LABORATORY NOTES

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Glass-capillary collimator for distance compensation and partial monochromatization at rotating-anode X-ray generators.

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Abstract

Access to the beam ports of rotating-anode X-ray generators is often obstructed by direct-coupled or belt-driven target drives. The construction of an easily adjustable stable glass-capillary collimator is described, which renders possible the unrestricted use of beam ports of these generators. Transmitted intensity and monochromaticity of the primary beam are sufficient for precession photographs of proteins after additional 20 µm Ni filtering as demonstrated by a precession photograph of hen egg lysozyme. The straight capillary collimator is now a routinely usable low-cost device for each X-ray laboratory.

1. Introduction

In modern rotating-anode high-power X-ray generators, the use of one beam outlet at the X-ray tube is often restricted by the target drive. In our laboratory, this problem arose with a direct-drive Rigaku Denki (Japan) RU H2R generator. Here the distance between the midpoint of the tube window and the housing of the 90 mm long driver unit is about 25 mm. Commercial graphite monochromators, mirror systems and application devices such as imaging plates or precession cameras cannot be adjusted at this port for spatial reasons. Simple distance tubes, recommended by the manufacturer, are expected to cause a considerable loss of intensity because the outlet for the direct beam has to be about 100 mm from the X-ray-tube window. The use of a straight glass-capillary collimator may be expected to avoid or reduce the loss of intensity. The use of hollow glass capillaries for guiding X-rays is based on the effect of total external reflection from optical smooth surfaces at grazing incidence.

Since Lely & van Rijssel (1951), a number of different authors have studied transmission of X-rays through capillaries (e.g. Hirsch & Keller, 1951; Marton, 1966; Damaschun & Weh, 1968; Mosher & Stephanakins, 1976; Chung & Pantell, 1977) both straight and curved. Capillaries were used in different fields, especially in X-ray fluorescence analysis apparatus (e.g. Rindby, 1986; Yamamoto & Hosokawa, 1988; Engström, Larsson, Rindby & Stocklassa, 1989). Furthermore, for focusing synchrotron radiation, tapered or straight mono- and polycapillaries were applied (e.g. Thiel, Bilderback, Lewis & Stern, 1992; Bzhaumikhov, Vartan'yants, Konleoev, Liev, Nikitina & Scherbakov, 1993; Arkadiev, Gruev & Kumakhov, 1993; Hoffman, Thiel & Bilderback, 1994; Bilderback, Thiel, Pahl & Brister, 1994). For standard X-ray crystallography devices, there has been no necessity for capillary optics up to now but the new design of rotating anodes requires the development of a simple standard device for routine laboratory use.

Here, we describe a simple device by which the divergence of the primary beam is reduced by total reflection in a single glass capillary usually implemented, with smaller diameter, in a polycapillary column of a so-called Kumakhov lens (Kumakhov & Sharov, 1992). In addition to the higher intensity of the beam at the outlet of the capillary in comparison with pin-hole collimation, partial monochromatization results from the reflection. With additional Ni filtering, the degree of monochromaticity is sufficient for various applications in protein crystallography. The construction of an easily adjustable collimator of high stability and radiation safety will be discussed, and its suitability is demonstrated by a precession photograph of hen egg lysozyme.

2. Methods

The experiments were made with a highly stabilized rotating-anode X-ray generator RU H2R (Rigaku Denki Co., Japan) with direct coupled target driver. Copper targets of 0.3 × 0.3 mm (44 kV, 110 mA) and 0.2 × 0.2 mm focal spots (44 kV, 68 mA) were used. The X-ray spectrum was registered by an energy-dispersive nitrogen-cooled lithium-drifted silicon detector (RÖNTEC GmbH, Berlin) in the energy region of 0–44 keV and plotted with Harvard Graphics©.

