

Comparative experimental study of single- and multi-walled carbon nanotube pellicles for EUV/soft X-ray space filters[☆]

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ABSTRACT

Freestanding carbon nanotube (CNT) thin films are attractive as highly transmissive, mechanically robust supports for extreme ultraviolet (EUV) and soft X-ray space mission applications. We compare single-walled (SW) and multi-walled (MW) carbon nanotube (CNT) pellicles grown by floating-catalyst chemical vapor deposition, combining X-ray absorption spectroscopy (XAS), EUV transmittance, UV–VIS–IR spectroscopy, and spherical indentation to link composition to transmittance, and explore mechanical properties.

In the 10–14 nm range, MW pellicles exhibit higher EUV transmission than SW pellicles with comparable visible transparency. At 13.5 nm, relevant for EUV lithography, representative MW samples reach transmittance $T \approx 0.80$, while SW films with similar optical density reach $T \approx 0.70$. XAS analyses show that this performance difference arises from compositional variations: SW pellicles contain a higher Fe:C atomic ratio, and the increased absorption, limited to the energy region above the Fe $M_{2,3}$ photoabsorption edge, accounts for the reduced EUV transmission.

Both types of pellicles display similar effective per-carbon extinction cross sections in the UV–VIS and low pellicle densities ($\rho_{SW} \approx 0.41$, and $\rho_{MW} \approx 0.31 \text{ g cm}^{-3}$). Mechanical tests yield higher elastic moduli for SW films (6.0–7.6 GPa) than for MW films (4.2–4.7 GPa), but comparable ultimate tensile strengths (≈ 276 –320 MPa). At fixed pellicle thicknesses, the lower stiffness of MW films results in higher rupture loads under out-of-plane indentation, as experimentally observed.

The higher mechanical resistance to rupture and higher transmission make MW pellicles promising as very light substrates for EUV filters and/or pellicles in high-energy astrophysics applications, as well as for EUV lithography.

1. Introduction

Thin free-standing pellicles are critical both as EUV/soft X-ray transmission filters in space instrumentation and as filters for particles used for cleanliness purposes in EUV Lithography [1–3]. In the field of astrophysics and space telescopes, these pellicles act as functional filters, blocking unwanted radiation while allowing the electromagnetic waves of interest to pass, and as barriers against contaminant particles. Past missions such as Solar Dynamics Observatory (SDO) [4] in the EUV and Chandra [5] in the X-rays are examples of missions that

employ filters for these wavelengths. Future missions such as NASA's *MUSE* [6–8], and ESA, or European Space Agency's *NewAthena* [9,10] – formerly *Athena* – will require large-area entrance filters (EFs) and filter wheel filters (FWFs) that need to be highly transparent in the EUV and X-ray domain, respectively, but that also reject visible and infrared light and survive the severe mechanical loads of launch [11–13]. Traditionally, for the aforementioned missions that have flown successfully, the EF/FWF roles have been fulfilled by multilayer thin films, usually comprising of either a free-standing polyimide (PI) film coated with thin metal layers such as aluminum or zirconium and supported by a

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metal mesh or an aluminum thin film directly supported by a very fine mesh [2,14,15]. PI is used in space for its proven technology readiness level for flight, mechanical robustness, high X-ray transmittance, and recent studies performed on both bare and aluminized PI pellicles have shown their fitness for applications in space and for the NewAthena mission in both the mechanical aspects [16] and EUV and X-ray aspects [17]. However, PI films at the relevant thicknesses for their application as space filter substrates (a few tens of nanometers) are close to their practical production limits, especially when scaled up to apertures of a few hundred cm^2 . Since next-generation instruments demand higher EUV/soft X-ray throughput, better out-of-band rejection, and large clear apertures, the study and identification of alternative and more transmissive filter materials to PI is highly beneficial.

