distance of 13 cm from the CRL. Thus, the demagnification was $M = .0114$. The spot size in the horizontal direction was measured to be $19 \text{ }\mu\text{m}$, which is larger, because the imaged source diameter was larger (1.7 mm , FWHM) in that dimension.

The CRL has an aperture with a Gaussian absorption profile, which causes stronger absorption of the extreme rays passing through the CRL outer radial regions, as compared to rays that pass through the less absorptive central region. The CRL aperture is much smaller than the source size, especially in the horizontal dimension. We obtained the transmission through the CRLs given in Table 3 by narrowing the x -ray beam to $50 \times 50 \text{ }\mu\text{m}^2$ and translating each CRL through the beam, thereby producing transmission profiles of the lenses. The absorption apertures (e^{-2} points, not FWHMs) were obtained from these figures. The calculated and measured peak transmissions (transmission at the lens axis) for the lenses are given in Table 3.

Given the measured transmissions and profiles, we determined the gains of these lenses. Both measured and calculated gains are given in Table 3 for all these lenses. The gain values varied between 3.5 to 25.5 . These gain values are primarily due to the large source size. If the same lens is placed on a beam line using a third generation X-ray source, the gain of the CRL can be substantially larger. These sources can possess spot sizes a factor

of 3 smaller (e.g. 0.5 by 0.5 mm²). Also, typical distances from insertion devices to end stations can be approximately 51 m, as compared to 16.8 m in our experiment. For these source parameters, the gain at 11 keV from the same lens is 138, a sizable increase of the intensity over that of the case without a CRL. Although the gains of these CRLs were small, larger gains can be achieved using smaller source sizes, larger CRL apertures, and longer object distances.

2.4 Measurement of the microcapillary refractive lens at ANKA Synchrotron Radiation Source

Parameters of the lens # 5-1 were measured at ANKA Synchrotron Source (Germany) (Dudchik et al., 2007a). The lens was designed in Institute of Applied Physics Problems of Belarus State University. The lens consists of 224 spherical epoxy microlenses formed inside glass capillary with curvature radius equal to 100 microns. Lens length is equal to 69 mm. The individual epoxy lenses inside of the glass capillary are spherical ones with the curvature radius equals to 100 microns. Spherical lenses may be characterized by the following set of parameters: lens focal length f , absorption aperture radius R_a (see formula 7), parabolic aperture radius R_p (see formula 15) , and the diffraction radius $R_{diff}=0.61 \lambda f/R_a$, that characterises diffraction blurring of the focused beam, where μ is linear absorption coefficient for the lens material.

The lens # 5-1 was used to focus X-rays with energy 12 keV and 14 keV. Calculations show that for 12 keV X-rays parabolic aperture radius of the lens is $R_p=27$ microns for the case of the discussed lens ($R= 100$ microns, $N= 224$). The absorption lens aperture radius R_a for the lens is equal to 69 microns. The same values for 14 keV X-rays are : $R_p=28$ microns, $R_a=94$ microns. Lens focal length f calculated by formula 5 for 12 keV and 14 keV is equal to 133 mm and 180 mm respectively. The lens length is equal to 69 mm and it is "thick enough" comparing to lens focal length. The focal length f_t of a thick lens may be calculated by the next formula (Pantell et al., 2003):

$$\frac{f_t}{f} = \frac{\left(\frac{t}{f}\right)^{1/2}}{\sin\left(\frac{t}{f}\right)^{1/2}}, \quad (18)$$

where t is lens length. Result of calculation of f_t : $f_t = 145$ mm for 12 keV X-rays and $f_t = 192$ mm for 14 keV X-rays.

