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Higher-order suppression in diffraction-grating monochromators using thin films

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Although a continuously tuneable source of photons is a very desirable feature of synchrotron radiation it has one main drawback: the contamination of the photon beam by higher-order diffracted light. Several elements have absorption edges which lie between 10 and 200 eV, a range prone to high second- and third-order content in XUV monochromators. They can, therefore, be used as transmission filters to reduce this higher-order content. This paper describes the use of thin filters to reduce the higher-order content in diffraction-grating monochromators. Their suppression efficiency, transmission and ageing have been characterized using photoelectron spectroscopy and compared with calculated values. The effect of oxide contamination on their performance has been assessed. Filters are now installed on eight XUV beamlines and have been in routine use for several years.

Keywords: monochromators; filters; higher-order suppression.

1. Introduction

Synchrotron radiation has proved to be an invaluable research tool in many areas of science. This is largely due to its tuneablility and intensity over an extremely wide photon energy range. However, the presence of higher orders in monochromated synchrotron beams is a universal problem. Double-crystal monochromators used above 2 keV can detune the second crystal slightly to combat this problem (Matsushita & Hashizume, 1983). For the diffraction-grating monochromators used below 2 keV no similar technique is available. Several approaches have been used at these lower photon energies: gas-absorption filtering (Suits *et al.*, 1995), selective reflectivity (Rehn, 1981; Miyake *et al.*, 1969; Peatman, 1990), diffraction-grating profile design (Neviere *et al.*, 1982). For existing beamlines where the design cannot readily be altered the simplest method involves the use of transmission filters.

2. Choice of filter

The choice of materials for transmission filters depends on the energy of the absorption edge, the mechanical strength (filters need to be thin but also pinhole free and self-supporting to some degree) and resistance to chemical alteration, such as oxidation. Having selected a suitable material, the filtering efficiency can then be calculated from the ratio of the transmission of first-order and higher order light.

2.1. *Calculation of filter efficiency*

The transmission, T, of a material at a particular photon energy E can be calculated from

$$T = \exp(-\mu_1 x),$$

where x is the thickness of the material and μ_1 (cm^{-1}) is the linear absorption coefficient and is dependent on the photon energy. Henke *et al.* (1982) have compiled sets of data for the mass absorption coefficient, μ (cm^2 g^{-1}), between 30 and 10 000 eV for 94 elements. The relation between μ and μ_1 is given by,

$$\mu = \rho\mu_1,$$

where ρ is the mass density of the absorber in c.g.s. units. Also tabulated are the scattering factors, f_1 and f_2. The relation between f_2 and μ is given by the Kramers–Kronig dispersion equation,

$$f_2 = \frac{E\mu A}{2r_0 hcN_0},$$

where A is the atomic weight, r_0 is the classical electron radius, h is Planck's constant, c is the velocity of light and N_0 is Avogadro's number. The scattering factors, f_1 and f_2, may also be calculated from the optical constants, n and k (see Palik, 1985). An estimate of how well the filters reduce second-order content of the beam at any photon energy, E, is given by the ratio of the transmission

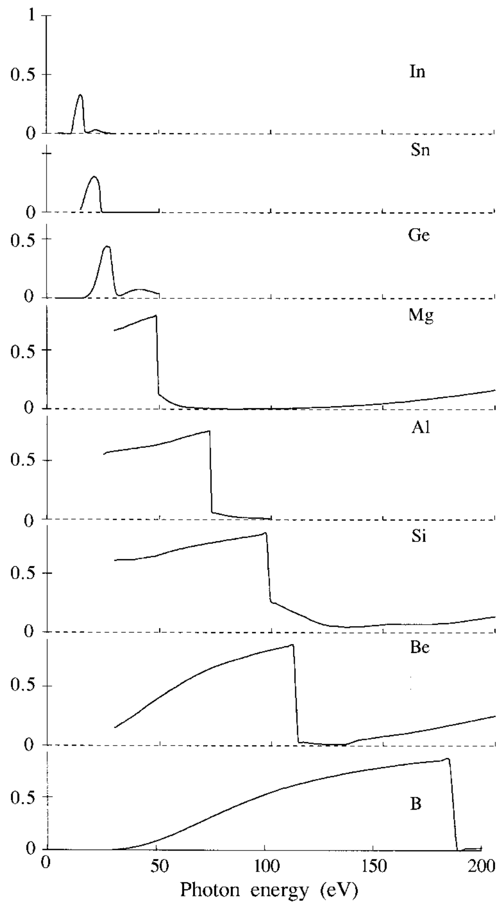

Figure 1
Calculated transmission of 0.16 μm In, 0.16 μm Sn, 0.16 μm Ge, 0.2 μm Mg, 0.15 μm Al, 0.12 μm Si, 0.15 μm Be and 0.2 μm B as a function of photon energy

for second- and first-order light and, herein, is called the rejection ratio, RR:

$$RR = T(2E)/T(E).$$

Fig. 1 shows the first-order transmission for boron, beryllium, silicon, aluminium, magnesium, germanium, indium and tin. For an efficient filter, the first-order transmission needs to be high while the rejection ratio should be low. Al, Mg and Si are the most generally useful elements and between them cover the photon energy range 27–99 eV.

