Published in final edited form as:

Nucl Instrum Methods Phys Res A. 2011 September 1; 649(1): 101–103. doi:10.1016/j.nima.2010.12.023.

A new beamstop for microfocus X-ray capillary beams

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Abstract

In order to accurately measure the photon flux and to assist in aligning the beam, we have designed a modified beam stop device based on a photo diode integrated with the beam stop. The beam stop contains a small CdWO₄ crystal that completely stops the X-rays and at the same time produces photoluminescence proportional to the X-ray flux. The light is then guided to a photosensitive diode, using a flexible light pipe, to monitor the flux. With this device we achieve the goal of stopping the primary X-ray beam and simultaneously monitoring the X-ray intensity, thus eliminating the need for integrating ion-chambers into the capillary or collimator mount.

Keywords

Synchrotron radiation; microbeam; beamstop

1. Introduction

With the intense X-ray beams from modern synchrotron sources, limiting radiation damage to the samples becomes an important concern. In order to plan experiments appropriately, it is necessary to calculate the amount of radiation a sample will have absorbed as a function of exposure time. Programs such as RADDOSE [1,2], can do this, and can determine how much exposure is tolerable before damage becomes so severe that the data are no longer reliable; to do the calculation, accurate knowledge of the X-ray flux (i.e. photons s⁻¹) at the beamline is required. At CHESS, a small ion chamber mounted on the tip of a collimator is used to measure flux; however, the accuracy of this measurement is low. It is primarily used for alignment of the optical table that holds the experimental setup, for which purpose only relative intensity readings are important; maximization of ion-chamber counts by translations and rotations of the table, around a “virtual center” at the position of the sample, assures optimal alignment of the beam.

When a microbeam is required, a simple collimator is unsatisfactory. The alternative used at CHESS is microfocusing glass capillaries with ellipsoidal profiles, developed by D. Bilderback's group [3]. These capillaries are suitable for applications ranging from materials science experiments looking at small-scale inhomogeneities to diffraction experiments on biomolecular specimens, including both small angle X-ray scattering (BioSAXS) and single

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crystal diffraction (macromolecular crystallography, MX) studies. [4] For macromolecular crystallography, MacCHESS routinely uses capillaries with focal spot sizes of $\sim 20 \mu\text{m}$ and intensity gains of a factor of 10, at the cost of a modest increase in beam divergence, to 2-4 mrad [5].

In the case of a capillary mount, integration of an ion chamber before the sample is severely hampered by space restrictions and the relatively high voltage needed in such a device, and has not yet been implemented. With no ion chamber, the flux cannot be measured during the experiment, and the automatic table alignment procedure does not work. Hence we sought an alternative means of measuring the X-ray flux when a capillary is in use.

Recent experiments have shown that the X-ray flux can be accurately measured with a high quality silicon PIN diode [6]. Space restrictions would make it difficult to mount such a device either on the capillary mount or on a separate support near the sample. However, there is another alternative.

X-ray experiments almost always require a beamstop, whose role is twofold: it blocks the direct X-ray beam to protect the detector from overexposure and possible damage to the detector surface, and also keeps the path of the beam in air short to reduce air scatter, which affects the background, especially in the low resolution region. Ellis et al. described the design of a beamstop using a PIN diode encapsulated into a tungsten epoxy composite mounted at the end of a tapered support cut out of a circuit board [7]. It had a diameter of only 1.5 mm and useful response down to an X-ray energy as low as 6 keV [7]. We have now designed a modified beamstop device based on the photoluminescence of cadmium tungstate, CdWO_4 , incorporating a light guide fiber optic conducting a signal to a photo sensitive diode. This device provides both accurate measurements of the photon flux and a means for aligning a capillary optic with the beam.

2. The Device

The device is based on the photoluminescence of cadmium tungstate. The material is highly stable in the X-ray beam, is non-hygroscopic and has excellent stopping power for the targeted X-ray energy range of 6 - 18 keV. A thin walled rigid steel tube (1.3 mm inner diameter, 0.075 mm wall thickness, about 100 mm long) is used as a support. At the tip of the tube a small window is cut out and a custom fabricated CdWO_4 crystal ($0.92 \times 0.92 \times 5 \text{ mm}^3$, Marktech Intl.) [8] is placed behind the window (figure 1a). From the other end of the tube a fiber optic filament with the same diameter as the inner diameter of the tube is inserted and pushed up against the small face of the CdWO_4 crystal. The tube is then flattened around the crystal, giving a total profile of about 1.1 mm. To prevent visible light from entering the window is covered with a thin layer of aluminized Mylar foil (figure 1b).

