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Synchrotron X-ray transmission measurements and modeling of filters investigated for Athena

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Abstract. Athena is a Large-class astrophysics space mission selected by ESA to study the theme “hot and energetic Universe”. The mission essentially consists of a large effective area X-ray telescope and two detectors: the X-ray Integral Field Unit and the Wide Field Imager. Both instruments require filters to shield from out-of-band radiation while providing high transparency to X-rays. The mission is presently in phase B, thus, in order to consolidate the preliminary design, investigated filter materials need to be properly characterized by experimental test campaigns. In this paper, we report results from high-resolution X-ray transmission measurements performed using different synchrotron radiation beamlines, to assess the filter calibration accuracy and mitigate the risk related to selecting a unique calibration facility. The main goals of these test campaigns are: (i) verify the compliance of the investigated filter design to the scientific requirements, (ii) develop an accurate X-ray transmission model, and (iii) start identifying suitable measurement facilities and achievable accuracy for the flight filters calibration program. In particular, the X-ray transmission model of X-IFU and WFI filters has been refined within the edges of Al, C, N and O, by deriving the optical constants from two reference samples measured by synchrotron light. The achievable filter calibration accuracy has been estimated by evaluating the agreement between the best fit according to the developed transmission model and the experimental data.

Keywords: X-ray transmission, astrophysics space mission Athena, X-IFU, WFI, optical and thermal filters.

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1 Introduction

In the Cosmic Vision 2015-2025 Science program of ESA (European Space Agency), Athena (Advanced Telescope for High-Energy Astrophysics) has been selected as a Large-class astrophysics mission to study the dynamical, physical, and chemical properties of hot plasma in the Universe and to understand the role of black holes in shaping the environment, from local accretion disks to galaxy cluster scales¹. The mission, with launch scheduled in 2033, is presently in phase B, ending with the Mission Adoption Review by ESA at the end of 2021.

Athena will be equipped with a large effective area ($\sim 1.5 \text{ m}^2$ at 1 keV) grazing incidence X-ray telescope with a focal length of 12 m and two interchangeable focal plane detectors: the X-ray

38 Integral Field Unit (X-IFU)², a microcalorimeter made of a hexagonal array of molybdenum-
39 gold transition edge sensors³, and the Wide Field Imager (WFI)⁴, a large array of depleted field
40 effect transistors (DEPFET) active pixels. The X-IFU is a cryogenic X-ray spectrometer
41 operating at a temperature of about 50 mK in the 0.2-12 keV energy range to offer high spectral
42 resolution (2.5 eV FWHM at 7 keV) over a field of view of 5 arcmin equivalent diameter. The
43 WFI instrument is based on an array of silicon detectors covering a field of view of 40×40
44 arcmin² with a small detector of the same type, featuring high count rate capability, to observe
45 very bright point sources in the 0.2-15 keV energy range over a wide field.

46 Both instruments require filters to attenuate out-of-band radiation, similar to optical and thermal
47 filters already used for other instruments, such as the HRC⁵ and ACIS⁶ detectors on Chandra⁷
48 and the SXS microcalorimeter on ASTRO-H (Hitomi)^{8,9}. Indeed, the X-IFU detector must be
49 shielded from incoming UV-Vis-IR radiation, due to both the optical load from bright UV-Vis
50 astrophysical sources (e.g., massive stars, AGN's) and the IR radiation heat load from the
51 instrument, which would degrade its energy resolution due to photon shot noise¹⁰⁻¹². The X-IFU
52 filters are each to be mounted on one of five thermal shields at the operating temperatures of
53 300K, 100K, 30K, 2K and 0.05K, thus becoming an integral part of the thermal engineering of
54 the cryostat. Likewise, the DEPFET active pixel sensors of the WFI¹³ are sensitive to UV-Vis
55 photons with energies above the Si bandgap (~ 1.1 eV).

56 The comprehensive filter stack in the X-IFU instrument should allow an X-ray transmission
57 compliant with the Q.E. (Quantum Efficiency) requirements¹⁴ in Table 1. Transmission
58 requirements¹⁵ are derived from the Q.E. for the whole X-IFU instrument, which is given by the
59 product of the filter stack transmission and the detector quantum efficiency. Since the X-IFU

60 Q.E. at low energy mostly depend on filter transmission, a filter must be extremely thin, while
61 still compliant with both out-of-band radiation attenuation and mechanical requirements.

62 Before launch, both onboard instruments must be calibrated. The resolution of X-IFU is expected
63 to be ~ 2.5 eV FWHM, thus, during calibration, the photoelectric absorption edge structure
64 should be resolved by a factor ~ 10 better than the resolution required, with an absolute energy
65 knowledge better than 0.4 eV. The calibration accuracy on the total X-IFU Q.E. (filters and
66 detector), currently identified in the calibration plan¹⁶, is the lowest between 4% absolute (0.04)
67 in a given energy bin and 3% relative to the transmission in that bin, over the full energy range of
68 interest, or a fixed value of 0.01 when Q.E. is less than 0.33. Calibration accuracy within edges is
69 required to be 3% on ground. However, the allocation budget on calibration accuracy between
70 the subsystems, namely, filters and detector, is yet to be defined, it is assumed to be mostly
71 allocated to the filter full stack at lower energies, where their Q. E. is lowest and the detector one
72 is highest, whereas the opposite is true on the higher end of the energy range. Calibration of
73 single filters can be performed, and the requirement is met by the square sum of all filters
74 calibration accuracies.

75 Given such demanding calibration requirements, we have started, as part of the research
76 activities for the design and development of Athena X-IFU thermal filters and WFI optical
77 filters, a campaign of X-ray transmission measurements on a set of representative filter samples
78 made of a thin layer of polyimide coated with aluminum, using different synchrotron radiation
79 facilities. Filter materials are chosen with a low atomic number Z , to have a high transmission in
80 the energy band of interest while shielding the IR and NIR energy ranges (polyimide), whereas
81 the aluminum coating has the ulterior purpose of reflecting the UV/VIS part of the spectrum.
82 Prior work was carried out on the calibration of aluminized polyimide films to be used as filters

83 for previous X-ray astronomy missions^{17, 18, 19}. The main goals of this campaign are: to verify the
84 compliance of the baseline filter design to the scientific requirements, to develop an accurate X-
85 ray transmission model for the filters, especially within element absorption edges and to start
86 identifying suitable measurement facilities.

87 **2 Materials and methods**

88 *2.1 Measured samples*

89 Samples, produced by LUXEL (USA), are made of a polyimide (PI: C₂₂H₁₀N₂O₄) layer of either
90 ~ 45 nm or ~ 150 nm, coated with a thin aluminum (Al) layer of either ~ 20 or ~ 30 nm. Samples
91 with a thin PI layer are representative of the baseline for thermal filters inside the detector
92 cooling system for the X-IFU instrument, whereas those with a thick PI layer are investigated as
93 the baseline for the optical blocking filter of the WFI instrument.

94 Henceforth, all samples will be denoted by the following label: Al thickness/PI thickness in
95 nanometers (e.g. 20/45 or 30/150). Filters are baselined to be further equipped with a honeycomb
96 Au/Ag plated stainless steel (SS) or Nb mesh with a 5 mm pitch between cells, cell bars 50-90
97 μm wide and a total blocking factor of about 2% (see Fig. 1a for a filter prototype). The mesh
98 serves multiple purposes, such as: structural strength, vibrational rigidity and improved thermal
99 conductivity. However, for ease of measurement, the samples used in these campaigns are
100 smaller, meshless versions mounted on a TF111/TF110 standard LUXEL frame (see Fig. 1b).

