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ABSTRACT

The X-Ray Integral Field Unit (X-IFU) detector on-board ATHENA is an array of TES micro-calorimeters that will operate at ~50 mK. In the current investigated design, five thermal filters (TF) will be mounted on the cryostat shields to attenuate IR radiative load and avoid energy resolution degradation due to photon shot noise. Each filter consists of a thin polyimide film (~50 nm thick) coated with aluminum (~30 nm thick).

Since the TF operate at different temperatures in the range 0.05-300 K, it is relevant to study how temperature affects their mechanical/optical performances (e.g. near edge absorption fine structures of the atomic elements in the filter material). Such results are crucial for the proper design of the filters as well as to establish the calibration program operating temperatures.

We report the preliminary results of visual inspections performed on test filters of polyimide/Al at different pressure and temperature conditions, IR transmission measurements (1-15 μm) performed in the temperature range 10-300 K, and X-ray Absorption Spectroscopy measurements (175-1650 eV) performed in the temperature range 130-300 K

Keywords: ATHENA mission, thermal filters, XANES

1. INTRODUCTION

ATHENA is a large astrophysical observatory[1] approved by the European Space Agency to address the science theme "Hot and Energetic Universe" (L2 mission with launch scheduled in 2028)[2]. One of the two instruments of ATHENA is the X-ray Integral Field Unit (X-IFU), an array of Transition Edge Sensor (TES) micro-calorimeters with spectral, imaging, and timing capabilities in the energy range 0.2÷12 keV [3][4].

The X-IFU will be cooled by a quite complex multi-stage liquid free cryostat down to nearly 50 mK [5]. In order to allow the focused X-ray beam to reach the detector, a clear path is opened in the cryostat thermal and mechanical shields. Thin windows, transparent to X-rays, will be mounted on such openings to minimize IR radiation heat-load onto the cold detector array, and to keep the photon shot noise well below the intrinsic energy resolution of the TES micro-calorimeters (ΔE_{FWHM} < 2.5 eV @ 6 keV) [6][7]. A few cryostat shields will operate as Faraday cages to protect the TES micro-calorimeters and SQUID electronics from Radio Frequency (RF) Electro-Magnetic Interferences (EMI) in the spacecraft environment (e.g. telemetry) [8]. The thermal filters mounted on such shields will, therefore, have to provide an attenuation of RF electromagnetic interferences. Since the aluminum layer is too thin with respect to RF electromagnetic field penetration depth, the use of fine metal meshes is foreseen to provide both RF attenuation and mechanical support.

In the current investigated design, five thermal filters will be mounted on the cryostat shields, each one consisting of a thin membrane of polyimide (approx. 50 nm) coated with aluminum (approx. 30 nm) [7]. Despite the

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small thickness and the use of low Z materials, the thermal filters become quite opaque at E < 1.0 keV, and they essentially define the low energy response of the X-IFU.

Since the main characteristic of the X-IFU is the high energy resolution, a detailed calibration program of the thermal filters will need to be performed with high energy resolution X-ray transmission measurements to properly characterize the low energy instrument response, and in particular near the absorption edges of the atomic elements present in the filter material (Al, O, N, C). Considering that the thermal filters operate at different temperatures ranging between 50 mK and 300 K, it is crucial to study whether temperature affects the optical performance of the filters (e.g. UV/VIS/IR transmission, near edge X-ray absorption fine structures of the atomic elements in the filter material). The results of this investigation are also relevant to define the filter calibration program.

The polyimide is known to be hygroscopic [9][10]. At room temperature in air polyimide films adsorb water and develop wrinkles which disappear when the filter is kept in high vacuum for a few hours. Considering that filters will operate in a ultra-high vacuum environment, wrinkles are not expected at room temperature operation in Space. On the other hand, as soon as the filters are cooled down, as discussed in section 3, wrinkles appear which might affect the performances of the filter.

In the following sections we will present preliminary results performed on small Polyimide/Al filter prototype samples manufactured by LUXEL corp. to identify the role of operational temperature on the performances of the X-IFU TF. Section 2 lists the procured samples, section 3 describes the visual inspection at different temperature and pressure, section 4 describes the IR transmission measurements performed at INAF-OAPA and UNIPA, section 5 summarizes the X-Ray Transmission measurements performed at ELETTRA synchrotron in Trieste.

2. FILTER SAMPLES

A first set of prototype filter samples (table 1) has been procured in November 2015 from LUXEL corp. to perform preliminary performance tests (mainly U/Vis/IR transmission measurements vs. Temperature, X-ray Absorpion spectroscopy of the absorption edges fine structures vs. Temperature, X-ray Photo-electron Spectroscopy of surface aluminum, RF attenuation measurements).

Table 1. List of procured filter prototype samples for preliminary performance investigations.

