The performance of transmission filters for EUV & soft x-ray astronomy

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ABSTRACT

Soft X-ray detectors for astronomical applications are often also sensitive to visible and UV photons. Filters of a new design have been developed, which could be used in future missions to suppress the flooding of X-ray detectors by low energy photons.

Three series of filters are discussed: Aluminium/Silicon multilayers, Niobium and Calcium/Boron multilayers. The filters are typically 100 to 200 nm thick and reject the IR/visible/UV from 10^{-4} to 10^{-6} . The transmission for soft X-rays is above 10% for wavelengths <30 nm, when combining the bandpasses of at least two filters. The optimum transmission band depends very much on the filter type.

The IR/visible/UV has been measured using standard laboratory spectro-photometers while the X-ray transmission characteristics have been determined using synchrotron radiation.

Keywords: X-ray filters, low energy X-rays, X-ray astronomy, optical opacity

1 INTRODUCTION

Novel thin filters, based on multilayers of optically opaque conductors (metals) and passivating materials on polyimide support structures, are being developed by the European Space Agency as VUV and soft X-ray filters. Good rejection of photons with wavelengths greater than 100 nm is required to prevent the flooding of many X-ray detectors with optical/UV photons.

The potential future space applications require that any such filters are the subject of an extensive environmental test program. Although only a fraction of a micron thick, the filters must sustain the conditions during launch and operation in space.

2 FILTER DESIGN AND PRODUCTION

2.1 Theoretical Modelling

A theoretical model of thin filters has been developed, which incorporates the most current material properties published in literature. The model considers both the absorption coefficients and interference effects.

An interface in a multilayer stack can be modelled using equation 1 for the forward and reverse electric field components (see figure 1 for notation)⁷.

Figure 1: Notation for forward and reverse electric fields at a boundary of two media.

$$\begin{bmatrix} E_o^+ \\ E_o^- \end{bmatrix} = \frac{1}{t_{of}} \begin{bmatrix} 1 & r_{of} \\ r_{of} & 1 \end{bmatrix} \begin{bmatrix} E_f^+ \\ E_f^- \end{bmatrix} = \hat{C}_{si} \begin{bmatrix} E_f^+ \\ E_f^- \end{bmatrix} \quad (1)$$

Where r_{of} and t_{of} are the Fresnel's complex amplitude reflection and transmission coefficients:

$$r_{of} = \frac{E_o^-}{E_o^+} = \frac{(n_o - n_f) + i(k_o - k_f)}{(n_o + n_f) + i(k_o + k_f)} \quad (2)$$

and

$$t_{of} = \frac{E_f^+}{E_o^+} = \frac{2(n_o + ik_o)}{(n_o + n_f) + i(k_o + k_f)}. \quad (3)$$

For a medium with an uniform index of refraction we have

$$\begin{bmatrix} E_o^+ \\ E_o^- \end{bmatrix} = \begin{bmatrix} e^{i\delta} & 0 \\ 0 & e^{-i\delta} \end{bmatrix} \begin{bmatrix} E_f^+ \\ E_f^- \end{bmatrix} = \hat{C}_{li} \begin{bmatrix} E_f^+ \\ E_f^- \end{bmatrix} \quad (4)$$

Where

$$\delta = \frac{2\pi(n + ik)d}{\lambda}, \quad (5)$$

and $n + ik$ is the complex index of refraction and d the layer thickness. The multilayer stack can be thus represented by the matrix

$$\hat{C}_r = \hat{C}_{so} \times \hat{C}_{l1} \times \hat{C}_{s1} \times \hat{C}_{l2} \times \hat{C}_{s2} \times \dots \times \hat{C}_{lf} \times \hat{C}_{sf} \quad (6)$$

The total transmission and reflection coefficients (for the electric field) can be calculated from equation 7.

$$\begin{bmatrix} 1 \\ r \end{bmatrix} = \hat{C}_r \begin{bmatrix} t \\ 0 \end{bmatrix} \quad (7)$$

The index of refraction data has been obtained from references⁸⁻¹⁰.

2.2 Filter Design

Using the model described above, many possible window designs were simulated. A filter was thereby allowed not only to be a single layer of a given material, but also to be a stack of different materials, so as to cope better with the many often conflicting requirements imposed on the filter.

Compatibility of the materials in contact with each other within the filter multilayers, corrosion aspects and feasibility of production were also considered in the design phase. For example protective layers are required when using metals like Calcium, which have very good soft X-ray photon transmission properties, but are quickly destroyed if exposed to air.

