Beamstop with integrated X-ray sensor

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A versatile beamstop with an integrated sensor has been developed at the Stanford Synchrotron Radiation Laboratory (SSRL) using non-specialized components. A diameter of 1.5 mm was achieved using a commercial subminiature surface mount PIN diode (Phillips BAP64) molded into a tungsten epoxy composite cup. The cup is supported on a thin fiberglass arm with printed circuit traces to transmit the signal from the diode. The assembly has an active area of approximately 100 μm in diameter. As the diode is encapsulated in plastic, the response diminishes with decreasing energy but is still useful at 6 keV.

Keywords: beamstops.

1. Introduction

In a typical macromolecular structure determination, a collimated X-ray beam is passed through a rotating crystalline sample and the resulting diffraction pattern is measured using an area detector. A small cup fabricated from a high-Z metal such as lead or tungsten is usually placed immediately following the sample to block the transmitted X-rays. As the sample absorbs little of the incident beam, this is essential to reduce background air-scatter and to protect the sensitive detector. Ideally, the 'beamstop' will be large enough to completely obstruct the direct beam, yet small enough to be placed close to the sample without obscuring the important low-angle data. It should also be sturdy to withstand repeated jarring such as often occurs during manual sample mounting, while easy to align with respect to the beam. At present the standard method is to hold the beamstop in place using thin kapton tape or a slender metal arm aligned with the rotation axis to place the shadow within the cusp of the diffraction pattern.

At the Stanford Synchrotron Radiation Laboratory (SSRL), although we have successfully used tungsten beamstops of the latter design for several years, they were subject to two problems: (i) alignment relied on the use of photographic film, which rendered the procedure very tedious; (ii) the long arm was easy to bend making them susceptible to misalignment. With the imminent SPEAR3 upgrade and the arrival of large-area mosaic CCD detectors, we had become increasingly concerned that accidental exposure to the direct beam resulting from a misaligned or damaged beamstop could result in serious detector damage. Consequently, developing a beamstop addressing the limitations of the previous system became a priority.

A small versatile X-ray beamstop with integrated sensor, mounted on a flexible fiberglass arm (Fig. 1), is described. During the experiment, the signal from this sensor may be monitored to protect the detector from prolonged direct-beam exposure and may be maximized during scans of the beamstop position for rapid alignment.

2. Design

The beamstop design consists of a cup assembly supported on a long fiberglass arm. The cup incorporates an X-ray photodiode and has an active area of approximately 100 μm in diameter. The photodiode is connected to an external current-to-voltage amplifier through traces incorporated in the arm. With a design diameter of 1.5 mm the cup can be placed 16 mm from the sample at the Se edge for a low-resolution cut-off of 20 Å.

The small cup diameter was achieved using an off-the-shelf subminiature surface mount PIN diode [Philips BAP64 (Philips Semiconductors, 2001)] molded into a tungsten epoxy composite cup. As the diode is encapsulated in plastic, the response diminishes with decreasing energy but is still useful at 6 keV. Unlike other solutions, this device directly measures the beam and does not require any additional membranes or foils. Furthermore, it is insensitive to visible light and thus requires no special shielding arrangements. It is currently in use at the SSRL macromolecular crystallography stations.

3. Fabrication

The beamstop is fabricated in two main steps: (i) constructing the fiberglass arm and attaching the diode; (ii) fabricating the composite cup.

The fiberglass arm was manufactured from a printed circuit board, 200 mm in length and 5.4 mm wide, with through-holes for mounting and for a connector at one end joined by traces to through-holes for the diode at the other. As the prototype device was found to oscillate by several hundred micrometers when the shutter (Crystal Logic, based on a high-speed rotary solenoid) was opened or closed, it was stiffened by laminating it on both sides with blank fiberglass material to a final thickness of 5 mm at the base, tapered to a thickness of 0.8 mm at the sensor end. With the experimental set-up currently in use at SSRL, the vibration induced by shutter operation has an initial peak-to-peak amplitude of 60 μm, dampened by a factor of four every second. Finally, the diode mounting through-holes were filled with solder, the end of the board was sectioned through the midpoints and the diode was soldered to the newly exposed surface (Fig. 2).

The final manufacturing process was fabrication of the cup surrounding the diode. This cup consists of a molded tungsten/epoxy composite surrounding the diode and a 1 mm-thick solid tungsten backplate. The composite consisted of a 7:1 mixture by weight of 2 μm tungsten powder and 30 min epoxy. It is approximately 1:1 tungsten:epoxy by volume and hence has about half the X-ray stopping power of solid tungsten but is considerably easier to form and has the additional advantage of being non-conductive.

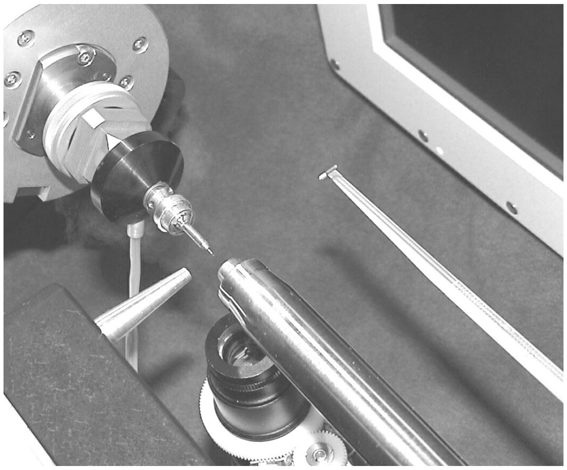

Figure 1
The new beamstop as mounted on SSRL beamline 11-1. As shown, the beamstop is 40 mm from the sample. Using the motorized z-translation, it can be moved in as close as 13 mm.

