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Robust infrared filters for X-ray spectroscopy ☆

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Abstract

One challenge to using cryogenic detectors for X-ray spectroscopy on a scanning electron microscope is the implementation of infrared blocking filters. In order to achieve high X-ray transmission, these filters can be as thin as 250 nm and consequently are extremely fragile. To avoid breaking the filters, the cryostat must be evacuated slowly and by a trained operator. In this presentation, we describe the filter system currently used at NIST. In addition, we describe recent efforts to build a more robust and easy-to-use filter system. We present initial efforts to strengthen conventional aluminum–parylene filters with micromachined silicon grids that only reduce X-ray transmission by 2%. We also describe an automated pump-out system based on a commercial mass-flow controller.
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1. Introduction

The integration of a high-energy-resolution X-ray spectrometer with a high-spatial-resolution scanning electron microscope (SEM) yields a powerful microanalysis instrument, essential to technology-intensive industries such as the semiconductor industry. Features of interest are continually shrinking, requiring new generations of instruments, such as the microcalorimeter energy-dispersive X-ray spectrometer developed at NIST [1]. One challenge of using this cryogenic detector is the implementation of infrared blocking filters.

The NIST microcalorimeter operates at temperatures near 100 mK. Therefore, it is necessary to shield the detector from infrared radiation, while still allowing for the transmission of X-rays. The present system has three sets of filters to accomplish this task, a commercially available 300 K filter, two 4 K filters and a 100 mK filter. To achieve high X-ray transmission, the 4 K and 100 mK filters must be very thin (150 nm aluminum on 100 nm parylene-N), which makes them extremely fragile and sensitive to pressure gradients.

A simple schematic of the cryostat is shown in Fig. 1. The 4 and 300 K vacuum spaces are separated by a serpentine pipe designed to let air pass but not radiation. The pipe creates an impedance between the chambers, which can cause a pressure difference during pumpout and venting. If the cryostat pressure is changed too quickly, the filters will break.

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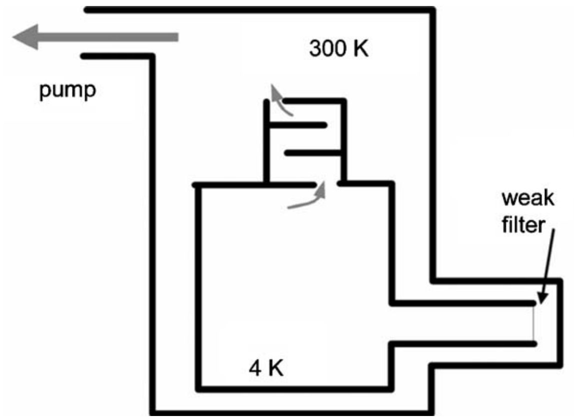

Fig. 1. Schematic of cryostat showing 300 and 4 K vacuum chambers connected by serpentine pipe.

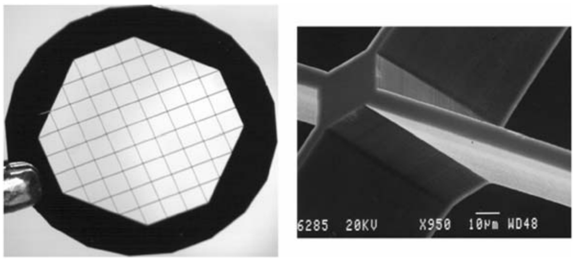

Fig. 2. Left: Picture of micromachined grid—diameter 1.27 cm (0.5 in); Right: SEM image showing intersection of two grid bars.

2. Micromachined grids

Micromachined silicon grids were used to strengthen aluminum–parylene filters (Fig. 2). The grids were fabricated using the Bosch process [2] in a commercial deep reactive ion etcher. The grid cells are 1000 μm on a side, the bars are 10 μm thick and ~100 μm deep. The grids reduce X-ray transmission by 2%.

3. Filter testing results

Filters were mounted on a chamber whose pressure could be increased above atmospheric pressure. Filters were tested until failure and recorded on video. Failure pressures were tested for thin (0.1 μm parylene) and thick (1.5 μm) filters, with and without supporting grids.

