Detailed characterization of polycapillary focusing x-ray lenses by a charge-coupled device detector and a pinhole

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A method to measure the detailed performance of polycapillary x-ray optics by a pinhole and charge coupled device (CCD) detector was proposed in this study. The pinhole was located between the x-ray source and the polycapillary x-ray optics to determine the illuminating region of the incident x-ray beam on the input side of the optics. The CCD detector placed downstream of the polycapillary x-ray optics ensured that the incident x-ray beam controlled by the pinhole irradiated a specific region of the input surface of the optics. The intensity of the output beam of the polycapillary x-ray optics was obtained from the far-field image of the output beam of the optics captured by CCD detector. As an application example, the focal spot size, gain in power density, transmission efficiency, and beam divergence of different parts of a polycapillary focusing x-ray lenses (PFXRL) were measured by a pinhole and CCD detector. Three pinholes with diameters of 500, 1000, and 2000 μm were used to adjust the diameter of the incident x-ray beam illuminating the PFXRL from 500 μm to the entire surface of the input side of the PFXRL. The focal spot size of the PFXRL, gain in power density, transmission efficiency, and beam divergence ranged from 27.1 μm to 34.6 μm, 400 to 3460, 26.70% to 5.38%, and 16.8 mrad to 84.86 mrad, respectively.

Keywords: polycapillary x-ray lenses, charge-coupled device detector, pinhole, performance characterization

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1. Introduction

Polycapillary x-ray lenses operating on the principle of total reflection is a widely used x-ray guidance optics.[1–3] They can be categorized as polycapillary focusing x-ray lenses (PFXRL) and polycapillary parallel x-ray lenses (PPXRL), depending on the function of the optics.[4] PFXRL are commonly used in laboratory x-ray source to focus divergent x-ray beams into a small focal spot with a high gain in power density. PPXRL convert divergent x-ray beam from the x-ray tube into quasi-parallel x-ray beam or focus the quasi-parallel x-ray beam from the synchrotron radiation into a focal spot. Since polycapillary x-ray lenses were proposed by Kumakhov in 1990, they have been widely used in confocal x-ray technology, micro x-ray fluorescence (μ-XRF) analysis, and x-ray imaging.[1,5–8]

The performance of the polycapillary x-ray lenses has a significant influence on applications. For instance, the PFXRL typically requires a small focal spot and high gain in power density for use in a μ-XRF spectrometer.[7] The focal spot size and gain in power density of the PFXRL determine the resolution and detection efficiency of the μ-XRF spectrometer. The size of the focal spot of the polycapillary x-ray lenses is also crucial to the confocal x-ray fluorescence setup. The size of the focal spot of the PFXRL and PPXRL determines the spatial resolution of the confocal XRF instrument.[9] The beam divergence must be considered when a polycapillary x-ray lenses is used in micro x-ray diffraction (μ-XRD) or small-angle x-ray scattering (SAXS) because it influences the resolution of both methods.[8,10–12] In x-ray imaging research, the size of the focal spot of the PFXRL affects the edge-enhancement-to-noise ratio of the x-ray phase imaging.[13,14] The beam divergence and the intensity of the output beam of the PPXRL used in x-ray absorption imaging influence the image quality.[15] Furthermore, it was reported that the intrinsic point defects of the PFXRL lead to the formation of multiple x-ray images of an object positioned in the focal plane.[16] Therefore, to use polycapillary x-ray lenses more efficiently, it is necessary to characterize the performance of the polycapillary x-ray lenses in detail.

Typically the performance of a polycapillary x-ray lenses is characterized by a detector that obtains the photon counts or spectroscopic data of the output beam of the optics.[2,17–19] The size of the focal spot of the polycapillary x-ray lenses is commonly measured using the knife-edge scanning method.[17] The gain in power density of the polycapillary x-ray lenses is the ratio of the intensity with and without the optics. The transmission efficiency of the polycapillary x-ray lenses is the proportion of photons transmitted by the optics. The gain in power density and the transmission effi-