The lens #5-1 has been characterized for 12 keV and 14 keV X-rays at the ANKA-FLUO experimental station situated at a bending magnet of the ANKA Synchrotron Light Source. The energy was monochromatized by a W/BC4 double multilayer monochromator with 2% bandwidth. For the measurement of the beamsize the lens was placed on a five axis positioning device and exactly oriented in the direction of the X-ray beam. The distance a between source and lens was equal to 12.7 m. The size of the source s : 800x250 μm^2 FWHM. The source size can be reduced by a 0.1mm x 0.1 mm² slit #1 placed at a distance 4.7 m to the source. There was one more slit #2 placed at 1m distance from lens. The slit size was 0.1mm x 0.08 mm². It was also possible to hold slits in opening mode.

Measured were beam size at different distance to the lens and lens transmission. The minimum value of beam size was considered as lens image distance. The beam size was derivated from knife edge scans conducted around the focus position derived with the x-ray camera. A 0.5 μm thin Permalloy structure was chosen and the edges have been scanned with 0.5 μm or 1 μm resolution. Characteristic K_{α} Fe-atom X-rays emitted by Permalloy structure were registered by X-ray camera. The measured profile of the edge is the convolution of the Fe concentration function (approximated by a step function) and the profile of the x-ray beam. As the step function converts the convolution in to a simple integration, the measured function is the error function if the beam profile is a Gaussian. Thus an error function (Fig. 9) has been fitted to the knife edge data. Fitting a Gauss function to the derivative is equivalent; nevertheless numerical derivating adds a considerable amount of noise to the data.

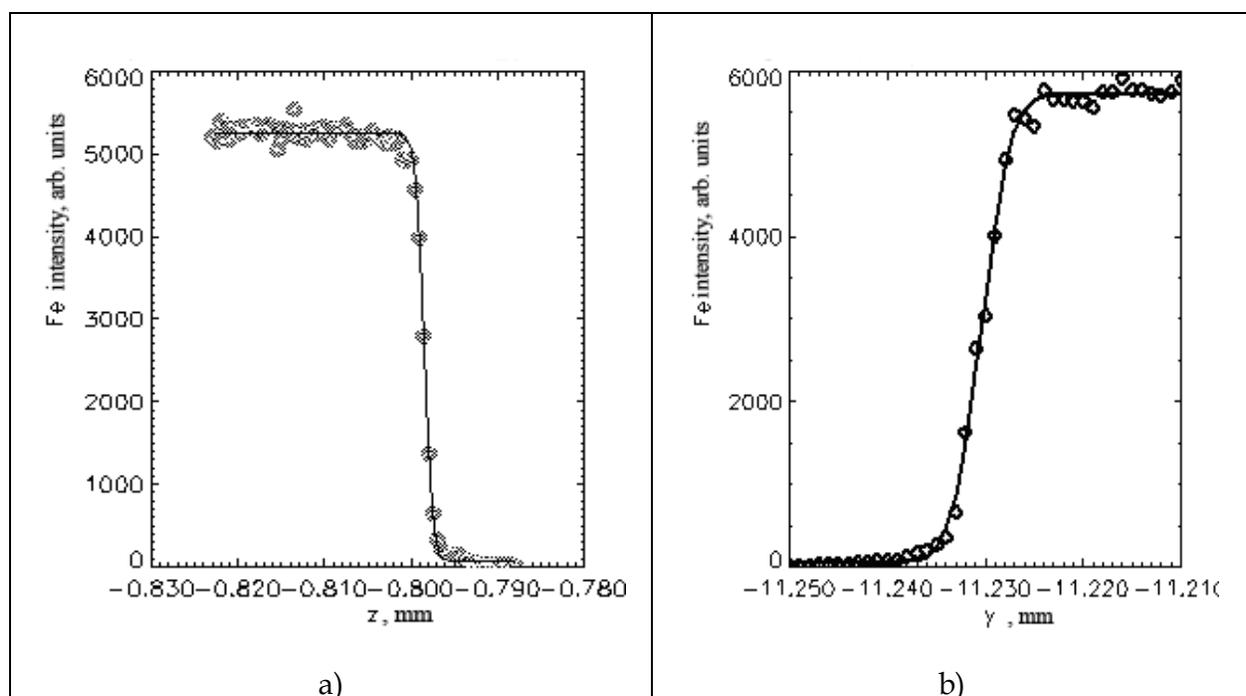


Fig. 9. Fit of error function to vertical (a) and horizontal (b) scan over lithographic structure