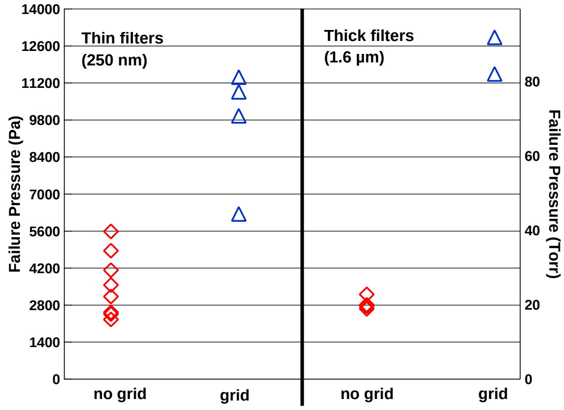

Fig. 3. Filter results showing increased strength when supported by grid.

The results of the filter tests in Fig. 3 show that grid-reinforced filters can withstand 3 to 5 times more pressure than freely-suspended filters. Grids increase the strength of both thin and thick filters. A surprising result is that unsupported thick filters are not necessarily more robust than unsupported thin filters.

The failure modes of the grid-supported filters contain information that can be used to improve future grids. Two failure modes are possible: 'grid failure' and 'filter failure'. Grid failure occurs when the whole grid fails (usually at the edges) and filter failure occurs when the parylene in a single or small group of cells fails. Filter failure suggests that the grid is stronger than the filter, while grid failure suggests the opposite. Of the four data points for thin filters with grids, the highest three were filter failures. The weakest point was a grid failure. Both the thick filters with grids were filter failures. With the exception of the single grid failure, these results suggest that the current grid design is stronger than the parylene in the individual cells. Hence, the filter strength can be further increased by reducing the grid cell size while leaving the bar thickness unchanged.

4. Optimizing pumpout

In order to protect the filters in the current system, the cryostat must be slowly evacuated by a trained operator. To save time and manpower, the optimal pumpout speed has been determined. The pressures of the 4 and 300 K chambers are P_1 and P_2, respectively (Fig. 1). The conductance between the chambers is determined by the serpentine pipe, $C_{1-2} = \alpha(P_1 + P_2)$, where α is a constant determined by geometry. The mass flow equations are

$$V_1\dot{P}_1 = -\alpha(P_1 + P_2)(P_1 - P_2) \quad (1a)$$

$$V_2\dot{P}_2 = +\alpha(P_1 + P_2)(P_1 - P_2) - F \quad (1b)$$

where F is proportional to the mass flow out of the cryostat. Set $P_1 - P_2 = P_{\mathrm{fail}}$ [3], where P_{fail} is a constant determined by the strength of the filter. This approximation yields the shortest pumpout time, but is unphysical at the beginning and end of a pumpout. With this assumption, Eq. (1a) and (1b) are satisfied by the following expressions:

$$P_1 = P_0 e^{-t/\tau} + P_{\mathrm{fail}} \quad (2a)$$

$$P_2 = P_0 e^{-t/\tau} \quad (2b)$$

$$F = \frac{1}{\tau}(V_1 + V_2)P_2 \quad (2c)$$

where $1/\tau = 2\alpha P_{\mathrm{fail}}/V_1$ and P_0 is the initial pressure. The form of Eq. (2c) is chosen for practical value. The mass flow from the cryostat has been expressed in terms of P_2, an easily measurable pressure, and is independent of pump speed or fore pressure. An important result is that pumpout time is inversely proportional to filter strength. Therefore, doubling the filter strength cuts the pumpout time in half.

A commercial mass-flow controller has been added to the pumping system to automate pumpouts [4]. A computer monitors P_2 and sends a signal to the flow controller to follow the flow curve defined by Eq. (2c).

5. Conclusion

We have shown that micromachined silicon grids significantly increase the strength of conventional aluminum–parylene filters. We also described a pumpout theory to minimize pumpout times and used a mass-flow controller to automate the process.

Future work will involve a redesign of the micromachined grids, increasing the number of support bars.

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