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Silicon nitride transmission X-ray mirrors

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Transmission X-ray mirrors have been fabricated from 300–400 nm-thick low-stress silicon nitride windows of size 0.6 mm × 85 mm. The windows act as a high-pass energy filter at grazing incidence in an X-ray beam for the beam transmitted through the window. The energy cut-off can be selected by adjusting the incidence angle of the transmission mirror, because the energy cut-off is a function of the angle of the window with respect to the beam. With the transmission mirror at the target angle of 0.22°, a 0.3 mm × 0.3 mm X-ray beam was allowed to pass through the mirror with a cut-off energy of 10 keV at the Cornell High Energy Synchrotron Source. The energy cut-off can be adjusted from 8 to 12 keV at an angle of 0.26° to 0.18°, respectively. The observed mirror transmittance was above 80% for a 300 nm-thick film.

Keywords: X-ray optics; white X-ray beam; X-ray windows; Laue diffraction.

1. Introduction

Synchrotrons are magnificent X-ray sources that use an extensive arsenal of X-ray optics to tailor the X-ray beam for a wide sampling of experiments. X-ray transmission mirrors (TMs) are one type of X-ray optic which has been used in the past, but they have not been used in 15 to 20 years because of mechanical and lifetime limitations. The first TM optics were made from soap-bubble films, which performed very well as TMs (Lairson & Bilderback, 1982). However, the major limitation of the soap-bubble TM was that it lasted only a few hours in the X-ray beam. This limitation made them difficult to use if the experiment lasted more than a few hours. The bubble TMs were used in time-resolved Laue diffraction experiments and in wide-bandpass X-ray fluorescent experiments (Bilderback *et al.*, 1984). Attempts at producing a better TM included employing a 0.5 µm-thick Mylar film which was stretched onto a frame (Iida *et al.*, 1985). This technique also had some limitations: the film was not uniform enough in thickness and only lasted for a day or two in the X-ray beam at the Photon Factory (T. Matsushita, personal communication). Since that time in the mid-1980s, and to the best of our knowledge, there have not been any further attempts at fabricating a more permanent TM.

TMs are very similar to total-external-reflection mirrors. The difference is that the TM is very thin, so the beam which is normally absorbed by the total-external-reflection mirror is transmitted instead. The total-external-reflection mirror reflects X-ray energies up to a sharp cut-off energy, which is set by the critical angle for total external reflection. Energies above this cut-off energy are transmitted into the mirror. Total-external-reflection mirrors make excellent low-pass energy filters because they have a reflection of close to 100% below the cut-off energy, and the reflected energy drops off rapidly above that cut-off energy. For those reasons, total-external-reflection mirrors are a staple optic used at synchrotron facilities. TMs work in just the same way, but the beam used is the transmitted beam, thereby switching the functionality into a high-pass filter. In the limit of having a very thin film, the critical energy cut-off is just as sharp as it is for the total reflection mirrors. It is this sharp cut-off energy, in conjunction with changing the cut-off energy (by changing their angle), which makes them very attractive high-pass filters compared with absorption filters.

There are a few critical parameters that determine the optical properties of the TM. The TM has to be thin, flat, smooth and uniform in thickness. All of these qualities have to be maintained over a fairly large surface area to allow usable beam sizes. The mirror has to be thin, from 0.5 µm to as thin as possible, to minimize the absorption of the film. Another way to minimize the effects of absorption is to have the mirror material made of a low-atomic-number material. The mirror must be flat so that the angle of incidence does not vary over 5% (Bilderback *et al.*, 1984). At an angle of 0.22° (3.8 mrad) this corresponds to a variation of 0.011° (190 µrad). By comparison, high-quality X-ray reflection optics generally have less than a 0.003° (10 arcsec or 50 µrad) variation. The mirror has to be smooth and uniform in thickness. Without a smooth flat and uniform boundary, X-ray mirrors do not work well. A small amount of roughness can be tolerated and may even have some advantages in dampening the amplitude of the Kiessig fringes of the transmitted beam.

2. Silicon nitride membranes

The most limiting feature of the original TM was its very short life span in the X-ray beam. One major reason for attempting to make TMs out of silicon nitride is that this material has proven itself to be a radiation-hard material in X-ray window applications, lasting virtually indefinitely in synchrotron X-ray beams. We have been able to test the life span of the silicon nitride membrane TM in two ways: (i) a TM was put in a white X-ray beam at CHESS's B1 bending-magnet station for 24 h; (ii) a TM was used for ten days intermittently in a pink X-ray beam (with a high-energy cut-off at about 13.5 keV) at the D1 bending-magnet station at CHESS. Both mirrors suffered no degradation over the time that they spent in the X-ray beam. We do not anticipate radiation damage to be a short-term failure mechanism.