To determine gain in intensity of the beam due to focusing by the lens next procedure was applied. The lens was removed and the fluorescence intensity resulting from a Permalloy square of 50 μm size was measured. This intensity was compared to the intensity of the focussed beam and the area of the focussed beam was calculated with $A=2\pi\sigma_x\sigma_y$ with being the Gaussian beams size (FWHM value/2.35). With closed front end slits the gain factor for a smaller source can be obtained. For the ANKA source however closing slits cannot improve the gain. Therefore two values for the gain are given in Table 4 and Table 5.

Investigations shown that tested lens is suitable to focus 12 keV-14 keV X-rays into some microns in size spots (Dudchik et al., 2007a). Calculated lens focal length is in a good agreement with measured one. The lens parameters may be improved by increasing lens transparency. Also lenses with shorter lens focal length may be formed inside capillary with inner diameter equals to 100 microns. In this case the lenses may be used for nano-focusing.

| | | |
|---|-------|-----------|
| Energy, keV | 12 | 12 |
| Size of slit #1, mm ² | 1 x 1 | 0.1 x 0.1 |
| Measured image distance, mm | 146 | 147 |
| Calculated image distance, mm | 147 | 147 |
| Calculated lens focal length f_t , mm | 145 | 145 |
| Measured horizontal focal size, μm | 10.4 | 4.1 |
| Measured vertical focal size, μm | 2.2 | 1.7 |
| Gain | 34/31 | 113/18 |
| Transmission | 9.5% | 9.5% |

Table 4. Parameters of spherical compound X-ray lens for 12 keV X-rays

| | | |
|---|-------|-----------|
| Energy, keV | 14 | 14 |
| Size of slit # 1, mm ² | 1 x 1 | 0.1 x 0.1 |
| Measured Image distance, mm | 195 | 196 |
| Calculated image distance, mm | 195 | 195 |
| Calculated lens focal length f_t , mm | 192 | 192 |
| Measured horizontal focal size, μm | 12.2 | 6.3 |
| Measured vertical focal size, μm | 3.0 | 2.1 |
| Gain | 43/40 | 162 /22 |
| Transmission | 21.5% | 21.5% |

Table 5. Parameters of spherical compound X-ray lens for 14 keV X-rays

2.5 X-ray imaging with compound refractive X-ray lens

X-ray imaging is a power tool to study inner structure of objects and materials. This method is realised with synchrotron and laboratory X-ray sources. A well-known in-line laboratory X-ray projection microscopy and microtomography is based on the using of microspot X-ray tube as a source of radiation. The system for imaging consists of a quasi-point X-ray source and a CCD-camera. The object for investigation is placed at a distance R_1 from the source, and the CCD-camera is placed at a distance R_2 from the sample. The spatial resolution of the method depends on the source size and is in range from 5 to 1 microns. The magnification is determined as $(R_1 + R_2)/R_1$ and may be 10 or higher. The disadvantage of the method of direct imaging is that the position of the point X-ray source is not stable in time. This disadvantage is remedied by using imaging optics for microscopy. There are some types of imaging X-ray optics: pin-hole, zone plate and compound refractive X-ray lens. We used previously discussed microcapillary refractive X-ray lens as an imaging device. In this case there is no limitation to the source size and ordinary X-ray tubes may be used.

