Status of the EPIC thin and medium filters on-board XMM-Newton after more than 10 years of operation I: laboratory measurements on back-up filters

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Status of the EPIC thin and medium filters on-board XMM-Newton after more than 10 years of operation: 1) laboratory measurements on back-up filters

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ABSTRACT

After more than ten years of operation of the EPIC camera on board the X-ray observatory XMM-Newton, we have reviewed the status of its Thin and Medium filters by performing both laboratory measurements on back-up filters, and analysis of data collected in-flight.
We have selected a set of Thin and Medium back-up filters among those still available in the EPIC consortium, and have started a program to investigate their status by different laboratory measurements including: UV/VIS transmission, X-ray transmission, RAMAN IR spectroscopy, X-Ray Photoelectron Spectroscopy, and Atomic Force Microscopy. We report the results of the measurements conducted up to now, and point out some lessons learned for the development and calibration programs of filters for X-ray detectors in future Astronomy missions.

Keywords: X-rays: XMM-Newton, X-rays: instrumentation, X-rays: filters

1. INTRODUCTION

In order to fully exploit the performances of the European Photon Imaging Camera (EPIC)[1][2] on board the XMM-Newton X-ray satellite, three type of filters, depending on target source, can alternatively be used to prevent optical and UV light to reach the cooled CCD X-ray detectors. The Thin and Medium filters developed by MOXTEK Inc. in Orem, Utah, consist of a polyimide membrane coated with aluminum on one single side, while the thick ones, developed at the Max-Planck-Institut fur Extraterrestrische Physik (WE, Germany), consist of a Polypropilene membrane coated with tin and aluminum on one side and aluminum on the other side.

After more than 10 years of operation, the performances of the EPIC filters may have degraded due to oxidation, contamination, irradiation doses, fractures/holes, etc. Such degradation might introduce changes in:

- UV/VIS transmission: strong impact on sources with bright optical/UV counterpart;
- X-ray transmission: strong impact at low X-ray energies;
- Spatial homogeneity: strong impact on extended sources, or in presence of bright UV/VIS off-axis sources.

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No degradation of the in-flight EPIC filters has been reported to date, but no specific tests have been recently performed, neither laboratory measurements have been recently performed on back-up filters. For this reason, within a research contract by Italian Space Agency to support the XMM-Newton operation, we have started an investigation on the status of the Thin and Medium filters, whose development program has been under the responsibility of the Italian research team in the EPIC consortium [3][4]. In particular, we have conducted a set of laboratory measurements on back-up filters (this paper), and analysis of in flight observational data in a companion paper presented in this conference[5].

Following the experience gained in the calibration program of the Chandra HRC UV/Ion shields[6], the high energy resolution X-ray transmission curves of one Thin (G18) and one Medium (G19) EPIC filters have been measured at Bessy synchrotron in Berlin (Germany) to derive the fine structure features near the absorption edges (XANES and EXAFS) of the atomic elements constituent the filter material (C, N, O, Al). These measurements have been used to complement the tabulated imaginary part of the atomic scattering factors[7] in order to derive the transmission model of the EPIC Thin and Medium filters in the full energy range of interest [4].

UV/Vis transmission measurements in the range 190-1000 nm have been performed between May 1997 and July 2002 on one Thin (T4) and one medium (G12) EPIC filters to monitor their time stability[7]. The filters have been stored in dry Nitrogen between April 1997 and December 1999, and in vacuum, after the launch of Newton XMM, between January 2000 and July 2002, to follow a story as close as possible to the flight filters. Such measurements have shown an increase in transmission in the first year of the monitoring activity, followed by a stabilization. According to our model the change in transmission cannot be interpreted simply with an increase of the aluminum oxidation layer. Among possible alternative explanation are: a change in the polyimide that reduces the intensity of the characteristic absorption bands at about 2500 Å, or an increase in the surface roughness of the aluminum that reduces the reflected component thus increasing the transmitted one.

In this paper we report the preliminary results of an on-going experimental program aimed at investigating the status of the EPIC filters after more than 10 years of mission operation, and more than 16 years from production. This paper is dedicated to laboratory measurements on back-up filters while a companion paper presented in this conference is dedicated to investigating the status of the in-flight filters through the analysis of observational data.

The experimental work conducted on back-up filters will also allow us to evaluate the capabilities of different experimental techniques to characterize filters made of a metallized thin plastic foil, for the development and calibration programs of focal plane filters for future high energy Astrophysics missions. In the next sections we briefly describe the experimental techniques used, the measurements performed and the preliminary results of the investigation.

2. FILTER SELECTION AND STORAGE

The EPIC and Medium filters manufactured by MOXTEX consist of a thin film of polyimide, with nominal thickness of 1600 Å, coated with a single layer of aluminum whose nominal thickness is 400 Å for the Thin and 800 Å for the Medium. The polyimide thin films are produced by spin-coating of a polyamic acid (PAA) solution obtained by dissolving two precursor monomers (an anhydride and an amine) in an organic polar solvent. For the EPIC Thin and Medium filters the two precursors are the Biphenyldianhydride (BPDA) and the p-Phenyldiamine (PDA) (Dupont PI-2610), and the solvent is N-methyl-2-pyrrolidone (NMP) and Propylene Glycol Monomethyl Ether (Dupont T9040 thinner). To convert the PAA into polyimide, the solution is heated up to remove the NMP and to induce the imidization through the evaporation of water molecules. The film thickness is controlled by spin coating parameters, PAA viscosity, and curing temperature[11]. The polyimide thin membrane is attached with epoxy onto a transfer ring and the aluminum is evaporated in a few runs, distributed over two-three days, each one depositing a metal layer of about 200 Å thickness.

The EPIC Thin and Medium flight qualified filters have been manufactured during a period of one year, from January ’96 to January ’97. Table 1 lists the full set of flight-qualified filters (Flight Model and Flight Spare) delivered to the EPIC consortium, together with their most relevant parameters. Along with the production of the flight qualified filters, prototypes, and qualification filters, not included in this list, have been manufactured and tested for the construction of

the filter transmission model and to assess the stability in time of the Optical/UV transparency (opacity). Among these qualification filters are T4, G12, G18, and G19 that have been previously mentioned.