Item	Id. Numbers	Description
1	TF111-2788 TF111-2768	45 nm LUXFilm ® Polyimide/30 nm Al film, meshless, mounted on TF111 anodized aluminum frame (I.D. = 16 mm).
2	TF111-2761	45 nm LUXFilm ® Polyimide (same as lot 1) without aluminum coating, meshless, mounted on TF111 anodized aluminum frame (I.D. = 16 mm).
3	TF111-2782 TF111-2677	150 nm LUXFilm ® Polyimide/30 nm Al film, meshless, mounted on TF111 anodized aluminum frame (I.D. = 16 mm).
4	TF111-2764	150 nm LUXFilm ® Polyimide (same lot as Item 3) without aluminum coating, meshless, mounted on TF111 anodized aluminum frame (I.D. = 16 mm).
5	TF111-2620	45 nm LUXFilm ® Polyimide/25 nm Al film on honeycomb Al grid: 1.4 μm thick, 8 μm bar width, 200 μm pitch, mounted on TF111 anodized aluminum frame (I.D. = 16 mm).
6	TF111-2648	25 nm Al film on honeycomb Al grid: 1.4 μm thick, 8 μm bar width, 200 μm pitch, mounted on TF111 anodized aluminum frame (I.D. = 16 mm).

3. COOLING DOWN IN ULTRA-HIGH VACUUM

The test filters are corrugated at ambient temperature and pressure, because of the water absorbed by the polyimide[9]. After a few hours in Ultra High Vacuum (UHV), at room temperature, the filters loose the water content and stretch. To evaluate the height of the wrinkles in air at room temperature, we measured the reflection angle of a laser beam coming from an angle of 45° with respect to the filter plane (Figure 1). The laser footprint on the filter had a diameter of about 2 mm and the filter was moved horizontally and rotated so that the beam was perpendicular to the axis of a chosen wrinkle. The width of the larger wrinkles is about 2 mm from valley to valley and the beam was aimed to illuminate entirely the fold, so the reflected light formed a vertical line on a projection screen, the length of which depends on the minimum and maximum reflection angles. We have repeated the test on different folds on the filter and, on average, we found a total angle difference of about 10° between the minimum and maximum fold slope. This value translates into a height from peak to valley of nearly 90 μm approximating the wrinkle with an isosceles triangle and 60 μm approximating it with a sinusoid profile.

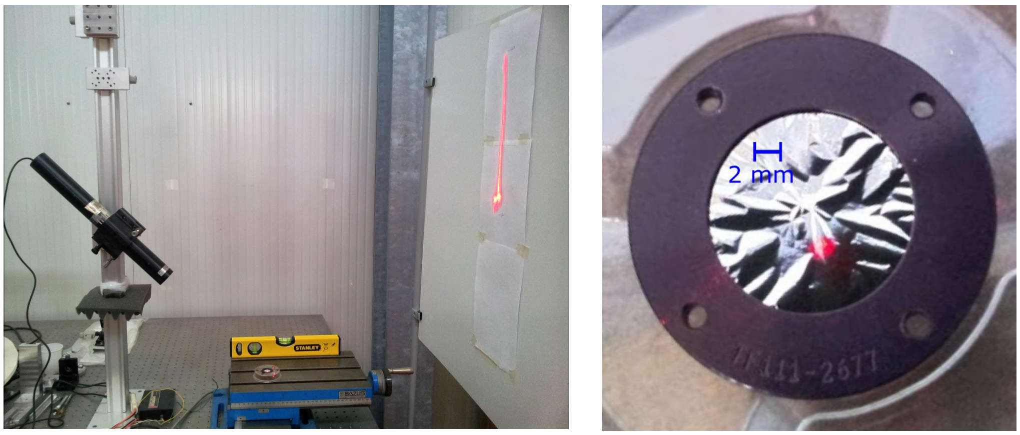

Figure 1. Experimental apparatus used to measure the slope of the wrinkles by laser beam reflection. Left: experimental set-up. Right: The laser beam spot diameter on the filter is about 2 mm. Test filter shown: TF111-2677.

When the polyimide films are cooled down, while kept in high vacuum, they corrugate again. Both bare polyimide and polyimide/aluminum filters are subject to the same kind of corrugation, while the sample filter filter TF111-2620 consisting of 45 nm Polyimide/25 nm Al with a honeycomb Al lithographic mesh (1.4 μm thick, 8 μm bar width, 200 μm pitch) do not present large scale size wrinkles because of the relative rigidity of the mesh; they rather show wider deformations involving also the mesh and possibly small-scale deformations confined within the mesh cells.

To examine the corrugation of the test filters at low temperatures we mounted them inside the UHV chamber of the Light Irradiation Facility for Exochemistry (LIFE) at INAF-OAPA [11]. The chamber, pumped by cryogenic and turbo-molecular pumps, reaches a ultra-high and clean vacuum, and is equipped with a cryostat to cool samples down to ~10 K. The base pressure of the chamber we reached before starting to cool the filters was 2.5 10^{-9} mbar. A cooling-down ramp was set with a speed of 3 K/min. At about 270 K the filters started to corrugate. Wrinkles became more and more pronounced continuing the cooling (Figure 2).