Diffraction patterns of hen egg lysozyme were recorded with an integrating precession camera (Enraf–Nonius, Netherlands). With a 0.3 × 0.3 mm focal spot, the following parameters were used: crystal-to-film distance $F = 75$ mm, layer-line screen $r_s = 20$ mm ($\Delta = 2$ mm), crystal-to-screen distance $s = 54.9$ mm, precession angle $\mu = 20°$. The film Curix RP1.000G is a product of AGFA Gevaert N.V. (Belgium).

3. Construction and alignment of the collimator

Although capillary optics are rapidly evolving from novel optical components into standard devices at synchrotrons, capillaries of high (optical) surface quality are still not easily produced and the optimization process is continuing. We used a glass capillary made by Roentgen Optical Systems (Moscow, Russia) that is usually (with smaller diameter) implemented in Kumakhov lenses. Its quality has been discussed recently by

Bzhaumikhov, Vartan'yants, Konleoev, Liev, Nikitina & Scherbakov (1993) in connection with the polycapillary semilens.

A detailed construction scheme of the collimator is presented in Fig. 1. The basic element is the glass capillary. Its length and diameter are chosen corresponding to the distance to be compensated and to the dimensions of the focal spot of the X-ray tube. This capillary is held by lead rings along the axis of a small brass tube. No vibration transmission from the driver unit upon the collimator could be measured. To change the capillary, the whole brass tube can be removed and replaced by another tube containing a capillary with the appropriate diameter. The brass tube is fixed by a Teflon ring with a lead shield at the outlet of a second, outer, brass tube which acts as radiation shield. The outer brass tube is mounted *via* a brass socket on the housing of the X-ray tube with an inclination angle of 84°, corresponding to the recommended take-off angle for Cu $K\alpha$ radiation. Because the diameter of the capillary is larger than the effective focus dimensions, no additional mercury (Engström, Larsson, Rindby & Stocklassa, 1989) or tungsten powder around the glass fiber is required to prevent X-rays outside the capillary from reaching the outlet. Four screws, each of them displaced by 90°, permit easy movement of the capillary entrance window along two orthogonal axes in a plane perpendicular to the beam direction towards the position for maximum transmitted intensity. The proper alignment of the collimator relative to the beam is monitored *via* a charge-coupled device (CCD) detector behind the precession camera. The detector distance of about 500 mm from the capillary outlet guarantees a highly resolved primary-beam spot that can be evaluated visually during the alignment. A shift of the focal spot can be compensated by the four-screw adjustment despite the fixing of the capillary outlet. The inlet and outlet of the outer brass tube can be closed by Ni foils. Two hose couplings permit flushing of the capillary with helium or evacuation in order to avoid absorption of the beam by air.

Here, capillaries of 135 mm length were used to span the length of the direct drive. The inner diameters were 0.44 and 0.7 mm for focal spots of 0.2 × 0.2 and 0.3 × 0.3 mm, respectively. The thickness of the capillary wall is about 0.03 mm, providing sufficient mechanical stability.

4. Results and discussion

An X-ray collimator is characterized by the gain in intensity (counts/time/area), exit-beam size, beam divergence and its monochromator capacity. The beam path and distances necessary for estimation of these collimator parameters are shown in Fig. 2. For a material-dependent critical external reflection angle of $\delta_E = 3 \times 10^{-3}$ rad for Cu $K\alpha$ radiation (Bzhaumikhov, Vartan'yants, Konleoev, Liev, Nikitina & Scherbakov, 1993) and focal-spot dimensions of 0.3 × 0.3 mm, the capillary of 135 mm length and 0.7 mm diameter is almost completely illuminated by characteristic radiation with an incident angle less than δ_E. The minimum distance between the capillary inlet and the focal spot is 55 mm. The shadowed area, starting 12 mm behind the inlet, marks the region of total reflection for Cu $K\alpha$ radiation. The path of that component, as it would be without reflection, is drawn with dotted lines in Fig. 2. A pinhole collimator with holes at the ends instead of a capillary would remove this part of the primary beam. For an approximately radially symmetrical distribution of the radiation intensity, an intensity reduction corresponding to the ratio of the squared distances, $r_1^2 : r_2^2$, would be observed. In the present case, the reduction is $r_1^2 : r_2^2 = 55^2{:}190^2 = 1{:}12$. The solid angle of the collected primary beam is effectively enlarged by reflection of the characteristic radiation rendering the ratio of the effective illuminated areas in the capillary exit plane to $0.72^2 : 035^2 = 4.2$. Thereby, the loss of intensity should be reduced from 1:12 to 1:3. The transmission efficiency for radiation with $\lambda = 0.1542$ nm, defined by the ratio of the flux at the capillary exit to the flux at the entrance, was measured as 0.49 (20). The intensity gain when using the fiber was experimentally determined by measuring the intensity with a pinhole collimator and with the glass-capillary collimator. The pinholes correspond to the inner diameter of the capillary hole and the ratio of the intensities is measured as 2.7 (1) for Cu $K\alpha$ radiation. That result is below the theoretical value, showing that some absorption caused by surface rugosity of the capillary is detectable.