The films for the windows were grown by vapour deposition on silicon double-sided polished 100 mm-diameter wafers. The films grown on the wafers had thicknesses of 300 nm and had a uniform thickness of within 2–5% over the entire wafer, with the thickness in the middle of the wafer being slightly thinner than the thickness at the edges. The roughness of typical silicon nitride films is 1.5 nm r.m.s. to well over 2 nm, measured by atomic force microscopy at the Cornell Nano-Scale Science and Technology Facility (R. Ilic, personal communication). Our films have not been measured by atomic force microscopy to verify their roughness. Each wafer had three windows of size 0.6 mm × 85 mm. One of the 0.6 mm sides was not supported by the silicon wafer in order to allow the transmitted beam to pass through. Thus, a piece of silicon wafer covered by silicon nitride was glued onto the top side of the silicon wafer with epoxy to support the free side of the silicon nitride window (Fig. 1). This unfortunately blocks the reflected beam from the transmission window. Other support methods have been tested to allow collection of both the transmitted and reflected beams from the silicon nitride membrane, but they have not yet produced a working TM.

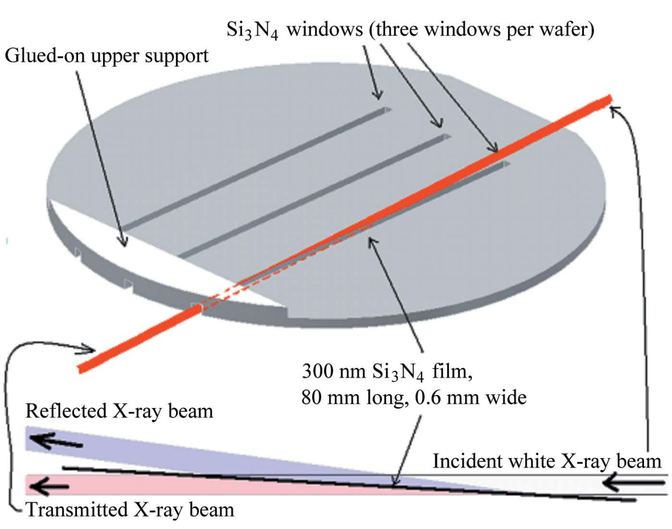

Figure 1
Drawing of silicon nitride TM windows on a silicon wafer. The upper support blocks the reflected beam.

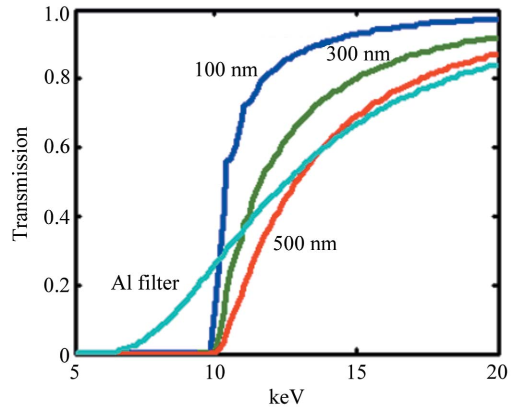

Figure 2
Calculated transmission of 100 nm-, 300 nm- and 500 nm-thick silicon nitride membrane at 0.22° with 5 nm r.m.s. roughness on the surfaces. A 200 µm aluminium attenuation filter is also shown for comparison. Calculated at http://www-cxro.lbl.gov/.

We wanted to have the membrane as thin as possible. Fig. 2 shows the predicted transmission curves for membranes of 100 nm, 300 nm and 500 nm thickness, at 0.22° and with 5 nm r.m.s. of roughness (Henke *et al.*, 1995–2007). The TM has a higher transmission efficiency than the aluminium absorber. The TM also has a desirable sharp change in its energy-response curve. The thinner windows have better transmission properties, especially near the cut-off energy. Silicon nitride windows are very strong for their thickness (100–1000 µm), but the windows become more fragile when the membrane is thinner or has a larger window size. We tried to make both 100 nm- and 300 nm-thick windows. For a window size of 0.6 mm × 85 mm, none of the 100 nm films survived the etching process. The 300 nm-thick windows, however, had roughly a 50% survival rate from the fabrication etching process.