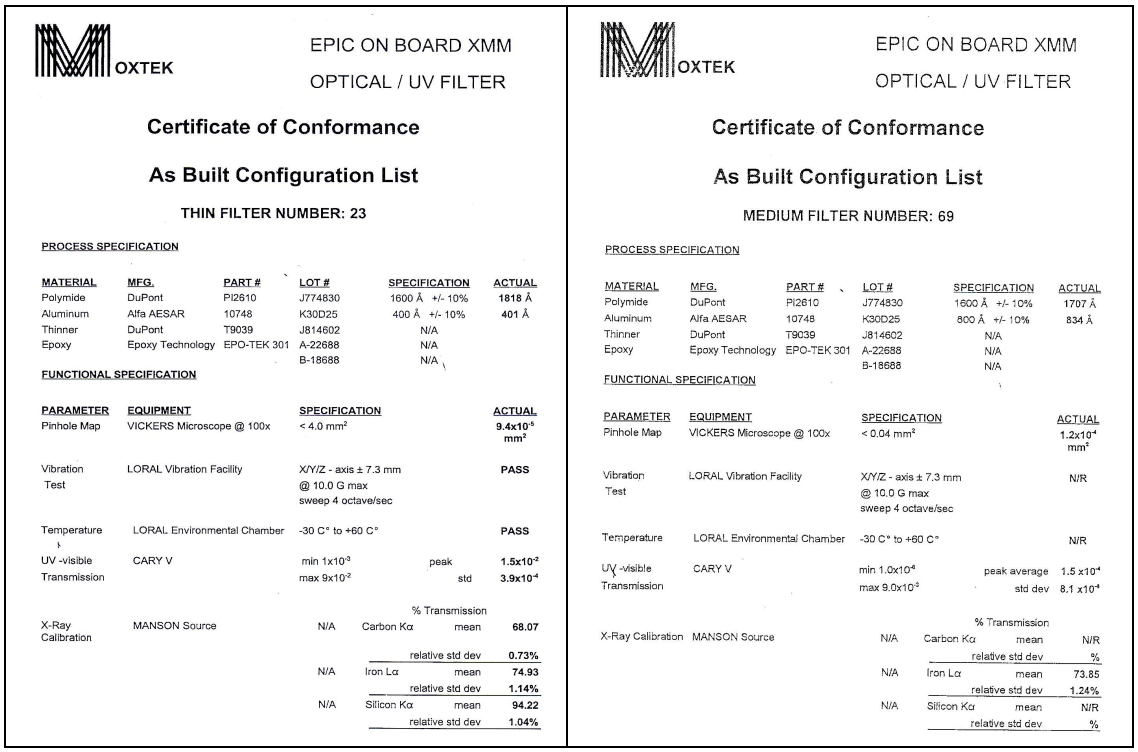
Table 1 – List of flight qualified Medium and Thin EPIC filters.

Num.	Type		Thickness (A) polyimide	Al	Production Date	Pinhole area ($x10^{-4}$ mm^2)	Transmission UV/VIS*	C Kα	Fe Lα	Si Kα
01	T	FM MOS1	1818	406	08/1996	2.7	1.1E-2	0.67	0.75	0.93
02	T	FS	1817	391	08/1996	0.51	2.3E-2	0.67	0.77	0.92
03	M	FS	1705	825	07/1996	2	1.1E-4	0.47	0.74	0.91
04	**M**	**FS**	**1705**	**825**	**07/1996**	**1.4**	**9.1E-6**	**0.49**	**0.74**	**0.89**
05	M	FM MOS2	1705	825	07/1996	4.7	5.4E-5	0.47	0.75	0.90
06	T	FS	1778	413	08/1996	0.69	1.1E-2	0.66	0.77	0.95
07	**T**	**FS**	**1737**	**391**	**08/1996**	**0.24**	**1.2E-2**	**0.67**	**0.78**	**0.91**
08	T	FS PN	1778	413	08/1996	0.38	1.3E-2	0.66	0.77	0.94
11	T	FS PN	1723	406	09/1996	0.12	1.2E-2	0.67	0.78	NA
12	T	FM PN	1818	406	08/1996	0.095	1.1E-2	0.66	0.78	0.94
13	T	FS	1778	413	08/1996	1.9	1.1E-2	0.67	0.77	0.94
14	T	FM MOS1	1778	404	08/1996	0.67	9.6E-3	0.66	0.77	0.96
15	**M**	**FS**	**1843**	**830**	**08/1996**	**0.83**	**8.4E-5**	**0.48**	**0.73**	**0.90**
17	M	FS	1626	834	08/1996	1.5	6.9E-5	0.49	0.71	0.89
18	T	FS MOS	1723	406	09/1996	0.11	1.2E-2	0.66	0.77	0.92
21	T	FM MOS2	1778	404	08/1996	5.1	1.1E-2	0.66	0.76	0.94
23	**T**	**FS**	**1818**	**401**	**08/1996**	**0.94**	**1.5E-2**	**0.68**	**0.75**	**0.94**
24	T	FM MOS2	1779	398	07/1996	23	1.3E-2	0.67	0.77	0.95
32	T	FS	1737	400	08/1996	0.44	1.2E-2	0.67	0.76	0.92
33	T	FS MOS	1800	388	08/1996	0.42	1.3E-2	0.66	0.74	0.93
40	T	FM PN	1723	406	09/1996	0	1.1E-2	0.66	0.77	NA
63	M	FS	1647	859	10/1996	0.64	1.2E-4	NA	0.74	NA
65	M	FM MOS1	1647	859	10/1996	0.31	1.3E-4	NA	0.72	NA
66	M	FM PN	1707	834	10/1996	0	1.2E-4	NA	0.75	NA
67	M	FS MOS	1707	834	10/1996	0.31	1.2E-4	NA	0.73	NA
68	M	FS PN	1707	834	10/1996	0.67	1.7E-4	NA	0.76	NA
69	**M**	**FS**	**1707**	**834**	**10/1996**	**1.2**	**1.5E-4**	**NA**	**0.74**	**NA**

* Peak value in the wavelength band 200-800 nm

We have identified all back-up Thin and Medium EPIC filters still available in the laboratories of INAF-IASF-MI and INAF-OAPA, we have collected the MOXTEX documentation, and have visually inspected their integrity. The five filters, stored inside their original shipping containers, namely 4, 7, 15, 23, and 69 are identified in bold in table 1.