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ciency are typically measured with a pinhole. In 2016, Bonfigli *et al.* first proposed the use of an LiF crystal radiation detector with nanoscale resolution to characterize the polycapillary x-ray lenses.[20] Due to the high resolution of the LiF crystal radiation detector, the two-dimensional (2D) and three-dimensional (3D) intensity distribution of the focal spot of the PFXRL and the fluorescence image at the exit of PPXRL could be obtained. This method intuitively reflects the beam transmission properties of the polycapillary x-ray lenses. Both above mentioned methods have unique advantages but only focus on the performance of the whole polycapillary x-ray lenses. In this study, a charge-coupled device (CCD) detector and a pinhole was proposed to characterize the performance of a polycapillary x-ray lenses and parts of the lenses. As shown in Fig. 1(a), the pinhole is located between the x-ray tube and the polycapillary x-ray lenses. The pinhole controls the area of the incident beam irradiating the polycapillary x-ray lenses to characterize the performance of the polycapillary x-ray lenses in detail. A method for characterizing the performance of the polycapillary x-ray lenses with a CCD detector is established in this study. As an application example, the performance of a PFXRL is characterized by a CCD detector and pinhole in detail.

2. Experimental setup and method

Figure 1(a) shows the schematic diagram of the experimental setup. A tungsten target micro-focus x-ray tube with a focus diameter of 20 μm [L9631, Hamamatsu, Japan] was operated at 30 kV and 500 μA. The distance of the focus of x-ray source to the beryllium window of the x-ray tube was 16.5 mm. Three pinholes with diameters of 500, 1000, and 2000 μm were used to obtain different x-ray irradiation regions of the optics. The PFXRL used in the experiment was designed and manufactured by the College of Nuclear Science and Technology of Beijing Normal University. The input focal distance (f_i), length (L), and output focal distance (f_o) of the PFXRL were 69, 71.5, and 14.6 mm, respectively. The size of the focal spot of the PFXRL measured by the knife-edge method was 36.5 μm at 17.4 keV. Figure 1(b) shows the schematic diagram of the input side of the PFXRL. The profile of the input side and output side of the PFXRL is hexagonal. The length of the side (l_s) of the hexagon on the input side and output side of the PFXRL was 2.42 mm and 1.79 mm, respectively. The PFXRL consisted of hundreds of one-time composite tube that is drawn by hundreds or thousands of capillaries, as shown in Fig. 1(b). A CCD detector [M11427-61, Hamamatsu, Japan] placed downstream of the PFXRL was used to detect the far-field pattern of the optics. The resolution of the x-ray imaging system was 10 μm. Three motorized translation stages were used in the optics detection system: two 3D stages and one five-dimensional (5D) stage. One 3D stage was used for controlling the position of the pinhole. The other stage was used for measuring the focal spot of the PFXRL using the knife-edge scanning method. The 5D stage was used to adjust the PFXRL.

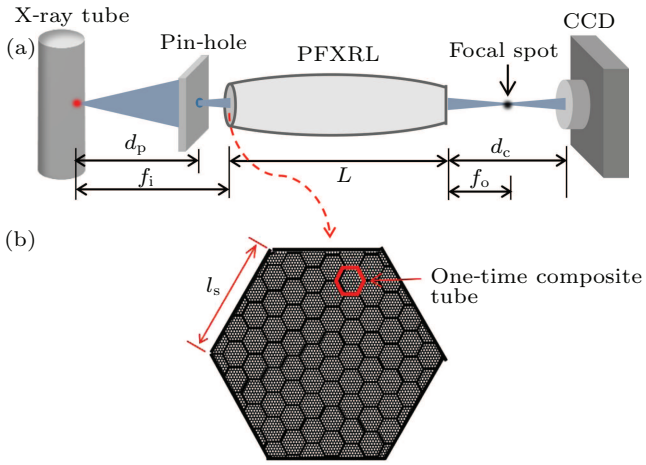


Fig. 1. (a) Schematic diagram of the experimental setup for the characterization of the polycapillary x-ray lenses. (b) Schematic diagram of the input side of the PFXRL.

In this study, the commonly used semiconductor detector or proportional counter tube was replaced by a CCD detector for the characterization of the PFXRL. Unlike the semiconductor detector or proportional counter tube, the CCD detector does not only obtain the intensity of the output beam of the optics but also acquires an output beam image. The intensity of the output beam of the PFXRL can be obtained by summing the photon counts of each pixel of the output beam image captured by the CCD detector. Similar to the characterization of a PFXRL by a semiconductor detector or proportional counter tube, the focal spot size can be measured by the knife-edge scanning method. This approach is convenient for the measurement of the beam divergence of the optics because the diameter of the output beam can be easily acquired from the output beam image obtained from the CCD detector. Under the monitor of the CCD detector, the location and area of incident beam irradiating on the surface of the input side of the PFXRL can be controlled by adjusting the pin-hole between the x-ray tube and PFXRL.