101 This choice is due to the fact that, in terms of X-ray transmission, the meshless samples can be
102 thought of as a single cell of the full-scale filter, since the beam spot never exceeds a diameter of
103 1.2 mm, and is entirely contained in a 5 mm mesh cell. Design is still ongoing on mesh materials
104 and geometrical properties. Nominal layer thicknesses and manufacturing tolerances are reported

105 in Table 2. Only two samples come from the same production batch, and measurements could
106 not be repeated on the same sample in different facilities, since samples underwent several types
107 of tests, some of which destructive, whereas synchrotron measurements span over three years.

108 2.2 Synchrotron facilities

109 Three synchrotron facilities were selected to measure the EUV-Soft X-ray transmission of
110 samples:

- 111 - CNR-IOM BEAR beamline at Elettra (Trieste, Italy), for its capability of performing
112 measurements at the C K edge (<http://www.elettra.trieste.it/elettra-beamlines/bear.html>);
- 113 - PTB-EUV beamline at BESSY II (Berlin, Germany), for its wide energy range including
114 the Al K edge (<https://www.ptb.de/cms/en/ptb/fachabteilungen/abt7/fb-71/ag-712.html>);
- 115 - METROLOGIE beamline at SOLEIL (Univ. Paris-Saclay, France), also for its wide
116 energy range (<https://www.synchrotron-soleil.fr/en/beamlines/metrologie>).

117 The energy ranges investigated include edges of Al, C, N and O, the elements present in each
118 filter representative sample (see Table 3).

119 An edge region usually spans from ~10 eV before the edge (pre-edge) to ~50-100 eV after it
120 (post-edge). Edge regions have to be included in the Q. E. requirements evaluation in calibration
121 tests. Beamline measurement conditions adopted in each campaign are summarized in Table 4.

122 2.2.1 Transmission measurement

123 The experimental procedure to obtain each filter transmission is described in this section with
124 reference to the BEAR beamline²⁰. At the end of this section, relevant differences between
125 beamline specific procedures are outlined.

126 The conceptual scheme of BEAR is reported in Fig. 2. The monochromator beam line is based
 127 on an entrance slitless PMPG²¹ optical layout. The inclusion angle was continuously changed
 128 along the scan according to a pre-set working curve to mitigate the higher orders contributions.
 129 In addition, a suite of high-energy cut-off filters was used, including an Al filter 0.2 μm thick in
 130 the range 40-72 eV, a Si filter 0.5 μm thick in the range 72-100 eV and two Ag filters, one 0.6
 131 μm thick in the range 250-320 eV and the other 1 μm thick in the range 1100-1600 eV.

132 The photon beam intensity $\Phi(\hbar\omega)$, both impinging and transmitted, were measured by an SXUV-
 133 100 photodiode. The reduction of incident photons per seconds was accomplished according to

$$\Phi(\hbar\omega) = I(\hbar\omega)/[e \cdot f(\hbar\omega)], \quad (1)$$

134 where $I(\hbar\omega)$ is the pico-ammeter current (I_S and I_0 for transmitted and impinging photon beam), e
 135 is the electron charge and $f(\hbar\omega)$ is the diode quantum efficiency.
 136

137 The I_S and I_0 currents were measured in two separate runs (acquisition times t_1 and t_2). The I_0
 138 signal was acquired removing the sample (sample in/out in Fig. 2) from the measuring position.
 139

140 The transmission (T) was obtained according to

$$T(\hbar\omega) = \frac{I_S(t1) - I_{Sd}(t3)}{I_{Sm}(t1) - I_{Smd}(t3)} \cdot \frac{I_{0m}(t2) - I_{0md}(t4)}{I_0(t2) - I_{0d}(t4)}, \quad (2)$$

141 where the currents with the m subscript indicate the simultaneous monitor signals of the
 142 transmitted/impinging beam, that is, the emission current of the refocusing mirror (REFO in Fig.
 143 2) or beam position monitoring (BPM) drain current (see Fig. 2) used in the regions of low
 144 impinging intensity (typically the C K-edge). Monitor signals allow to normalize I_S and I_0 , taking
 145 into account any impinging flux variation with time, mainly due to synchrotron beam decay
 146 (Elettra works in top-up mode, typically percent over hours), together with any beam fluctuation
 147 due to beam steering.
 148
 149

150 The d subscripts in Eq. 2 indicate dark currents from reading pico-Ammeters. The consequent
151 correction is crucial at the low intensity regions, especially in the C K edge region, where optics
152 reflection is reduced by carbon contamination of optical surfaces. A schematic of the BEAR
153 beamline setup is reported in Fig. 2.

154 A similar procedural scheme was adopted at Metrologie and PTB-EUV, with their particular
155 instrumentation solutions and setups²²⁻²⁴. The light spot at sample and energy step for all
156 beamlines are reported in Table 4. The C K edge was not acquired at PTB-EUV, and I_0 signal
157 was recorded before and after each I_s signal and averaged out, whereas at Metrologie no
158 correction for dark current was implemented, a fact negatively affecting the signal to noise ratio
159 in low intensity regions such as the C K edge.

160 **3 Quantitative modeling of filter transmission**

161 In this section, a numerical modeling of a filter transmission is presented, together with a method
162 for recovering optical cross sections from experimental data. The model achievable accuracy in
163 predicting filter transmission over the energy range of interest is assessed, which is important to
164 plan the calibration activity of the flight filters.

165 A filter is a multi-layer of thin films, differing for thickness, atomic species and relative
166 concentration, its transmission curve can be divided in two regions: outside and inside edges.
167 Reliable literature values of the optical constants can be found for outside edge regions²⁵,
168 however, this is not the case inside edge regions. Thus, the optical constants within edges were
169 obtained from experimental data, after fitting outside element edges using a model of the filter
170 transmission. The filter is described as a stack of thin films made of three materials: aluminum,
171 polyimide, and aluminum oxide. A layer of native aluminum oxide is introduced, since its
172 presence is known from the literature²⁶⁻²⁸, with a stoichiometry Al_2O_3 assumed henceforth.

173 The filter transmission $T(E)$ can be approximated as the product

$$174 \quad T(E) = \prod_1^n e^{[-\mu_i(E) \cdot x_i]} \quad (3)$$

175
176 where x_i is the height of the volume occupied by the i_{th} atomic specie and μ_i is its corresponding
177 linear attenuation coefficient. The linear attenuation coefficient is proportional to the imaginary
178 part f_2 of the atomic scattering factors.

179 On the basis of Eq. 3, we develop a model for filter transmission, involving directly the atomic
180 elements without any further constraint related to the specific material, such as its density and
181 stoichiometry (they shall however be considered afterwards to estimate each layer thickness).

182 The model allows to fit experimental transmission data, provided that the atomic scattering
183 factors are known. Outside absorption edge regions, atomic scattering factors in Henke et al.
184 1993²⁵ can be used, however, within edges, we use the model to obtain *effective* values for f_2 of
185 each element from data. Specifically, the fit function is based on the product of the transmission
186 due to each element and has four free parameters, each element volume height in the layer:

$$187 \quad T(E) = T_{Al} T_C T_N T_O = e^{[-\mu_{Al}(E) \cdot x_{Al}]} e^{[-\mu_C(E) \cdot x_C]} e^{[-\mu_N(E) \cdot x_N]} e^{[-\mu_O(E) \cdot x_O]} \quad (4)$$

188
189 In Eq. 4, we disregard H, since it has a negligible absorption in the energy range of interest.