3. Silicon nitride membranes in a white beam

The silicon nitride TMs were tested in the B1 station white beam at CHESS with the full energy spread from a bending magnet with 10 keV critical energy. The only other elements in the beamline were two beryllium windows between an air/vacuum and vacuum/vacuum environment, a few Kapton windows on helium-filled flight tubes, and the TM chamber. The beam was slitted down to 0.2 mm × 0.2 mm and was then put through the TM. The X-ray beam travelled 2.25 m in a helium flight path, then through a 1 mm-diameter lead clean-up aperture, and finally through a 0.5 mm-thick film of Kapton tape. An XFlash diode detector was used to measure the Compton scattering from the Kapton tape in order to observe the energy spectrum of the beam. The effects of transmission of windows, flight paths, detector efficiency *etc.* were normalized by taking a scan with the TM in the beam, then dividing it by a scan taken without the TM in the beam. Fig. 3 shows the observed transmission curves, along with the predicted transmission from two different mirrors. Fig. 3(*a*) shows the transmission curves for a 300 nm-thick silicon nitride

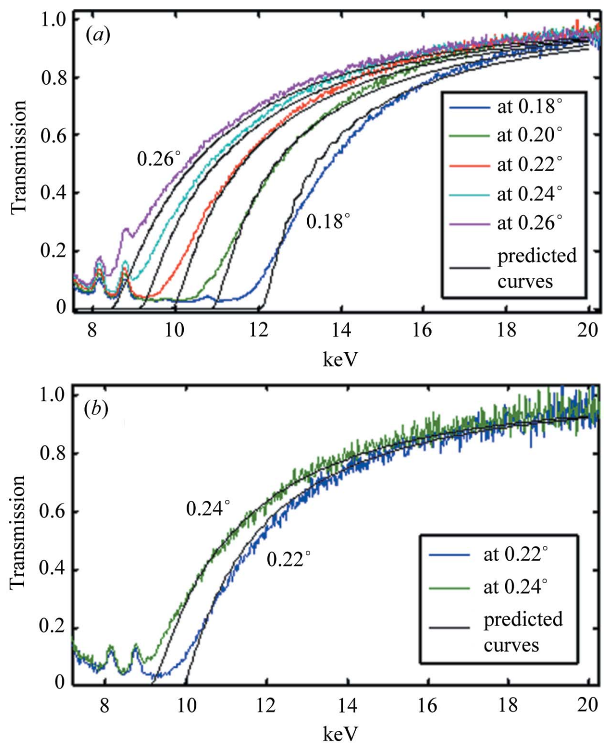

Figure 3
(*a*) Transmission of a 300 nm-thick silicon nitride film at angles ranging from 0.18° to 0.26°. (*b*) Transmission of a second 300 nm-thick silicon nitride film at angles 0.22° and 0.24°. The small peaks between 8 and 9 keV in the transmission curves are a result of both copper and zinc fluorescent contamination in the signal that we were unable to eliminate, which most likely originated as trace elements within the Kapton tape.

membrane at angles from 0.18° to 0.26° in steps of 0.02°, along with the predicted curves, which assume a 5 nm r.m.s. roughness. The actual curves track well with the predicted curves. Angles below 0.18° were not reasonable because the beam started to be clipped by the glued-on silicon wafer support. We did not go above 0.26° because the cut-off edge started to be obscured by impurity X-ray fluorescent signals from the scattering Kapton tape. Fig. 3(*b*) shows a curve from a second window at 0.22° and 0.24°, which is also well matched to the predicted transmission curves.

From these transmission curves we can confirm that the flatness of the silicon nitride membrane is of the order of 0.01° (119 μrad) or below. The consequence of this lack of flatness is that the transmission edge is not as sharp as the predicted sharpness. The observed quality of the TM transmittance in Fig. 3(*b*) is slightly better than for the TM in Fig. 3(*a*). This can be seen by observing the match between the predicted and actual transmission curves close to the cut-off energy. Also, to explain the absence of Kiessig fringes in the predicted transmission curves, the mirrors need a roughness of 5 nm r.m.s. The roughness estimated here is based solely on eliminating the Kiessig fringes to match the measured transmission curves. The actual roughness may be lower, with the Kiessig fringes most likely eliminated by a combination of effects, such as a combination of surface roughness of less than 5 nm and the variation in flatness at a level of 0.01° or less. Single oscillations of the Kiessig fringes for a membrane of thickness 300 nm are of the order of 100–150 eV FWHM (Henke *et al.*, 1995–2007). Because the fringe energy spacing is close to the resolution of the detector, and the fringe modulation is about 5% or less, we were not able to observe them with the X-flash detector (resolution of 130 eV to 170 eV between 10 and 15 keV, respectively). Thus, from the fitting work, we estimate that the membranes have a 5 nm r.m.s. roughness or less.

4. Conclusions

We have successfully demonstrated the value of silicon nitride TMs as permanent high-pass energy filters for synchrotron applications. They are adequately thin, flat, smooth and uniform in thickness for working TMs. They have been able to last in an X-ray beam for several days, and could last a great deal longer. We plan on using them in conjunction with traditional reflection X-ray mirrors to establish a wide tunable X-ray band-pass for experiments which need a wide band-pass beam, such as for Laue X-ray diffraction.

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