3. Experiment and results

3.1. Positioning of the PFXRL

The input focal spot of the PFXRL was a finite region where the focal spot of the x-ray source was located. In this configuration, the PFXRL exhibits the best performance. Before the characterization of the PFXRL, it was necessary to place the focal spot of the x-ray source to the input focal spot of the PFXRL. For the collimation of the optics, the distance between the x-ray source and CCD detector was fixed, and the position of the x-ray source relative to the PFXRL was changed. The start position should be close to the known f_i of the PFXRL; this distance can be estimated by the shape curve

of the PFXRL. In every position, the PFXRL was adjusted to perform optimally using the accurate 5D translation stage.

The change in the far-field pattern of the output beam of the PFXRL for different distances between the PFXRL and x-ray source is shown in Fig. 2(a). The distance corresponding to the largest output beam area designates the input focal distance of the PFXRL. The far-field pattern of the output beam of the PFXRL reflects the configuration of the one-time composite tube. As images shown in Fig. 2(a), the greater the deviation from the input focal distance, the smaller the area of the far-field pattern of the output beam is, suggesting that the marginal capillaries of the PFXRL do not operate efficiently if the x-ray source deviates from the optimal position (f_i). Figure 2(c) shows a schematic diagram of the PFXRL transmission of the x-rays from the x-ray source for different distances, explaining the above phenomenon. The apertures of the capillaries of a well-made PFXRL should all point to the input focal spot. The x-ray source deviated from the input focal spot increases the angle of the incident x-ray and the inner surface of the capillaries of the PFXRL, which leads to the filature of the x-ray propagation. This influence is stronger closer to the edge of the optics. This visual method is appropriate for a rapid evaluation but may generate inaccuracies that directly affect the performance of the PFXRL. To improve the accuracy of the determined the input focal distance, the intensity of the output beam of the PFXRL shall be calculated for the x-ray source placed on at several distances close to the input focal distance (the black line in Fig. 2(b)). The input focal distance in Fig. 2(b) can be specified by the values of the distance between PFXRL and x-ray source corresponding to the maximum of the intensity curve.

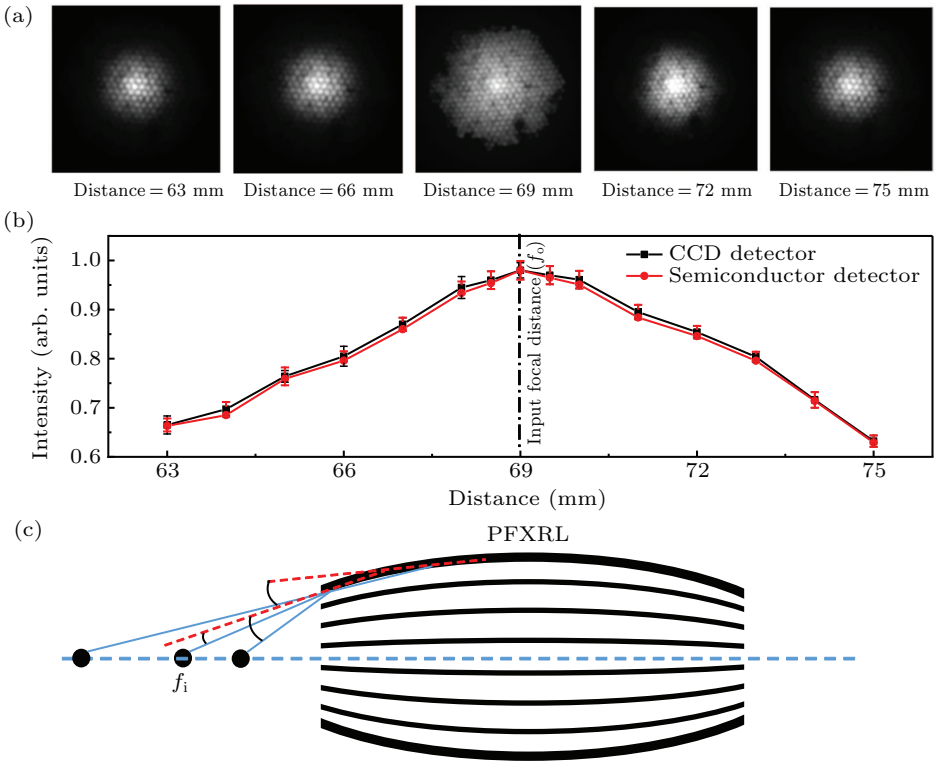


Fig. 2. (a) Images in the figure is the output beam of the PFXRL that was placed at different distances from the x-ray source. (b) Determined the position of the input focal spot of the PFXRL by the CCD detector and silicon drift detector. (c) Schematic diagram of the x-ray source deviation from the input focal spot of the PFXRL increased the angle of the incidence x-ray and the inner surface of the optics.