For these reasons, CNT pellicles have been proposed and studied as an alternative substrate material for high-energy astrophysics, inspired by current trends in EUV Lithography (EUVL). Early work on free-standing CNT films tailored for EUVL applications as filter pellicles demonstrated EUV transmission up to $\sim 96\% - 99\%$, with transmission non-uniformity over large scales below $<0.4\%$, limited deflection under differential pressure, and thermal stability under high EUV source powers, highlighting CNTs as a promising pellicle material for the 13.5 nm generation of EUVL [18]. More recent studies in the semiconductor industry context have extensively explored CNT membranes for EUVL [19,20], showing tunability in CNT type (multi-walled, MW, or single-walled, SW), bundle density, and composition. EUV pellicles made with these materials are employed due to their essential properties to protect EUV masks under high thermal loads, such as surprising mechanical strength, high transparency, thermal resistance, [21,22], and scalability [23]. Recent developments are also in their quality assessments during mass production [24], underpinning their relevance as a valid alternative under study to other EUV pellicles employed in the semiconductor industry in general. A treatment usually employed for these EUV pellicles is post-growth high-temperature purification, employed to reduce the pellicles' metal content [25] used as a catalyst during thin-film growth: though no studies in literature explore the mechanical properties changes before and after treatment, and degradation of the mechanical performance is likely – thus unfavorable for astrophysics applications as supports. Beyond applications as EUV pellicles in EUVL, and as alternative substrates for astrophysics – the primary focus of this paper – recent studies have also shown interesting applications of SWCNT thin films for thin-film transistors [26]. These studies show that composition and morphology in CNT films are relevant and their physical properties depending on the synthesis conditions might be tailored for different applications: for example, in high-energy astrophysics, the highest amount of carbon per iron catalyst particle yield would be beneficial to increase as much as possible the EUV transmittance, while iron removal might be detrimental because of the potential (unexplored, yet) mechanical performance degradation of the pellicle which could hinder the resistance of the pellicles to vibration tests simulating launch conditions for space missions; while in the EUVL industry, maximization of the EUV throughput is the most important driving factor for the pellicles, with a preference for the post-treatment processes removing metal content, and for efficient chip manufacturing.

As briefly discussed, with proper synthesis parameters and processing, CNTs form a fibrous web of bundles of nanotubes – either single-walled (SW) or multi-walled (MW) [23] – that can be fabricated into large and free-standing membranes, combining the high tensile strength of individual tubes with the scalability of a two-dimensional network. Prior studies in the space field have shown that free-standing CNT pellicles can be more transparent in the EUV and soft X-ray ranges than PI films, both bare and when used as substrates for Al or Zr coating [13,27,28]. A comparative study by Mehew et al. [22] on the thermal properties of dual walled pellicles (DWCNT) compared to the SW ones, showed the higher thermal conductivity efficiency of the DWCNT pellicles, with no estimations of the difference in content of

systematic contaminating particles (such as Fe, or other atomic species depending on synthesis conditions) between the two kinds of pellicles. More generally, the number and diameter of CNT walls couple with the catalyst chemistry and growth conditions of CNT pellicles. For instance, during direct spinning syntheses, controlling sulfur promoters and their contact time with iron catalyst particles allows one to switch between CNT fibers composed mainly of single-, double- or multi-walled nanotubes [29]. The metallic catalyst content and CNT wall structure are synthesis-process-dependent variables, which are relevant when interpreting the properties and trends observed between SW and MW pellicles.

In this work, we directly compare the properties of free-standing SW and MW films produced by floating-catalyst chemical vapor deposition (FCCVD) with post-densification and without thermal treatments to remove residual catalyst. In particular, we measure the same kind of films with the following techniques and methods:

- EUV and soft X-ray transmittance measurements along with X-ray absorption spectroscopy (XAS) to quantify the elemental areal densities (C, O, Fe) and extract atomic fractions of (Fe, O) atoms with respect to C.
- broadband UV-Vis-IR transmission spectroscopy to extract effective per-carbon extinction cross sections, identifying π -plasmon absorption bands and van Hove features as characteristic fingerprints of these carbon structures
- spherical indentation of $\sim 60\text{--}100$ nm thick free-standing pellicles to extract in-plane elastic moduli and ultimate tensile stress (UTS)

The thicknesses of the membranes studied using spectroscopic techniques will primarily be of interest for space applications as substrates for the MUSE or NewAthena missions. In contrast, we employ thinner pellicles for their mechanical properties evaluation.

