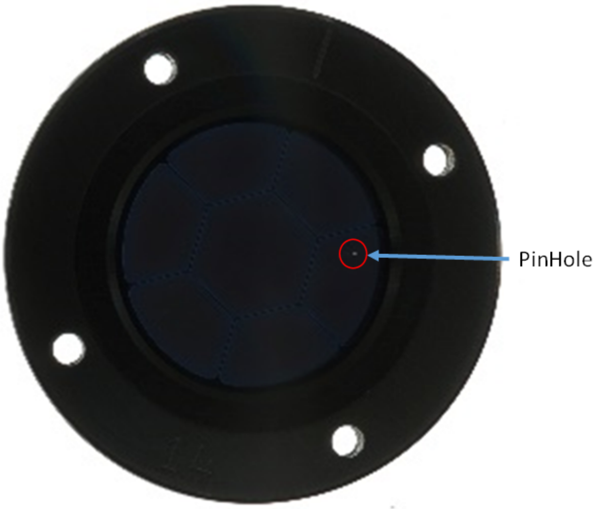

Fig. 65 – LAOF-S1-C1B5-W2-F4 after MS1 shake level with particle size 27-31 um have one pinhole.

After the application of the MS2 level with the same particle size 27-31 um, the filter LAOF-S1-C1B5-W2-F4 presents 3 broken cells (See figure 66). Is it worth to note that the same pinhole generated on the previous run did not propagate into a crack.

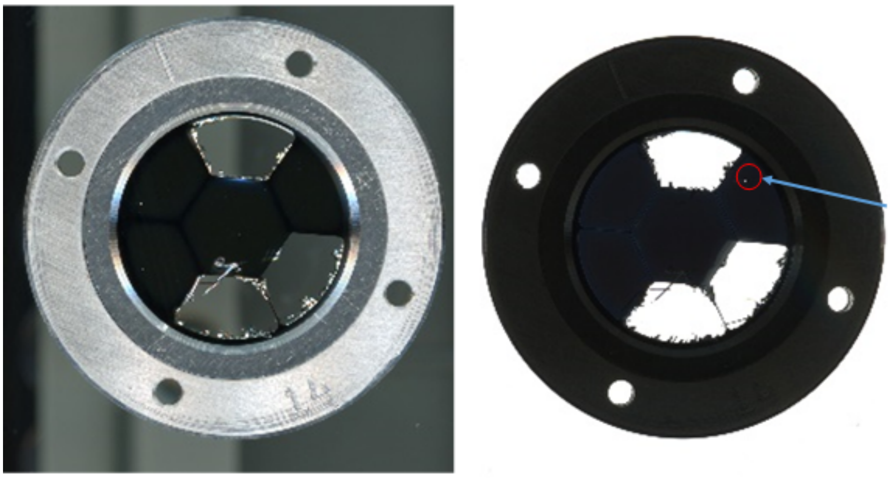

Fig. 66 - LAOF-S1-C1B5-W2-F4 after MS2 application with particle size 27-31 um.

The broken S/C# LAOF-S1-C1B5-W2-F4 was replaced by the Polyimide/Al supported by polyimide mesh sample OIT–F1111–84 manufactured by OXFORD, starting from the MS1 level with particles 57-67 µm in diameter.

The filter LAOF-CNT2-C1B2-F01 was damaged during the manipulation activity before the shaking test with the 57-67 um particle (see figure 67). It is worth noting that the filter was in perfect condition before the damage occurred during the manipulation.

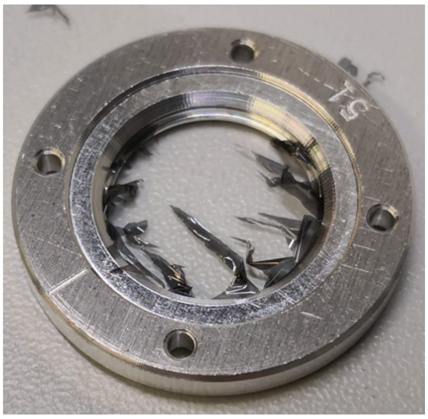

Fig. 67 - LAOF-CNT2-C1B2-F01 filter damaged during manipulation.

The LAOF-CNT3-C1B2-F01 sample was replaced by the LAOF-CNT2-C1B2-F03 starting from the MS1 level with particle 57-67 um. From this run (MS1 level with particle 57-67 um) to the last run (MS3 level with particle 95-105 um) all the four filter samples mounted on the interface passed the test without damage.

In the last three rows of table 45 we report the total time each filter was loaded with a specific MS level. Table 46 summarizes the TMV vibration tests for each filter.

Table 46: Resume of the TMV test with the time each filter was subject to the MS load [in seconds]

Vibration level (in air)	**Time each filter was subject to Sine (Sx), Random (Rx) and Shock (SKx) load (seconds)**			
	W3a#03	LAOF-CNT1-C1B1-F07	LAOF-CNT3-C1B2-F01	W3b#12
S2	65	65	65	\
R2	30	30	30	\
S4 (qualif)	130	130	130	130
R4 (qualif)	150	150 (broken for debonding on inner frame)	150 (broken)	150
SK3	0.020	\	\	\

The W3b#03 filter passed all planned runs until the qualification level S2, R2, S4, R4 and SK3 without any damage. In figure 68 a scan of the filter after the test is shown.

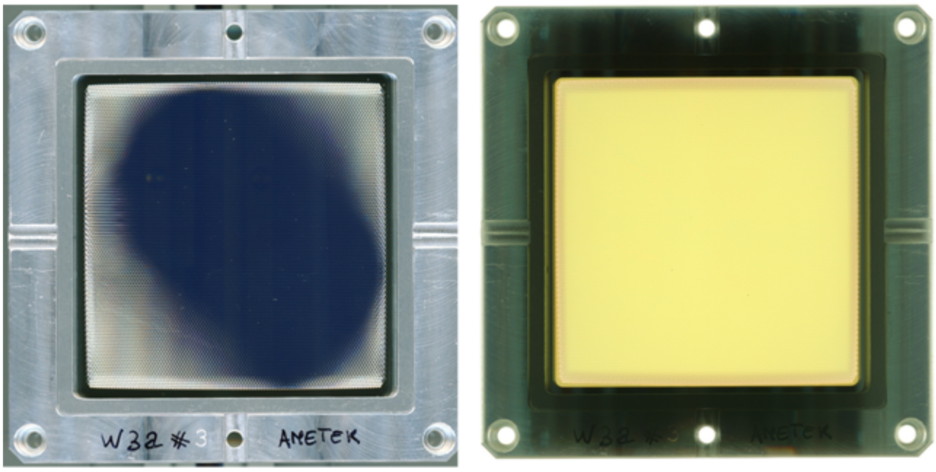

Fig. 68 - scan of W3a#03 filter in reflection (left) and transmission (right) after the last run.