190 A preliminary fit was performed outside each element edge region on two reference samples,
191 using Henke tabulated values for f_2 . Focusing on one element at a time, e.g. Al, we then modeled
192 the transmission curve within its edge region due to all the other elements, using the preliminary
193 best fit parameters x_i . We then divided the experimental transmission (T_{exp}) by the modeled
194 transmission of all the other elements, given by the product $T_C \cdot T_O \cdot T_N$ in this example. The linear
195 attenuation coefficient (μ_{Al}) can then be calculated by dividing the logarithm of this ratio by the
196 specific element volume height obtained from the preliminary fit. The f_2 inside a specific element

197 edge region are proportional to these values, which are smoothed with a polynomial and
 198 interpolated over the energy range, and can be derived as in Eq. 5 (example for Al):

$$199 \quad (f_2)_{Al} \propto \mu_{Al}(E) = \frac{1}{x_{Al}} \cdot \ln\left(\frac{T_{exp}}{T_C \cdot T_N \cdot T_O}\right), \quad (5)$$

200
 201 These *effective* values for f_2 , calculated from the experimental data, are atomic in nature, but take
 202 into account each atom local environment within the material. For instance, since Al is present
 203 both in the metallic and aluminum oxide compounds, the f_2 calculated for Al include both
 204 components. This method can be safely applied if edges of different elements do not overlap
 205 over the same energy range, as is the case for our samples. Having obtained *effective* values for f_2
 206 inside each edge, the fit can be performed again, over the whole energy range, now including
 207 edge regions. From these best fit parameters, all layer thicknesses can be estimated, introducing
 208 two quantities *a posteriori*: material density and stoichiometry. For instance, PI thickness can be
 209 estimated from the best fit parameter of either C or N. Starting from x_N , and dividing it by the
 210 product of PI density (1.4 g/cm^3) times the nitrogen mass fraction f_N (PI molecule $\text{C}_{22}\text{H}_{10}\text{N}_2\text{O}_4$),
 211 PI thickness $x_{PI(N)}$, can be calculated as in Eq. 6:

$$212 \quad x_{PI(N)} = \frac{x_N}{\rho_{PI} \cdot f_N}, \quad \text{with} \quad f_N = \frac{2A_N}{22A_C + 10A_H + 2A_N + 4A_O} \quad (6)$$

213
 214 In principle, the same calculation can be carried out using x_C , but oxygen cannot be used since it
 215 is also involved in aluminum oxide. However, knowing the PI layer thickness from N, the Al_2O_3
 216 layer thickness can be derived from the remaining number density of O atoms.

217 Finally, to define a 3σ confidence interval (CI) and assess the accuracy achievable by the
 218 transmission model, the percent relative error (*%err*) between best fit and data was calculated
 219 and the corresponding CI estimated by modeling the maximum and minimum fit curves
 220 according to the best fit.

221 4 Results and Discussion

222 Experimental transmission data are shown in Fig. 3 for thin samples (X-IFU thermal filters) and
223 in Fig. 4 for thick samples (WFI optical blocking filters), comparing results obtained at different
224 beamlines. Notice that samples PN 3203 measured at Elettra and PN 3204 measured at SOLEIL
225 come from the same batch and thus the corresponding transmission data are closer, whereas
226 sample PN 2472 has slightly less Al (see Al L edge in Fig. 2). The opposite is true for the thick
227 samples, where sample PN 2460 measured at BESSY shows a slightly thicker coating of Al.

228 In Fig. 5, the fit performed outside edge regions using tabulated scattering factor is plotted, for a
229 reference filter (PN 3096). The experimental data of this filter was used to obtain the *effective*
230 values for f_2 of elements C, N and O in the ranges: 280 eV-375 eV, 395 eV-450 eV and 527 eV-
231 570 eV. A different reference filter (PN 2460) was used to obtain *effective* values for f_2 of Al
232 inside its L and K edges: 72 eV-220 eV and 1550 eV-1670 eV.

233 After obtaining the *effective* values for f_2 in all the elements edge regions from the reference
234 samples, a best fit was performed for all the samples, now including edges, to find each layer
235 thickness (see Table 5, where a star indicates reference samples).

236 From Table 5, the sum of Al and Al₂O₃ thickness is just a few nm less than the nominal value
237 provided by the filter manufacturer. This remains true when comparing the equivalent Al amount
238 (equiv. Al in Table 5), calculated from the total number of Al absorbers divided by the Al
239 density 2.7 g/cm³. Possibly, the actual Al coating density could be slightly lower than expected,
240 because of the nanometric scale of the layer and the technological process involved. The Al₂O₃
241 thickness is in line with what is expected if the spontaneous oxidation process occurred on both
242 surfaces of the Al layer, since the value found in the literature is 3-4 nm per surface²⁶. A slight
243 variability in Al₂O₃ thickness is observed, probably due to different ages of the samples, ranging

244 from a few months to four years old. The thickness of the PI layer estimated from N is in overall
245 good agreement with the nominal thickness, except for the reference sample where it is
246 underestimated. The authors have no clear explanation about this slight discrepancy, possibly the
247 fact that this sample was used as the reference sample to retrieve the *effective* f_2 may have
248 influenced the result. In any case, the PI layer thickness from C is consistently overestimated.
249 There are at least two possible explanations: the first one is that samples, which have been
250 exposed to air in some instances, have some C surface contamination; the second is that there is
251 either a different stoichiometry of the PI or carbon-based molecules trapped somewhere within
252 or between layers. We have a few X-ray Photoelectron Spectroscopy (XPS) measurements,
253 which preliminarily indicate the presence of C surface contamination.

254 In Figs. 6-8, datasets of thin samples with their best fit are shown, whereas a plot with the *%err*
255 along with its CI is plotted in Fig. 9 for a thin sample, with a red vertical line to signal the low
256 energy threshold for the nominal sensitivity of the Athena X-ray sensors (200 eV), and two
257 horizontal lines to display the filter calibration requirement of $\pm 1\%$ relative error.

258 Data at $E < 200$ eV, where the sample has a lower transmittance, are still important to accurately
259 estimate the Al layer, since the L edge is very sensitive to changes in the number of Al absorbers.
260 The best fit is overall in good agreement with all datasets, even inside edge regions. Regarding
261 BESSY II and SOLEIL, the agreement of the fit to the data is very good, with a percent relative
262 error in transmission below 1% everywhere, except in the C, N, and O K-edges, where *effective*
263 f_2 were obtained from the thick sample measured at Elettra. Looking at the C K-edge measured at
264 SOLEIL, the shape is noticeably different from the same edge measured at Elettra, making this
265 measurement less reliable (as expected). The N K-edge measured at SOLEIL is also quite noisy,
266 whereas at BESSY the energy step is one order of magnitude bigger with respect to the Elettra

267 one. Regarding the O K edge, the scattering factors were recovered from a thick sample, where
268 the relative amount of O in PI with respect to Al_2O_3 is bigger, also causing a difference in edge
269 shape between thin and thick samples as measured by all beamlines.

270 For what concerns Elettra, the fit is also in good agreement with the data, with a percent relative
271 error in transmission below 1.5% in the energy range of interest for Athena, excluding the C, O
272 and Al K-edges. Within these edges, the model reproduces the data less well, due to:

273 - A ~ 0.1 eV shift in the energy calibration of the C K-edge between thin and thick samples.

274 Since the reference sample from which *effective* f_2 were recovered is the thick one, this
275 shift also reflects on the fit accuracy for the thin sample in this region;

276 - BEAR performance at the high energy end of its range, where it is currently suffering an
277 important contamination of beam purity ascribed to **stray zero-order** light coming from
278 diffuse scattering at the grating and causing, in data reduction, an unphysical decrease of
279 the transmission as the energy increases. Empirical numerical correction is possible and
280 has been proven of use for spectroscopy purposes, though this fact does not make BEAR
281 the first choice for a quantitative measurement of the transmission in this range.

282 The agreement between best fit and data, in terms of percent relative error, is summarized in
283 Table 6, grouped by energy range. The N, O and Al K edges are reported separately, whereas the
284 more problematic C K edge is not included.

285 Overall, the model is in good agreement with the data, in terms of percent relative error.

286 However, some improvement is necessary to meet the X-IFU strict calibration requirements,
287 especially within C and O K-edges, both in terms of obtaining improved *effective* f_2 and of
288 developing an **absolute energy scale calibration strategy**. The *effective* f_2 , however, appear well
289 suited to model each element edge region, thus increasing the fit agreement with the data.