We show that MW films show a higher transmission than SW films with the same optical transmittance at the 550 nm wavelength in the 10–14 nm band, including 13.5 nm and 10.8 nm – respectively of relevance to EUVL and to solar astrophysics as the emission line of Fe_{XIX} in the flaring corona and hot flare plasma [6]. We attribute this difference in EUV transmittance performance to a systematically lower iron fraction in the MW networks. By analyzing the absolute interaction cross section per-carbon-atom, and confirming that it is similar between the two pellicles and behaves similarly to the case of graphene-like structures, we show that the higher Fe:C ratio is the dominant measured compositional factor explaining the reduced EUV throughput of the SW pellicles in the investigated energy range, and that this is a general property of the material. We also show that SW films exhibit a higher Young's modulus, whereas both pellicle types exhibit similar UTS. They have properties comparable to those of other materials, such as PI, with a lower effective density ($\sim 0.3\text{--}0.4 \text{ g cm}^{-3}$). By providing values of Young's moduli, UTS along with the pellicles densities – estimated in Table B.4 and Appendix B – we provide valuable input to multiphysics simulations to be used in the space context, as these are really important in the development of instrumentation for space missions in general [30,31], closing the gap to materials with higher usage in space and literature coverage such as PI. Overall, our results suggest that CNT pellicles, particularly MW-based networks, could provide a robust and promising route to next-generation free-standing thin-film filters and pellicles, meeting the ever-increasing demand for technological performance with improved EUV throughput and mechanical endurance.

Previous studies on carbon nanotube films generally considered EUV transmission, mechanical performance, or composition separately, often focused on a single type of CNT morphology or application [18, 19,26,27]: for example, Gubarev et al. [32] mainly focused on characterizing a more densely packed structure of SWCNTs. Here, we adopt an integrated approach tailored primarily for CNT pellicles for space applications. We systematically compare SW and MW CNT pellicles