The W3b#12 filter passed the S4, R4 runs without any damage. For the lack of time the S2 and R2 runs were skipped in order to test only with the qualification level (S4 and R4). In figure 69 a scan of the filter after the test is shown.

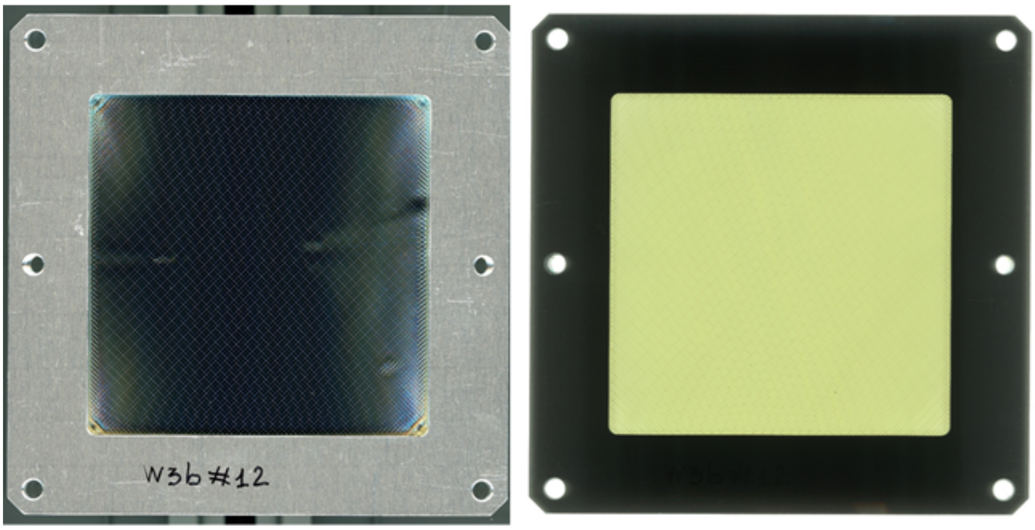

Fig. 69 - Scan of W3b#12 filter in reflection (left) and transmission (right) after S4 and R4 runs.

The LAOF-CNT1-C1B1-F07 and LAOF-CNT3-C1B2-F01 passed the S2, R2, and S4 (qualification sine level) runs without damage.

During the R4 run a metallic noise was coming from the interface, the ringing noise was due to the debonded inner frame of the LAOF-CNT1-C1B1-F07 that broke the CNT membrane.

The LAOF-CNT3-C1B2-F01, broke at the same run R4 probably for the same reason (a partial debond of the inner frame). The gluing of the inner frame of the LAOF-CNT3-C1B2-F01 need to be checked. In figure 70 a picture of the broken LAOF-CNT1-C1B1-F07 and LAOF-CNT3-C1B2-F01 filters after the R4 run is reported.

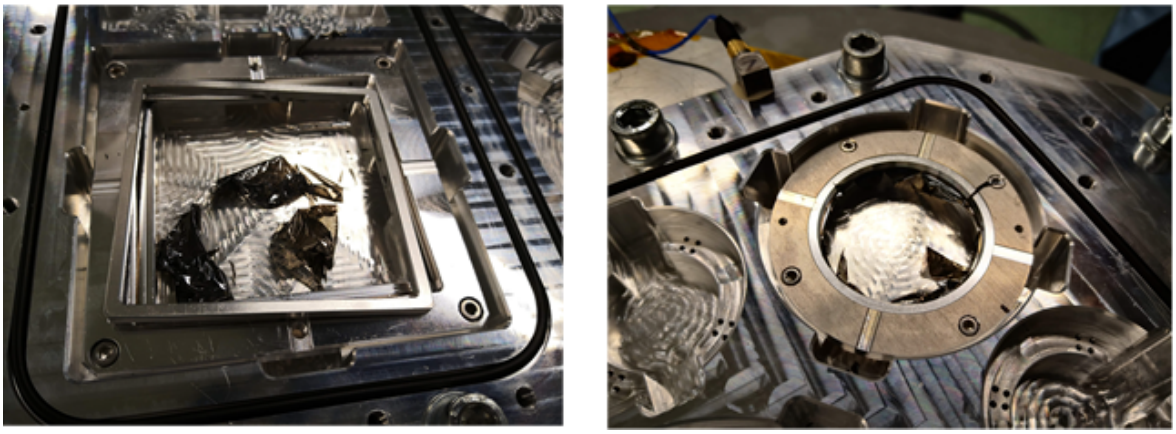

Fig. 70 - Picture of the broken LAOF-CNT1-C1B1-F07 and LAOF-CNT3-C1B2-F01 filters after the R4 qualification level; on the left the LAOF-CNT1-C1B1-F07 with the debonded inner frame, on the right the LAOF-CNT3-C1B2-F01with the broken membrane.

10.2 Acoustic Tests

The Athena WFI detector will be launched in atmospheric pressure with filters subject to acoustic loads, for this reason partially representative filter samples, mounted inside a mock-up of the filter wheel assembly developed in Poland by CBK, have been tested with ARIANE V qualification acoustic loads.

Two acoustic test campaigns have been performed on filter samples manufactured within this contract. The first one was performed in April 2018 on Si_3N_4/Al filters with Si mesh mounted on TF112 frames while the second one was performed on polyimide/Al filters with polyimide mesh on July 2019.

