A Conical-Type X-ray Guide Tube for Diffraction Experiments with Small Crystals

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(Received 4 May 1985; accepted 13 July 1986)

Abstract

The divergence and intensity distribution of X-rays transported through a conical-type X-ray guide tube (XGT) were measured. Diverging X-rays from a point source were condensed by use of the conical-type XGT. The parallelism of X-rays through the XGT was better than that encountered for a cylindrical-type XGT proposed previously [Nakazawa (1983). J. Appl. Cryst. 16, 239-241]. The intensity of the X-ray beam around the central axis of the conical-type XGT was exceedingly high in comparison with that measured without the tube and almost uniform over a cross-sectional area 60 µm in diameter. The high intensity provides the possibility of performing diffraction experiments with crystals as small as 20 µm in diameter with a conventional X-ray diffraction system.

Introduction

The use of an X-ray guide tube (XGT), through which X-rays are transported by total reflection, can provide a more intense X-ray beam than other methods (Nakazawa, 1983) and various applications of an 'X-ray light pipe' have been suggested (e.g. Vetterling & Pound, 1976). Cylindrical glass tubes were used as the XGT in those studies. The use of an XGT seems to be advantageous in particular for measurements of X-ray diffraction intensities from very small crystals. However, there remain some technical difficulties: (1) The divergence of an X-ray beam through the 'cylindrical' XGT is twice the critical angle of total reflection (e.g. 2 × 3.9 mrad at the wavelength of Cu Kα radiation). (2) The intensity distribution in a cross section of the X-ray beam is not uniform, resulting in an uncertainty in diffraction intensity measurements. A 'conical-type' XGT developed in the present study can eliminate these difficulties. A similar idea was proposed previously for an X-ray collimator which consists of two plates facing each other at a small angle (Lely & van Rijssel, 1949).

Experimental

The experimental arrangement is shown in Fig. 1. X-rays emitted from a rotating Cu target (20 kV accelerating voltage, 2 mA tube current and 0.1 × 0.1 mm focus size at a take-off angle of 6°) were transported through a conical-type XGT (0.25 mm inner diameter at the entrance, 0.45 mm at the exit and 220 mm in length). The XGT was made of a pyrex glass tube drawn with a lathe. For comparison, a cylindrical-type XGT (0.5 mm inner diameter with the same length) was also employed. The XGT was aligned by an X-Z stage to an accuracy of 10 µm. The X-ray beam thus obtained was examined from the following three aspects.

(I) The divergence through the conical-type XGT. This was compared with that attained by the cylindrical-type XGT by taking photographic images on the film placed at a distance of 1500 mm from the X-ray source.

(II) The intensity distribution in a cross section of the beam. This was measured by using a receiving slit (50 µm in horizontal width and 12 µm in vertical width) positioned in front of a SSD. The X-ray beam was traversed by the slit horizontally in steps of 60 µm.

(III) The applicability of the conical-type XGT to diffraction experiments with small crystals. This was examined by using a small pinhole (20 µm in diameter) placed at the center of a four-circle diffractometer. X-ray beam intensities through the pinhole were measured with and without the XGT for comparison.

Fig. 1. Experimental arrangement for examining the performance of the XGT. Units of distance are mm. Broken lines show X-ray paths. 2θ is the apical angle of the cone. Inset, a ray diagram shows the definition of the angles θ, α, β, and φ.

0021-8898/86/060453-03$3.50 © 1986 International Union of Crystallography
Results and discussion

1. Divergence of the X-ray beam

The divergence of the X-ray beam passing through a conical-type XGT is restricted by the geometry for total reflection of X-rays at the inner wall of the XGT (Fig. 1). Consequently, the divergence angle of the X-ray beam is given by $2(\omega - 2\varphi)$, where $2\omega$ is the divergent angle of the incident beam and $2\varphi$ the apical angle of the cone (Fig. 1). Thus, the conical-type XGT serves as a collimator. The size of the X-ray beam through the conical-type XGT was compared on a photographic film with that through the cylindrical-type XGT (Fig. 2). The angle of divergence for the former is $3.6$ mrad, which is about half that for the latter. This value is, however, about twice as large as the calculated one, while the value of the cylindrical-type XGT is in agreement with the calculated value. This shows that the conical-type XGT used has a shape somewhat deviating from an ideal cone.

2. Intensity distribution of the X-ray beam

The measured intensity distribution of the X-ray beam through the conical-type XGT is shown in Fig. 3 together with that measured without the XGT. The ordinate is the number of photons through the slit for a period of 128 s. The abscissa is the horizontal distance of the slit from the central axis of the X-ray beam. The results are summarized as follows.

(I) The profile of the observed intensity distribution is sharp in peak shape as expected but broader than calculated (inset, Fig. 3). The peak height is about three times as large as the intensity measured without the XGT. However, the peak height observed is considerably reduced in comparison with the calculated value. This may also be due to the deviation in the shape of the XGT from an ideal cone.