2.2. *Description of filters*

A useable filter should transmit about 50% of the first-order light, requiring material thickness of 0.2 µm or less for the XUV region. Such thin foils need to be supported to survive the operating environment of a typical beamline. Luxel Corporation manufactures filters of the required thickness cemented to a high transparency nickel mesh (Powell *et al.*, 1990). As these can survive rocket launches, they were considered robust enough for use on SRS beamlines. The filters chosen for the initial tests on the surface-science beamlines were 0.12 µm Si, 0.15 µm Al, 0.2 µm Mg and 0.16 µm Sn. They were all 16 mm diameter mounted on standard 82% transmitting nickel mesh in circular frames. Except for the Mg filter, these were the standard sizes and thicknesses available. The Mg filter was over- and under-coated with 0.05 µm of Al to reduce the build up of magnesium oxide. The filters were mounted on linear drives and placed just after the exit slits of the monochromators, where the photon beam is relatively small, thus reducing the size (and cost) of the required filter.

3. Filter performance

3.1. *Transmission measurements*

As an XUV monochromator produces both the desired first-order and higher order light, the filter transmission cannot be measured by a simple transmission experiment.

The end stations on beamlines 1.2, 3.3, 6.1 and 6.2 are photoelectron spectroscopy stations, enabling the higher order content to be measured by setting the monochromator to an energy E and taking photoelectron energy spectra for $KE = 0$ to $2E$ with and without filters present. The first-order transmission, $T_1(E)$, is given by the ratio of the count rates (CR_1) for the peaks produced with a filtered and an unfiltered photon beam with photons of energy E,

$$T_1 = CR_1(\text{filtered})/CR_1(\text{unfiltered}).$$

Similarly, the second-order transmission, $T_2(2E)$, is given by the ratio of the filtered and unfiltered count rates, CR_2, for the peaks produced by photons with energy $2E$. The effect of electron-energy analyser transmission and cross section for production of photoelectrons (different for E and $2E$) cancel out. This is not the case when measuring the exact second-order content, and because of the difficulty in measuring these unknowns, the ratios given here for second-order content are uncorrected for any of these variables. The rejection ratio, RR, is then

$$RR = T_1/T_2.$$

On beamline 6.1, spectra were taken using a cleaned Cu(111) crystal. The electron energy analyser was a VSW 100 mm mean radius hemispherical analyser. Data from beamline 6.2 were taken using a VG ADES 400 spherical sector analyser and a cleaned Cu(111) crystal. Fig. 2 shows sample photoelectron spectra taken using the plane grating monochromator on beamline 6.1 at a photon energy of 60 eV. With an Al filter, the number of 120 eV photons was reduced to less than 1% of the incident beam while the number of 60 eV photons was only reduced by 50%.

3.2. *Performance of filters*

The results for Sn, Mg, Al and Si are shown in Fig. 3. As can be seen from the data, the filters can provide a significant rejection of second-order light while still transmitting between 25 and 60% of the first-order light. Sn shows useful performance over a narrow range, giving a rejection ratio less than 0.1 between

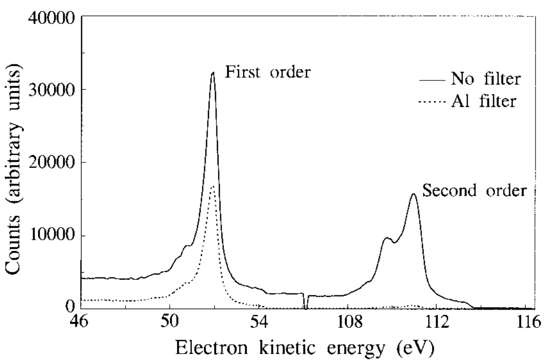

Figure 2
Photoelectron spectra taken with a 100 mm radius hemispherical analyser on beamline 6.1 at a photon energy of 60 eV. The second-order content at this energy is about 50%; this is reduced to 1% by an aluminium filter.

