



# CRYO-DIFFRACTION CHAMBER WITH LARGE DIFFERENTIALLY PUMPED KAPTON WINDOW

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## Introduction

The FEMTO group at the microXAS beamline at the Swiss Light Source (SLS) operates a femtosecond slicing source since 2006 to perform laser/THz-pump and X-ray probe diffraction experiments with time resolution 200 fs.

The required accuracy of the sample for position is 1 micron and for rotation 0.001 degree at a temperature as low as 30 K. The X-ray detectors are operated in air which requires vacuum compatible laser- and X-ray windows of different sizes.

The present chamber consists of the following components:

- Cylindrical vacuum chamber (volume 0.02 m<sup>3</sup>) with turbo pump (700 l/s, Hipace, Pfeiffer GmbH) and LN<sub>2</sub>-cold trap (VAb GmbH)
- 6-axes high-load hexapod (M850K190, PI GmbH) sample manipulator and vacuum-feedthrough sample servo-rotation (ALMA GmbH)
- Closed-cycle He-cryocooler (CTI-CRYOGENICS Inc.)
- Cryo-sample holder with manual adjustment for alignment with thermocouple and heater
- Large cylindrical-section and differentially pumped double-wall exit window (2 x 125 micron thick Kapton) coated with aluminum (3-10 micron). Size: 450 cm<sup>2</sup> (inner window) and 550 cm<sup>2</sup> (outer window)

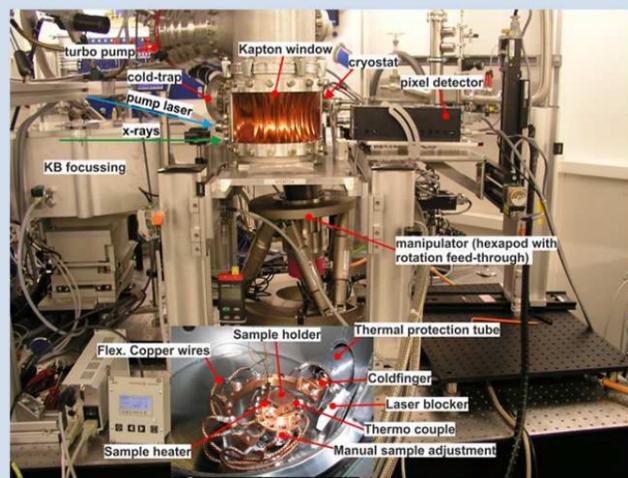


Fig. 1: Femto diffraction setup at the microXAS beamline. Inset: sample assembly



Fig. 2: Cold-trap inside the vacuum chamber



Fig. 3: Nitrogen connectors of the cold trap

## Vacuum

The large Kapton window compared to a welded Beryllium window has the advantage that it can conveniently be opened to allow access for laser pre-alignment. However Kapton is not completely vacuum tight. To reach low temperatures, the vacuum has to be as low as possible.

For a single-wall Kapton window a vacuum of  $2 \times 10^{-6}$  mbar is reached with the turbo pump and  $2 \times 10^{-7}$  mbar when the Nitrogen cold trap is used in addition. When the sample is cooled to 30 K, the vacuum is  $5 \times 10^{-8}$  mbar. This condition can be kept for 7 days. Nonetheless, a tiny ice layer on the sample can gradually grow and block the laser beam. In this case the sample has to be heated up and cooled down again.

## Burst tests

When working with a large vacuum window, for safety reasons it is essential to determine the burst safety margin. To perform burst tests the evacuated chamber has been mounted in a pressure tank to apply overpressure. On average the burst occurred at an absolute pressure of 3.8 bar (i.e. safety margin 2-3).

Upon baking the chamber where the Kapton foil had a temperature of 58 °C, the burst occurred at an absolute pressure of 2.2 bar. This means although the tensile strength of Kapton critically depends on temperature, the chamber can moderately be baked to improve the vacuum without risking a burst of the large Kapton window.