The design phase also considered mechanical stability of the filters. Filters must be handled during production, characterisation and use, potentially including launch and exposure to space. As a baseline hexagonal polyimide grids were selected as the most promising support structure. The pitch and mesh strength were optimized, trading off open fraction against mechanical strength. Two designs were chosen for production:

- 7% covered area, 400 μ m pitch
- 10% covered area, 400 μ m pitch

For the filter membranes the following designs have been selected:

- Si/Al/Si (protective layers of Si on Al optical absorber).
- Nb and polyimide/Nb (Nb shows an X-ray transmission window where Al absorbs X-rays; polyimide backing considered due to the brittleness of Nb)
- B/Ca/B/Ca/B (protective layers of B on Ca as an optical absorber; with the splitting of the Ca layers to decrease the number of pinholes)

For the transmission measurements presented here, a Si(30nm)/Al(150nm)/Si(30nm) multilayer was chosen as Al/Si filter. For the Nb filter a nominal thickness of 250nm was selected, and the Ca/B filter nominal layer thicknesses were B(25nm)/Ca(120nm)/B(15nm)/Ca(150nm)/B(25nm).

2.3 Filter Production

The fabrication process of the filters is based on the polyimide grid technology developed by Metorex International Oy. The process consists of the following steps:

- Spin casting a layer of polyamic acid precursor and curing it to form a 100 - 400 nm thick BPDA-PPD polyimide layer $[C_{22}H_{10}N_2O_4]_n$ on a silicon wafer.
- Spin casting and prebaking of a 20 μ m thick layer of photosensitive polyimide on the thin polyimide layer.
- UV exposure of the photosensitive layer through a grid-pattern mask.
- Chemical development to remove the unexposed parts of the grid pattern, followed by a high-temperature cure.
- Detachment of the grid-supported membrane from the substrate by chemical etching.
- Rinsing and drying the membrane
- Coating the membrane with sputtered, evaporated or spin-cast materials.
- Optionally etching some or all of the polyimide layer in O_2/Ar plasma, if e.g. pure metal/multilayer foils are to be fabricated.

As the surface purity of the polyimide membrane is essential for the quality of the subsequent sputtered/evaporated layers, it has been found necessary to add a second polyimide coating on the base membrane in order to add a clean coating over the possible residue originating from the etching/rinsing process.

For hard coating materials, like Nb, adjusting the internal stress to zero, or even slightly compressive, greatly increases the yield and reliability of the foils. The stress adjustment can be done by adjusting sputtering pressure and geometry.

Figure 2: Optical transmission of the Al/Si multilayer filter and the Nb filter, both with spectrometer background subtracted.

3 FILTER CHARACTERIZATION

3.1 IR/visible/UV Transmission

3.1.1 Experimental

Optical transmission measurements were performed using standard laboratory spectrometers. The Al and Nb filters were measured with a spectrometer having a continuous range of 400 to 1100 nm.

3.1.2 Results

The transmission was found to be sufficiently small for most applications in the case of the Al/Si and the Nb filters, see figure 2. The transmission of the Ca/B filter turned out to be above the goal of 10^{-6} , as shown in figure 3. This is assumed to be due to oxidation of the Ca layer during production, and improvements in the fabrication are being examined.

In all cases the general shape of the calculated transmission function corresponds reasonably well with the measurements. In the case of the Al-filter, the measured transmission level is higher than expected by two orders of magnitude, probably due to oxidation of the Al-layer during the production steps. The measured optical transmission level corresponds to that of 105 nm thick layer of pure Al. For the Nb-filter, the transmission level agrees well with calculations, indicating little or no oxidation. In the case of the Ca-filter the measured optical transmission is two orders of magnitude higher than calculated for the region 350 to 600 nm, where published

Figure 3: Optical transmission of the Ca/B multilayer filter, with spectrometer background subtracted. The Ca filter was measured using two adjacent ranges on the spectrometer, with the changeover occurring at 1000 nm. Due to a small inter-calibration error a jump is therefore visible in the transmission data gathered for the Ca filter.

optical transmission data is available. Again, oxidation during the manufacturing is assumed to be the cause of the increased transmission.

3.2 X-ray Transmission

3.2.1 Experimental

The filters have been measured at the PTB reflectometry facility at BESSY using the reflectometer⁵ behind a plane grating monochromator.⁶ Two geometries were used during the measurements. The first consisted of 'wide beam' illumination, using an X-ray pencil beam of $1.3 \times 1.5 \text{ mm}^2$. The beam size was chosen such to be small compared to the expected filter non-uniformities, but at the same time large enough to average over the support grid structure. The second geometry consisted of a 'fine beam', which was chosen to be small enough to fit into one support grid hexagon, avoiding interference with the grid. Due to the much higher flux in the wide beam configuration, which uses the full acceptance of the monochromator, the measurement statistics are better in the energy regions of low transmission. The influence of the polyimide support structure can be verified by comparison with the fine beam measurements.