In order to verify effectiveness of acquiring the intensity of the output beam by summing the counts in each pixel of the far-field pattern captured by the CCD detector, a silicon drift detector (AXAS-M, Amptek, Germany) was used to the measure the intensity of the output beam of the PFXRL (the red line in Fig. 2(b)). The intensity of the output beam of the PFXRL was measured by the method proposed in Ref. [17]. A plastic plate was attached on a linear step stage that moved in the cross-direction of the output beam of the PFXRL after the CCD detector captured the image of the output beam spot. The silicon drift detector oriented on the plastic plate obtained the scattered spectrum reflecting the intensity of the output beam of the PFXRL. The intensity curves obtained from the CCD detector and silicon drift detector are highly similar, indicating the feasibility of characterizing the polycapillary x-ray lenses by the CCD detector.

3.2. Detailed characterization of the PFXRL

Three pinholes with diameters of 500, 1000, and 2000 μm were used as a dimmer placed between the x-ray source and PFXRL to control x-ray beam illuminating on definite region of input surface of the PFXRL. Due to a homemade shield

attached to the exit of the x-ray source to protect the beryllium window, the adjustable range of the distance (d_p) between the x-ray source and PFXRL was 31.5 mm to 71.6 mm (f_i). Therefore, three pinholes were alternate used to make sure that adjust the irradiation range of the circular beam spot from 500 μm to the entire surface of the input side of the PFXRL. Capturing the output beam spot of the PFXRL with the CCD detector ensures that the incident beam accurately illuminates the center of the input surface of PFXRL in the experiment. The size of the irradiation area can be controlled by adjusting the relative distance between the pinhole and the x-ray source to characterize the performance of different parts of the PFXRL in detail, as shown in Fig. 3.

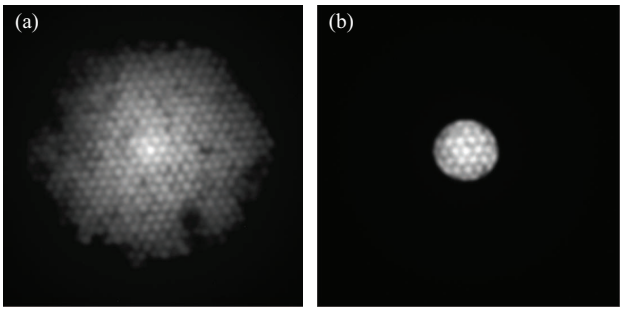


Fig. 3. Output beam image of the PFXRL captured by CCD detector with (b) and without (a) a pinhole.

3.2.1. Measurement of the focal spot size

Before measuring the size of the focal spot (d_f), the output focal distance of the PFXRL was determined by scanning the output beam diameters along the exit direction. The output focal distance of the optics is defined as the distance from the output side to the focal spot of the optics. The diameter of the output beam for different distances from the output side of the optics is shown in Fig. 4(a). The output focal distance of the PFXRL is the distance at the minimum point of the curve. The output focal distance measured by the CCD is 14.8 mm, which is close to the measurement result obtained from the silicon drift detector.

The size of the focal spot of polycapillary x-ray optics is commonly defined by the full width at half maximum (FWHM) of the scanning curve of the focal spot. The scanning curve used to measure the size of the focal spot of the PFXRL is shown in Fig. 4(b). The scanning curve indicates that the focal spot size is 34.6 μm. The deviation of the focal spot size of the PFXRL obtained from the CCD and silicon drift detector is attributed to the different energy of the incident x-ray. In the working energy range, the size of the focal spot of the PFXRL decreases with increasing energy.[2,17] The diameter of the incident x-ray beam irradiating the center of the input side of the PFXRL can be controlled by changing the pinhole and the position of the pinhole between the x-ray source and the PFXRL. The scanning curves of the focal spot of the PFXRL for incident x-ray beams with different diameters are shown in Fig. 4(c). The focal spot size of the PFXRL illuminated by incident x-ray beams with different diameters is shown in Fig. 4(d). The focal spot size increases with the increasing diameter of the incident x-ray beam. The maximum focal spot size is 34.6 μm without a pinhole, and the minimum focal spot size is 27.1 μm with an incident x-ray beam with a diameter of 500 μm. Furthermore, when the diameter of the incident x-ray beam is in the range of approximately 500 μm to 1250 μm, the focal spot size remains almost constant.