April 2018 Test Campaign

Two medium size Si_3N_4 filter samples on TF112 frames, manufactured by AMETEK Finland, have been mounted inside a vacuum tight aluminum box with plexiglass cover (figure 71, right panel), namely: ReLAX WP4L1 W17 K4 n. 17 and ReLAX WP4L1 W14 K6 n. 3. Both filters consist of 40 nm thick Si_3N_4 coated with 15 nm thick Al on each side supported by a honeycomb Si mesh with pitch size = 1 mm, thickness = 30 µm. The two samples differ only for the mesh bar width which is 40 µm in sample W17 K4 (OA = 92%), and 60 µm on sample W14 K6 (OA = 88%).

The box has been placed inside the reverberation chamber at AGH University of Science and Technology in Krakow (figure 71, left panel) and has been exposed to the Ariane 5 acoustic reference load. Different tests have been performed with filters kept at pressure < 1 mbar, 10 mbar, 100 mbar and 1000 mbar pressure. Figure 72 shows the measured acoustic spectrum outside the box (red line) and inside the box at atmospheric pressure (blue line). The reference Ariane 5 acoustic reference spectrum is also shown (orange line).

Both tested filters have survived the acoustic load in all mentioned pressure conditions.

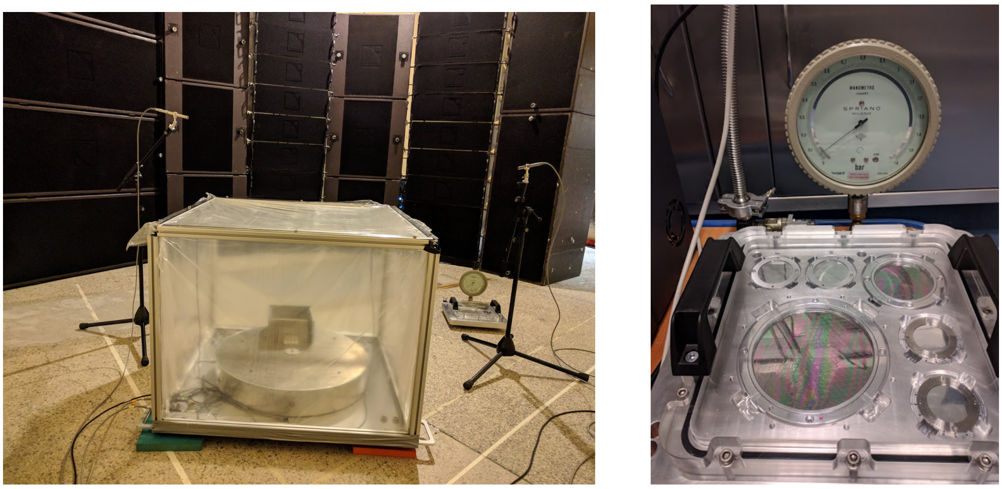

Fig. 71 - Test equipment inside the AGH reverberation chamber (left panel). The plastic tent in foreground covers the filter wheel mock-up used to test filter samples of the ATHENA Wide Field Imager, while the vacuum tight box used for the X-IFU filters acoustic tests is in the background. The right panel shows the tested X-IFU filter samples mounted inside the vacuum tight box.

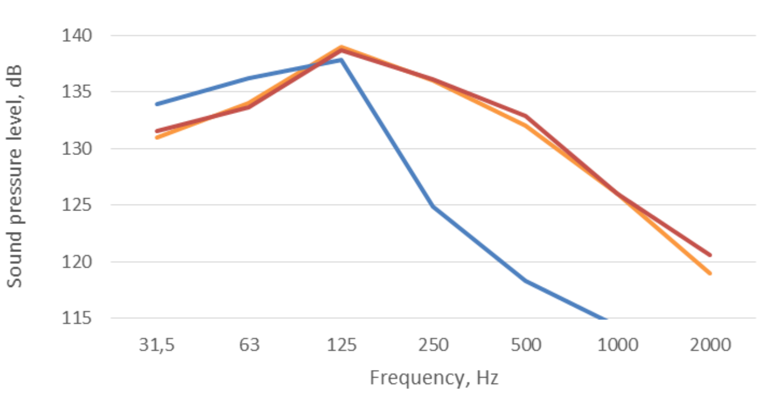

Fig. 72 - Sound Pressure Level measured inside (blue) and outside (red) the vacuum tight box. The reference Ariane V qualification level is in orange.

July 2019 Test Campaign

Acoustic tests have been performed at AGH Acoustic Laboratories in Krakow adopting the filter wheel (FW) configuration shown in figure 73 named “balanced configuration”.

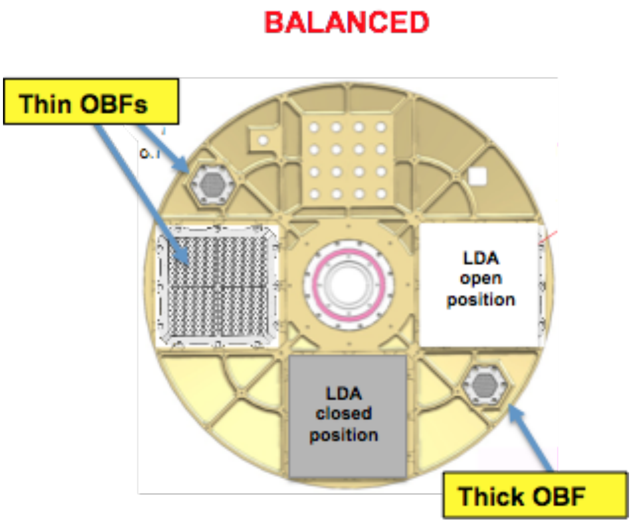

Fig. 73 - Filter wheel configuration adopted for the acoustic tests.

Different parameters have been measured during the tests to make comparisons with simulations and identify areas of improvement in the FW design, namely:

- Vibration levels in different positions on the filter wheel;
- Acoustic spectrum above and below the filter (in different locations).
- Acoustic pressure time histories above and below the filter (in different locations) to derive the differential acoustic pressure induced by acoustic loads.