290 5 Conclusions

291 The two onboard instruments of the astrophysics space mission Athena, X-IFU and WFI, have
292 severe calibration requirements. In order to meet the goals of the filters calibration plan, we have
293 started several X-ray measurement campaigns, using different European synchrotron radiation
294 facilities. Our aim is to characterize filter materials, presently investigated for the filters
295 preliminary design, and to get some confidence on the measurement accuracies achievable in the
296 flight filters calibration program. More than one facility is needed in this regard, to mitigate the
297 risk of potential unavailability of the beamline during calibration measurements on flight filters.
298 Transmission data were acquired for six filter samples, made of a thin polyimide membrane
299 coated with aluminum, at three different synchrotron facilities: CNR-IOM BEAR at Elettra
300 (Trieste, IT), PTB-UV at BESSY II (Berlin, DE) and METROLOGIE at SOLEIL (Univ. Paris-
301 Saclay, FR). A model was developed, based on the transmission of a gas of elements Al, C, O,
302 N, with four free parameters, to determine each layer thickness. New *effective* values for the
303 imaginary part of the scattering factors were obtained by a preliminary fit of two reference
304 samples, with the goal of refining the fit accuracy within edge regions. The layer thicknesses
305 obtained from the best fit parameters are in general in good agreement with the nominal ones.
306 The percent relative error between best fit and data was evaluated, to estimate the model
307 accuracy in reproducing the data, in view of satisfying the strict calibration requirements of the
308 X-IFU instrument. A good agreement between the measured transmission and best fit was found
309 for all the sample filters. Finally, actions should be undertaken in acquiring more accurate data,
310 such as developing an absolute energy calibration strategy specific for our samples, which takes
311 into account the very strict calibration requirements of X-IFU filters, to be run prior to each
312 experimental campaign, and investigating filter surface C contamination.

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325

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387

388

389 **Biographies**

390

391 Elena Puccio, Master Degree in physics, obtained her Ph.D. in Physics in 2017 working on Big
392 data, Network theory, correlation and cluster analysis. She is currently a post-doc researcher at
393 the University of Palermo (Italy) with a three years fellowship, affiliated with INAF-OAPa, to
394 work on the design, development, experimental characterization and modeling of optical
395 blocking filters and thermal filters for the WFI and X-IFU instruments on board the large
396 astrophysics mission Athena funded by ESA.

397

398 Michela Todaro is a physicist, with a Ph.D. in Material Science and Nanotechnology (2018). She
399 won a post-doc scholarship at the Astrophysics National Institute (INAF-OAPA) in 2018, on
400 remote sensing through aerostatic devices. She currently has a Post-Doctoral fellowship at the
401 University of Palermo, on the experimental characterization of the spectral and morphological
402 properties of prototype filters of the X-IFU and WFI instruments onboard the ATHENA mission
403 of ESA.

404

405 Ugo Lo Cicero, PhD in Electronic Engineering, is a senior technologist at INAF. He works on
406 the development of new technologies for Astrophysics, in particular related to X-ray
407 instrumentation. He has worked on the design and development of microcalorimeter arrays,
408 active optics, X-ray sources and on the design and extensive characterization of cryogenic X-ray

409 filters. He is System Manager for the “Thermal Filters” subsystem of the XIFU instrument on
410 board ESA flagship mission Athena.

411

412 Luisa Sciortino obtained a Ph.D. in Chemistry in 2012. She has been working on the
413 development and design of the thermal and optical blocking filters of the X-IFU and WFI
414 instruments on board the Athena mission. Her expertise is the spectroscopic characterization of
415 materials and nanomaterials. She is currently a researcher of the Department of Physics and
416 Chemistry of the Università degli Studi di Palermo.

417

418 Philippe Laurent is working at the CEA Astrophysical Department in France. He is specialized in
419 High Energy Astrophysics and got his PHD in 1992, studying Black Holes X-ray binaries high-
420 energy emission. In parallel, he developed a theoretical model of radiation transfer in the
421 environment of black holes. He participates to the ESA/INTEGRAL gamma-ray mission since
422 1995, and is now the IBIS telescope Co-PI. He is also co-chair of the ATHENA background
423 studies topical group.

424

425 Philippe Ferrando obtained a PhD in high energy astrophysics in 1987. He has been working on,
426 and was Co-I of, cosmic-rays instruments onboard HEAO3, Voyager, and Ulysses. In 1995 he
427 turned to X-rays and was responsible for the calibration team of the EPIC cameras on XMM-
428 Newton. He has been P.I. of several mission proposals and phase A studies, in particular of the
429 SIMBOL-X hard X-rays telescope. He is currently deputy-head of the CEA Astrophysics
430 Department.

431

432 Angelo Giglia obtained his degree in Engineering of Telecommunications in 1999 and the PHD
433 in 2006 in Engineering of Materials and Environment. He is technologist and beamline scientist
434 for experiments for absorption X, reflectivity and photoemission spectroscopies at the
435 synchrotron beamline BEAR@Elettra of the CNR IOM Laboratory, from april 2002 with a
436 permanent position. His activity consists mainly in the use of UV-soft X-rays synchrotron Light
437 for the characterization of materials and devices.

438

439 Stefano Nannarone graduated in Physics in 1967, University Roma La Sapienza. Presently
440 retired from the University of Modena-Reggio Emilia (professor of General Physics), associated
441 fellow of CNR and scientific responsible of IOM-CNR synchrotron beamline BEAR (Elettra,
442 Italy). Scientific activity covered electronic and structural properties of solids, surfaces and
443 interfaces studied by optical (lab and synchrotron) and electron scattering techniques. Teaching
444 experience included General and Solid state Physics, Solid state Devices and elementary
445 Quantum Chemistry.

446

447 Marco Barbera (Ph.D. in Physics) is associate professor of Astrophysics at University of
448 Palermo, Italy (2004). He is affiliated with INAF-OAPa with responsibilities in the operation of
449 X-ray Astronomy Calibration and Testing facility for development/test of instrumentation for X-
450 ray Astronomy missions (filters, optics, detectors) such as Chandra, Newton-XMM, Hinode,
451 Chang'E-1, Coronas Photons, Athena and eXTP. He is responsible for the development of filters
452 of the WFI and X-IFU instruments on board ESA space mission Athena.

453

454 **Tables**

455

Table 1 X-IFU Q. E. and filter transmission requirements for the comprehensive filter stack.

Energy	X-IFU Q.E.	T _{filters}
0.35 keV	≥ 17 %	≥ 21 %
1 keV	≥ 67 %	≥ 76 %
7 keV	≥ 72 %	≥ 89 %
9.5 keV	≥ 50 %	≥ 89 %

456

Table 2 Nominal layer thicknesses of Al and PI for each sample with its **tolerances from the manufacturer** displayed in parenthesis. Next to each sample, in parenthesis, is indicated the synchrotron facility where it was measured.

457

Sample (synchrotron)	Part Number (PN)	Al (nm)	PI (nm)
20/45 (Elettra)	3203	20.6 (2.5)	45.5 (5)
20/45 (SOLEIL)	3204	20.6 (2.5)	45.5 (5)
20/45 (BESSY II)	2472	21.5 (2.5)	42.7 (5)
30/150 (Elettra)	3096	31.1 (5)	149.7 (5)
30/150 (SOLEIL)	2677	28.8 (5)	153.4 (5)
30/150 (BESSY II)	2460	30.4 (5)	157.8 (5)

458

459

Table 3 Atomic L- and K-edges indicative energy ranges in the measured samples.

Element	L-edges	K-edge
Al	73 eV, 118 eV	1560 eV
C	-	284 eV
N	-	402 eV
O	-	532 eV

460

Table 4 Measurement parameters adopted for the campaigns at BEAR (Elettra - Trieste, IT), PTB-EUV (BESSY II - Berlin, DE), and METROLOGIE (SOLEIL - Paris, FR) beamlines.