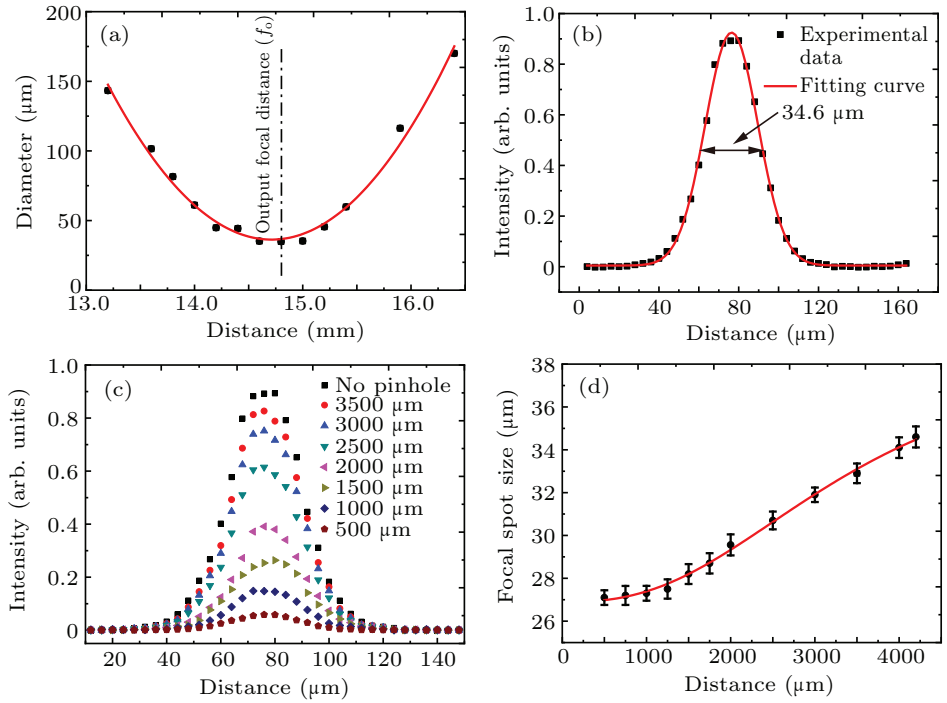


Fig. 4. (a) The process of determining the output focal distance; (b) focal spot size obtained from the knife-edge scanning method; (c) scanning curves of the focal spot of the PFXRL for incident beams with different diameters; (d) the focal spot size of the PFXRL for different part optics used.

3.2.2. Measurement of the gain in power density

The gain in power density (G) of the PFXRL is a parameter that significantly affects the efficiency of micro x-ray analysis. G is defined as the ratio of the photon flux with and without the optics at the focal spot:

$$G = \frac{F_{\text{with}}}{F_{\text{without}}}. \tag{1}$$

The x-ray flux without optics (F_{without}) can be measured directly by the pinhole or calculated since the intensity is inversely proportional to the square of the distance from the source. The x-ray flux with optics (F_{with}) in the focal spot can be calculated by the total number of photons transmitted by the PFXRL divided by the area of the focal spot. The total number of photons can be acquired from the far-field image of the output beam of the PFXRL obtained from the CCD detector. The diameter of the focal spot has been measured previously. The gain in power density of the PFXRL decreases with the diameter of the incident beam. As shown in Fig. 5, the gain in power density increases from 400 as to 3460 as the diameter of the incident x-ray beam increases from 500 μm to the entire surface.

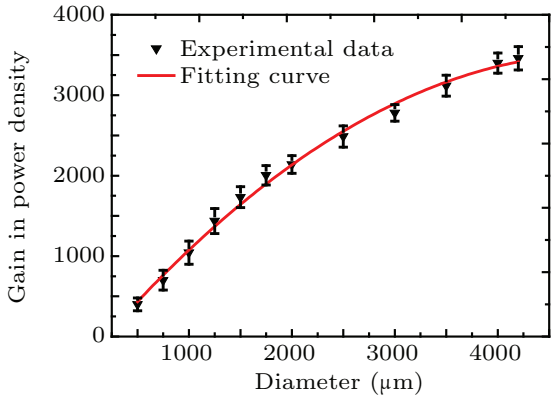


Fig. 5. Gain in power density of the PFXRL for different diameters of the incident x-ray beam.