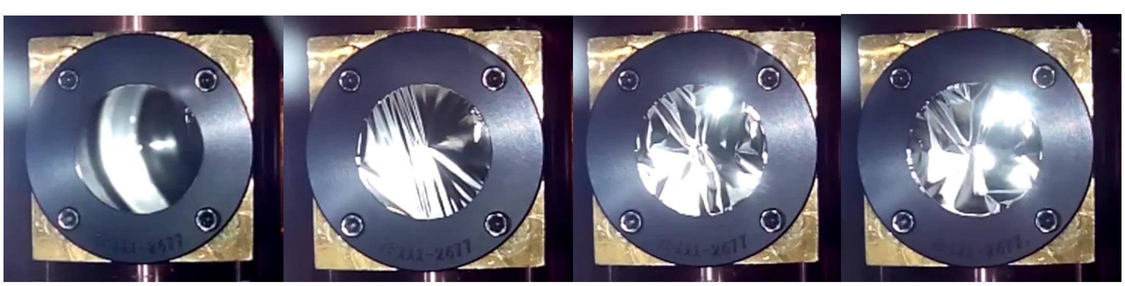

Figure 2. Test filter TF111-2677 in UHV (P = 2.5 10-9 mbar) at 300 K, 250 K, 220 K, 158 K from left to right. Pictures are taken from a side view port of the vacuum chamber.

We could not set-up the laser reflection measurement through the viewing port of the vacuum chamber to measure the height of the wrinkles at low temperature, however, we noted that the filter corrugation observed in air at room temperature was visually similar to that one observed in UHV at about 220 K (Figure 2). Then we calculated the expected wrinkles height that would result from the differential contraction between the al frame and the polyimide film with a $\Delta T = 80$ K. For this calculation, we assumed: a coefficient of thermal linear expansion (CTE) equal to 3 10^{-6} K^{-1} for the polyimide (LUXEL private communication) and 23 10^{-6} K^{-1} for aluminum, and a sinusoid with $\lambda = 2$ mm for the wrinkles profile. The calculated value for the wrinkles height peak to valley is 60 µm which is in very good agreement with the slope reflection measurements at room temperature, thus allowing to relate the corrugation in UHV at 220 K entirely to differences in thermal expansion between the two materials. The same calculation for a $\Delta T = 250$ K brings to a calculated value of 110 µm for the wrinkles height.

4. INFRARED TRANSMISSION

Two campaigns of InfraRed (IR) measurements were carried out in transmission geometry at different temperatures in the range 10-300 K. The first one was performed in low vacuum (P ~ $4*10^{-3}$ mbar) using a JASCO FT-IR 410 instrument in the 1000-7800 cm^{-1} range with a resolution of 2 cm^{-1} and 25 averaged scans. The second one was performed in ultra-high vacuum (P ~ $2*10^{-9}$ mbar) using a Bruker Vertex 70 instrument in the 600-8000 cm^{-1} range with a resolution of 2 cm^{-1} and 64 averaged scans. In both campaigns the filter samples were mounted on a cryostat cold finger with a cylindrical radiation shield leaving open only the beam optical path (Figure 3).

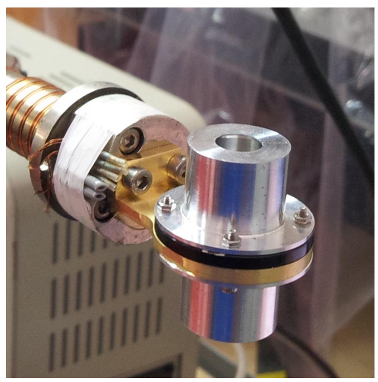

Figure 3. Filter sample mounted on the colt finger of the JASCO FT-IR 410 cryostat. The sample (black anodized aluminum frame) is thermally anchored to the gold plated copper fixture and protected by two aluminum radiation shields opened only along the beam optical path.

Before mounting the sample to be measured, the system is pumped down to measure the reference beam which is needed to derive the filter transmission. Two samples have been investigated, namely: TF111-2761 (Polyimide 45 nm), and TF111-2677 (Polyimide 150 nm/Al 30 nm). The left panel of figure 4 shows the transmission measurements performed on sample TF111-2761 (Polyimide 45 nm) at 6 different temperatures in the range 50-300 K. In this wavelength range Polyimide is nearly transparent. The measured transmission, however, shows a small steady decrease with decreasing temperature.

In order to verify whether changes in transmission with temperature occur in specific regions of the IR spectrum we plot also the ratio between the transmission measured at different temperatures with respect to the transmission measured at 300 K (Figure 4, right panel).