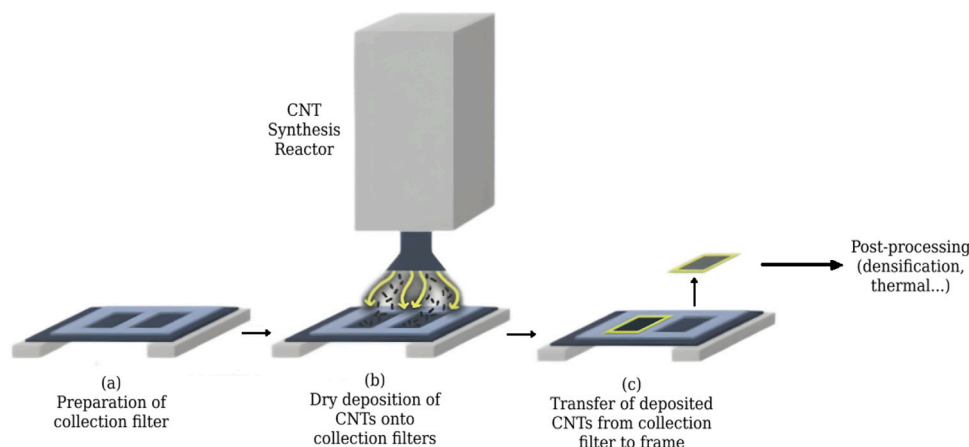


Fig. 1. General scheme of the Floating Catalyst Chemical Vapor Deposition (FC-CVD), i.e., the dry deposition fabrication process of CNT pellicles employed by CANATU Oy for the synthesis of the samples used in this work. Various steps are required in the process: (a) Preparation of a perforated collection filter mounted on a rigid support; (b) Dry deposition of CNTs growing in a carrier gas from the synthesis reactor onto the collection filter, forming a uniform CNT network. (c) Transfer of the deposited CNT film from the collection filter to the final frame, followed by post-processing to obtain the final freestanding CNT pellicle. The latter processes depend on the application of the pellicle. Alcohol-based densification is applied to astrophysical filter samples in this paper, whereas thermal purification treatments, which remove Fe contaminants for EUVL, are not used. Adapted and used with permission. Credit: CANATU Oy/J. Etula et al. [23].

grown by the same FCCVD process and matched in visible transparency, and we analyze EUV/X-ray, broadband optical and mechanical data on the same kind of samples. This allows us to directly correlate Fe content with EUV transmission, and also to find the density and mechanical properties associated with the different CNT pellicles under study. This approach provides a comparative account of the electromagnetic, mechanical, and physical properties of these pellicles, highlighting multi-walled films as ultralight alternatives to conventional polymers and aiding future efforts to optimize designs for space applications, while also providing valid inputs for ongoing research in EUV lithography. To our knowledge, this is the first work to correlate Fe content and EUV transmission at 13.5 nm, along with mechanical performance evaluations, on SW and MW FCCVD-grown CNT pellicles.

2. Materials and methods

2.1. CNT pellicle synthesis and samples

The freestanding pellicles employed in this work were manufactured at Canatu Finland Oy, using the floating-catalyst chemical vapor deposition (FCCVD) technique with the direct, dry collection of built CNT-bundles mass into a freestanding sheet, along with further steps involving alcohol-bath densification, also described in Mitin et al. [33]. We provide a schematic of the CNT pellicle fabrication process in Fig. 1.

The relevant review by Sehrawat et al. [34] details the main driving parameters of products from the FCCVD technique. By fine-tuning the various knobs relevant in the synthesis production, such as (and not limited to) catalyst precursors, environment, carrier gas, temperature of the process, and residence time, it is possible to fine-tune the properties of resulting CNT pellicle films, in the form of predominant presence of SW or MW CNTs, their bundle size, and thickness. We provide examples of experimentally observed SW/MW CNTs in Fig. 2.

SW and MW freestanding CNT-pellicle implementations similar to those of this work are described in Etula et al. [23] and Barbera et al. [27]. Throughout the paper, we will use the terms film, pellicle, and membrane interchangeably. The SW/MW designation reflects the main kind of CNT produced by the employed growth recipe: we will discuss the differences between the SW/MW pellicles in terms of chemical composition in Section 3.2 and in their optical absorption differences in Section 3.3. The effective thickness is evaluated from the pellicle visible transmittance at 550 nm, T_{550} by using a Beer–Lambert

calibration curve established for the specific FCCVD batches, following the methods employed by Ermolaev et al. [35]. The relative uncertainty of this effective thickness estimation is 10%, and its accuracy is within the same value when compared to the direct thickness measurement via Atomic Force Microscopy (AFM) step-height measurements done on samples deposited on Si wafers, as discussed in the original work [35].

Twelve samples with distinct frame geometries are employed. The first six witness samples (nominal $T_{550} \sim 40\text{--}55\%$) are in standard Luxel TF110 frames with a clear aperture of order 1 cm, suitable for synchrotron X-ray/EUV transmission measurements and optical/infrared spectroscopy laboratory work. The other six samples (nominal $T_{550} \sim 65\text{--}77\%$) were supplied on a frame (named push-pin frame) with a 21 mm circular free aperture (radius $a_m = 10.5$ mm), suitable for spherical-indentation mechanical testing.