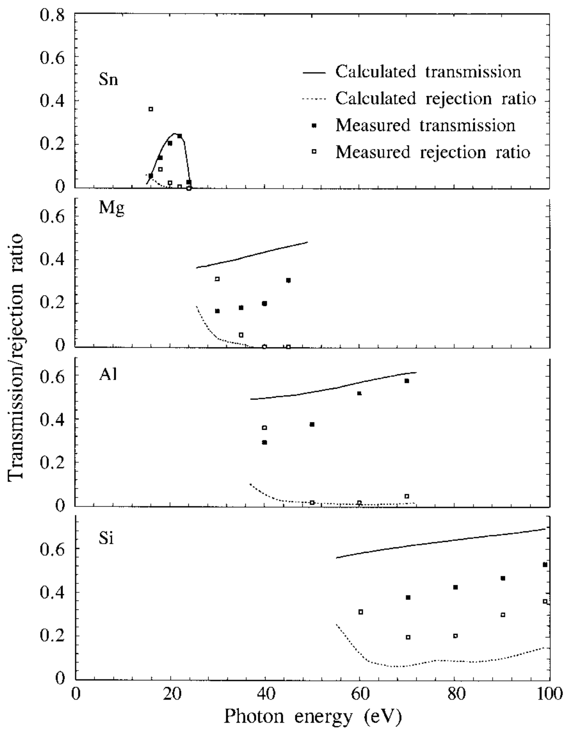

Figure 3
Results for Sn, Mg, Al and Si filters. Solid lines denote the calculated first-order transmission, dashed lines show the calculated rejection ratio for second-order transmission, solid squares show measured first-order transmission, open squares show measured second-order rejection ratios.

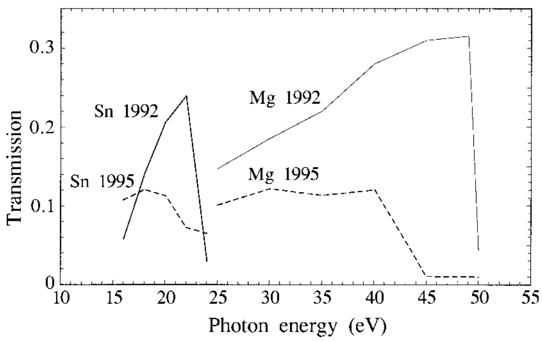

Figure 4
First-order transmission for Mg and Sn filters. The data were taken on beamline 6.2. The rejection ratios are similarly degraded.

17 and 23 eV. However, the transmission of first-order light is low, at only 20% at best. The Mg filter has a useful range from 25 to 49 eV, giving similar second-order rejection and first-order transmission. Al gives the best filter performance with a wide useful range of 36–72 eV. The rejection ratio is less than 0.1 while the first-order transmission increases from 30 to 58% towards the absorption edge. The Si filter tested has a transmission of 30–50% over the range 55–95 eV; however, the rejection ratio is disappointingly high at around 0.2–0.3.

3.3. *Oxide layers*

For Mg, Al and Si filters, the data show that the transmission is significantly below the calculated value; this is most likely owing to the presence of an oxide coating. From the difference between the calculated and measured values for the Al filter, and assuming there to be no other factor affecting the transmission, the thickness of the aluminium oxide layer has been estimated to be 0.008 μm. (This assumes the stoichiometry of the oxide layer to be Al_2O_3, and the density to be 3.8 g cm^{-3}.) The effect of this oxide coating is to degrade the filter performance significantly.

3.4. *Effect of ageing*

Several of the filters have been remeasured after 3 years use. These data show significant deterioration in transmission and reduction in filtering efficiency after a period of 3 years (Fig. 4). However, this performance monitoring has not been repeated sufficiently frequently to distinguish between gradual ageing resulting from residual gas and synchrotron radiation beam exposure, and deterioration as a result of a single event. Nevertheless, it gives an indication of the expected lifetime in a beamline at a pressure of 1×10^{-8} mbar. The section of beamline containing the filters is periodically baked at ~423 K for optic maintenance.

4. Reduction of higher-order content

The benefit of using these filters is shown in Fig. 5. This shows the second-order content for beamline 6.1 with and without thin metal filters.

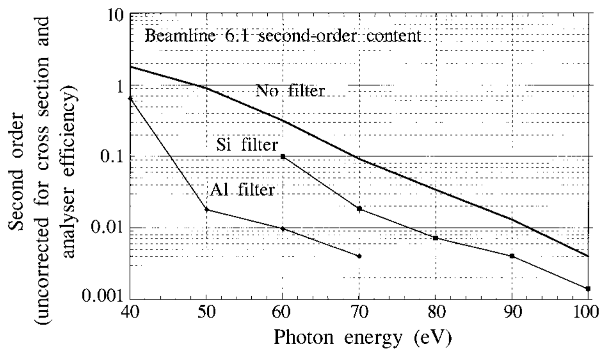

Figure 5
The second-order content of beamline 6.1 is plotted without filters, with an Si filter and with an Al filter. The data are uncorrected either for the variation of analyser transmission with electron kinetic energy or for the difference in photoionization cross section with the different photon energies.

This plot clearly shows how second-order content can be reduced to a negligible fraction of the first-order light over a wide photon-energy range. This has been of great benefit, particularly to the study of condensed matter systems where the photoelectron spectrum can be highly complex and the effect of higher orders can be to render the spectrum impossible to interpret.

5. Conclusions

Thin metal foils deposited on highly transparent nickel mesh and mounted on a retractable drive have been shown to reduce second-order content by a factor of 10 with respect to the first-order content over the photon-energy range 15 to >100 eV. They have been successfully used on several SRS beamlines for a number of years, proving to be robust enough to withstand use in vacuum (including bakeout at 423 K). Some filters have shown deterioration which is likely to be a result of oxidation after a period of 3 years.

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