The main absorption bands typical of cured Polyimide films at ~5.8 µm, ~6.6 µm, and ~7.4 µm [12][13]present a small blue shift going towards lower temperature, indicative of increasing strength of the associated bonds. Beside these and few other minor features, it is clear that transmission ratios are nearly constant as a function of wavelength in the investigated range, suggesting that the effect of decreasing transmission with temperature can be ascribed to a change in optical depth.

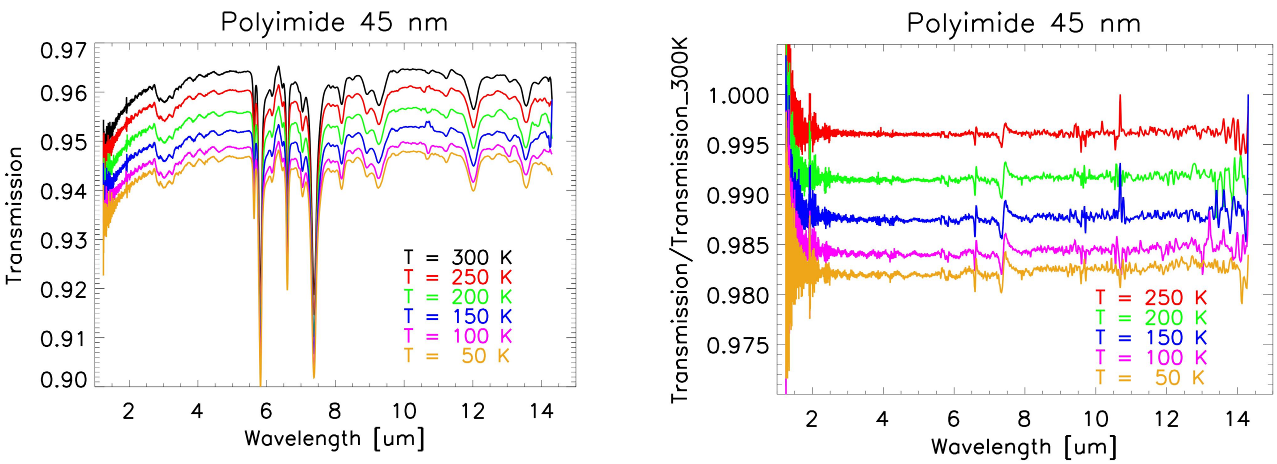

Figure 4. Left panel: IR Transmission measurements performed on sample TF111-2761 (Polyimide 45 nm) at 6 different temperatures in the range 50-300 K. Right panel: ratio between the transmission measured at different temperatures and the transmission measured at 300 K.

One possibility is that this is due to the occurrence of wrinkles (see section 3) which cause an increase of the effective thickness of the film. The measured change in transmission between 300 K and 50 K, is consistent with an increase of the thickness close to a factor 1.5. Such large increase of effective thickness is consistent with wrinkles with typical wavelength of 2 mm and amplitude ~ 800 μm peak to valley. This amplitude is quite large and cannot be fully explained in terms of a differential contraction of polyimide film and aluminum frame according to our best knowledge of the CTE of the two materials (see section 3).

Another effect that can increase the absorption is an increase of the density of the material due to the temperature contraction. Only the surface contraction has to be taken into account since the thickness contraction does not produce an increase in optical depth. Considering a CTE of polyimide equal to 3 10^{-6} K^{-1} [LUXEL private communication], and a $\Delta T = 250$ K, the derived increase of optical depth is approximately a factor 1.002 and thus negligible with respect to the estimated increase of a factor ~ 1.5.

Figure 5 shows the transmission measurements performed on sample TF111-2677 (Polyimide 150 nm/Al 30 nm) at 4 different temperatures in the range 14-300 K. The superimposed black line is the modeled transmission derived by the use of the matrix formulation of the boundary conditions of the electromagnetic field[14], with the refractive index of aluminum derived from Smith (1985) [15] and that one of polyimide extrapolated from Cavadi et al. (2001) [16]. In the model we assumed the presence of a layer of 6 nm of aluminum oxide [17] that is transparent in the IR spectral region.

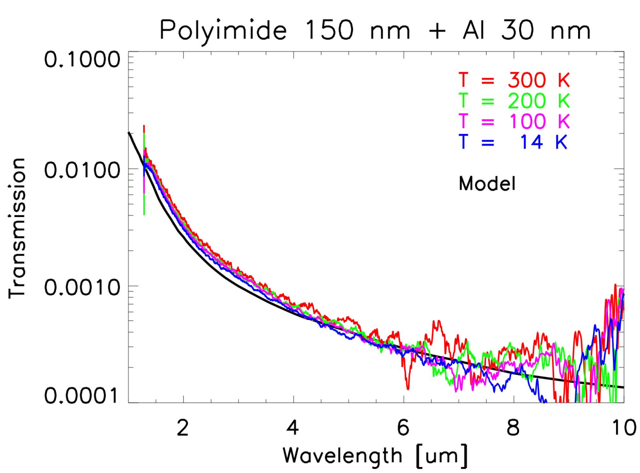

Figure 5. IR Transmission measurements performed on sample TF111-2677 (Polyimide 150 nm/Al 30 nm) at 4 different temperatures in the range 14-300 K. Superimposed is also the modeled transmission (black solid line).