461

	BEAR at Elettra	PTB-EUV at BESSY II	METROLOGIE at SOLEIL
Spot size	0.4x0.7 mm ² (VxH)	1.2x1 mm ² (VxH)	0.6-1x1 mm ² (VxH)
Spectral resolution	E/ΔE ~ 2000	E/ΔE > 1000	E/ΔE > 1000
Energy range	40-1650 eV	55-1800 eV	30-2000 eV
Energy step	0.05 eV	2-20 eV outside edge regions 0.2-0.4 eV inside edge regions	1-2 eV outside edge regions 0.2-0.5 eV inside edge regions
Number of data points	~ 32000	~ 800	~ 3000

462

463

Table 5 Layer thickness of each material (Al, Al₂O₃, and PI) with its 3σ statistical uncertainty in parenthesis, as obtained from the best fit parameters.

464

Sample (synchrotron)	Best fit thicknesses (nm)					Nominal thickness (nm)	
	Al	Al ₂ O ₃	equiv. Al	PI (from N)	PI (from C)	Al (nm)	PI (nm)
20/45 (Elettra)	9.4 (0.2)	8.5 (0.2)	16.00 (0.02)	45.5 (1.1)	57.8 (0.1)	20.6 (2.5)	45.5 (5)
20/45 (SOLEIL)	9.1 (0.6)	8.5 (0.7)	15.61 (0.06)	49 (4)	58.3 (0.5)	20.6 (2.5)	45.5 (5)
20/45 (BESSY II)	9.3 (1.0)	7.4 (1.1)	15.0 (0.1)	46 (6)	55.9 (1.1)	21.5 (2.5)	42.7 (5)
30/150 (Elettra)*	20.6 (0.2)	9.4 (0.3)	27.90 (0.03)	137.5 (1.3)	177.3 (0.2)	31.1 (5)	149.7 (5)
30/150 (SOLEIL)	18.4 (1.5)	11 (2)	26.8 (0.2)	146 (10)	178.0 (1.3)	28.8 (5)	153.4 (5)
30/150 (BESSY II)*	23 (3)	10 (4)	30.6 (0.5)	164 (20)	197 (3)	30.4 (5)	157.8 (5)

465

* reference samples used for the determination of the *effective* values for f_2 inside edges.

466 **Table 6** Percent relative error between data and best fit for 20/45 samples grouped by energy range (in eV). K edges
 467 are listed separately and the C K edge is not reported.

Sample (synchrotron)	Percent relative error (%)					
	0.2<E<0.4 keV	N K edge	0.4<E<0.6 keV	O K edge	E>0.6 keV	Al K edge
20/45 (Elettra)	<±1.5	<±1	<±1	>3	<±1	<±2.7
20/45 (BESSY II)	<±1	<±1.2	<±1	>3	<±1	<±1
20/45 (SOLEIL)	<±1	<±1.7	<±1	>3	<±1	<±1

468
 469

470 **Figure Caption List**

471
 472 **Fig. 1** Sample filters with a thin PI membrane, coated with Al. (a) Filter sample with a 5 mm
 473 pitch Au plated SS honeycomb mesh mounted on a TF130 standard frame. (b) Meshless filter
 474 sample mounted on a TF110 standard frame, as used in the campaigns here reported.

475 **Fig. 2** Conceptual scheme of the BEAR beamline, downstream from synchrotron (Elettra) on the
 476 right (not shown): beam position monitor (BPM), polarization selector (not used, fully open in
 477 these runs), PGPM monochromator (two gratings, GNIM not used in these runs) and G1200, exit
 478 slit and filter wheel, light shutter, REFO refocusing mirror, sample stage (cooling system not
 479 used in these runs) with sample mounted and beam in/out movements in the experimental
 480 chamber (base pressure low 10^{-9} mbar) together with light detector (AXUV diode). Instruments
 481 indicate p-Ammeters measuring monitor current, sample drain current (not used in these runs)
 482 and diode current. BPM plates drain current p-Ammeters not shown in figure.

483 **Fig. 3** Comparison between experimental transmission data acquired at Elettra (orange line),
 484 SOLEIL (blue line) and BESSY II (black line) on 20/45 thin samples. Data in the C K-edge is
 485 absent for BESSY II. Insets show zooms of edges element-wise.

486 **Fig. 4** Comparison between experimental transmission data acquired at Elettra (orange line),
 487 SOLEIL (blue line) and BESSY II (black line) on 30/150 thick samples. Data in the C K-edge is
 488 absent for BESSY II. Insets show zooms of edges element-wise.

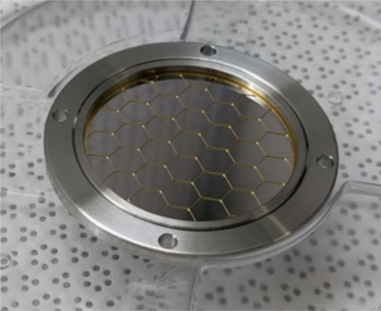
489 **Fig. 5** Experimental data (black dots) of a reference thick sample measured at Elettra (PN 3096),
490 used to obtain *effective* values of f_2 for C, N and O (red line).

491 **Fig. 6** Dataset of 20/45 sample (black dots) measured at Elettra with its best fit (red line).

492 **Fig. 7** Dataset of 20/45 sample (black dots) measured at SOLEIL with its best fit (red line).

493 **Fig. 8** Dataset of 20/45 sample (black dots) measured at BESSY II with its best fit (red line).

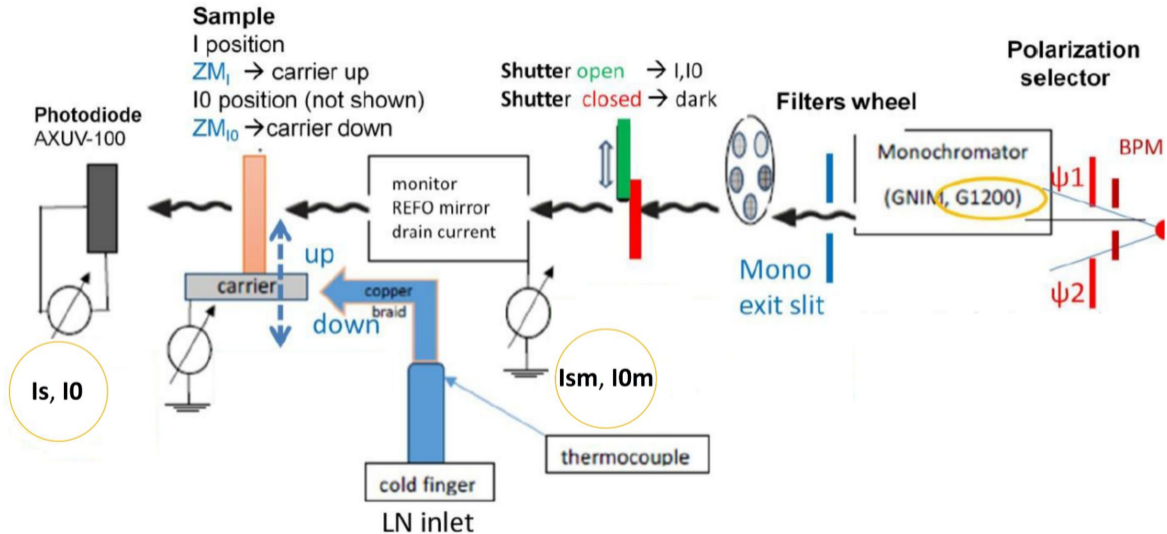
494 **Fig. 9** Percent error ($\%err$) between data and best fit (blue squares) for sample 20/45 measured at
495 BESSY II with its CI (grey area). Red lines define the range of interest for filter calibration.