We have arbitrarily chosen to start the verification study on the two filters 23 and 69 representative, respectively, of the two types Thin and Medium. Figure 1 provides the main characteristics of the two filters reported in the MOXTEX datasheets. The Thin filter 23 has a measured thickness of polyimide and aluminum, respectively, of 1818 Å and 401 Å, and a peak UV transmission in the 200-800 nm wavelength range of $1.5 \cdot 10^{-2}$. The same parameters for the Thick filter 69 are: 1787 Å, 834 Å, and $1.5 \cdot 10^{-4}$.



MOXTEK

EPIC ON BOARD XMM

OPTICAL / UV FILTER

Certificate of Conformance

As Built Configuration List

THIN FILTER NUMBER: 23

PROCESS SPECIFICATION

MATERIAL	MFG.	PART #	LOT #	SPECIFICATION	ACTUAL
Polymide	DuPont	PI2610	J774830	1600 Å +/- 10%	1818 Å
Aluminum	Alfa AESAR	10748	K30D25	400 Å +/- 10%	401 Å
Thinner	DuPont	T9039	J814602	N/A	
Epoxy	Epoxy Technology	EPO-TEK 301	A-22688	N/A	
			B-18688	N/A	

FUNCTIONAL SPECIFICATION

PARAMETER	EQUIPMENT	SPECIFICATION		ACTUAL
Pinhole Map	VICKERS Microscope @ 100x	< 4.0 mm²		9.4x10⁻⁵ mm²
Vibration Test	LORAL Vibration Facility	X/Y/Z - axis ± 7.3 mm @ 10.0 G max sweep 4 octave/sec		PASS
Temperature	LORAL Environmental Chamber	-30 C° to +60 C°		PASS
UV -visible Transmission	CARY V	min 1x10⁻³	peak	1.5x10⁻²
		max 9x10⁻²	std	3.9x10⁻⁴
			% Transmission	
X-Ray Calibration	MANSON Source	N/A Carbon Kα	mean	68.07
			relative std dev	0.73%
		N/A Iron Lα	mean	74.93
			relative std dev	1.14%
		N/A Silicon Kα	mean	94.22
			relative std dev	1.04%

MOXTEK

EPIC ON BOARD XMM

OPTICAL / UV FILTER

Certificate of Conformance

As Built Configuration List

MEDIUM FILTER NUMBER: 69

PROCESS SPECIFICATION

MATERIAL	MFG.	PART #	LOT #	SPECIFICATION	ACTUAL
Polymide	DuPont	PI2610	J774830	1600 Å +/- 10%	1707 Å
Aluminum	Alfa AESAR	10748	K30D25	800 Å +/- 10%	834 Å
Thinner	DuPont	T9039	J814602	N/A	
Epoxy	Epoxy Technology	EPO-TEK 301	A-22688	N/A	
			B-18688	N/A	

FUNCTIONAL SPECIFICATION

PARAMETER	EQUIPMENT	SPECIFICATION		ACTUAL
Pinhole Map	VICKERS Microscope @ 100x	< 0.04 mm²		1.2x10⁻⁴ mm²
Vibration Test	LORAL Vibration Facility	X/Y/Z - axis ± 7.3 mm @ 10.0 G max sweep 4 octave/sec		N/R
Temperature	LORAL Environmental Chamber	-30 C° to +60 C°		N/R
UV -visible Transmission	CARY V	min 1.0x10⁻⁴	peak average	1.5 x10⁻⁴
		max 9.0x10⁻³	std dev	8.1 x10⁻⁴
			% Transmission	
X-Ray Calibration	MANSON Source	N/A Carbon Kα	mean	N/R
			relative std dev	%
		N/A Iron Lα	mean	73.85
			relative std dev	1.24%
		N/A Silicon Kα	mean	N/R
			relative std dev	%

Figure 1. Certificate of conformance of filters 23 and 69 issued by MOXTEX.

A small vacuum chamber has been set-up at the XACT facility of INAF-OAPA[9] to store the two filters during the investigation period. The chamber is evacuated by a scroll pump providing a clean vacuum in the high 10^{-2} mbar range. Figure 2, shows pictures of the vacuum chamber, the vessel to hold the filters inside the vacuum chamber, and the two inspected filters 23 an 69.

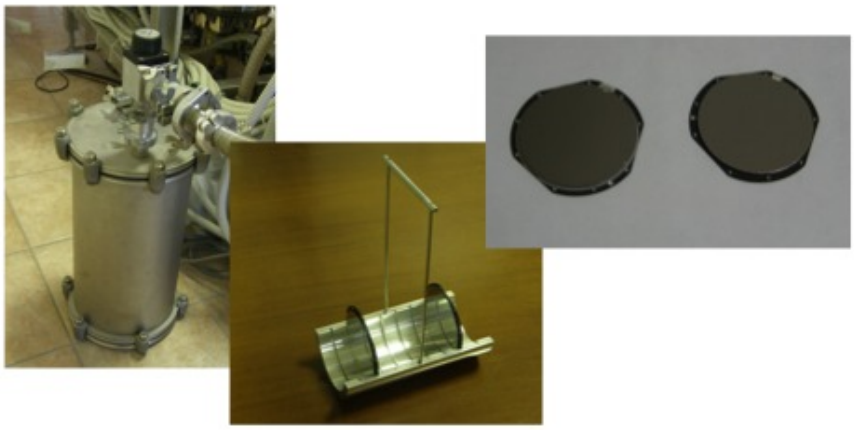


Figure 2. From left to right: the small vacuum chamber set-up at the XACT facility of INAF-OAPA to host the two EPIC filters 69 and 23; the vessel for safe handling of the filters in/out of the chamber; the two filters on a laminar flow bench

Vacuum and venting procedures are performed very slowly to prevent turbulent motion of residual particles in the chamber to damage the filters. The samples are kept in atmosphere only during the measurement sessions, and transported inside the MOXTEX original shipping containers from/to the different laboratories.