Figure 5 shows that the temperature in the range 300-15 K has negligible effect on the IR transmission on Al coated Polyimide films essentially because it is dominated by Aluminum reflection. Furthermore, the measured IR transmission in the 1-15 μm wavelength range is in very good agreement with the model.

5. X-RAY ABSORPTION SPECTROSCOPY

X-ray transmission of the investigated filter samples was measured in ultra-high vacuum (P ~ $7*10^{-9}$ mbar) at the BACH beamline of the Italian synchrotron ELETTRA in the 175-590 eV and in the 490-1645 eV ranges at different temperatures (300, 200, 135 K). The adopted energy step was 2 eV except for the ranges 273-278 eV, 278-290 eV, 290-310 eV, 360-425 eV, 510-560 eV, and 1540-1645 eV, where the energy steps were 0.2, 0.1, 0.2, 0.2, 0.2, and 0.5 eV, respectively. The transmitted beam flux is derived by measuring with a pico-ammeter the photoelectric current induced in a gold plate absorber. The angle between the incident beam and the sample is fixed at 30° in the employed setup (Figure 6), therefore the effective optical path is twice the thickness of the filter.

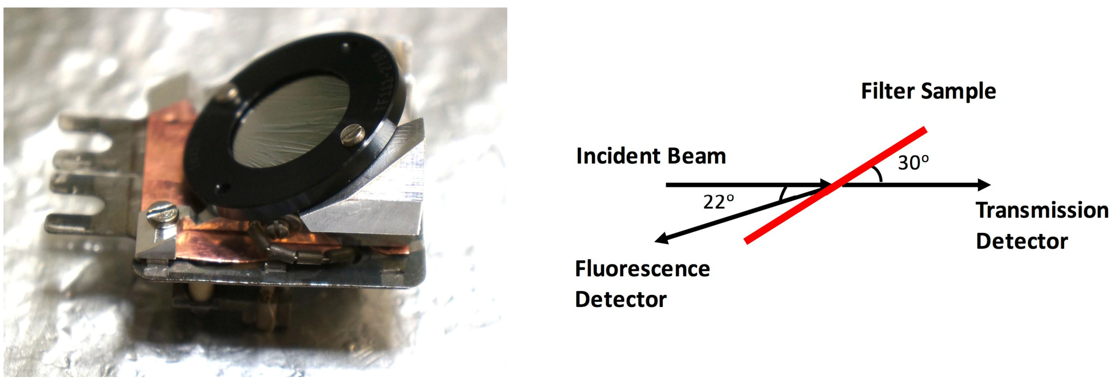

Figure 6. Left panel: filter sample TF111-2768 (Polyimide 45 nm/Al 30 nm) mounted on the sample holder of the BACH beamline. The filter plane has an inclination angle of 30° with respect to the incident beam. Right panel: schematic configuration of the set-up adopted at the BACH beamline for the X-Ray Absorption Spectroscopy.

For a few samples, the X-ray Absorption Near Edge Spectroscopy (XANES) has been also performed measuring the fluorescence yield using a micro channel plate detector placed at 22° with respect to the incident beam direction (Figure 6, right panel).

In order to derive the X-ray transmission of the investigated filter sample, the reference beam flux is measured with the same experimental configuration (monochromator, undulator, slits, etc.) before the measurement of the transmitted beam flux.

The X-ray transmission near the absorption edges of the atomic elements present in the filter material (C, N, O, Al) have been measured at three different temperatures, namely 135 K, 220 K, and 300 K (Figure 7).

The measured transmissions plotted in Figure 7 have been corrected for the effective thickness due to the 30° inclination of the filter in the sample holder, and have also been shifted to match the nominal transmission value before the absorption edge in order to compare only details of the fine structures. The black line is the model transmission based on the nominal filter thicknesses (45 nm Polyimide + 24 nm Al + 6 nm Al_2O_3) and the use of the imaginary part of the atomic scattering factors by Henke et al. (1993) [18].

Such plots present small differences in the absorption edge fine structures at different temperatures in the investigated range 300-135 K. The differences being a little more pronounced in the Carbon Kα edge.

The fluorescence yield measured on the same filter sample (Figure 8) shows essentially no major change with temperature for the O, and Al edges while small changes in the amplitude of some features are present at the N and C Kα edges.