Fig. 4: Diffraction chamber prepared for burst test



Fig. 5: Diffraction chamber after burst test

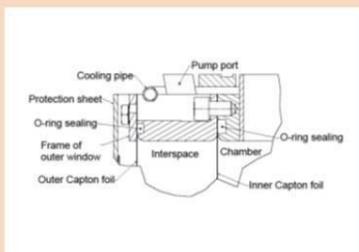


Fig. 6: Cross section of the double Kapton wall

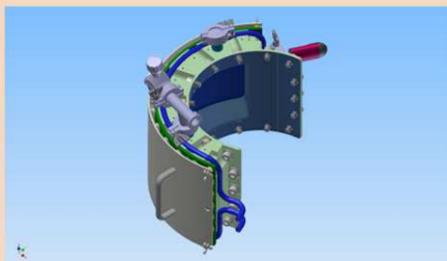


Fig. 7: CAD model of the double Kapton wall



Fig. 8: Frame with inner window

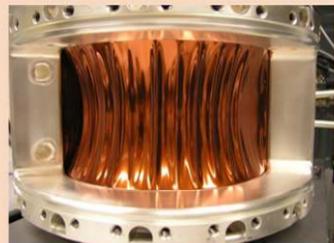


Fig. 9: Inner window under vacuum

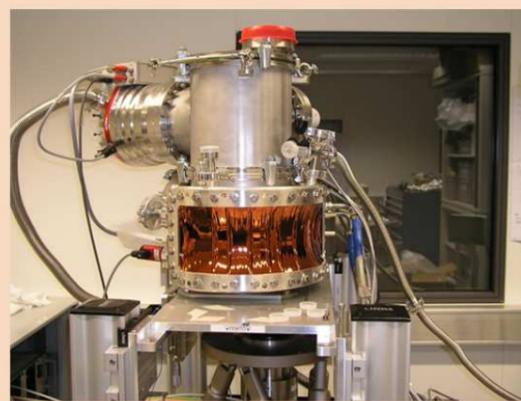


Fig. 10: Double window under vacuum

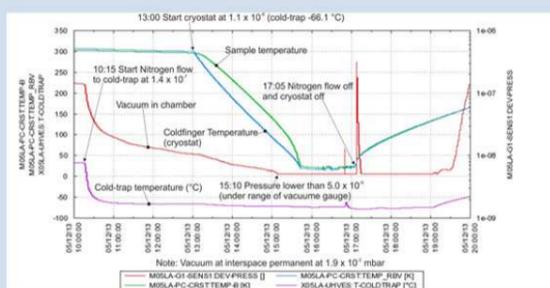
## Double-wall Kapton window

To reach ultra-high vacuum (UHV) after bakeout we developed a differentially pumped double-wall Kapton-window which is water-cooled at 21 °C. With turbo pumping a vacuum in the 10<sup>-8</sup> range is reached after 24 hours. Because now the inner Kapton foil is not under tension and the outer one is water-cooled, bakeout becomes more feasible.

We applied bakeout at 150 °C for 72 hours. A vacuum of  $5 \times 10^{-9}$  mbar has been reached after cool down of the sample to 30 K and in the upper 10<sup>-10</sup> mbar range when in addition LN<sub>2</sub>-cold trap pumping has been applied.

## Conclusions

To reach UHV vacuum conditions in a scattering chamber with a large window, a bakeable, differentially pumped double-wall Kapton window is an alternative to a much more expensive Beryllium window which must be welded and therefore is less flexible. Such a chamber allows to operate large 2D pixel detectors in air or in a helium environment whereas the sample is cooled to low temperatures.



Graph 1: Performance after bake out with cold-trap and cryostat cooling

Pos.	Description	Vac. [mbar]	Waiting time	Notes
1	Only inner window, cryo off, no bake out, cold-trap off	4.3x10 <sup>-5</sup>	10 min after turbo pump on	
2a	Both windows, cryo off, no bake out, cold-trap off	2.3x10 <sup>-6</sup>	10 min after turbo pump on	Vacuum interspace: 4x10 <sup>-2</sup>
2b	Both windows, cryo off, no bake out, cold-trap off	5.3x10 <sup>-8</sup>	4.5 days after turbo pump on	Vacuum interspace: 4x10 <sup>-2</sup>
3	Both windows, cryo off, <b>baked out</b> , cold-trap off	1.2x10 <sup>-8</sup>	1 h on room temp. after bake out	
4	Both windows, <b>cryostat 20K</b> , baked out, cold-trap off	5x10 <sup>-9</sup>	9 h after cryo on	
5	Both windows, <b>cryostat 70K</b> , baked out, <b>cold-trap on</b>	< 5x10 <sup>-9</sup>	2 h after cryo on	Under range Vacuum gauge. Tendency still decreasing.

Table 1: Vacuum pressures (Note: Changes are marked)