Three different sound pressure levels have been identified for the tests and are shown in table 47 and figure 74 as measured inside the FW assembly close to the Large Area Detector optical blocking filter. The low level (red) simulates the effect of launching the Athena WFI with a valve closing the entrance baffle of the FW, the qualification level (green) is the reference Ariane V acoustic load at launch with the FW in presently identified optimal configuration, and the stress level (blue) is the sound pressure spectrum in a non-optimal FW configuration (unbalanced) providing higher acoustic load onto the optical blocking filters.

Table 47: Three different pressure levels used in the tests.

Low Level LL (balanced)		
Differential pressure (mbar)		SPL (dB)
RMS	PEAK	TOTAL
0.1	1.2	134.3
Qualification Level QL (balanced)		
Differential pressure (mbar)		SPL (dB)
RMS	PEAK	TOTAL
0.4	4.6	137.3
Stress level SL (unbalanced)		
Differential pressure (mbar)		SPL (dB)
RMS	PEAK	TOTAL
0.8	7.9	142.0

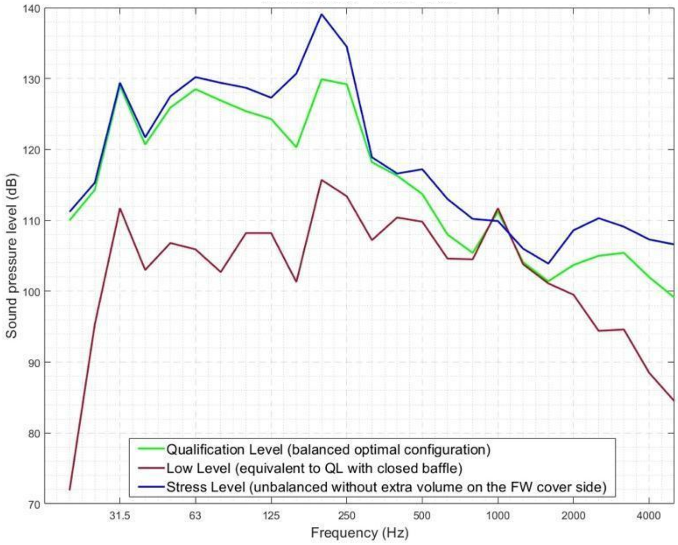

Fig. 74 - Sound pressure level spectra: qualification level (green line), equivalent to the qualification level with the closed baffle (red line), and stress unbalanced level (blue line).

Two filter samples in polyimide/Al supported by polyimide mesh manufactured by Oxford Instruments, Finland have been tested with low and qualification sound pressure level previously specified. The two filters, different only for the mesh geometry, are: W3a-#03 (hexagonal mesh) and W3b-#12 (square mesh). Figure 75 shows a picture of one of the two filters mounted inside the FW assembly. Both filter samples have survived the acoustic tests without any damage.

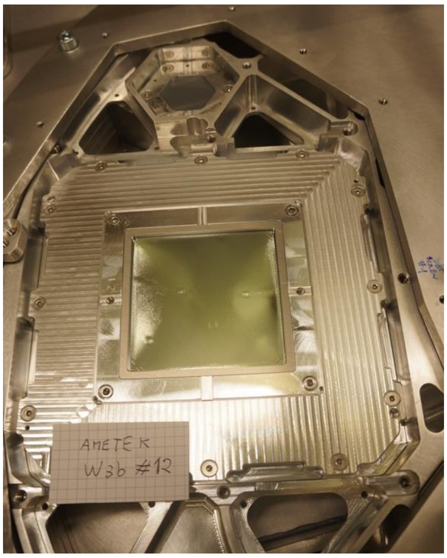

Fig. 75 - Filter sample W3b#12 mounted inside the Athena WFI filter wheel mock-up before entering the acoustic tests reverberation.

11. Atomic Oxygen Tests

The goal of this activity is to verify the effect of the atomic oxygen, a residual species of the atmosphere in Low Earth Orbit, on some witness filters.

Tests at THE LEOX facility in the Materials and Electrical Components Laboratory of ESA have been postponed to a date still to be notified due to the COVID-19 pandemic. In the standard set-up twenty samples (about 2 x 2 cm^2) are integrated into the sample holder that can be rotated to any beam axis angle of attack, so from 0° up to 90°.

12. Thermo-Vacuum

The goal of this activity is to test the compatibility and differential CTE effects of the materials used to make a representative filter (plastic foil, silicon nitride, Si or metal meshes, frame) over the full operational temperature range. Tests were performed in March 2020 in a custom facility set-up at the X-ACT facility of the Istituto Nazionale di Astrofisica (INAF-OAPa) in Palermo on the two filter samples listed in table 48.

Table 48: Polyimide film/polyimide mesh filter samples under test

Sample code	Frame	Al (nm)	Film (nm)	Mesh
W3a#03	DWG No. WFI-UP-ASD-132110-000-02-001	30÷40	Polyimide 140	Polyimide honeycomb thickness: 17.4 μm ÷18.4 μm bar width: 20 μm, OA 95%
LAOF-CNT-C1B1-F08	ATHENA-01-01_TEST	ALD3	CNT 120-150	

Fig 76 shows the Oxford W3A#03 (left panel) and AMETEK LAOF-CNT-C1B1-F08 (right panel) sample filters mounted on single quadrant WFI LDA OBF aluminum square frames (open area 82 mm x 82 mm) placed in the thermal cycling vacuum chamber.