For small uncertainties in the transmission measurements a high spectral pureness of the radiation is required. Appropriate beamline filters can be used to reduce the amount of false light. This in turn requires, that the total available energy range is split into separated regions using different suppression filters. An overview of these energy regions and filters as well as the remaining content of false light achieved is given in table 1. Most of

Table 1: X-ray transmission measurements: energy ranges and false light contributions.

<i>Energy Range /eV</i>	<i>Suppression Filter</i>	<i>Remaining Content of False Light</i>
40–70	0.75 μm Al	$\leq 1\%$
60–110	0.25 μm Be	$\leq 1\%$
110–140	no filter	$\leq 1\%$
140–250	no filter	1–5%
250–450	1 μm Ti	$\leq 1\%$
450–700	0.5 μm Fe	$\leq 1\%$
700–900	0.5 μm Cu	$\leq 1\%$
900–1500	7 μm Be (0.75 μm Al)	$\leq 1\%$
1500–1950	30 μm Kapton	$\leq 5\%$

the regions are contaminated with false light by less than 1%, hence we estimate the total uncertainty for the transmission to be about 1%. Transmission measurements above 1500 eV, using filters with different suppression powers, showed that these fairly transparent window materials seem to suffer less from straylight. Therefore the effective uncertainty for energies above 1500eV is far lower than suggested from the false light content listed in table 1.

A further contribution to the measurement error is the effect of harmonics. Depending on the filter transmission characteristics, the second order monochromator transmission is sometimes much less absorbed by the filter than the first order photons. Since a non-energy dispersive detector, a Silicon diode, is used in the experiment, the harmonics contribute to a different extent to the sample and reference measurements. This error is in principle recoverable, to a large extent, provided the beamline characteristics are known well enough. The data presented here has not been corrected for this effect. For the current filters, this error is most prominent at the lowest energies, where the filter transmission is also low. The relative error is worst for the Ca-filter and reaches $\approx 10\%$ for $\lambda \geq 10\text{nm}$. However in terms of absolute transmission the error amounts to $\leq 1\%$.

3.2.2 Results

The measured X-ray transmissions for the Al, Nb and Ca filters are shown in figures 4, 5 and 6. Also plotted are the calculated transmissions, determined using the model described above. In all cases the nominal filter layer thicknesses have been used, and no additional contaminations, oxide layers or polyimide residuals have been assumed.

For the Nb filter the deviations between prediction and measurement are rather small. The Nb-filter has an effective bandpass from 6 - 16 nm and additionally transmits below 1.7 nm, using 10% as the threshold level. Some Nb oxides are present in the sample, as evident from the oxygen absorption edge.

The Al/Si filter seems to contain a residual polyimide layer, indicated by a much better fit, if a 40 nm polyimide layer is added in the model using the nominal layer thicknesses. The Al and Nb filters complement each other very well, with the Al filter transmitting in the band 16 to 31 nm and below 6 nm.

The Ca filter model deviates most from the measured response, but we must however be aware that the Ca filter has the broadest measured transmission window: it transmits below 14 nm. Note, that there is a considerable difference in the optical constants reported in the literature for Ca, which was used in the model,¹¹ and others,¹² at wavelengths above 12 nm. A reasonable fit was obtained by using Ca-data from reference,¹² assuming an

Figure 4: Measured X-ray transmission of Al/Si multilayer filter, solid curve. The dotted curve is the calculated transmission, using a nominal filter design described in the text.

Figure 5: Measured X-ray transmission of Nb filter, solid curve. The dotted curve is the calculated transmission, using a nominal filter design described in the text.

Figure 6: Measured X-ray transmission of B/Ca multilayer filter, solid curve. The dotted curve is the calculated transmission, using a nominal filter design described in the text.

additional polyimide layer and uniform oxygen layer, but some deviations still persist above 12 nm wavelength. This will require further thin film material diagnostics coupled to further refinements of the model. The oxygen residues are caused by gettering during evaporation and by oxidation through the faults in the protective layers.

The polyimide residues can be caused by underestimations of the original polyimide layer thickness due to interaction between the two successively spin-cast layers, and by a partial conversion of the carbon in the polyimide into its graphitic form during the Ar/O₂ plasma etching, leading to diminished etching speed for the last tens of nanometers.

3.3 TEM Analysis

3.3.1 Sample Preparation and Imaging

Transmission Electron Microscopy (TEM) cross-sections have been made of one Si/Al/Si filter and one Nb filter. Sample preparation was carried out at MRIC research and the images were obtained with the Manchester University TEM. To prepare the samples, 6 mm squares were cut out from the centre of the windows, which were sandwiched under pressure between two slabs of silicon wafer using epoxy resin, and cured to a high hardness at a temperature of 120° C. The sample from the Al/Si multilayer was finished with a low voltage (2kV) ion-beam milling step once a hole in the sample was obtained, to reduce ion damage to the electron transparent part of the sample surrounding the hole. Sample preparation for the Nb filter was hampered by the large difference in hardness between the Nb in the filter and the epoxy and silicon used for sample support.