3.2.3. Measurement of transmission efficiency

The transmission efficiency (ε) of the PFXRL is the ratio of the x-ray photons focused by the PFXRL to the x-ray photons incident at the entrance of the PFXRL and characterizes the transmission ability of the optics for the x-ray beam. The transmission efficiency of polycapillary x-ray optics is influenced by geometric parameters, such as the glass fiber dimensions (diameter, length), bending radius, surface smoothness, and glass material.[20] Therefore, the transmission efficiency differs in different areas of the PFXRL.

The transmission efficiency of the PFXRL can be easily measured using a CCD detector and a pinhole. The transmission efficiency of the PFXRL for different diameters of the incident x-ray beam irradiating the center of the PFXRL decreases significantly with an increase in the diameter of the incident x-ray beam (Fig. 6). The maximum value of the transmission efficiency is 26.7% with a diameter of the incident x-ray beam of 500 μm. The minimum value of the transmission efficiency of 5.38% was obtained without the pinhole. It can be readily calculated the transmission efficiency different annular areas of the PFXRL (Fig. 7). Distribution of the transmission efficiency shown in Fig. 7 can visually demonstrate the transmission performance at different regions of the PFXRL.

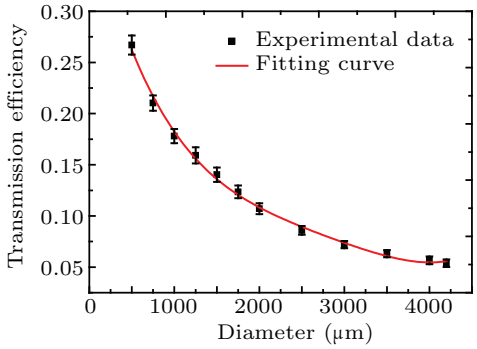


Fig. 6. Transmission efficiency of the PFXRL for different diameters of the incident x-ray beam.

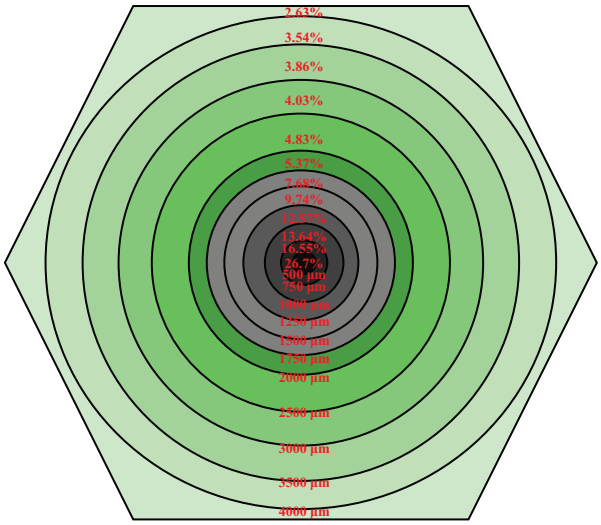


Fig. 7. Distribution of the transmission efficiency in different regions of the PFXRL.

3.2.4. Measurement of beam divergence

Generally, the beam divergence is determined by measuring the output beam diameter of the PFXRL along the beam direction using the knife-edge scanning method. In this study, this procedure is more convenient due to the CCD detector. Compared with the conventional method, several knife-edge scans are replaced by the images of the output beam of the PFXRL captured at different distances from the focal spot of the PFXRL. The far-field pattern of the output beam of the PFXRL captured by the CCD detector has 2048×2048 pixels. As shown in Fig. 8(b), the scanning curve of the output beam of the PFXRL in the horizontal direction can be obtained by summing the intensity in the columns. The scanning step is the resolution of the CCD detector. The full width at half maximum (FWHM) of the scanning curve indicates the diameter of the output beam of the PFXRL. The beam divergence of

the output beam of the PFXRL can be calculated based on the slope of the fitting curve of the diameter of the output beam (Fig. 8(c)). The beam divergence of the PFXRL increases from 16.8 mrad to 84.86 mrad with increasing diameter of the incident x-ray beam from 500 μm to the entire surface (without the pinhole).