3. LABORATORY MEASUREMENTS

3.1 UV/VIS Transmission

UV/VIS transmission measurements in the wavelength range 190-900 nm have been performed every three months, from March 2011 to July 2013 (10 measurements), using a JASCO V560 spectrophotometer. The first measurement session was carried out on filters previously stored inside the MOXTEX containers. The second session was conducted after the filters have been maintained in vacuum for about a month. Since then, filters have been always kept in vacuum.

The measured transmission curves are shown in Figure 3 together with two set of measurements performed on similar filters, namely T4 (Thin) and G12 (Medium), in September 1997 and July 2002 in a previous monitoring program[7].

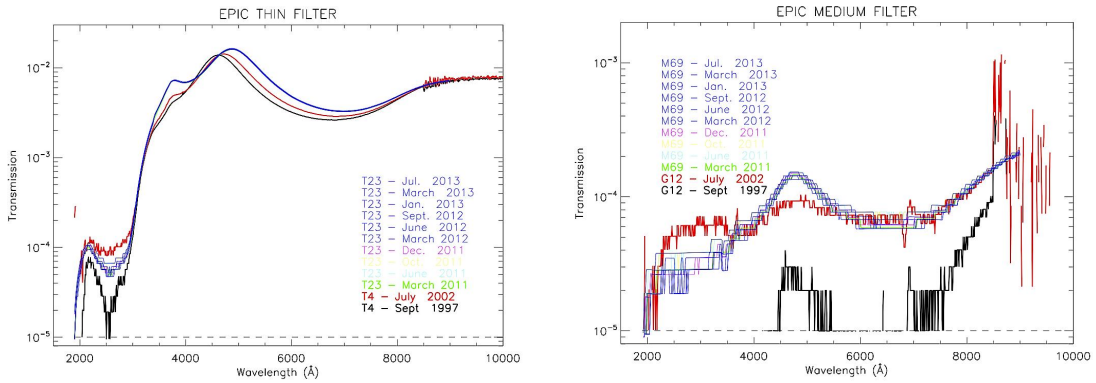


Figure 3. UV/VIS transmission curves measured on the EPIC filters 23 (Thin, left panel) and 69 (Medium, right panel). Superimposed are also the transmission curves measured on similar filters in a previous monitoring program.

All measurements performed on both filters 23 and 69 are showing no evidence of change with time during the over two years period of monitoring. The first measurement performed when the filters where still stored in the original MOXTEX containers are indistinguishable from the other measurements performed after the filters have been stored in vacuum.
The measured transmission curves are consistent with the values reported in the MOXTEX datasheet (Figure 1) and in good agreement with the transmission curves measured in 2002 for G12 and T4 filters, when the performance of those filters had already stabilized. The small shift of the resonance peak at about 4500 Å of the Thin filter 23 with respect to T4 is due to a slightly different nominal thickness of polyimide for the two compared filters, namely 1818 Å for T23, and 1656 Å for T4. A small difference is also observed at about 2500 Å in the transmission curves of both Thin and Medium filters with respect to the filters measured in 2002. The origin of these small discrepancies will be further investigated.

3.2 X-ray Transmission

X-ray transmission measurements will be completed in fall 2013 and are not here reported. A multi anode Manson model 5 electron impact X-ray source is used to produce X-rays dominated by the characteristic fluorescent K and L lines of different anodes including B, Be, C, Mg, Al, Si, Ti, Cr, Fe, Cu. A transmission grating monochromator is used to select the lines and remove most of the Bremsstrahlung continuum. X-rays are detected by a microchannel plate and a gas flow proportional counter, as done in previous calibration programs[10]. We plan to derive the transmission curve vs. energy as well as to perform X-ray shadowgraphs to investigate the spatial uniformity of the filters.

3.3 Raman IR Spectroscopy

The formation of Al-C and Al-O bonds between a subatomic monolayer of aluminum and the polyimide film have been observed experimentally using the High Resolution Electron Energy Loss (HREEL) technique that induces vibrational bands[12]. Based on this experimental evidence we have decided to investigate the occurrence of interfacial polymer-Al bonds in our filters using the micro-Raman technique.

Micro-Raman measurements were carried out at room temperature using a Bruker-Senterra micro-Raman spectrometer equipped with a 532 nm laser diode excitation operated at a power of 20 mW. Confocal measurements were done in the

range 50÷4478 cm^{-1} with a spectral resolution between 9, and 15 cm^{-1}. Scans perpendicular to the filter surfaces were also executed to highlight specific spectral features. Furthermore, different points on a given surface were examined in order to evaluate the repeatability of the obtained spectra. Both the polyimide and the aluminum side of the filters 23 (T) and 69 (M) have been investigated. The measured spectra obtained by illuminating the Al side are dominated by a strong continuum, likely luminescence, which does not allow to detect IR lines. The measured Stokes Raman spectra, obtained illuminating the polyimide side of the filters, are shown in Figure 4.

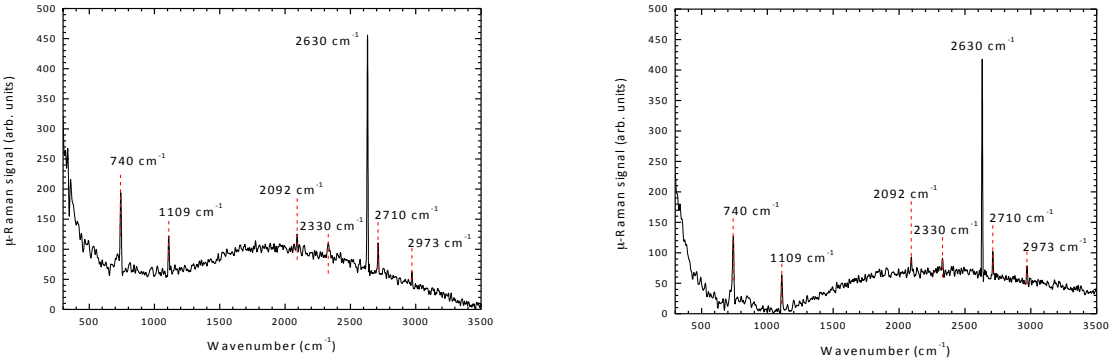


Figure 4. Raman spectra of the Epic Thin (left panel) and Medium filters (right panel) obtained in confocal geometry

The observed spectra are very repeatable on the same filter, and they present the same major features on both Thin and Medium filters. However, the main characteristic features that are identified in the literature on similar materials, mainly the C-C, C-O, and C-N stretching modes between 1100 cm^{-1} and 1800 cm^{-1}, are not present in our spectra[13][14][15]. This can be likely due to one of the following reasons: 1) the thickness of the polyimide is too small and the actual investigated area is the interface, or 2) the laser power is too high and degrades the polymer. On the other hand, we find some clear features that we have not yet been able to identify unambiguously. We plan to continue this investigation by operating the laser at lower power and by using new samples of polyimide with and without aluminum coating.