3. Implementation

The steel tube is mounted onto the beamstop bracket along the phi axis of the goniometer (figure 2) so that the shadow of the tube only affects reflections along the rotation axis with high Lorentz factor. Figure 3 illustrates this fact depicting a typical diffraction pattern of bovine trypsin. A motorized x/y stage supports the beamstop bracket and allows for accurate alignment of the beamstop in front of the beam. The fiber optic connects the CdWO_4 crystal to a photo diode that can be placed 30 cm or more from the goniometer with little loss of sensitivity. When the CdWO_4 crystal is exposed to X-rays light is emitted and is transferred via the fiber onto the diode. The amount of photoluminescent light from the crystal is proportional to the amount of X-ray photons that hit the crystal. After calibrating the device for the specific energy of the beamline, the length of the fiber and the characteristics of the

photo diode, absolute flux measurements for the individual samples are available. The device is currently prototyped at the CHESS F1 station for macromolecular crystallography.

In order to test the performance of the beamstop we measured the response of the device at various X-ray fluxes at 13.5 keV, the operational energy of the F1 station. To vary the X-ray flux aluminum filters of defined and increasing thickness were placed in front of the beamstop. With these filters the flux was attenuated from 100% (no filter in the beam) to 70% (5 mil=127 μ m aluminum filter) [9]. For comparison the beam flux was also measured using an ion chamber under the same conditions.

The normalized transmitted X-ray flux for various filter thicknesses is shown in figure 4. The data points measured by the beamstop diode are close to the theoretical X-ray transmission curve (Beer-Lambert law). The data scatter for the diode response is noticeably less than the corresponding values measured using an ion chamber.

4. Summary and Conclusion

A new X-ray monitoring device based on the photoluminescence of CdWO₄ has been designed and incorporated into a beamstop. With this device we achieve the goal of effectively stopping the primary X-rays and simultaneously monitoring the X-ray intensity, thus eliminating the need for integrating a bulky, high-voltage, ion-chamber into a capillary mount. The device can also be used with standard collimated beams or slits. Since its response does not depend on ionization of air, it is unaffected by changes in the atmosphere, most notably the humidity. Further testing is scheduled for the upcoming fall X-ray period at the macromolecular crystallography stations.

Acknowledgments

The authors would like to thank Marian Szebenyi for helpful comments on the manuscript and all members of MacCHESS for critical discussions. This work is based upon research conducted at the Cornell High Energy Synchrotron Source (CHESS), which is supported by the National Science Foundation and the National Institutes of Health/National Institute of General Medical Sciences under NSF award DMR-0936384, using the Macromolecular Diffraction at CHESS (MacCHESS) facility, which is supported by award RR-01646 from the National Institutes of Health, through its National Center for Research Resources.

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Fig1a

Fig1b

Figure 1.

- (a) A view of the steel tube with a window cut out at the end. Just behind the opening a $0.92 \times 0.92 \times 5$ mm CdWO_4 crystal is inserted. The steel tube is flattened to firmly hold the crystal in place and reduce its profile.
- (b) The window is covered with a small piece of aluminized Mylar foil to prevent light from entering the fiber.

Figure 2.
The goniometer with the beamstop at the left of center, mounted in line with the goniometer phi axis. X-rays enter through the collimator from the right (here shown with an ion chamber).

Figure 3. A diffraction pattern of bovine trypsin (cell dimensions $55 \times 55 \times 106 \text{ \AA}^3$, hexagonal symmetry) taken at the CHESS F1 station (energy = 13.5 keV), showing the size of the shadow from the beamstop when moved close to the sample. The size of the shadow can be reduced by increasing the sample to beamstop distance.

Figure 4.

The graphical analysis of the normalized transmitted X-ray flux vs. thickness of the aluminum filter shows a smooth response for both types of monitoring devices: ion chamber (black squares) and the new photodiode-coupled CdWO_4 crystal (red circles). Both follow closely the ideal line described by the Beer-Lambert law (red line). Data taken at the CHES F1 station at 13.5 keV.