The optical scheme of the system for imaging with refractive X-ray lens is shown in Fig.10. The object for imaging 3 is exposed by X-rays from X-ray tube 1. The lens 2 forms decreasing

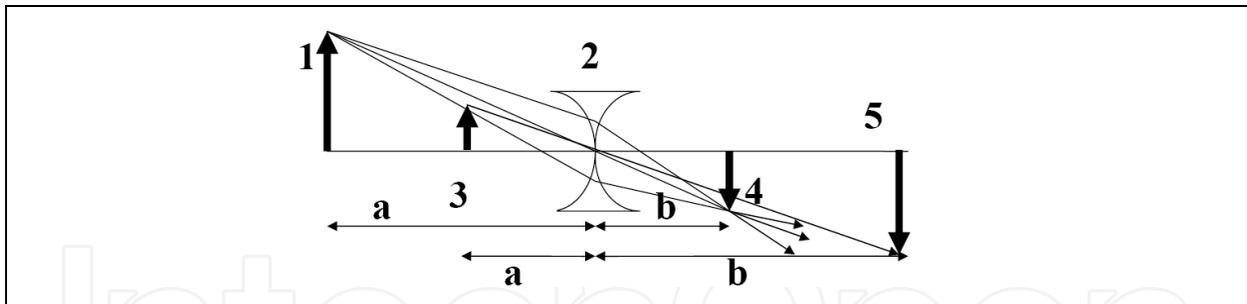


Fig. 10. Schematic of X-ray imaging with refractive lens. 1- X-ray source (tube); 2- compound refractive X-ray lens; 3- object; 4- source image; 5- object image.

image of the X-ray tube focal spot 4 and increased image of the object 5. X-ray CCD-camera is placed at the position of object image. The object, lens and CCD-camera are placed in-line at distances from one another that satisfied the lens formula:

$$\frac{1}{a} + \frac{1}{b} = \frac{1}{f}, \quad (19)$$

where a is the distance from the object to the lens; b is the distance from the lens to CCD-camera; and f is the lens focal length.

The imaging system (microscope) was designed in the Institute of Applied Physics Problems of Belarus State University (Dudchik et al., 2007b; Dudchik et al. 2007c). The system photo is shown in Fig. 11. The microscope consists of X-ray tube 1, X-ray lens 2 in a holder and goniometer 5, CCD X-ray camera 3. The object for imaging 4 is placed between the X-ray tube and the lens.



Fig. 11. X-ray microscope. 1- X-ray tube; 2- X-ray lens in a holder; 3- CCD X-ray camera; 4 - object for imaging; 5- goniometer for X-ray lens.

A water-cooled copper-anode X-ray tube (Russian model # BCV-17) with tube focal spot of 0.6 mm x 8 mm was used as a source of X-rays.

The image of the object was recorded by a Photonic Science camera (model FDI VHR) with 4008 x 2670 pixels, and 4.5 microns pixel size.

The X-ray lens used for imaging consists of 161 individual spherical, biconcave, microlenses, each with 50-microns curvature radius R . The CRL length is equal to 18 mm. The lens photo is shown in fig. 12. The lens focal length, calculated in accordance to the formula 5, is equals to 41 mm for 8 keV X-rays.

Gold meshes #1000 with 5 μm wires separated by 20.4 μm was used as object for imaging.