3.4 Atomic Force Microscopy

Tapping mode amplitude modulation AFM measurements[16][17][18][19] were performed by a Multimode V (Veeco Metrology) scanning probe microscope equipped with a conventional piezo-scanner and a four-segment photo-detector for cantilever deflection monitoring. PointProbe®Plus Silicon-SPM-probes were used with aluminum backside reflex coating, resonance frequency about 300 KHz, and tip apical radius 5÷10 nm. All the acquired images had a resolution of 512×512 pixels, and were obtained with a tip velocity on the surface of about 2 μm/s. Quantitative information on images were extracted using the GWYDDION software[20].

The measurement apparatus does not allow to perform a direct measurement on one of the large format EPIC filters, furthermore, there would be a significant risk of damage if the measurement was performed on a free-standing thin filter. For this reason we have conducted our AFM investigation on a small fragment of the Medium filter G12 that was investigated during the previous aging monitoring program between 1997 and 2002, and was unfortunately damaged while handling it in a transfer from the vacuum chamber. Two samples of few square mm size have been prepared, one with the aluminum side facing up and the other one with the polyimide side facing up. The sample with aluminum facing up, similarly to T23 and M69 filters, show a slightly yellowish color, likely due to oxidation, well distinct from the polyimide side that presents a shining silver color.

Figure 5 and 6 show images and Z-profiles of the AFM measurements performed on the two samples with aluminum facing up and polyimide facing up, respectively. The actual image size is 2×2 μm^2, while the full Z scale is ± 25 nm for the aluminum and ± 2.5 nm for the polyimide. As it is evident the aluminum, which has been deposited by evaporation, presents a granular structure with typical size of the particles of few tens nm. A reliable estimate of the particle size, which takes into account the size of the tip, gives an average diameter of the aluminum particles of 50 nm with a statistical variance of the distribution of about 10 nm. The polyimide is much smother than aluminum.

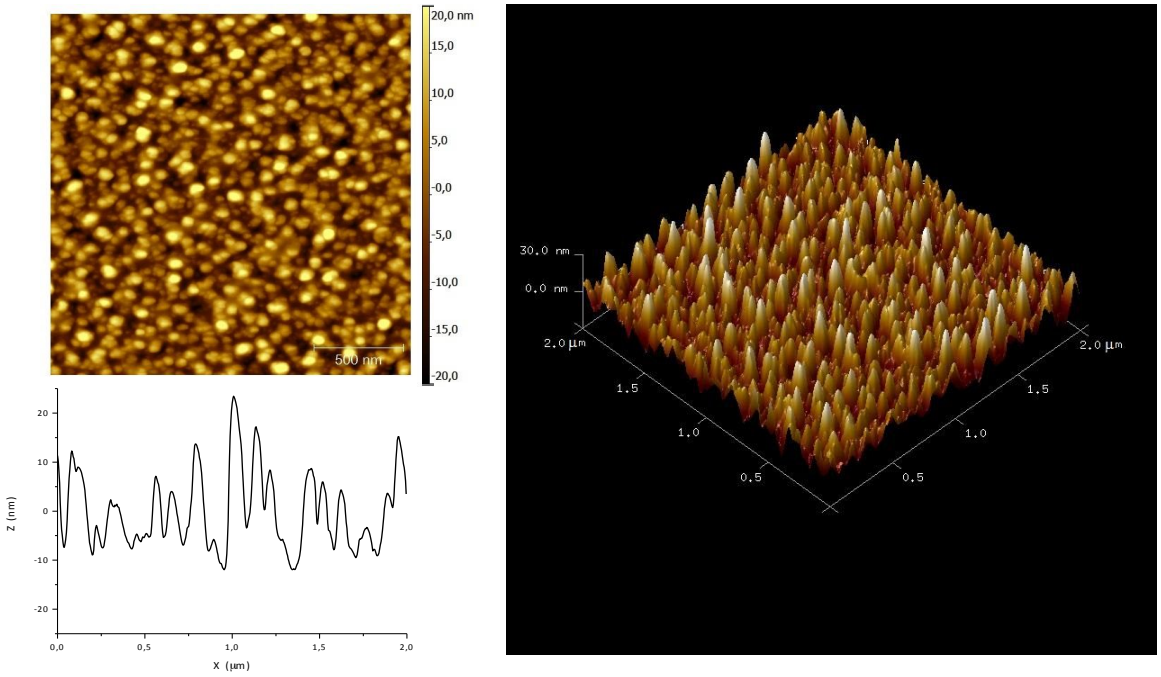


Figure 5. 2-D (top-left) and 3-D (right) AFM image of the aluminum side of filter G12. The image has a resolution of 512×512 pixels. The Z-profile (bottom-left) has been measured on the central horizontal line of the AFM image.