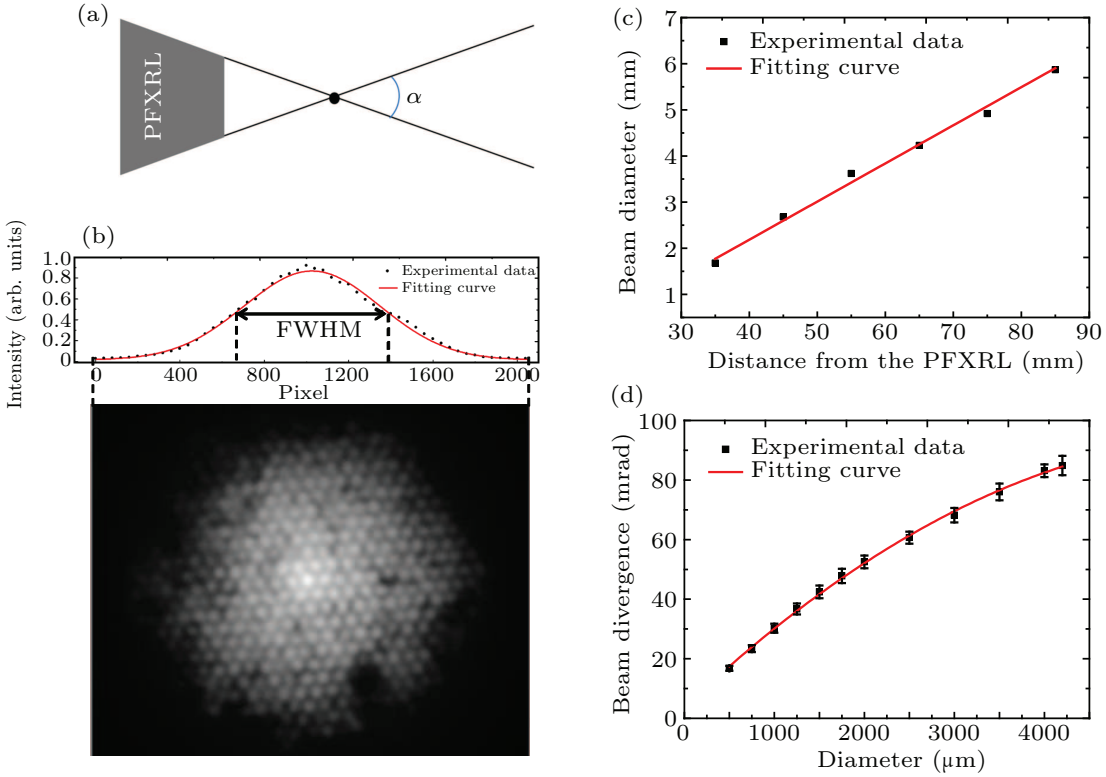


Fig. 8. The measurement of the beam divergence of the PFXRL by the CCD detector; (a) schematic diagram of the beam divergence of the PFXRL; (b) extracting the beam diameter from the far-filed image acquired by CCD detector; (c) output beam diameter of the PFXRL at different distances from the exit of the PFXRL; (d) beam divergence for different diameters of the incident x-ray beam of the PFXRL.

4. Discussion

4.1. Focal spot size

Geometrically, the exit apertures of the capillaries comprising the PFXRL point to the position of the focal spot. The cone-shaped x-ray beams emitted from the aperture of the capillaries converge at the focal spot of the PFXRL. The overlap of the cone-shaped x-ray beams forms the focal spot of the PFXRL. Theoretically, the size of the dispersion area of the focal spot can be expressed as

$$F_{\mathrm{d}} = d_{\mathrm{g}} + 2f_{\mathrm{o}}\theta_{\mathrm{c}}, \tag{2}$$

where d_{g} is the diameter of the capillary, f_{o} is the output focal distance of the PFXRL, and θ_{c} is the critical angle of the total x-ray reflection. The FWHM of the dispersion area is the size of the focal spot of the PFXRL. In practice, the curvature of the capillaries is not ideal. In the PFXRL, the curvature of the capillary gradually increases from the center outward. Generally, in the manufacturing process, capillaries with larger design curvature tend to produce greater bending deviation. As shown in the schematic diagram in Fig. 9, the bending deviation increases the focal spot size. As shown in Fig. 4(d), the focal spot size of the PFXRL remains nearly constant when the diameter of the incident x-ray beam is less than 1250 μm. The reason is that the curvature of the capillaries in the center part of the PFXRL is relatively small, and the corresponding bending deviation of the capillaries has a negligible influence on the size of the focal spot of the PFXRL. Conversely, the bending deviation of the outer capillaries is relatively large, and the focal spot size of the PFXRL increases with the increasing diameter of the incident x-ray beam, as shown in Fig. 4(d).