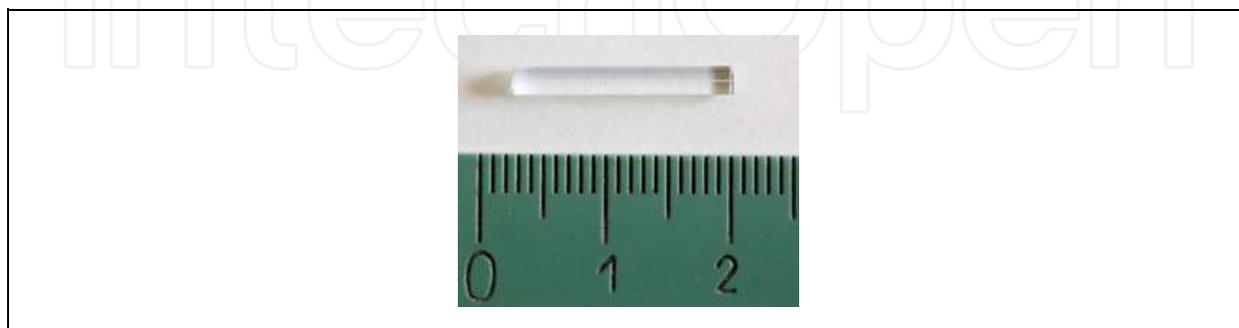


Fig. 12. Photo of microcapillary refractive X-ray lens with 161 concave spherical microlenses inside of glass capillary

The tube voltage was set to 20 kV and the current -14 mA, resulting in a standard bremsstrahlung and 8 keV characteristic-line spectra from the tube without filtering. The mesh was placed at distance $a = 45$ mm to the lens. The X-ray CCD-camera was placed a distance $b = 440$ mm to the lens in according to the lens formula (19), magnification $M = b/a = 9.8$. Fig.13 shows images of mesh #1000 recorded by the CCD-camera at different exposition equals to 5 min and 7 min.

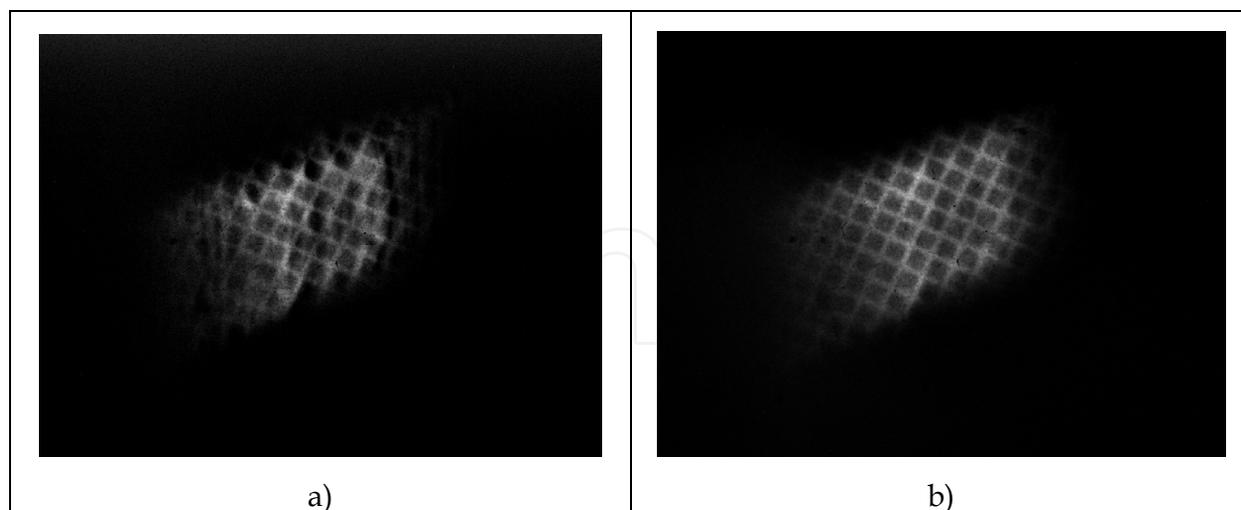


Fig. 13. X-ray image of mesh # 1000 at magnification 9.8. a) 5 min exposition time; b) 7 min exposition time

As it can be seen from Fig. 13, 5 μm gold wires of mesh #1000 are recognized by the CCD-camera, which means the spatial resolution of the simple X-ray imaging system is not worse than 5 μm . In according with calculations of lens parameters, presented in Table 1 and Table 2,

better spatial resolution may be achieved by using monochromatic X-rays and diaphragm to decrease spherical aberrations.