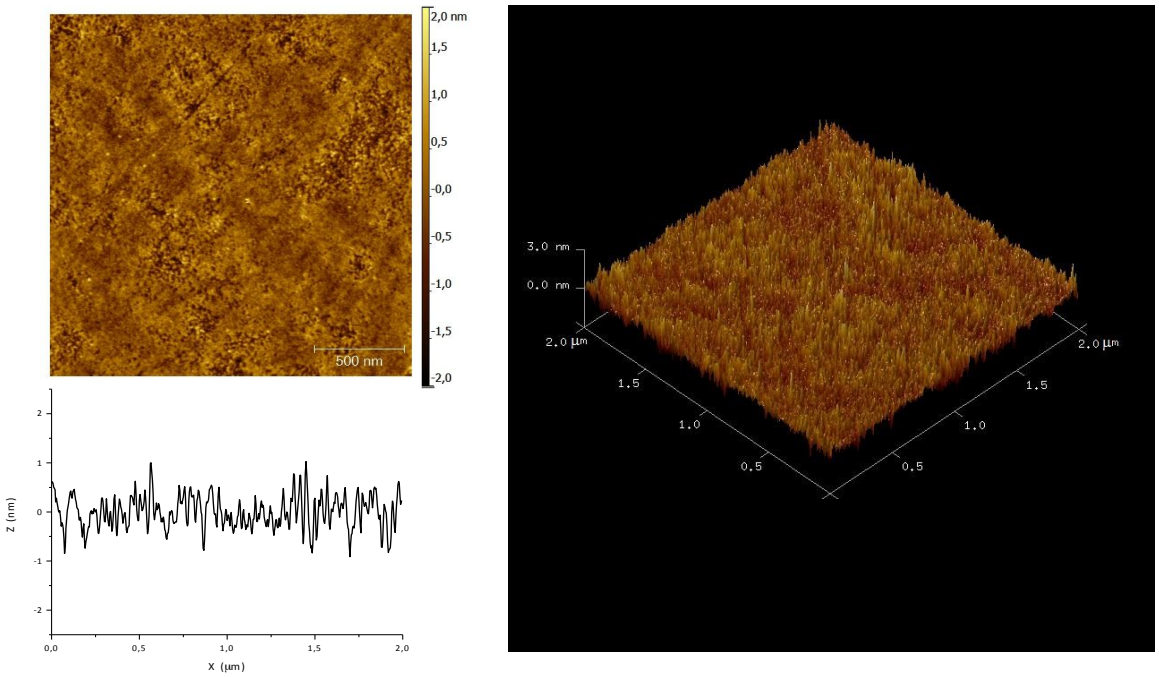


Figure 6. 2-D (top-left) and 3-D (right) AFM image of the polyimide side of filter G12. The image has a resolution of 512×512 pixels. The Z-profile (bottom-left) has been measured on the central horizontal line of the AFM image.

3.5 X-Ray Photoelectron Spectroscopy

The X-ray photoelectron spectroscopy analyses were performed with a VG Microtech ESCA 3000 Multilab, using the non-monochromatic Al Kα source (1486.6 eV) run at 14 kV and 15 mA. The pass energies of 50 eV and of 20 eV were used in the hemispherical electron energy analyser for the survey and the individual peak energy regions, respectively. As it is the case for the AFM measurements, XPS investigation has been performed on small fragments of the G12 EPIC Medium back-up filter. Two samples were mounted with double-sided adhesive tape onto a stainless-steel holder. In one case the membrane was mounted exposing the aluminum side (side up), in another case exposing the polyimide side (side down). Binding energies were referenced to the C 1s binding energy of adventitious carbon set at 285.1 eV. The software provided by VG was used for peak analyses and for the calculation of the atomic concentrations. The precision on the binding energy and on the atomic percentage values was respectively ± 0.15 eV, and ± 10%.

The thickness of the oxide film formed over the aluminum metal was calculated using the formula developed by Strohmeier[21] and Carlson [22] defined as:

$$d = \lambda_{ox} \sin\vartheta \ln\left[\frac{N_m \lambda_m I_{ox}}{N_{ox} \lambda_{ox} I_m} + 1\right] \qquad (1)$$

where θ is the photoelectron take off angle which in the present instrument configuration is 90°, I_{ox} and I_m are the percentage areas of the oxide and metal peaks, N_m and N_{ox} are the volume densities of the metal atoms in the metal and oxide, respectively, and λ_m and λ_{ox} are the electron inelastic mean free path (IMFP) of the metal and oxide, respectively, obtained from the kinetic energies of the photoelectrons according to the algorithm of Penn [23].

Figure 7 shows the two wide survey scans, characterized by a low energy resolution and lower intensity, of the two sides of the filter. The side up spectrum contains mainly the peaks due to aluminum (Al2p and Al2s), carbon (C1s) and oxygen (O1s and O Auger KLL). Some minor components due to the silicon from the adhesive tape are also present. The side down spectrum contains hardly visible Al peaks and an additional N1s peak at 400.4 eV typical of an imide nitrogen.

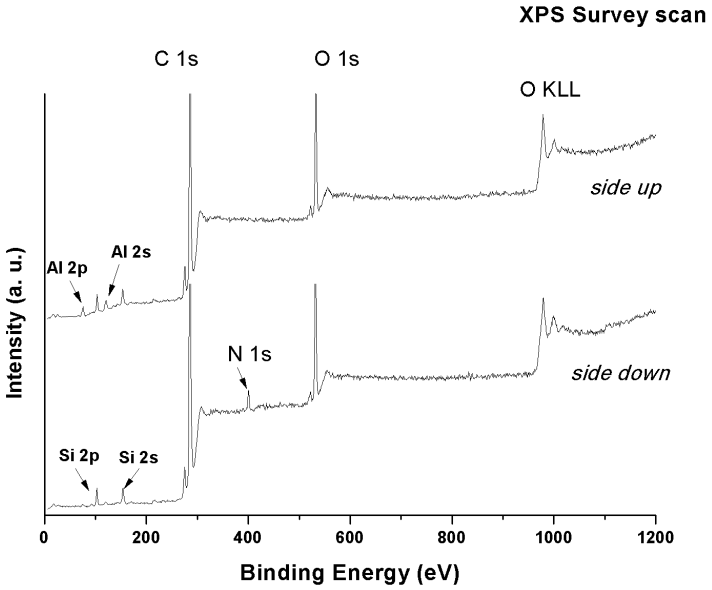


Figure 7. XPS Survey scans of the two different sides of the filter sample.

Figure 8 shows the experimental Al2p region spectra with the fitted curves of both sample sides. Each spectrum contains the two components, one due to metallic aluminum at 72.7 eV, and the other due to the amorphous aluminum oxide at 75. 5 eV. In the case of the side down sample the intensity of the aluminum peak with the two components is rather low since aluminum is underneath the polyimide film 160 nm thick. It is likely that attaching the very thin and fragile sample onto the bi-adhesive tape a small fraction of it is wrapped, and thus small portions of the aluminum coating are facing-up. Using the formula given above and the peak areas of the oxide and metal components, the thickness of the oxide film was estimated in 5.9 nm in both cases.