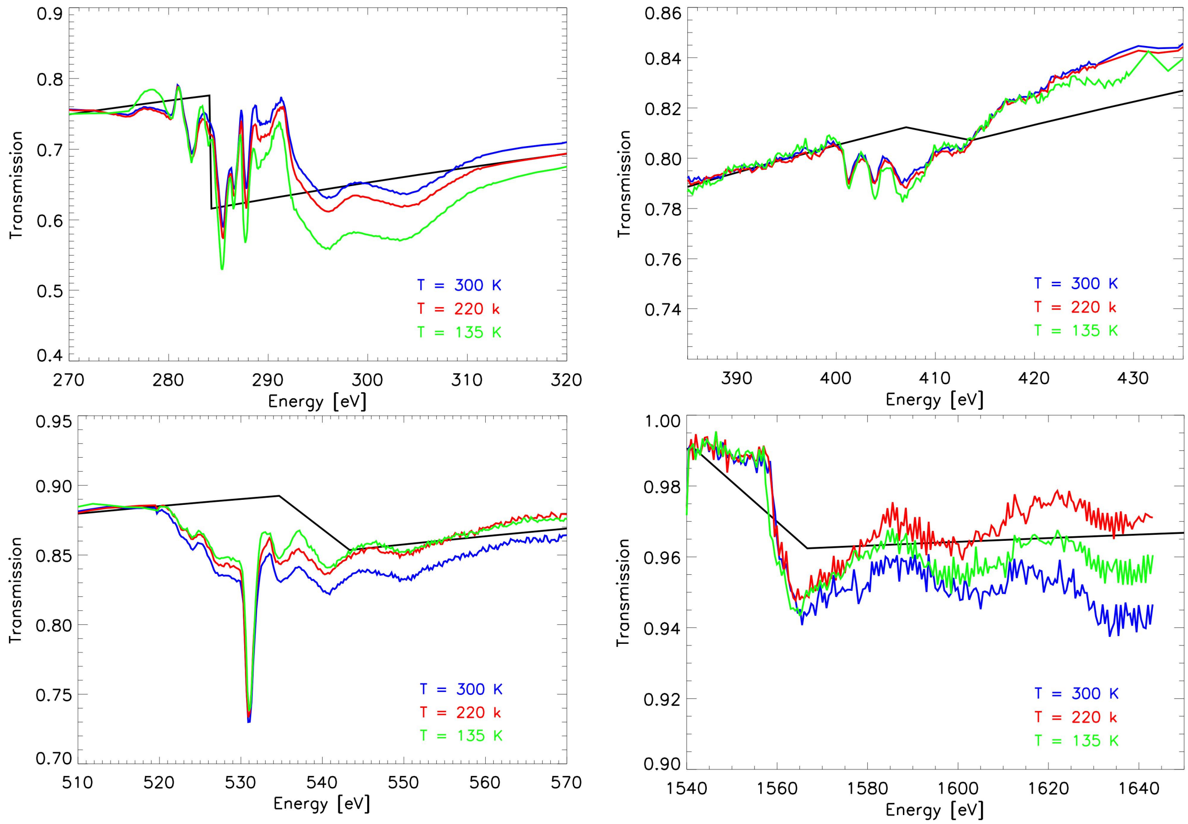

Figure 7. X-ray transmission near the absorption edges of the atomic elements present in the filter material (C, N, O, Al) measured at three different temperatures, namely 135 K (green), 220 K (red), and 300 K (blue). The black line is the model transmission based on the nominal filter thicknesses (45 nm Polyimide + 24 nm Al + 6 nm Al_2O_3).