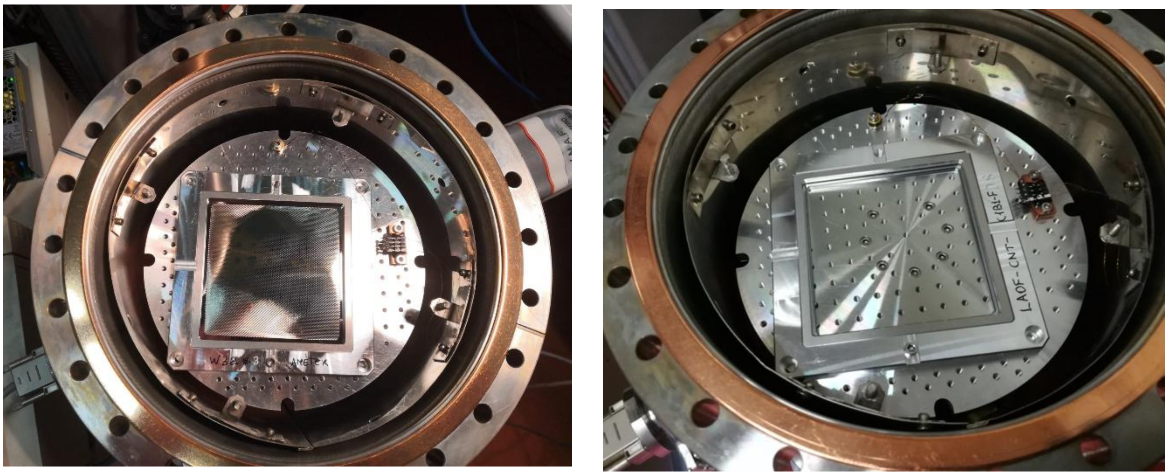

Fig. 76 - Test filter samples OXFORD W3A#03 (left panel) and AMETEK LAOF-CNT-C1B1-F08 (right panel) mounted inside the thermal cycling vacuum chamber

The experimental setup consists of a test chamber, a vacuum pump (turbo+scroll), a mechanical cooling system based on He cycle and a heater powered by a dedicated controller. Two thermometers are available, one measuring the cooling system cold head temperature and the other measuring the sample holder temperature. A custom computer software, interfaced with the temperature controller, allows to set, perform and monitor the thermal cycling. The sample is placed within the chamber, then brought into vacuum and finally the controller unit starts the T cycle as programmed, while a sensor within the chamber measures the sample T. A camera is placed on top of the chamber window, to acquire pictures of the sample at the beginning of each cycle segment. Table 49 summarizes the thermal cycle parameters used during the test.

Table 49: Test parameters for 'Large' W3A#03 sample.

Minimum temperature	Tmin	253 K
Maximum temperature	Tmax	320 K
Temperature ramp	Tramp	2.5 K/min
Hold time	Ht	21 min
Number of cycles	N cycles	100
Pressure in the chamber	P	3 x 10^{-5} mbar

Fig. 77 shows a plot of a part of the thermal cycle (100 cycles between Tmax and Tmin), where the stability of the single cycle, the T ramp slope and holding time can be checked. 100 thermal cycles were correctly completed on both filter samples and they showed no macroscopic damage. A visual inspection with an high resolution scanner was performed afterwards, and no damages/alterations of the filter surfaces were observed.

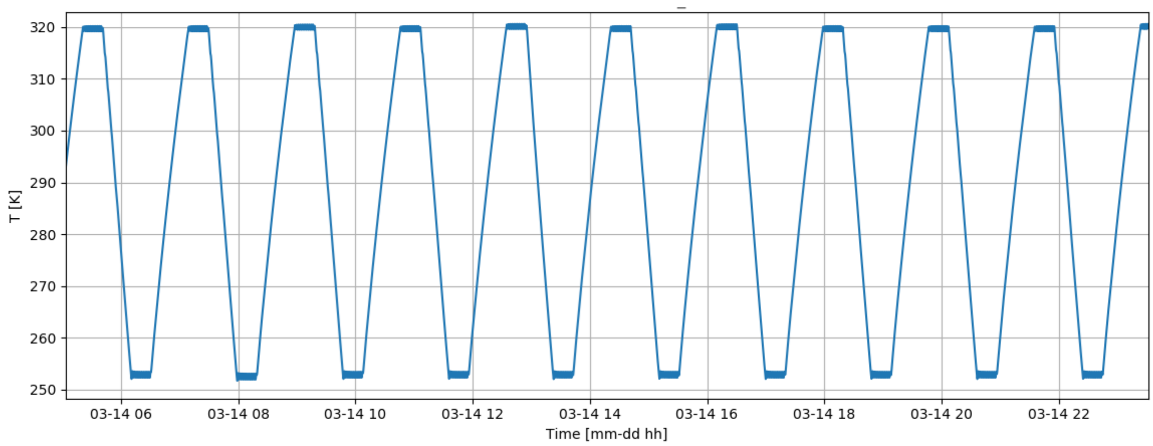

Fig. 77 - Part of the thermal cycle test, showing the maximum and minimum temperatures reached, the temperature ramp and the holding time.

13. Differential Pressure

The goal of this activity is to evaluate the mechanical behavior of the filter when loaded with a differential static pressure. A deflected profile of the membrane is acquired for each static differential pressure load-step. The test can also be run reaching the failure limit of the membrane. The outcomes of the test are useful for the design optimization, validating the numerical model, analyzing the failure mechanism and for the further generation of pass-fail criteria.

The experimental setup comprises a custom motorized X-Y-Z table, a filter holder with a pressure chamber, an optical measuring system and a pressuring device, with the aim of applying a static differential pressure (see Fig. 78). To perform the test a filter interface, ensuring the sealing between the two faces, have to be designed and manufactured for each filter frame geometry (TF 111 in this case).

It is worth noting that, with the current equipment, to perform the test the filter must be airtight, since a leakage would not allow to apply a static differential pressure.

The tests are conducted at the X-ACT Laboratory of the INAF – National Institute of Astrophysics of Palermo, in the Microtechnology laboratory ISO 7 cleanroom. A list of the filters that have been tested is shown in table 50.

Table 50: List of small size samples tested under static differential pressure.