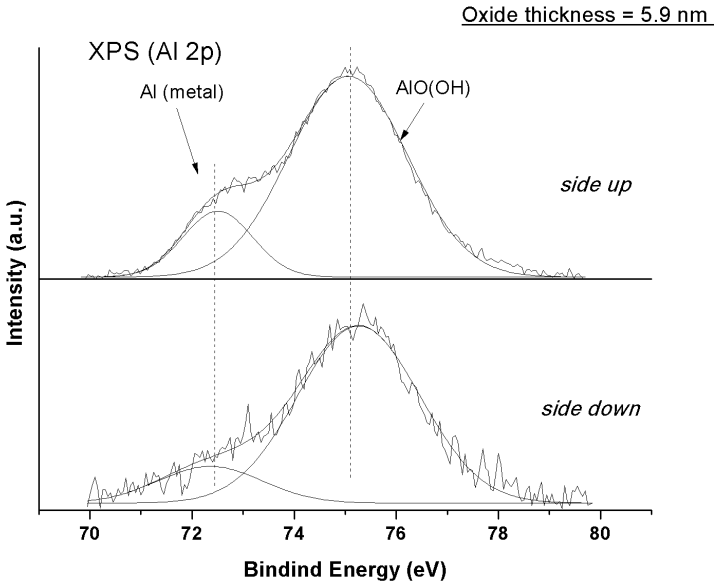


Figure 8. XPS scan of the Al 2p binding energy region for the two different sides of the filter sample.

4. SUMMARY AND CONCLUSIONS

In this paper we have reported the preliminary results of an on-going experimental program aimed at investigating the status of the EPIC Thin and Medium filters after more than 10 years of operation of the XMM-Newton observatory in Space, and more than 16 years from production of the filters by MOXTEX.
We have selected two representative filters of Thin and Medium types, flight qualified and stored as spares in the laboratories of the EPIC consortium. The two filters, namely 23 (Thin) and 69 (Medium), were initially stored in their original shipping container built by MOXTEX. A small chamber has been set-up at the XACT facility of INAF-OAPA to store the two filters in vacuum during the investigation.

In order to verify the status of the two filters, we have used different experimental techniques, mainly non-destructive, not all of them commonly used for the investigation of thin X-ray filters, but each one potentially capable of providing useful information, namely:

1. UV/VIS transmission measurements provide direct information on the out of band opacity, which is one of the critical science requirement to maintain a low background of the CCD detectors. These measurements provide also some hints on the time stability of the surface aluminum oxidation;

2. X-Ray transmission measurements provide a direct verification of the filters response curve in the energy range where the EPIC experiment has been designed to be sensitive;
3. IR Raman Spectroscopy can be useful to identify and monitor bonds between Al and C, O, and N at the interface between polyimide and aluminum as well as provide structural information on the polymer;
4. Atomic Force Microscopy allows to measure the surface roughness of the filter and turns out to be particularly useful to determine the characteristic size of the granules of metal deposited onto the polymer membrane by evaporation or sputtering, which may affect the oxidation process, and the out of band opacity.
5. The X-ray Photoelectron Spectroscopy is a very sensitive technique of surface chemical analysis, which can be used to determine the amount of oxide of aluminum or other metal coatings, as well as identify the presence of surface contamination.

The UV/VIS transmission measurements performed on both filters 23 and 69 are showing no evidence of change with time during the over two years period of monitoring. The first measurement performed on March 2011, when the filters where still stored in the original MOXTEX containers, are indistinguishable from the other measurements performed after the filters have been stored in vacuum. The measured transmission curves are in good agreement with the transmission curves measured in 2002 for similar filters.
The RAMAN IR spectroscopy needs further measurements to be performed in order to clearly identify the spectral features that are present and repeatable in both filter samples. The potentials of the technique are, however, evident.
The AFM measurements have shown that the evaporated aluminum is structured in granules with average diameters of 50 nm and a typical height, as derived from linear profiles, between 10 and 30 nm. These dimensions are of the same scale size as the nominal thickness of aluminum in the Thin (40 nm) and Medium (80 nm) filters.
The APS measurements allowed to quantify the thickness of the surface aluminum oxide which turns out to be 5.9 nm ± 10%. This result is in good agreement with the aluminum oxide thickness derived from the analysis of high resolution X-ray transmission measurements performed at Synchrotron facilities[6].

In conclusion, the results obtained up to now on back-up filters stored in laboratory confirm that the Thin and Medium filters built by MOXTEX for the EPIC experiment on-board the XMM-Newton mission are still in good shape after 16 year from production. They are fully compliant with the scientific requirements, and not significantly modified since the previous measurement campaigns. Aluminum coated polyimide filters are robust and reliable in time confirming that such materials are presently still one of the best choices for future X-ray experiments in Space missions.

We point out that none of the explored techniques provide information on the aging of the mechanical strength of the filters, which is also a very critical parameter in the operation in Space for long duration missions. Mechanical tests are not trivial on such fragile filters and are likely to be destructive, for this reason they should have been properly planned in advance with the construction of a set of small size witness samples of the flight filters specifically built for the purpose. We can only say that the filters T23 and M69, investigated in this work, have been subject in these two years to a few tens of vacuum/vent cycles, and have been transported many times from/to different laboratories to conduct experimental tests showing no mechanical failure.

We plan to further investigate the potentials of these experimental techniques in the near future on other samples of thin filters for X-ray detectors, in particular, samples with bare polyimide, or samples with freshly evaporated aluminum. The results of this investigation will allow us to better plan the development programs of filters for future high energy Space missions such as those presently under study for the LAD detector of LOFT[24], an X-Ray timing mission presently in phase-A study in the European Space Agency Cosmic Vision program.

Finally, we point out that all of these investigated experimental techniques, used to test the performance and status of the thin filters, are more useful if measurements are performed not just ones, but at different phases during the life of filters stored in properly controlled environments.