All of the samples are listed in Table 1. Sample names are as follows: the first two letters mark the material type (SW or MW), clearly indexing either single- or multi-walled carbon nanotubes, while the two subsequent digits indicate the nominal direct transmittance T_{550} at 550 nm in % units, which, as stated, is linked to their effective thickness. The table also contains the effective thickness information as retrieved from the aforementioned empirical Beer–Lambert-like formula. Even though the effective thicknesses of the samples probed for UV–VIS–IR and X-ray spectroscopy analyses were not similar in value to those of the pellicles used in the samples for spherical indentation tests, the conclusions of this work are drawn at the material level (SW vs. MW): materials are deposited using very similar processes and the deposition time is the main driving factor of a larger thickness; from the optical side, measurements of samples with different thicknesses — expected from the Beer–Lambert model to absorb more in general — will be normalized to the carbon density of absorbers with the procedure detailed in Section 2.3 to compare with a fair effective extinction cross section the various results; from the X-ray and EUV transmittance analysis side, we will also obtain chemical information normalized to the amount of carbon absorbers, as will be described in Section 2.2. Finally, regarding the mechanical properties, the effective thicknesses probed in this work are in the range between 60 and 100 nm: within this interval, we do not observe systematic dependencies of the fitted Young's modulus and UTS on thickness beyond our experimental uncertainties. This is consistent with previous works showing that polymeric freestanding pellicles with thicknesses ranging between 45 and 400 nm did not show significant changes in the elastic (i.e. Young's) modulus [16,36], and any deviation that could occur in the ultra-thin regime ($t \lesssim 20$ nm) are outside the scope of this work.

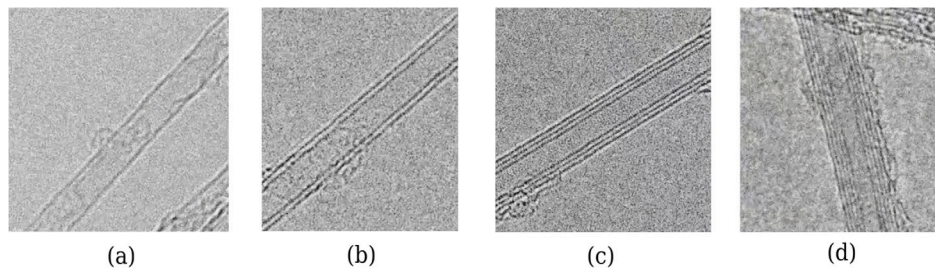


Fig. 2. Transmission electron microscopy (TEM) images of CNTs synthesized by floating-catalyst CVD with different wall counts. (a) predominantly single-walled, (b) double-walled, (c) three-walled, and (d) five-walled nanotubes. These examples illustrate how the CNT wall number can be tuned by growth conditions, enabling control over the surface properties of CNT networks. Adapted and used with permission. Credit: CANATU Oy/J. Etula et al. [23].

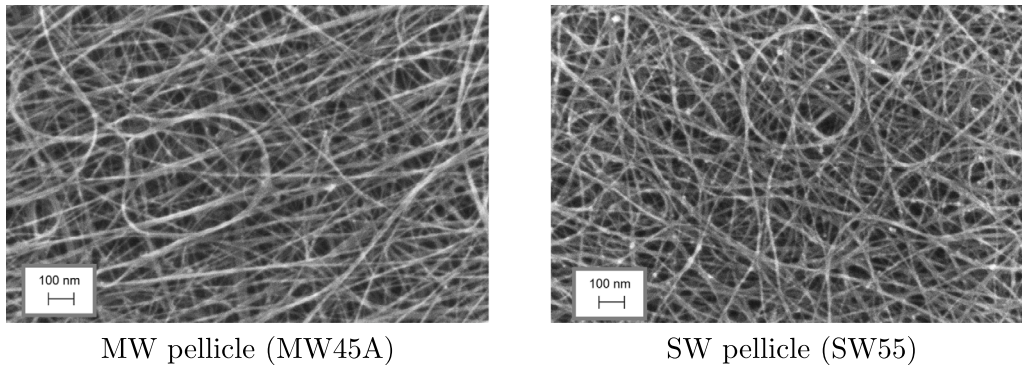


Fig. 3. Representative SEM appearance of MW (left) and SW (right) pellicles, for two pellicles in particular used in this work: MW45A and SW55. CNT bundles are visible, each containing multiple singular MW or SW CNTs. Both were acquired with identical settings (ZEISS Gemini FE-SEM, 5.00 kV, InLens, working distance of 3.7 mm; scale bar 100 nm), and, for visualization purposes, processed with uniform contrast enhancement (+35%) and sharpening (+40%).