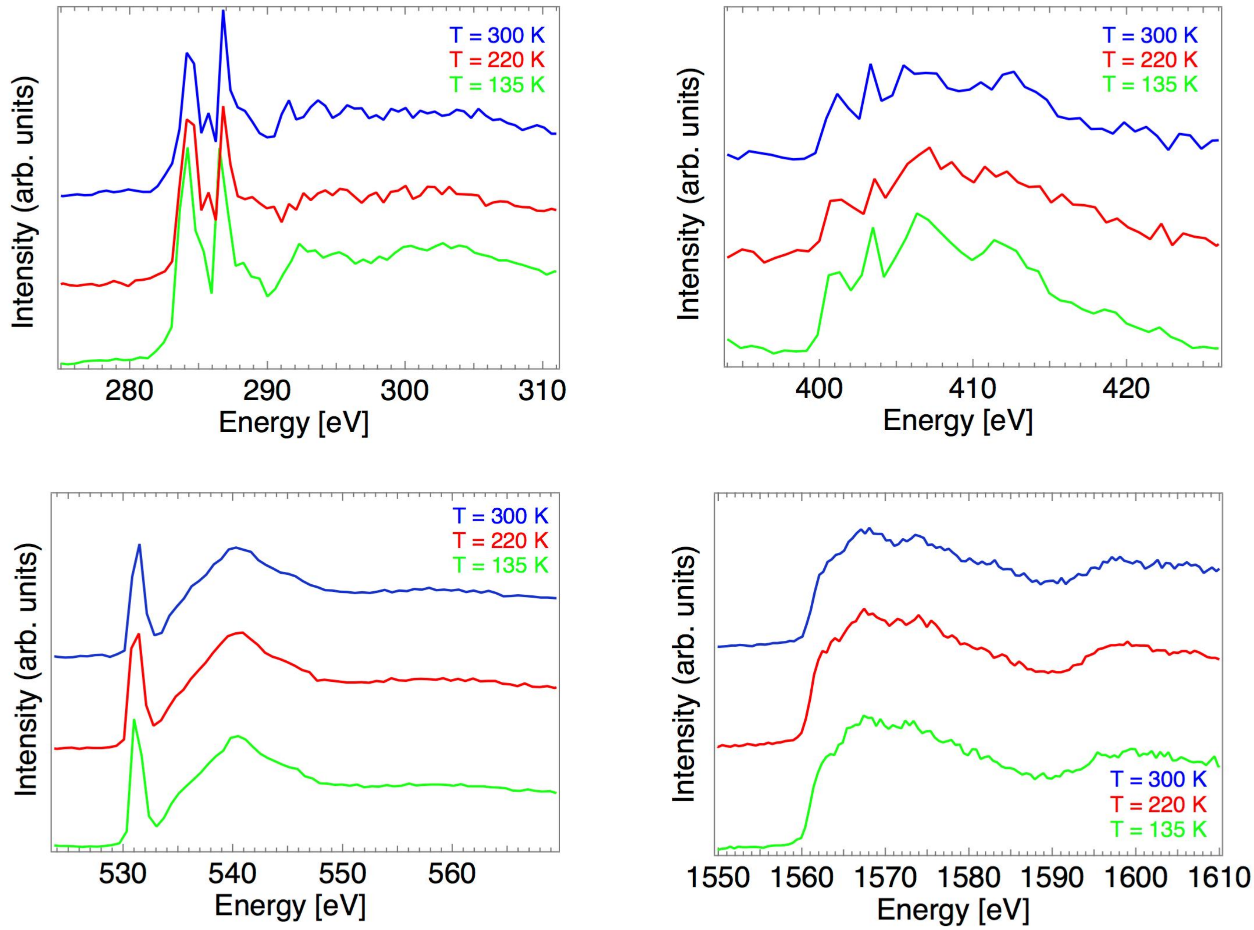

Figure 8. X-ray fluorescence yield in the energy range of the absorption edges of the atomic elements present in the filter material (C, N, O, Al) measured at three different temperatures, namely: 135 K (green), 220 K (red), and 300 K (blue). The plots are shifted to allow easier comparison.

Figure 9 shows the measured transmission near the Oxygen K_α absorption edge without any correction to match the nominal transmission before the edge. The transmission curve measured at 135 K is significantly lower than those measured at higher temperatures. In principle, this discrepancy could again be interpreted as an effect of increasing effective thickness as wrinkles build-up at decreasing temperature. However, since the X-ray beam is much smaller than typical scale size of the observed wrinkles, the measured transmission can be dependent on the local shape of the wrinkles which changes with temperature and thus a steady decrease in transmission with decreasing temperature is not consistently observed in other energy intervals. As for the IR measurements, however, the amplitude of the wrinkles necessary to justify the change in transmission, should be larger than what expected only on the basis of differential contraction between the Al frame and the Polyimide film.

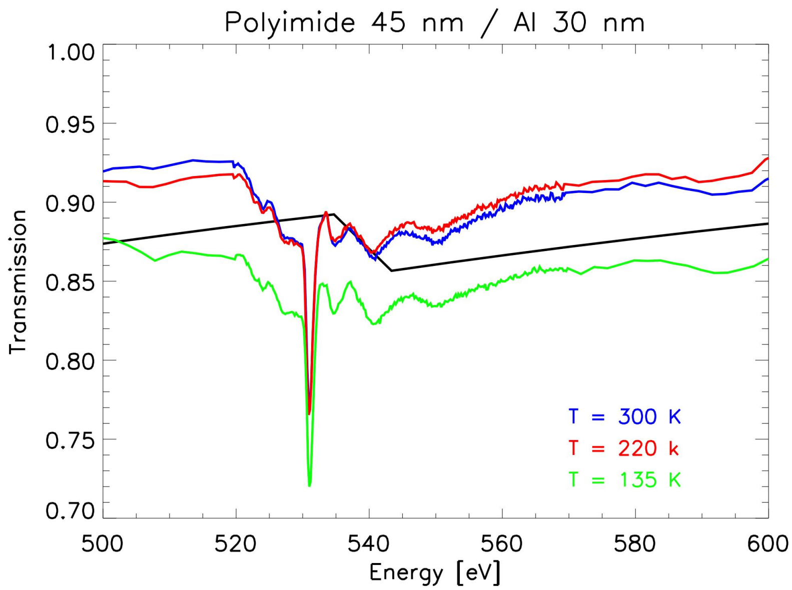

Figure 9. X-ray transmission near the absorption edge of the Oxygen K_{α} measured at three different temperatures, namely 135 K (green), 220 K (red), and 300 K (blue). In this case no normalization has been applied to the measured data.