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REFERENCES

[1] Turner M.J.L., et al., (2001). The European Photon Imaging Camera on XMM-Newton: The MOS cameras, Astronomy & Astrophysics, vol. 365, p. L27-L35, doi: 10.1051/0004-6361:20000087

[2] Strüder L., et al. (2001). The European Photon Imaging Camera on XMM-Newton: The pn-CCD camera, Astronomy and Astrophysics, v.365, p. L18-L26, doi: 10.1051/0004-6361:20000066

[3] La Palombara N., Musso C., Conte M., Barbera M. (1996). The filters for EPIC: Optimized design. Astrophysics and Space Science, vol. 239, p. 281-296, doi: 10.1007/BF00645782

[4] Villa G.E, Barbera M, Collura A, La Palombara N, Musso C, Serio S, Stillwell R, Tognon P, Turner C (1998). The Optical/UV Filters for the EPIC experiment, IEEE Transactions on Nuclear Science, vol. 45, N.3, p. 921-926, doi: 10.1109/23.682670

[5] Gastaldello F., Barbera M., Collura A., La Palombara N., Lo Cicero U., Tiengo A. (2013). Status of the EPIC thin and medium filters on-board XMM-Newton after more than 10 years of operation: II - analysis of in-flight data, Proc. SPIE 8859-41, this conference.

[6] Barbera M., Collura A., Dara A (1996). Calibration of the AXAF-HRC UV/Ion shields at Osservatorio Astronomico di Palermo G.S.Vaiana: III - Synchrotron measurements of XANES in aluminum coated Lexan film samples, Proc. SPIE, 2808, 120-133

[7] Henke B.L., Gullikson E.M., and Davis J.C. (1993). X-Ray Interactions: Photoabsorption, Scattering, Transmission, and Reflection at E = 50-30,000 eV, Z = 1-92, Atomic Data and Nuclear Data Tables, Volume 54, Issue 2, p. 181-342, doi: 10.1006/adnd.1993.1013.

[8] Barbera M, Collura A, Artale A, Varisco S, Peres G, Sciortino S, Serio S, Villa G E (2003). Monitoring the stability of thin and medium back-up filters of the Newton-XMM EPIC camera, Proc. SPIE, 4851, 264-269, doi: 10.1117/12.461592.

[9] Barbera M, Candia R, Collura A, Di Cicca G, Pelliciari C, Sciortino S, Varisco S. (2006). The Palermo XACT facility: a new 35 m long soft x-ray beam-line for the development and calibration of next-generation x-ray observatories, Proc. SPIE, 6266, 3F, doi: 10.1117/12.673004.

[10] Barbera M, Artale A, Candia R, Collura A, Lullo G, Peres G, Perinati E, Varisco S, Bookbinder J A, Cheimets P N, Cosmo M L, DeLuca E E, Golub L, Weber M A (2004). Calibration of the XRT-SOLARB flight filters at the XACT facility of INAF-OAPA, Proc. SPIE, 5488, 423-439, doi: 10.1117/12.553127

[11] S. Diaham, M.L. Locatelli and R. Khazaka (2012). BPDA-PDA Polyimide: Synthesis, Characterizations, Aging and Semiconductor Device Passivation, in "High Performance Polymers: Polyimides Based - From Chemistry to Applications", ed. by Marc Jean Medard Abadie, doi: 10.5772/53994

[12] J. J. Pireaux , M. Vermeersch , C. Grégoire , P. A. Thiry , R. Caudano, and T.C. Clarke (1988). The aluminum-polyimide interface: An electron-induced vibrational spectroscopy approach, J. Chem. Phys. 88, 3353, doi: 10.1063/1.453930

[13] Jason J. Ge, Gi Xue, Fuming Li, Kevin W. McCreight, Shy-Yeu Wang, Frank W. Harris, Stephen Z. D. Cheng, Xiaowei Zhuang, Seok-Choel Hong, Y. R. Shen (1998). Surface studies of Polyimide thin films via surface-enhanced Raman scattering and second harmonic generation, Macromol. Rapid Commun. 19, 619–623, doi: 1022-1336/98/1212–0619

[14] X. J. Gu (1993). Raman spectroscopy and the effects of ultraviolet irradiation on polyimide film, Appl. Phys. Lett. 62 (13), 1568-1570, doi: 10.1063/1.108643

[15] John P. LaFemina , G. Arjavalingam , and G. Hougham (1989). Electronic structure and ultraviolet absorption spectrum of polyimide, J. Chem. Phys. 90, 5154-5160, doi: 10.1063/1.456558

[16] G. Binning, C. F. Quate, and C. Gerber, Atomic Force Microscope (1986). Atomic Force Microscope, Phys. Rev. Lett. 56, 930, doi: 10.1103/PhysRevLett.56.930

[17] R. Garcia, R. Perez. (2002). Dynamic atomic force microscopy methods, Surf. Sci. Rep., 47, 197-301

[18] F.J. Giessibl (2003). Advances in atomic force microscopy, Rev. Mod. Phys. 75, 949-983, doi: 10.1103/RevModPhys.75.949

[19] Y. Seo and W. Jhe (2008). Atomic force microscopy and spectroscopy, Rep. Prog. Phys. 71, 016101, doi: doi:10.1088/0034-4885/71/1/016101

[20] http://gwyddion.net/ (2010)

[21] B. R. Strohmeier (1990). An ESCA Method for Determining the Oxide Thickness on Aluminum Alloys, Surf. Interface Anal., 15, 51-56, doi: 10.1002/sia.740150109

[22] T. A. Carlson, G. E. Mc. Guire, J. (1972/1973). Study of the x-ray photoelectron spectrum of tungsten-tungsten

oxide as a function of thickness of the surface oxide layer, Journal of Electron Spectroscopy and Related Phenomena, vol. 1, issue 2, 161-168, doi: 10.1016/0368-2048(72)80029-X
[23] D. R. Penn (1987). Electron Mean-Free-Path Calculations Using a Model Dielectric Function, Phys. Rev. B, 35, 482, doi: 10.1103/PhysRevB.35.482
[24] Feroci M, et al. (2011). The Large Observatory for X-ray Timing (LOFT), Experimental Astronomy, Volume 34, Issue 2, p. 415-444, doi: 10.1007/s10686-011-9237-2