Filter code	T @ 550nm	Frame	Density	Coating and thickness
LAOF-CNT2-C1B3-F11	64.5	TF111	High	Al 11nm/CNT 210~270nm/Al 11nm
LAOF-CNT2-C1B3-F19	64.5	TF111	High	Al 14nm/CNT 210~270nm/Al 14nm
LAOF-CNT2-C2B2-F04	90	TF111	High	Al 40 nm /CNT/Al 0 nm

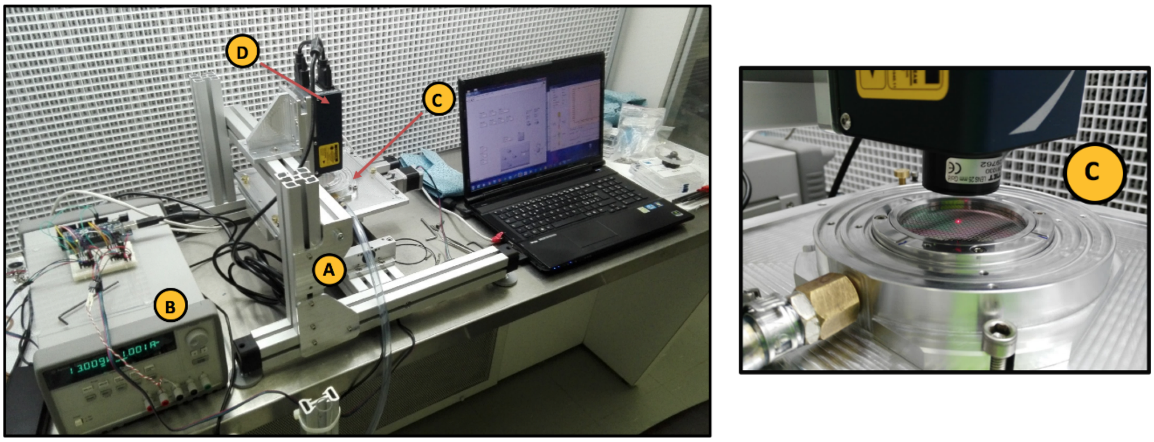

Fig. 78 – Set-up to measure the deformation and resistance of filter samples under static differential pressure. A) Motorized X-Y-Z table, B) Power supply, microcontroller and stepper motor drivers, C) Pressure chamber, D) Distance sensor.

Fig. 79 shows a picture of one of the filters mounted on the interface; an o-ring touching the outer frame of the TF111 sample holder seals the filter membrane, provided that the inner frame/outer frame bonding is continuous and leak tight.

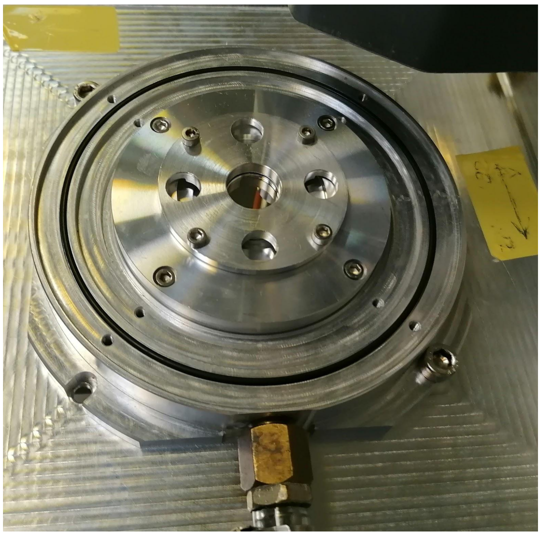

Fig. 79 – Picture of one of the filters mounted on the interface ready to be tested.

We have tried to put under pressure the three samples, however, none of them was able to keep even a moderate overpressure. Either the CNT network membranes are porous or an air leak is present between the outer and inner frames of the TF111 mounting. In order to perform differential pressure tests with not air-tight filters we need to modify our system to actively control the pressure. We plan to implement this upgrade within the LAOF-CCN contract.

14. Summary and Conclusions

A wide range of characterization tests have been performed on different filter samples manufactured/procured by AMETEK Finland within this contract, including:

- vibration tests at Max-Planck-Institut fuer Extraterrestrische Physik, Garching (D);
- acoustic tests at AGH Univ., Cracow (P);
- XAS at two PTB beamlines of Bessy II synchrotron, Berlin (D);
- proton irradiation at the Van der Graaf accelerator of the JWG Univ., Frankfurt (D);
- XAS at the BEAR beamline of ELETTRA synchrotron, Trieste (I);
- XPS at the BACH beamline of ELETTRA synchrotron, Trieste (I);
- X-ray mapping at the XACT facility beamline of INAF-OAPA, Palermo (I);
- UV/VIS/NIR spectroscopy at Univ. of Palermo (I);

In addition, we have designed, developed and used successfully three new experimental set-up to characterize thin large area filters for Athena and other high energy space missions, namely;

- Differential static pressure measurements set-up;
- Thermo-vacuum system to cycle filters down to 10 K and up to 325 K;
- Radio Frequency resonance chambers (1-20 GHz) to measure filter attenuation;

As an example, we point out that the activity performed within this contract to investigate the role of thin Al and of metallic meshes in attenuating the radio frequencies EMI from the telemetry and spacecraft operation, has been very successful and has provided critical indications in the design of the thermal filters of the Athena X-IFU. The performed test campaigns have proven that a metal mesh with pitch $\leq$ 4 mm exhibits a good attenuation of RF below 7 GHz while an aluminum film with thickness > 30 nm performs well at higher frequencies. The combination of the two provides > 30 dB attenuation in the full frequency range 30 MHz – 18 GHz (Athena X-IFU requirement for THF2 and THF300)

The first part of the contract has been dedicated to investigate Si_3N_4/Al membranes supported by Si fine meshes. In the second part, the investigation has been extended to polyimide/al supported by polyimide fine meshes. In the last months a new material based on CNT networks has been studied motivating a CCN specifically dedicated to this material. A few more tests are on-going or scheduled within the next weeks and will be reported in a final version of this report at the FR.

All the characterization tests and specifications of location, time, and investigated materials are summarized in table 51

Table 51: Performed characterization tests with specifications of location, time, and investigated filter materials.