6. SUMMARY AND CONCLUSIONS

The Thermal filters of the X-IFU detector will be thermally and mechanically anchored to the thermal shields of the cryostat and focal plane assembly, and therefore will operate at different temperatures in the range 50 mK – 300 K. In this paper we present some preliminary results on an investigation aimed at verifying if and how temperature modifies the performance of the TF and how this may affect the calibration activity.

A polyimide film in air at room temperature corrugates because it is hygroscopic. Keeping it in high vacuum for many hours it desorbs water and stretch back. We have measured the typical height of the wrinkles occurring at room temperature in air by measuring the reflection of a laser beam on the aluminized surface of the filter samples. With this method we estimated wrinkles height peak to valley of ~ 60 µm under the assumption that wrinkles follow a sinusoidal profile.

When the polyimide films are cooled down, while kept in high vacuum, they corrugate again. Observing the behavior of polyimide/Al filter samples as a function of temperature inside an Ultra-High vacuum chamber, we estimate the corrugation of the filter at ~220 K to be similar to that one observed at room temperature in ambient atmosphere. The wrinkles peak to valley amplitude, that we would expect by the differential contraction between the aluminum frame and the polyimide film, for a $\Delta T = 80$ K is consistent with the measured value of ~ 60 µm at room temperature. As temperature is lowered below 220 K wrinkles become more and more pronounced. At the moment we do not have a direct measurement of the wrinkles peak to valley amplitude at low temperatures. We plan to modify the experimental set-up to be able to perform the laser beam reflection measurement through the view port of the vacuum chamber.

Since the main role of the TF is to attenuate the IR radiation to reduce the radiative load onto the detector we have performed transmission measurements of bare polyimide and polyimide/Al samples in the wavelength range 1-15 µm where a 300 K black-body emission peaks. Measurements performed on a bare polyimide sample 45 nm thick show a small steady decrease of transmission at decreasing temperature from 300 to 50 K. The change in transmission is essentially wavelength independent and could be interpreted as a simple increase in optical depth. The estimated increase of optical depth, derived from the experimental decrease in transmission, turns out to be a factor ~ 1.5 at 50 K with respect to room temperature. If this increase in optical depth was due to the filter corrugation observed at low temperature, the wrinkles should have a peak to valley amplitude of the order of 800 µm which cannot be justified entirely by the differential contraction between the aluminum frame and polyimide film, according to our best knowledge of the CTE of the two materials.

Temperature in the range 300-15 K has negligible effect on the IR transmission of Al coated Polyimide films essentially because it is dominated by Aluminum reflection. Furthermore, the measured IR transmission in the 1-15 μm wavelength range is in very good agreement with the model.

The high energy resolution X-ray transmission measurements performed at the BACH beam-line of the synchrotron ELETTRA, present small differences in the absorption edge fine structures at different temperatures in the investigated range 300-135 K. The differences being a little bit more pronounced in the Carbon Kα edge.

The fluorescence yield measured on the same filter sample shows essentially no major change with temperature for the O, and Al edges while small changes in the amplitude of some features are present at the N and C Kα edges.

In addition, the overall transmission changes with temperature, although not always steadily decreasing with decreasing temperature. This discrepancy could again be interpreted as an effect of increasing effective thickness as wrinkles build-up at decreasing temperature. As for the IR measurements, if the change in transmission was entirely due to the filter corrugation, the peak to valley height of the wrinkles should be significantly larger than expected on the basis of the differential contraction between the aluminum frame and the polyimide film.

The measurements conducted at ELETTRA in transmission geometry suffer from quite large uncertainties as it is evident from the differences observed in the same energy intervals using different monochromator grating and undulator configurations. Despite these results are not conclusive on the effective temperature dependence of X-ray transmission near the absorption edges, we do have some indications that make it valuable to repeat the XAS measurements to confirm the results and make more quantitative estimate and modeling of the observed temperature dependence, as well as to extend them to lower temperatures to cover the full interval of interest for the X-IFU TF.

In conclusion, the preliminary results of the measurement performed on prototype samples of the X-IFU TF at low temperatures, namely IR transmission, X-ray transmission, visual inspection, indicate that the performance of the filters is affected by temperature. Part of this dependence is due to the film corrugation induced by differential contraction between the aluminum frame and the polyimide film. The corrugation seems less significant when a mesh is attached to the film.

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