Cross-sectional images of the two window samples have been taken at magnifications between 10000 and 350000

Figure 7: TEM image of the Al/Si multilayer filter cross-section.

times. In addition, selected-area electron-diffraction (SAED) patterns have been produced for both samples. Two representative cross-sectional images (with original magnifications of respectively 85000 and 42000 times) are shown in figures 7 and 8 for the Al/Si multilayer and the Nb monolayer, respectively. In both pictures the grey background is the epoxy and the density gradient across the image reflects the thickness gradient towards the hole in the sample, which is located close to the filter cross-section. In figure 8, the Nb sample depth was selected such as to show the actual thickness and surface quality. Other TEM images were taken to determine the grain structure.

3.3.2 Analysis

The TEM images confirm that the actual thicknesses of the layers are close to specification. The true dimensions of the Al/Si multilayer are: 27 nm Si, 98 nm Al and ≈ 27 nm of Si again, which is in close accordance to the nominal widths of resp. 30, 100 and 30 nm. Both Si layers were found to be amorphous, while the Al layer is poly-crystalline. The Nb monolayer has a thickness of 238 nm, again very close to the nominal thickness of 250 nm. Images of the electron transparent region of the Nb layer reveal that its structure is poly-crystalline. Note that the nominal Al layer thickness of the TEM-sample is 100 nm, while the filters measured for X-ray and optical transmissions have 150 nm Al layers.

In both windows the surface that was produced first by deposition onto the polyimide substrate is smoother than the subsequent surfaces and interfaces. The peak-to-valley fluctuations in the height on the rough side of the Al/Si multilayer are of the order of 40 nm. On the smooth side the fluctuations are small (few nm peak to valley) and seem mainly due to the fact that the filter is bent somewhat between the beams of the polyimide strongback. The Nb film seems even smoother: on the rough side the peak-to-valley undulations are estimated to be 5 nm, while on the smooth side the roughness is comparable to that of the first Si layer in the other film. It is likely, that the Nb surfaces are covered by thin (few nm) layers of oxides, possibly also traces of nitrides. This is also supported by other TEM images and the X-ray transmission measurements.

The grains in the Al are larger than those in the Nb. The grain sizes in the Al seem to vary between 40 nm and 200 nm, with a 'typical' value of 90 nm. Many grains extend from one Si layer to the other. Occasionally, an

Figure 8: TEM image of Nb filter cross-section.

Al grain sticks through the covering Si layer. The estimated surface density of such 'mountains' is about 4 per square micron. The grains in the Nb are more columnar with apparent widths in the range from 10 to 40 nm.

The SAED patterns do not reveal any preferred orientations for the Al or the Nb filter layers. Some reflections in the pattern cannot be ascribed to either Al or Si, indicating that some contamination is present in the layer. The same is true for the Nb layer, although here the unidentified reflections are fairly weak.

3.4 Environmental Tests

The Nb and Al/Si filters have been radiation, vibration, pressure and oxygen corrosion tested and in addition they have also undergone thermal cycling in vacuum. In some of the filters an increase in the number and size of pinholes was observed, but this was clearly connected to the localized process residue on the polymer membrane on which the metal membrane was deposited. The occurrence of pinholes was most abundant in the radiation tests, especially in the Nb filters without a polyimide membrane. These pinholes increased in size or formed fractures during the vibration test, which can be explained by the brittleness of the Nb foil as compared for instance to Al. The pinholes also tended to grow in the atomic oxygen corrosion test, especially when there was polyimide left in the membrane structure. This is not an unexpected result, since the polyimide is etched off in lateral direction around the pinholes, thus weakening the edges of the pinhole.

Al/Si filters as well as Nb filters with an unetched polyimide layer survived relatively well in the tests. Generally it can be said that the filters survived the environmental tests provided their surface is free of defects.

Note that the pressure tolerance of the filters was between 20 and 30 mbar, depending on the support grid type.

4 CONCLUSIONS

With the filters described herein transmission of more than 10% of the incident X-ray photons is possible for wavelengths shorter than ≈ 30 nm, if at least two filters are combined, e.g. mounted on a filter wheel. The filters at the same time reject optical photons with an efficiency of 10^6 or better, with the exception of the Ca based filter.

Such filters could be particularly important for the next generation of detectors which will be used in future space missions, like STJs, advanced soft X-ray CCDs and bolometers. These filters will not only prevent flooding of detectors by optical light photons, but also reduce the noise and thermal load on the coolers and increase the overall stability of the systems.

Improvements in uniformity and size, reduction of pinhole size and numbers, will eventually produce windows with an increased pressure tolerance and leak tightness.

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