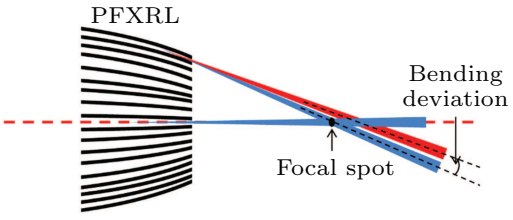


Fig. 9. Bending deviation increases the focal spot size of the PFXRL.

The PFXRL is primarily used in micro x-ray analysis. The focal spot size of the PFXRL determines the spatial resolution of the micro x-ray analysis instrument. By adjusting the pinhole, the size of the focal spot of the PFXRL can be changed within a certain range, increasing the application range of the PFXRL.

4.2. Gain in power density

The gain in power density of the PFXRL influences the detection efficiency of the micro x-ray analysis instrument. Since the diameter of the input section of the PFXRL is much smaller than the input focal distance, the intensity of the incident x-ray beam irradiating the PFXRL is approximate evenly distributed in this study. Therefore, the gain in power density can be estimated as

$$G = \left(\frac{f_{\rm i}+L+f_{\rm o}}{f_{\rm i}}\right)^4 \left(\frac{d_{\rm b}}{d_{\rm f}}\right)^2 \varepsilon, \tag{3}$$

where $d_{\rm b}$ is the diameter of the incident x-ray beam of the PFXRL. An increase in the focal spot size and a decrease in transmission efficiency slowed down the growth rate of the gain in power density of the PFXRL (Fig. 5).

4.3. Transmission efficiency

The experimental results showed that the transmission efficiency of the PFXRL decreased rapidly from the center of the optics to the outside. For the PFXRL used in this study, the transmission efficiency of certain areas of the capillaries of the optics mainly depended on the curvature. The higher the curvature of the capillaries, the more total reflections required for the x-rays to travel through the capillaries. Due to the roughness of the inner surface of the capillaries, more total reflections reduced the probability of x-ray photons transmitted through the tubes. The reason for the high transmission efficiency in the center of the PFXRL is that the curvature of the capillaries is small, and a large proportion of x-ray photons pass through the tube without being reflected. Optics grinding caused damage to the marginal capillaries of the PFXRL, thereby is decreasing the transmission efficiency at the margin of the PFXRL (Fig. 7).

4.4. Beam divergence

The PFXRL has potential applications for μ-XRD analysis. The resolution of the lattice spacing of the μ-XRD instrument is influenced by the beam divergence of the PFXRL. The smaller the beam divergence of the PFXRL, the higher the resolution of the μ-XRD instrument is. In practice, a pinhole can be used to reduce the beam divergence of the PFXRL and improve the resolution of the μ-XRD instrument.

4.5. Comprehensive discussion

Previous studies mostly focused on the characterization of the overall performance of polycapillary x-ray optics. In this study, the performance of different parts of the PFXRL was characterized using a pinhole and CCD detector. Parts of the PFXRL were characterized by blocking the incident x-ray beam using the pinhole, allowing for an adjustment of the performance of the PFXRL. Therefore, the PFXRL with the pinhole provides more experimental flexibility than that without the pinhole. For instance, the focal spot of the PFXRL can be decreased by adjusting the pinhole to obtain an improved 2D scanning map. Similarly, a focal spot with a high gain in power density can be obtained by omitting the pinhole, thereby increasing the detection efficiency. Furthermore, information on the performance of different areas of the lenses of polycapillary x-ray optics improves the design and manufacturing process of the optics. For example, a large range of the focal spot size of the PFXRL indicates that the focal spot size can be decreased by reducing the bending deviation of the capillaries of the PFXRL. For the design of the polycapillary x-ray optics, the transmission efficiency at the margin of the optics is an important reference index for the tradeoff between cost and performance improvement by increasing the diameter of the optics.

5. Conclusions

A method for the detailed characterization of a PFXRL using a pinhole and CCD detector was proposed. With the monitor of the CCD detector, partial capillaries of the PFXRL can be selected in use by adjusting the pinhole placed between the optics and the x-ray source. The size of the focal spot, gain in power density, transmission efficiency, and beam divergence of different parts of the PFXRL were characterized. Information on the performance of different parts of the PFXRL can improve the design and manufacturing process of the PFXRL. Furthermore, the use of the pinhole allows for diverse applications of the PFXRL.

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