X-ray Guide Tube for Diffraction Experiments

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(Received 8 March 1982; accepted 25 October 1982)

Abstract

For diffraction experiments with an extremely small crystal or with very long distance from X-ray source to detecting instrument, the use of an 'X-ray guide tube' (XGT) is proposed on the basis of a simple consideration and experiment with a pyrex-glass tube. X-ray intensities through an XGT of glass and also through a pin hole were measured as a function of photon energy and compared to derive the gain by use of XGT. At wavelengths between Cu Kα and Mo Kα radiations, the observed gain in brightness is about thirty to twenty for the present setup, corresponding to about 50 to 80% of the maximum possible gain calculated on the assumption that the reflectivity of X-ray total external reflection is 1.0.

Introduction

In X-ray diffraction experiments, there are some cases where the specimen is forced to be a long distance from the X-ray source, e.g. the use of a large χ circle on the four-circle diffractometer or use of synchrotron radiation (SR). A focusing mirror has recently been constructed for the concentration of SR applying the phenomenon of X-ray total external reflection (XTER) (e.g. Ehrenberg, 1949; Rehn, 1978; Rosenbaum & Harmsen, 1978). The X-ray mirror involves, however, technical difficulties in its construction and use, especially at the wavelength of 'hard' X-rays. The mirror does not function as a matter of course for diffraction experiments with a usual X-ray source.

An X-ray guide tube (XGT) is a logical tool for such a case, avoiding technical difficulties, because it does not require imaging. Since the intrinsic width of a Bragg spot caused by the crystal mosaicity is considered to be the order of milliradians for usual crystals (Renninger, 1934; Miyake, 1969), an angular resolution of experimental geometry better than this value is not necessary for the intensity collection of Bragg spots for structure analysis or related studies. The order of milliradians is comparable with the critical angles of XTER for usual glass at the wavelength of the 'hard' X-rays. This similarity in angle suggests that a laboratory glass tube such as pyrex glass may be used as an XGT, since the surface roughness of the inside wall of a glass tube is rather better than that of other polished materials.

The idea of an XGT using a laboratory glass tube was not entirely original: Pound & Rebka (1959) suggested the idea for the test of gravitational red shift. Vetterling & Pound (1976) reported recently that the reflection efficiency of γ radiation from a 37Co source was over 80% for the inside wall of a laboratory glass tube. Cylindrical or conical glass capillaries have previously been used as a tool in the X-ray microbeam technique (e.g. Peiser, Rooksby & Wilson, 1960).

In the present study, the gain by use of an XGT has been measured for white X-rays in the energy range from 1.5 to 50 keV. Based on the advantageous result of its use, an XGT of external-extrusion-die flow glass tube is proposed as an experimental tool for X-ray diffraction experiments which require 'stronger' X-rays and do not need angular resolution higher than some milliradians.

Experimental and results

A pyrex-glass tube of 0.5 mm inside and 6.0 mm outside diameters was utilized as an XGT. No means was used to reduce air scattering and absorption. A simple device used to align the XGT was made of a brass cylinder and two sets of four micrometer heads opposed horizontally and vertically (Fig. 1). A fine-focused X-ray source was used (0.2 × 0.2 mm, viewed at the take-off angle of 6°). The distance between the X-ray source and the beginning of the tube was 85 mm due to the geometrical restriction of the apparatus used. At the beginning of the tube, the maximum angle between the tube wall and X-rays entering the tube was 2.9 mrad corresponding to the critical angle of XTER for a wavelength of 1.15 Å.

The main interest is in the efficiency of energy-dependent transmission of X-rays through an XGT. This can easily be known by photon counting and comparing the intensities of transmitted X-rays through an XGT and those through a pin hole having the same diameter as that of the XGT. A solid-state detector (SSD, pure Ge) and a long glass tube (630 mm) were set up with the alignment device as shown in Fig. 1. The pin hole was placed at the same position as the end of the XGT when it replaced the XGT.
Because the X-ray intensities of the two paths were so different that these could not be measured under the same conditions of the photon-counting system and X-ray generation, the size of the pin hole was enlarged to 1.0 mm in diameter. The collected data were numerically normalized to the same diameter of 0.5 mm. A small difference in the dead time of the photon-counting instrument was also corrected.

The intensity ratio of X-rays through the XGT to that through the pin hole can be calculated by simple division of the observed number of photons at each channel (Fig. 2, curve A). The observed ratio indicates the obvious advantage of the XGT. In the present setup, it is over thirty for the wavelength of Cu Kα and twenty for that of Mo Kα which are those usually used for X-ray diffraction experiments.