(II) The ratio of the integrated intensity of the reflected X-rays to that of the 'direct beam' is about four. The 'direct beam' means an X-ray beam passing through the XGT without total reflection, whose contribution to the total intensity is estimated from the intensity measured without the XGT and the divergence of the beam itself. The observed ratio is very close to the value of 4.3 calculated on the assumption that the reflectivity at the inner wall of the XGT is 1.0 for Cu Kα radiation.

The intensity increases sharply in the vicinity of the central axis (Fig. 3). This can reasonably be explained by the following geometrical consideration. For simplicity, the combination of an ideal point X-ray source and ideally conically shaped XGT is assumed. X-rays emitted with an angle in the range between $\omega_{+}$ and $\omega_{-}$ are reflected by the XGT and reach a vertical plane, supposed to be at the position of the slit, to form a hatched circle of radius $r$, as shown in the inset to Fig. 1. The radius $r$ can be given in terms of $\omega_{+}$ and $\omega_{-}$ as $r = f(\omega_{+}) = -f(\omega_{-})$, where the function $f(\omega)$ is defined as $f(\omega) = [d_{-} - d_{+} \tan \omega/(\tan \omega - \tan \varphi)] \times \tan(\omega - 2\varphi) - d_{-} \tan \omega/(\tan \omega - \tan \varphi)$. $d_{-}$ and $d_{+}$ are the distances from the X-ray source to the plane and from the source to the apex of the XGT cone, respectively. The solid angle between the two cones of the X-rays with apical angles of $2\omega_{-}$ and $2\omega_{+}$ is $2\pi(\cos \omega_{-} - \cos \omega_{+})$. Thus, the total intensity of

![Fig. 3. X-ray intensity distribution in the cross section of the beam transported through the conical-type XGT (solid circles). The intensity is shown as the photon number measured for 128 s through a slit (12 × 50 µm) positioned just in front of the SSD. The intensity measured without use of the XGT is shown by triangles. The contribution of 'direct' X-rays is shown by the hatched area. Inset, the intensity distribution calculated by assuming a point X-ray source and an ideal cone shape of the XGT is shown for comparison, where the ordinate scale is logarithmic. The hatched area represents the calculated contribution of the direct X-rays relative to that of the reflected X-rays.](image)
X-rays within the circle of radius \(r\) is given by

\[ I_r = 2\pi B_0 (\cos \omega_r - \cos \omega_y), \]

where \(B_0\) is the intensity of the X-rays emitted from the source (photons s\(^{-1}\) sr\(^{-1}\)). The intensity per unit area at any part of the circumference with radius \(r\) is given by \(B_r = (dI_r/dr)Ar/2\pi rAr\), i.e.

\[ B_r = B_0/r[1/(df/d\omega)_0 \sin \omega_r + 1/(df/d\omega)_0 \sin \omega_y]. \]

Therefore the intensity of X-rays through the slit can be given by integrating \(B_r\) over the slit area. A calculated intensity is shown for slit size \(10 \times 2.4 \mu m\) in the inset to Fig. 3. This is different from the size of the slit used in the experiment, but the profiles of the intensity distribution for both cases are basically the same. The calculated intensity increases sharply in the vicinity of the central axis of the beam. [Note that

\[ \lim_{r \to 0} B_r = \lim_{r \to 0} 2(B_0 \sin \omega_0/r)/(df/d\omega)_0, \]

where \(\omega_0\) is the angle of the X-ray beam reaching the central point of the plane \((r = 0)\).] This is because X-rays radiated at an angle \(\omega_0\) are reflected by the XGT and focused to the small area around \(r = 0\). Thus, there is a considerable gain in intensity in the vicinity of the central axis of the X-ray beam in comparison with the case without the XGT.

3. Applicability of the conical-type XGT

A pinhole of 20 \(\mu m\) diameter was placed at the center of a four-circle diffractometer. The intensities of X-rays through the pinhole were measured with and without the conical-type XGT for comparison. The intensity ratio gives an effective gain of intensity expected for a diffraction experiment with a crystal as small as 20 \(\mu m\) in diameter employing the conical-type XGT. The X-ray spectra obtained are shown in Fig. 4. The peak height of Cu \(K\alpha\) radiation through the XGT is 17 times larger than without the XGT.

The intensity distribution in a cross section of the X-ray beam through the XGT was measured by traversing a pinhole (20 \(\mu m\) diameter) in steps of 30 \(\mu m\). The result given in the inset to Fig. 4 indicates that the intensity in the cross section is almost uniform within a circle of about 60 \(\mu m\) diameter.

Data collection for the crystal structure analysis of inorganic materials is usually carried out using single crystals larger than 50 \(\mu m\) in diameter with a conventional X-ray generator and diffractometer. The results of the present investigation suggest that the use of an XGT can considerably lower the limit of crystal sizes for diffraction intensity measurements even with a conventional system.

The authors thank Mrs A. Sato and Y. Masuda (NIRIM) for their help in the construction of the experimental setup with the XGT. They also thank Dr P. F. Okamura (NIRIM) for his critical reading of the manuscript.

References