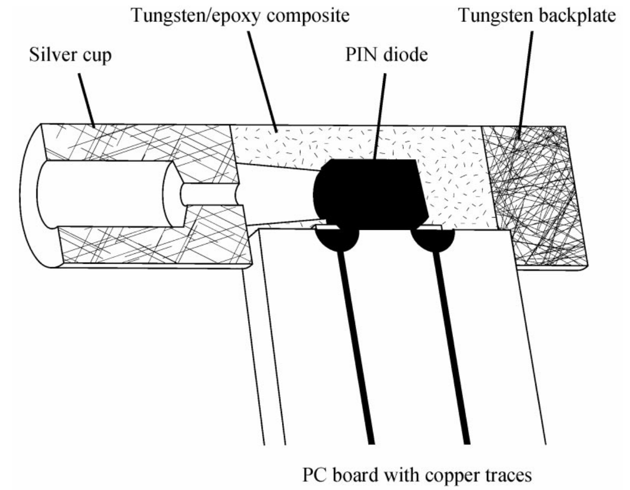

Figure 2
Cutaway diagram illustrating the construction of the beamstop cup assembly.

A mold was manufactured from acetal, consisting of two halves to form the outside of the cup and a plunger to form the tapered hole leading to the sensor. All components were attached in a single operation by positioning the arm to place the diode in the center of one half of the mold, placing the tungsten backplate behind the diode, filling the mold with the unset composite, clamping the two halves of the mold together and inserting the plunger until it rested against the front face of the diode.

Finally, a machined silver cup was glued to the front of the beamstop. This piece has a small through-hole (500 µm diameter) to allow the beam to reach the diode. It serves to shield the sample fluorescence detector from tungsten fluorescence.

4. Field trials

One of the two mounting holes in the beamstop arm fits snugly over a 0.125″-diameter dowel pin and the second accommodates a 4-40 UNC thumb screw allowing easy removal and accurate repositioning. The beamstop is connected to a motorized (x, y, z) stage enabling automatic centering on the direct beam and control of the sample–beamstop distance. The diode photocurrent is amplified by an SRS570 current-to-voltage amplifier before being digitized and used by the *Blu-Ice* beamline control software (McPhillips *et al.*, 2002). The system has been in operation on SSRL beamlines for more than a year with no noticeable loss of diode function from radiation damage.

The energy response was measured at SSRL beamline 9-2 from 6 keV to 16 keV incident photon energy (Fig. 3). Over this energy range, the response per photon changes by approximately three orders of magnitude. We attribute this to the plastic encapsulating the diode. Despite this variation, a useful signal is available at all energies.

Since being installed on the beamlines, the beamstop sensor has also proven useful in ways not considered when it was designed. Examples include examining the direct-beam profile, characterizing the experimental shutter timing, and measuring the absorbance spectra from foils for energy calibration.

As the z-translation on the motorized stage enables the beamstop to be moved as far away as 300 mm from the sample, the sensor has been used to measure the vertical and horizontal beam profile at the detector distance for focusing beamline optics. This method produces more reliable parameters than the fluorescent screens which are commonly used for this purpose.

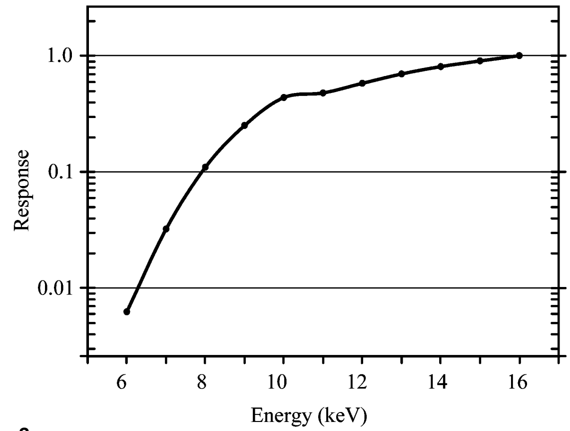

Figure 3
The energy response of the beamstop sensor. The response is plotted on a logarithmic scale relative to the signal at 16 keV.

The sensor has also been used to measure the time taken for the experimental shutter to open and close by recording the shutter control signal and the signal from the PIN diode on a storage oscilloscope. This set-up revealed unexpected shutter bounce in a faulty unit.

Finally, the sensor has been used to measure the absorbance spectra of various foils. This has proven particularly useful during monochromator testing as there is normally only one Se foil and six or seven Al attenuators at each of the SSRL beamlines. Although additional foils could be inserted into the beam prior to the final ion chamber, this requires breaking the He path, necessitating a time-consuming purge. With the new sensor, an edge can be easily measured by placing the foil at the sample position and calculating the absorbance as $-\log(I_{\rm beamstop}/I_2)$.

5. Conclusions

We have described a versatile beamstop with an integrated sensor fabricated from cheap non-specialized components. It is a drop-in replacement for the traditional SSRL beamstop and forms the basis of an area-detector protection system to be deployed before the SPEAR3 upgrade.

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