The beam size at the capillary exit of 0.7 mm is given by the diameter of the single capillary. At the spindle axis of the precession camera (80 mm from the exit) it is 1.15 mm, and at a distance of 500 mm it is 3.5 mm (divergence 0.006 rad). This value can be diminished by the additional standard pinhole collimator of the precession camera.

If the inlet and/or outlet of the capillary are not well defined circles but notched or, if the capillary is bent, the primary-beam spot shows marginal shell-like reflections (insert in Fig. 3), caused by missing or doubled total reflection of the $K\alpha$ radiation or longer waves. A symmetrical halo around the central spot is a criterion of high quality of the capillaries, since it exists only with undisturbed doubled total reflection. However, these shells become visible only at large distances from the capillary outlet

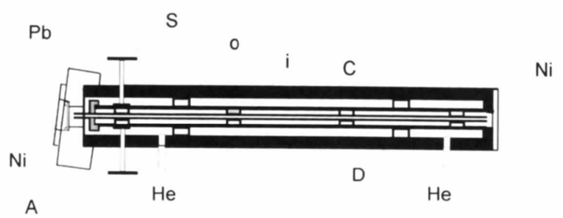

Fig. 1. Glass capillary collimator–schematic representation. A brass socket; *o*, *i* outer, inner brass tube; *S* adjustment screws (horizontal screws not shown); *C* capillary; *D* distance rings; Pb lead shield; Ni nickel foil; He hose couplings.

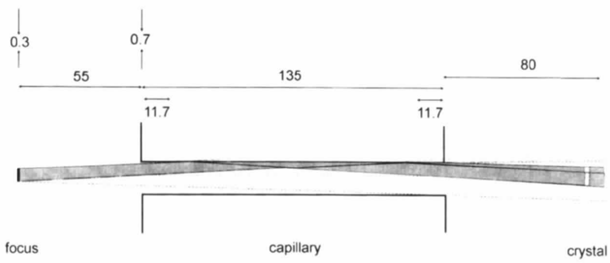

Fig. 2. Schematic beam path within the glass-capillary collimator. Shadowed region: Cu $K\alpha$ region of total reflection in the upper half plane. —— Direct non-monochromatized beam; ...... beam if no total reflection had occurred.

(*ca* 500 mm) and do not play a role because of low intensity, especially when using further pinhole collimation in application devices or monochromators.

The degree of monochromaticity of the beam is radially symmetrical for ideal capillaries. In Fig. 3, X-ray spectra are shown that were recorded in a plane 500 mm from the outlet of the collimator across the spot. The beam intensity was additionally reduced by 100 μm Ni filtering to protect the silicon detector. The beam passing directly through the capillary has a spot diameter of 3.5 mm in the detection plane and its quality is influenced only by this Ni filter. The reflected Cu $K\alpha$ radiation is distributed over the whole spot up to $r = 1.5$ mm. In