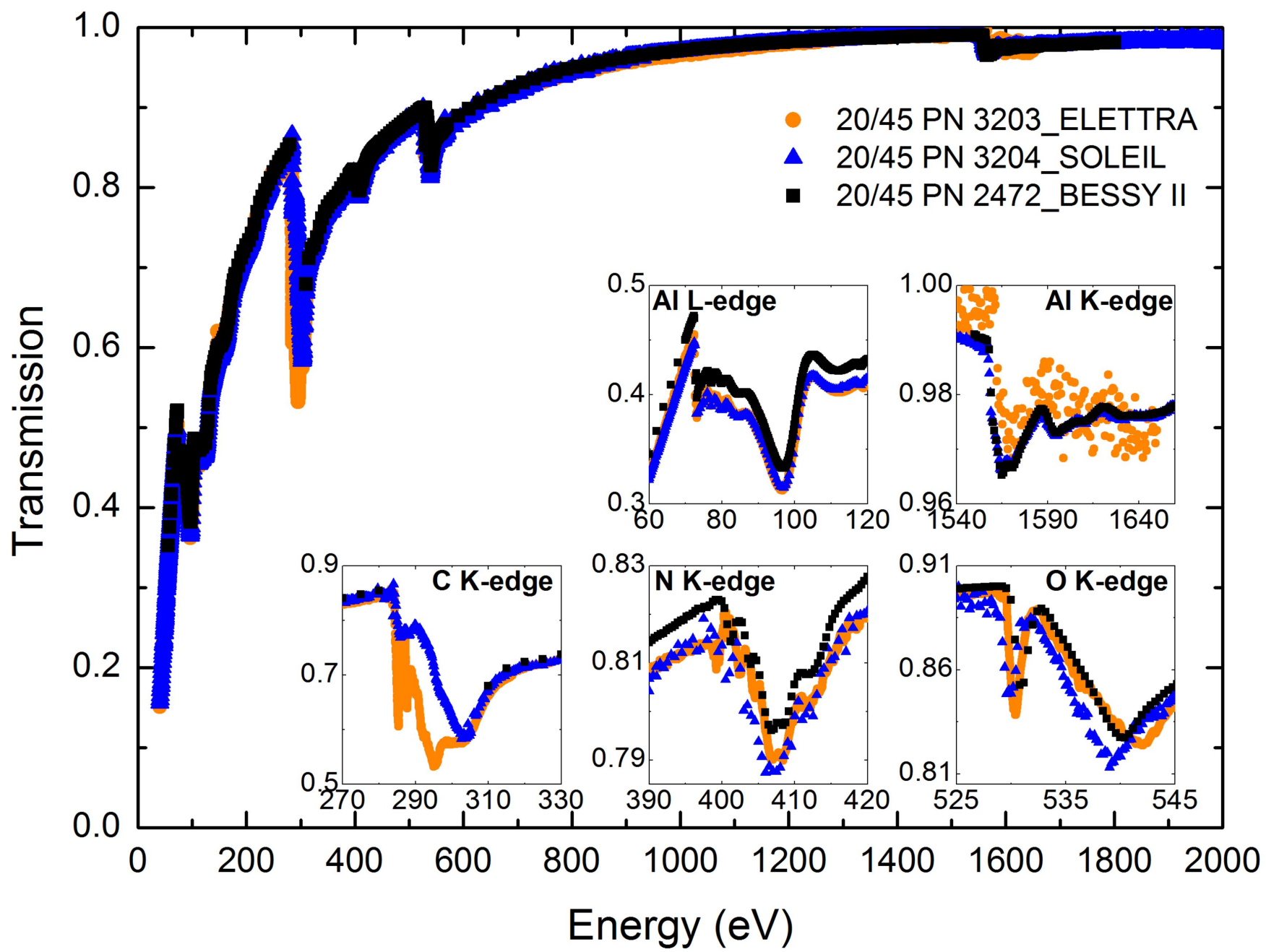


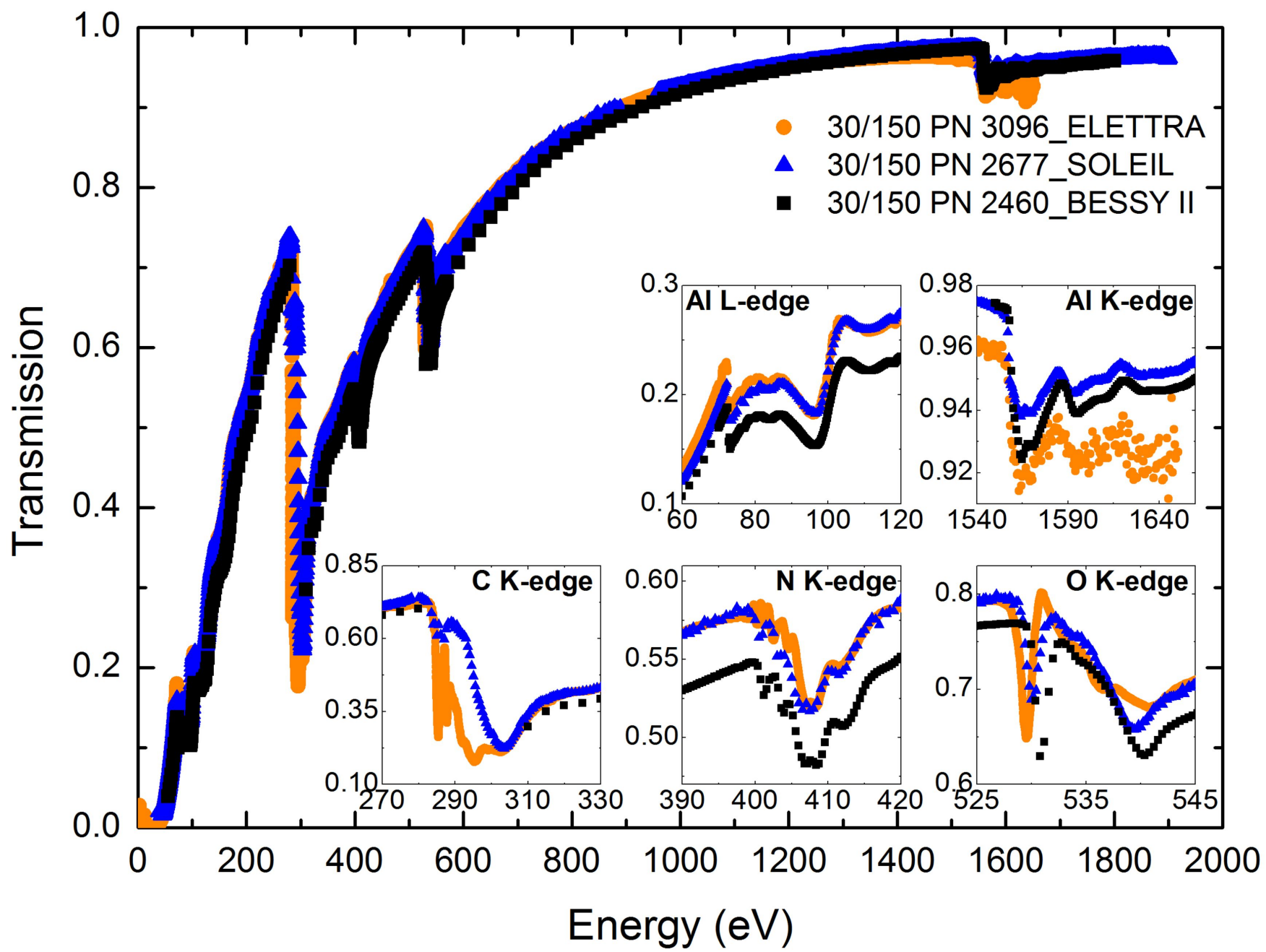
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