To improve spatial resolution of the system imaging experiments were accomplished on the National Synchrotron Radiation Laboratory (China) (Dudchik et al., 2010). The experiments were done on X-ray diffraction and scattering beamline (U7B). Synchrotron radiation (SR) from the Wiggler source was focused by a toroidal mirror. Focused SR was monochromized with a double-crystal monochromator and selected photon energy was 8 keV. The optical scheme of the experiments was the same as is shown in Fig. 10. The only difference was that the toroidal mirror was placed between X-ray source and the lens. Microcapillary X-ray lens in the form of glass capillary filled by 147 concave epoxy microlenses with 50 microns curvature radius each was used. The lens focal length is equal to 45 mm. Gold mesh #1500 with 5.5 microns wires were used as an object for imaging. Fig.14 shows images of gold mesh #1500 obtained with 8-keV monochromatic synchrotron X-rays at magnification $M=11.6$ (a) and $M=18.6$ (b).

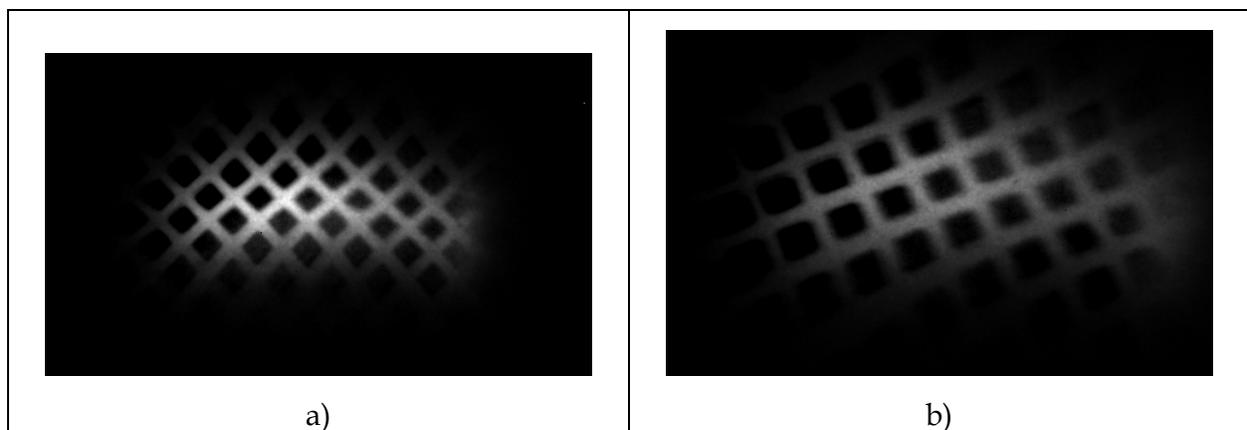


Fig. 14. X-ray images of mesh #1500 obtained with 8-keV monochromatic synchrotron X-rays at magnification $M=11.6$ (a) and $M=18.6$ (b).

Comparing images of gold mesh shown in Fig. 13 and Fig. 14 we may conclude that using monochromatic X-rays give significant improvement of spatial resolution of the system.

In conclusion, imaging experiment shows that the spherical compound refractive lens is a promising imaging optical element for hard x-rays, giving better than 2- 5 μm spatial resolution.

3. Conclusion

We have fabricated and tested compound refractive lenses (CRL) composed of micro-bubbles embedded in epoxy. The bubbles were formed in epoxy inside glass capillaries. The interface between the bubbles formed spherical bi-concave microlenses. The lenses were named as microcapillary refractive lenses or "bubble lenses". When compared with CRLs manufactured using other methods, the micro-bubble lenses have shorter focal lengths with higher transmissions for moderate energy X-rays (e.g. 7 - 12 keV). The lenses were tested at the Stanford Synchrotron Radiation Laboratory (SSRL) and ANKA Synchrotron Source. We used beamline 2-3 at the SSRL to measure focal lengths between 100-150 mm and absorption apertures between 90 to 120 μm . Transmission profiles were measured giving, for example,