Another interest in the XGT is the possibility and the limitation of guiding X-rays through a non-straight configuration (in analogy with an optical fiber). The intensity change of X-rays through an inclined (or actually bent) XGT was measured at every 0.1 mm shift of the tube end in the same experimental setup. Because the curvature of the bent XGT was not precisely known, it could only be concluded that it is possible to bend the XGT of the order of milliradians without much loss of X-ray intensity. This indicates, however, a convenient feature of the XGT: an easy alignment of the XGT and a possibility of joining several units of an XGT of glass tubes for long transportation of X-rays.

**Discussion**

As a result of the present experiments it is worth while to attempt a practical use of an XGT of a glass tube for an X-ray diffraction experiment which requires stronger X-rays and does not need angular resolution higher than the order of some milliradians.

If an 'ideal' arrangement of an XGT is supposed as that of an ideal point source which lies on the center axis of a straight XGT, the absolute maximum possible gain in brightness at the end of the tube is given by $G$, described below for the X-rays of which the critical angles of XTER are equal to or larger than half of the visual angle of the tube wall from the source:

$$G = \frac{\theta_1^2 + \theta_2^2}{\theta_1^2},$$

where $l_1$ = distance from source to start of tube and $l_2$ = length of tube. In the present arrangement, X-rays with wavelengths longer than 1.15 Å satisfy the above condition and the maximum possible gain is $G = (85 + 630)^2 / 85^2 = 71$ so that the presently observed gain, 30–40, is extremely good. To get a similar gain for X-rays of shorter wavelength, the inner diameter of the XGT must be smaller: e.g. 0.3 mm for Mo Kα radiation.

Because the critical angle of XTER is a function of X-ray energy, i.e. $\theta_c = k \sqrt{\rho / E}$ ($k = 2 \times 10^{-2}$ rad keV, $\rho$ = density of SiO₂ = 2.5 and $E$ = X-ray energy in keV), the maximum possible gain for X-rays with shorter wavelength than 1.15 Å in the present arrangement is given by $G = (85 + 630)^2 / l_1^2$, where $l_2 = (1/2)d/\theta_c(E)$ in mm and $d = 0.5$ mm (the diameter of the tube hollow), representing the distance from the source to the position from which XTER takes place for X-rays with energy of E(keV). The energy dependency of the maximum possible gain is shown in Fig. 2 as curve B to compare with the observed intensity ratio of X-rays through an XGT to those through a pin hole.

Although the agreement of observed and calculated gains is rather good, at least for their dependencies on X-ray energy, an interesting feature may also be seen: the ratio of observed to calculated gains increases with increasing X-ray energy, and, at above 25 keV, the observed gain is even slightly higher than that calculated on the assumption that the reflectivity of
XTER is 1.0. This may qualitatively be explained by the following reasons: (1) X-rays in the solid angle of $\pi 0.5^2/l^2$ are only taken into account for calculation of the gain, assuming that the tube wall is perfectly smooth and straight. In practice, the surface of the wall is, however, more or less irregular so that part of the X-rays which hit the wall with larger solid angle would be reflected and additionally guided to the tube end. (2) Even though the wavelength is a few ångströms or less and no surface is smooth to that degree, XTER takes place at the wall. The surface may, therefore, appear smoother than it is, when the surface is viewed at a glancing angle. This effect of glancing angle may be larger for higher-energy X-rays because of their smaller critical angles. The reflectivity of higher-energy X-rays may, therefore, be closer to the ideal one than that of lower-energy X-rays. (3) The number of reflections, which is larger for lower-energy X-rays (e.g. four times the number for X-rays less than 12.7 keV compared with those higher than 29.5 keV), is a factor to reduce preferentially the intensities of lower-energy X-rays.

More detailed calculation such as a ray tracing is required to interpret quantitatively the observed gain by using realistic parameters of source size and smoothness of the tube wall. What the present result directly indicates is, however, that the advantage of the use of an XGT is quite obvious. The experiment of a non-straight-tube configuration indicated that a misalignment less than some milliradians attenuates X-rays not so much that the advantage of an XGT is largely reduced. This suggests an easy alignment of an XGT and a possibility to join several units of glass pipe for long transportation of X-rays.

The author wishes to thank Dr M. Iwata, National Institute for Research in Inorganic Materials for helpful discussions to initiate this experiment, Dr O. Shimomura, NIRIM, for his help in data processing, Dr O. Fukunaga, Dr S. Yamaoka, NIRIM, and Dr M. Andow, National Institute of High Energy Physics for their continued encouragement and Dr M. Glazer, University of Oxford for his critical reading of the paper.

References