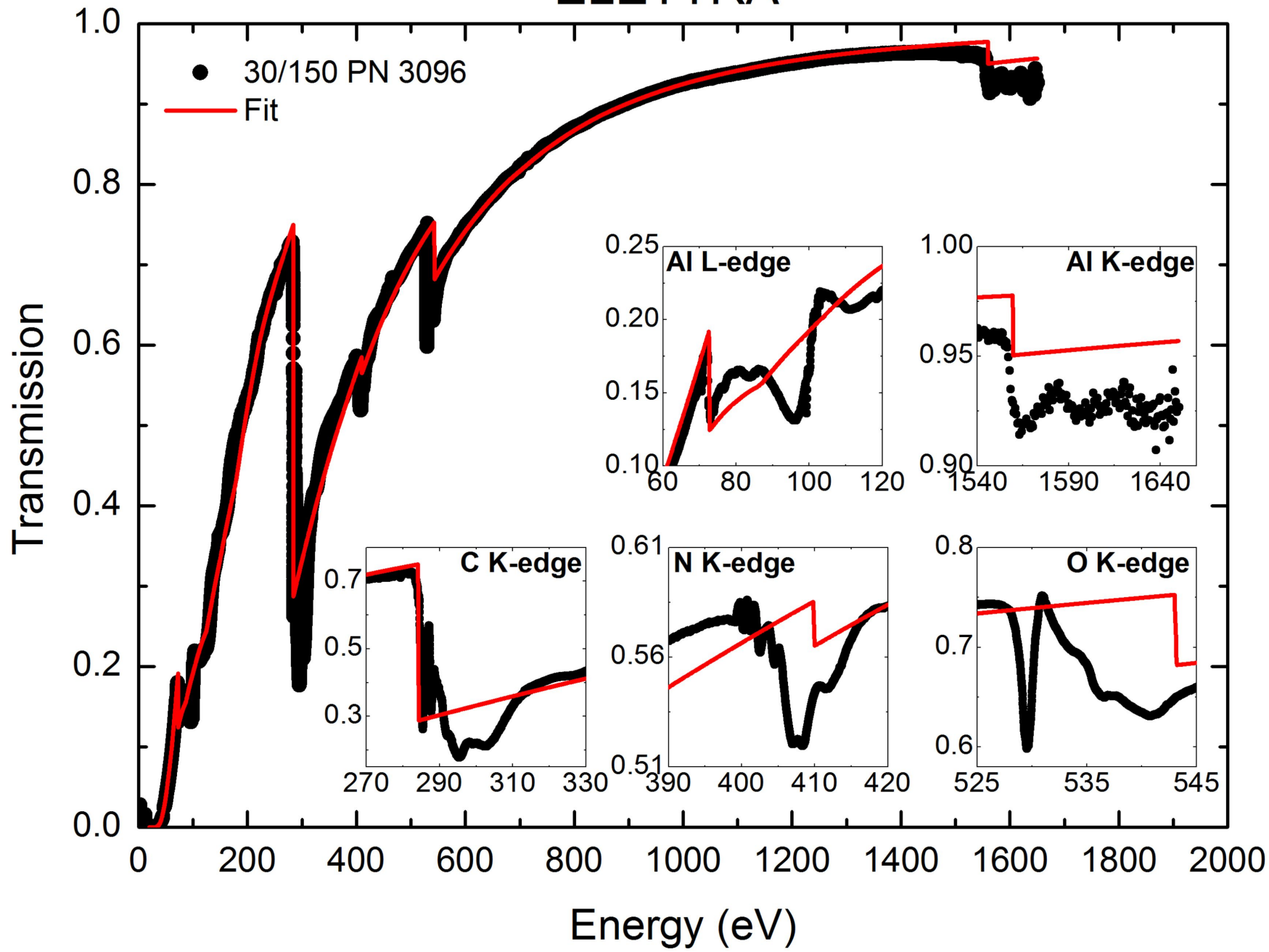
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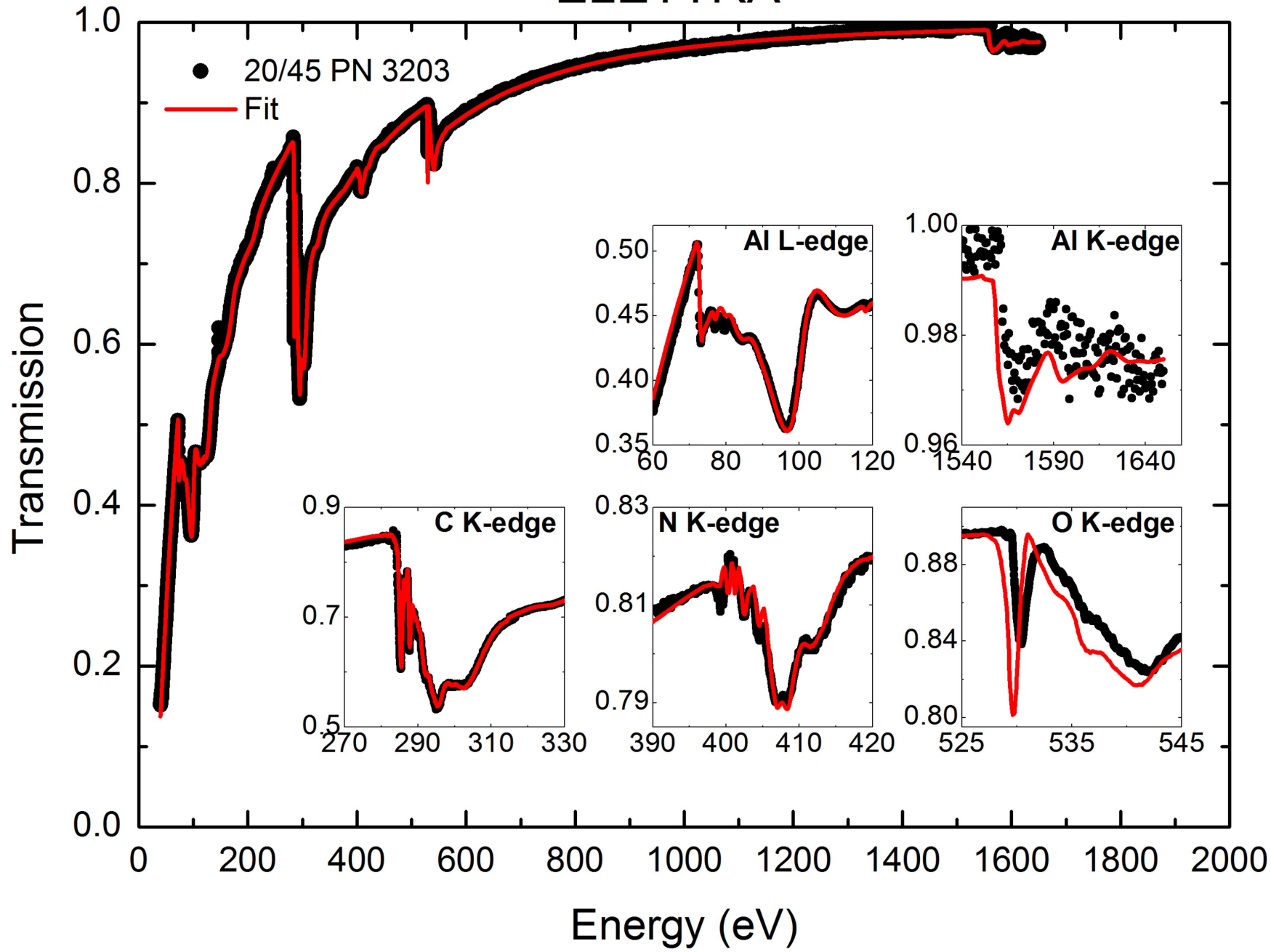