a peak transmission of 27 % for a 130-mm focal length CRL at 8 keV. The focal-spot sizes were also measured yielding, for example, an elliptical spot of $5 \times 14\text{-}\mu\text{m}^2$ resulting from an approximate 80-fold demagnification of the $0.44 \times 1.7 \text{ mm}^2$ source. Experiments at ANKA Synchrotron Source shown that the designed lens with 145 mm focal length focuses 12 keV-rays into $2.2 \times 10.4\text{-}\mu\text{m}^2$ spot.

The lenses are imaging device and may be used as objective for X-ray microscope. A simple microscope consisting of the X-ray tube, microcapillary refractive X-ray lens and X-ray CCD-camera was designed at the Institute of Applied Physics Problems of Belarus State University. The X-ray lens consists of 161 individual spherical, biconcave microlenses, each with 50-microns curvature radius. The lens focal length is equals to 41 mm for 8 keV X-rays. It was shown that the spatial resolution of the microscope is better than 5 microns when unfiltered X-ray beam from copper anode X-ray tube is used. Better spatial resolution (about 2-3 microns) was obtained in the experiments on the National Synchrotron Radiation Laboratory's (China) were monochromatic 8-keV X-ray beam was used.

The micro-bubble technique opens a new opportunity for designing lenses in the 8-9 keV range with focal lengths less than 30-40 mm.

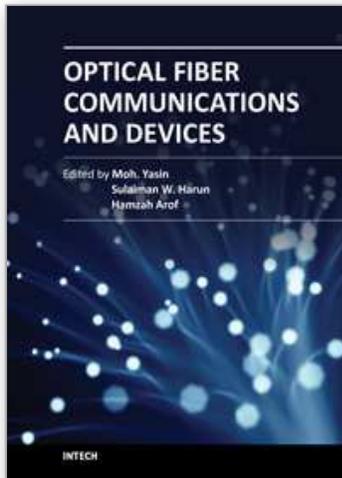
4. Acknowledgment

I would like to acknowledge my colleague Dr. N.N. Kolchevsky, who spent a lot of time to improve parameters of the microcapillary lenses when he was PhD student in the Institute of Applied Physics Problems of Belarus State University. I would like to acknowledge my colleagues Dr. M.A. Piestrup, Dr. C.K. Gary, Dr. J.T. Cremer from Adelphi Technology, Inc., who did a lot of experiments on testing microcapillary lenses for focusing and imaging with synchrotron and laboratory X-ray sources. Prof. T. Baumbach and Dr. R. Simon were so kind to invite me for taking part in experiments on focusing X-rays at ANKA Synchrotron Radiation Source. Prof. Zhanshan Wang, Dr. Baozhong Mu, Dr. Chengchao Huang, Prof. Guoqiang Pan invited me to take part in imaging experiments with microcapillary lenses at the National Synchrotron Radiation Laboratory (China). I am grateful to all of them for continues interest to this research and useful comments.

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Optical Fiber Communications and Devices

Edited by Dr Moh. Yasin

ISBN 978-953-307-954-7

Hard cover, 380 pages

Publisher InTech

Published online 01, February, 2012

Published in print edition February, 2012

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In order to correctly reference this scholarly work, feel free to copy and paste the following:

Yury Dudchik (2012). Design and Application of X-Ray Lens in the Form of Glass Capillary Filled by a Set of Concave Epoxy Microlenses, *Optical Fiber Communications and Devices*, Dr Moh. Yasin (Ed.), ISBN: 978-953-307-954-7, InTech, Available from: <http://www.intechopen.com/books/optical-fiber-communications-and-devices/design-and-application-of-x-ray-lens-in-the-form-of-glass-capillary-filled-by-a-set-of-concave-epoxy>

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