Characterization test	**Location**	**Time**	**Filter materials**
Visual inspection	Internal	after each delivery	SiN/PI/CNT
XAS	Bessy (Berlin) BEAR Elettra (Trieste)	11-18.06.2018 03-06.06.2019 15-19.11.2017 03.2019 26.11-01.12.2019 04-10.12.2019	SiN SiN SiN SiN SiN/CNT PI
X-ray mapping	INAF-OAPA (Palermo) Bessy (Berlin)	20.10.2019 30.10.2019 03-06.06.2019 07.2019	PI CNT PI CNT
XPS	Bach Elettra (Trieste)	26.11-01.12.2018 26.11-01.12.2019	SiN SiN/CNT
AFM	UNIPA (Palermo)	14.01.2019	SiN
UV/VIS/NIR	UNIPA (Palermo)	20.12.2018 11.01.2019 11.10.2019 15.09.2020	SiN SiN CNT CNT
Acoustic stress	AGH University of Science and Technology (Krakow)	03-04.04.2018 15-16.07.2019	SiN PI
Vibration tests	Max Plank-Insitut fuer extraterrestrische Physics (Garching)	06-11.11.2017 09-14.12.2019	SiN SiN/PI/CNT
Static pressure	INAF-OAPA (Palermo)	09.07.2020	CNT
Thermo-vacuum	INAF-OAPA (Palermo)	06-20.03.2020 29.09-09.10.2020	PI CNT
Radiation damage	Van der Graaf accelerator at the JW Goethe-Universität Frankfurt am Main	06-10.08.2018	SiN

Project: Large area high-performance optical filter for X-ray instrumentation	**Document:** Filter Characterization Report	Document Code: LAOF-TN-10

Si_3N_4 Samples

Si_3N_4 filter samples manufactured by AMETEK have been characterized by X-ray Absorption Spectroscopy, X-Ray photoelectron Spectroscopy and UV/VIS/NIR spectroscopy to verify compliance to req. 2, 3, 4, 6 and 16 of the SoW. The following results have been obtained:

- Si_3N_4 films coated with aluminum show a thin layer of native aluminum oxide (approx 4.0 nm), similarly to polyimide.
- The total amount of Al_2O_3 on Al deposited on Si_3N_4 filter samples, measured by XAS at BESSY, varies from sample to sample, probably due to the fit being less accurate around the O K-edge and by an uncertainty introduced by the presence of a fine Si mesh with pitch similar to the x-ray spot size. In general the total amount of Al_2O_3 seems slightly lower than in polyimide, likely because the former material is less permeable to oxygen than polyimide.
- The Al oxide thickness is not significantly different for pristine and proton irradiated samples.
- The transmission in the UV region of Si_3N_4 samples is affected by high irradiation fluences though not changing significantly the overall out of band attenuation.

Figure 80 shows a comparison of modeled UV/VIS/NIR between two filters of Polyimide and Si_3N_4 coated with aluminum. The models show that Al coated Si_3N_4 provides similar out of band attenuation as Al coated polyimide, which is the current baseline filter material for Athena. On the other hand, as shown in figure 81the two materials of the same thicknesses have significant difference in soft x-ray transmission showing that Si_3N_4 can be competitive with polyimide for space applications only if it can be manufactured with thickness as low as 20-30 nm and still large diameters.

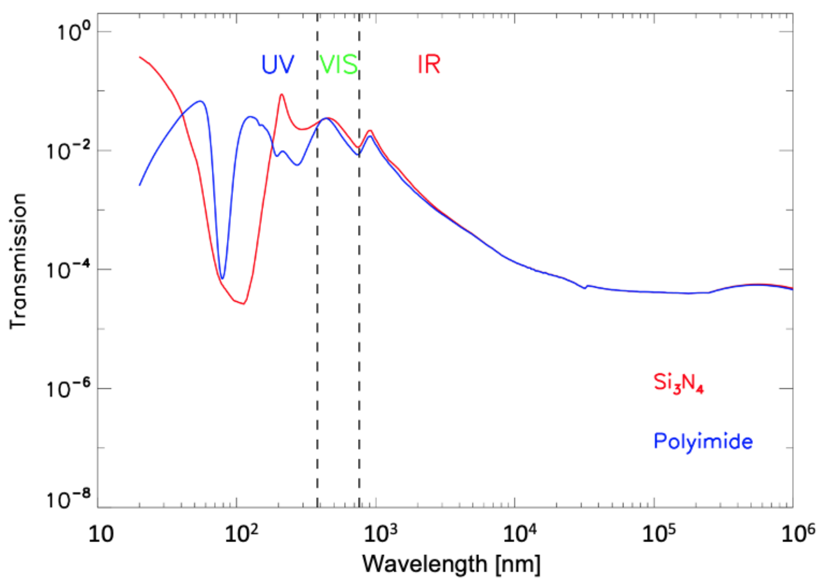

Fig. 80 - UV/VIS/NIR modelled transmission for two filters of Polyimide and Si_3N_4 both 50 nm thick

coated with 30 nm of aluminum.

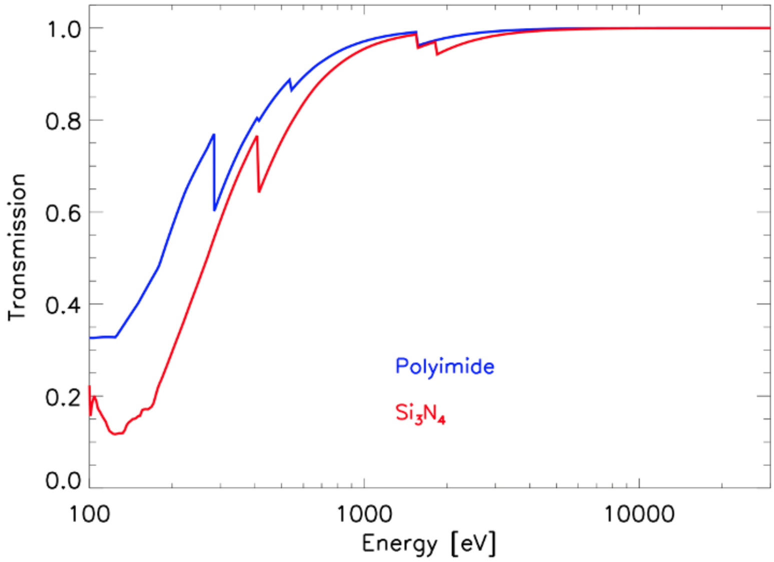

Fig. 81 - X-ray modelled transmission for two filters of Polyimide and Si_3N_4 both 50 nm thick coated with 30 nm of aluminum.