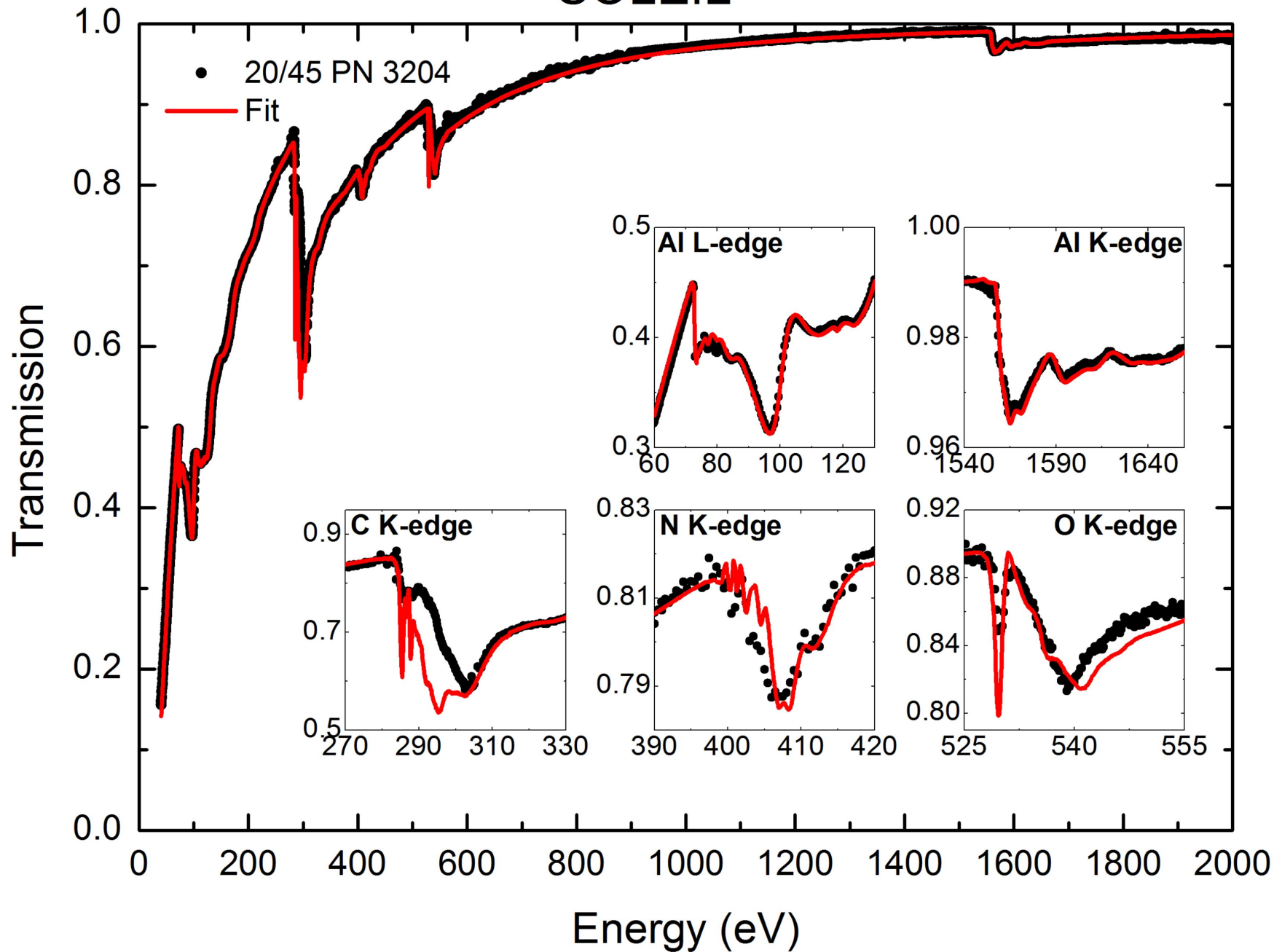
ELETTRA



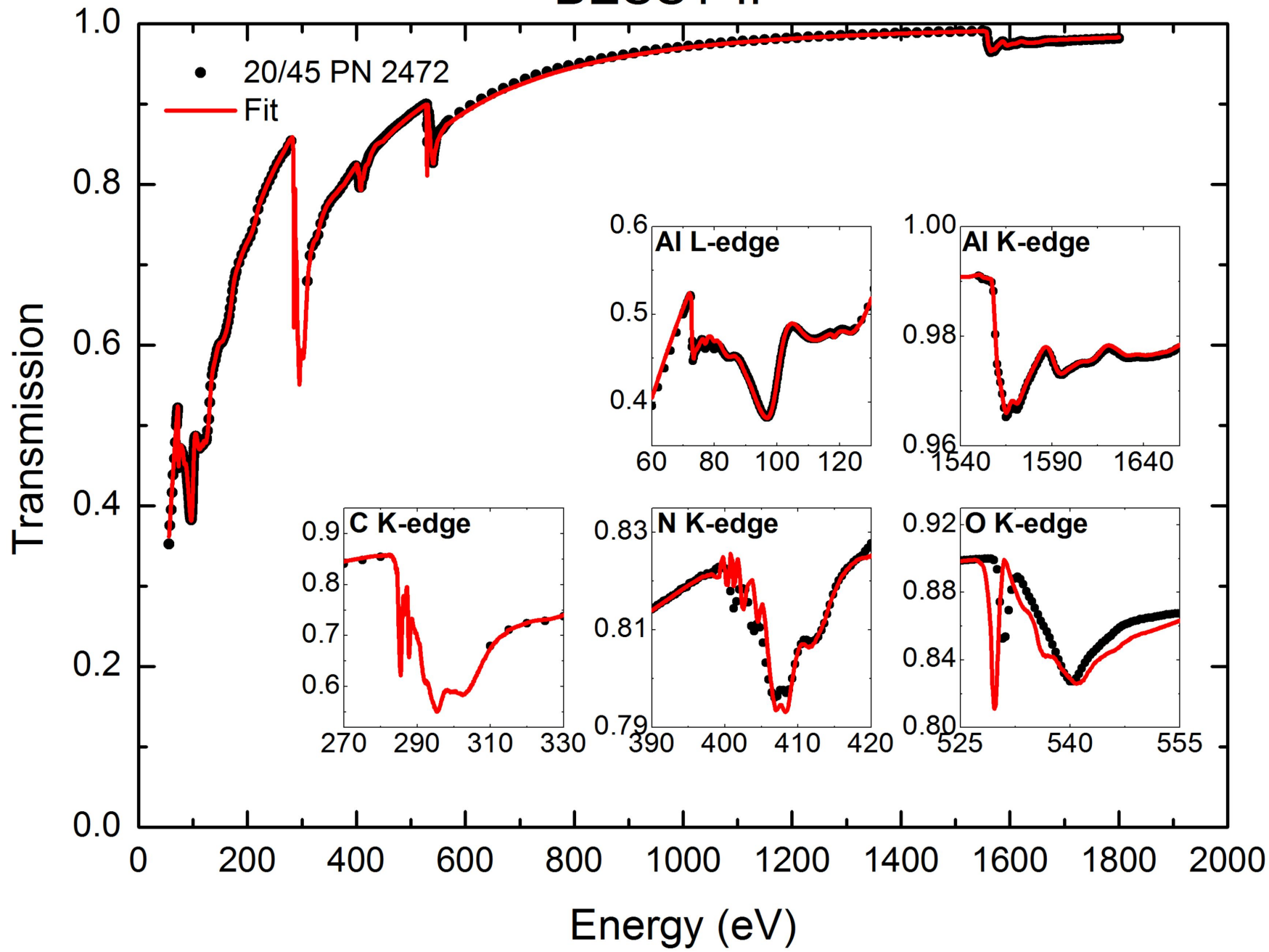
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SOLEIL



BESSY II



BESSY II