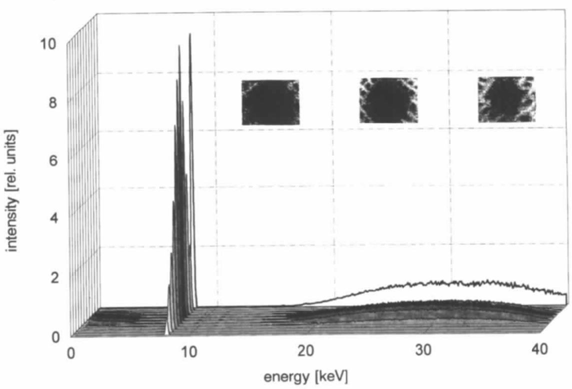

Fig. 3. X-ray spectra measured with an energy-dispersive silicon detector across the primary beam 500 mm behind the collimator and 100 μm Ni foil. The normalized spectrum of a non-collimated beam behind an 100 μm Ni foil is drawn in the background. Insert: left spot – well colliminated beam; middle spot – shell-like reflections from poorly defined ends of the capillary; right spot – poorly adjusted capillary. The diameter of the spot is 3.5 mm.

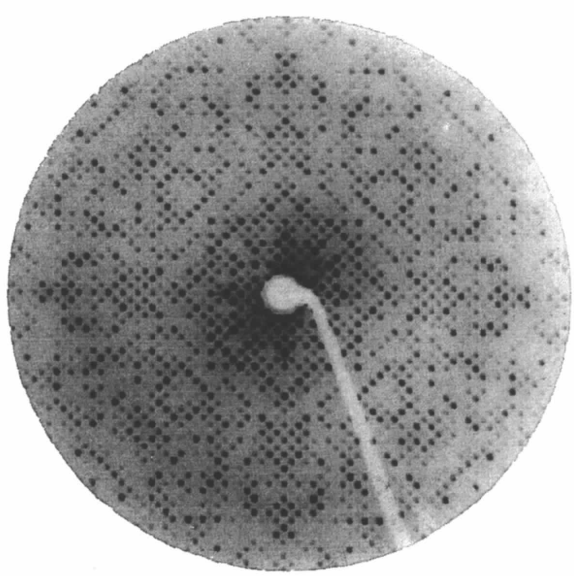

Fig. 4. Precession photograph of the (*hk*0) plane of tetragonal hen egg lysozyme. Focal spot 0.3 × 0.3 mm; copper target; 44 kV, 110 mA; 20 μm Ni filter; collimator diameter 0.7 mm, crystal size 0.6 × 0.4 × 0.4 mm.

this reflected beam, all wavelengths $\lambda_{\min} \leq 0.054$ nm are absent, since the minimum angle between the beam edges and the capillary wall is 1.05×10^{-3} rad. For comparison, in the background of Fig. 3, a spectrum is drawn which was normalized to the maximum $K\alpha$ intensity but registered without the capillary. After passing the collimator, the short-wave-lengths contribution is markedly reduced, $K\beta$ is suppressed by the Ni filter and the long waves are somewhat increased by the enlarged take-off angle in comparison with the $K\alpha$ radiation.

The resulting monochromaticity is sufficient for precession photographs as shown in Fig. 4 for a small crystal of hen egg lysozyme. The photograph was made using the 0.3 × 0.3 mm focal spot and the collimator 0.7 mm in diameter with 20 μm Ni filter in 5 h. The 13% absorption by air along the path through the glass capillary has been removed completely by helium flushing of the collimator.

5. Summary

An easily adjustable radiation-safe stable-glass-capillary collimator is used to overcome the restrictions in use of an X-ray tube window at the drive-shaft side of rotating-anode X-ray generators. The capillary adjustment shows brilliant long-time stability and is easy to rebuild after filament or cathode exchange. Intensity and monochromaticity of the collimated primary beam are sufficient for precession photographs of proteins. By shortening the collimator to 100 mm and coupling with a graphite monochromator, devices for recording complete diffraction data sets can now also be installed at this port.

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