Filter samples have been manufactured within this contract with thickness as low as 20 nm supported by a fine Si mesh with diameters ranging from 10 up to ~50 mm. Filter samples of these materials have been subject to different environmental testing including proton irradiation, vibrations, acoustic load. Si_3N_4 have proven to be radiation hard to the doses well above the expected ones over the lifetime of a space mission. Medium size filter samples have also survived the acoustic load typical of an Ariane 5 launch, however more samples have proven to be very fragile under vibration testing. This evidences and the encountered difficulty to extend the manufacturing process of very thin membranes to larger diameters significant for Athena (up to 160 mm diameter) have suggested to concentrate more efforts on other materials of potential interest.

Polyimide/Al supported by Polyimide mesh

OXFORD Instruments Oy Finland has developed in the past a thin filter technology based on nanometric films of polyimide supported by a thick (~20 µm) photo-lithographic polyimide mesh. This technology has some advantages (e.g. no need for a glue between mesh and polyimide film, higher transparency at high energy) and disadvantages (poor thermal conductivity, poor RF attenuation at low frequencies < 6 GHz) with respect to the Athena filter baseline design based on polyimide films coated with aluminum supported by metallic meshes. UV/VIS/NIR attenuation and soft x-ray transmission are similar to the baseline material of Athena.

A few filter samples have been manufactured by OXFORD to investigate this technology. Filter samples of this technology have been manufactured by OXFORD both of small (diam ~ 10-15 mm), medium (square 82 mm x 82 mm, circular with diam. ~ 50 mm) and large sizes (circular with diam. = 160 mm). A few of these samples presented a few pinholes and small defects at the border of the filters. Medium size filters with polyimide thickness ~ 150 nm and mesh OA ~ 96% have been investigated in XAS at BESSY and full x-ray imaging at INAF-OAPA showing a quite good spatial uniformity. The maximum relative difference in transmission for the Al/polyimide/Al medium size samples (WFI LDA single quadrant OBF) measured at BESSY between two mapped points is about 9%, partly due to the variable intersection between the small x-ray beam spot and the mesh wires.

Medium size filter samples representative of a single quadrant of the WFI LDA OBF have passed the Ariane 5 launch qualification levels vibration tests, and have also been successfully tested in the acoustic environment inside the WFI filter wheel during a simulated Ariane 6 launch. Another similar sample was subject to thermo vacuum cycles showing no damages at the end of the tests. Differential pressure tests have been performed on single quadrants of the WFI LDA OBF, however, we have not been able to pressurize the filter samples likely because the joint between the inner and outer frame is not leak tight. A large size filter sample representative of an X-IFU FW OBF (diam. = 160 mm) has been manufactured at the end of the contract and delivered to Palermo in June 2020. We plan to perform vibro-acoustic tests on this sample in the coming months but unlikely before the FR given the present restrictions in travelling and access to laboratories.

The overall results of characterization performed on these filter technology is quite encouraging and suggests to further investigate these filter material in particular for applications as Optical Blocking Filters in Filter Wheels, such as those present on both the Athena X-IFU and WFI instruments, where large size thin filters are required while good thermal conductivity or RF attenuation is not a requirement.

CNT/Al

A few characterization tests have been also performed on preliminary samples of CNT/Al manufactured in the last months of the contract. The first batch of manufactured samples named "low density" coated with aluminum present a lower attenuation in the UV/VIS with respect to Polyimide/Al samples of similar thicknesses, this is partially due to the Al coating not being continuous and also because of a very significant aluminum oxidation also verified by XPS measurements we have performed at the BACH beamline in ELETTRA. Figure 82 shows the comparison between a measured x-ray transmission curve of a bare "low density" CNT, performed at BESSY on July 2020, and a modelled transmission of a 45 nm thick film of Polyimide (baseline for the Athena X-IFU thermal filters). These preliminary measurements shows that a significant gain in soft x-ray transmission can be obtained by the use of CNT thin films, provided that we are able to prove that these material can respond to the main performance and technical requirements for Athena.

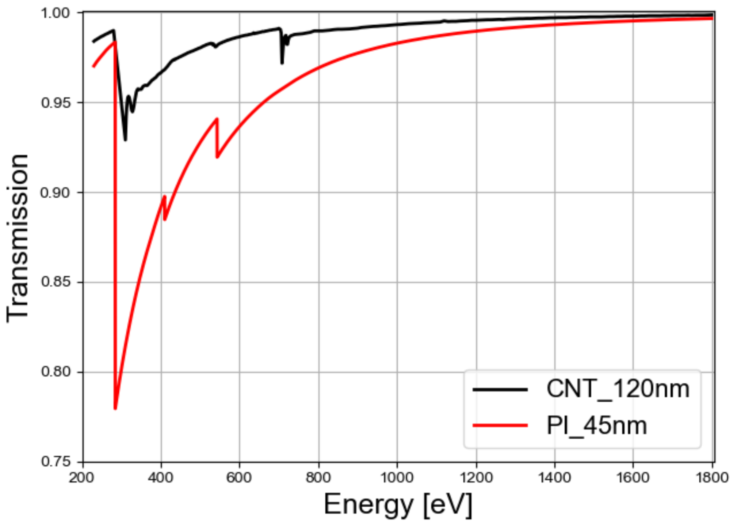

Fig. 82 - . Comparison between a measured x-ray transmission curve of a bare "low density" CNT, performed at BESSY on July 2020, and modelled transmission of a 45 nm thick film of Polyimide

New samples named "medium density" and "high density" with reduced network gaps thus providing a more uniform substrate for Al layer deposition have been manufactured recently, furthermore, passivating layers of AlN and ALD aluminum have been deposited on a few samples to reduce the aluminum oxidation. These new samples are currently being investigated in UV/VIS/NIR spectroscopy at UNIPA, XPS at BACH beamline in ELETTRA as part of the ongoing CCN.

Annex 1 includes the list of all filter samples manufactured and delivered to Palermo along the contract together with some relevant information on the samples and characterization tests performed.