Table 1

The CNT freestanding pellicles sample list. Naming scheme is multi-walled (MW) or single-walled (SW) with two digits as the $T_{50\%}$ pellicle nominal transmittance; Thickness is retrieved from the Beer–Lambert-like law based on the strategy by Ermolaev et al. [35]. Effective thickness uncertainty relative error of the cited strategy is 10%. Clear apertures measure the diameters of the freestanding membranes, excluding contributions from any inner or outer frames.

Sample	Type	Effective Thickness t	Frame (clear-aperture)
MW45A	MWCNT	(188 ± 19) nm	TF110 ($\varnothing 10$ mm)
MW45B	MWCNT	(188 ± 19) nm	TF110 ($\varnothing 10$ mm)
SW55	SWCNT	(141 ± 14) nm	TF110 ($\varnothing 10$ mm)
SW54	SWCNT	(145 ± 14) nm	TF110 ($\varnothing 10$ mm)
SW43	SWCNT	(199 ± 20) nm	TF110 ($\varnothing 10$ mm)
SW44	SWCNT	(194 ± 19) nm	TF110 ($\varnothing 10$ mm)
MW68A	MWCNT	(91 ± 9) nm	Push-pin ($\varnothing 21$ mm)
MW68B	MWCNT	(91 ± 9) nm	Push-pin ($\varnothing 21$ mm)
MW68C	MWCNT	(91 ± 9) nm	Push-pin ($\varnothing 21$ mm)
SW65	SWCNT	(102 ± 10) nm	Push-pin ($\varnothing 21$ mm)
SW71	SWCNT	(81 ± 8) nm	Push-pin ($\varnothing 21$ mm)
SW77	SWCNT	(62 ± 6) nm	Push-pin ($\varnothing 21$ mm)

To qualitatively assess the morphology of the samples, we acquired two representative SEM images on a ZEISS Gemini FE-SEM (5.00 kV, InLens, working distance 3.7 mm; scale bar 100 nm) and represented them in Fig. 3, juxtaposing one MW pellicle (MW45A) and one SW pellicle (SW55). At this magnification, the two families of bundles look essentially the same — an entangled fibrous network.

2.2. EUV and X-ray transmittance characterization and analysis

The EUV and X-ray measurements were carried out at the METROLOGIE beamline's soft X-ray branch of the SOLEIL synchrotron (proposal #20231204), allowing for the measurement of absolute transmittance

$T(E)$ over the 30–1600 eV range. The specific experimental parameters employed in the beamline are listed in Appendix A, Table A.3. TF110-mounted samples (Table 1) were mounted at normal incidence in dedicated vacuum-compatible sample holders manufactured at the Osservatorio Astronomico di Palermo (INAF-OAPA). For each energy E , we recorded the incident (*open beam*) and transmitted fluxes, $I_0(E)$ and $I_t(E)$, translating the holder between an open reference aperture and the sample using the beamline motion stages. The transmittance was then retrieved as $T(E) = I_t(E)/I_0(E)$, in accordance to previous works [2,17,28]. Dark currents were monitored and found negligible; typical relative uncertainties on T with these measurements are ~ 0.3 –1% (1σ).

The chosen range between 30 and 1600 eV for the transmittance scans allowed the measurement and retrieval of two main results: (i) study and compare the EUV transmittance in relevant wavelengths in the 10–14 nm range, i.e. ~ 124 –89 eV,¹ to compare pellicle throughput in a band containing wavelengths of interest to both solar astrophysics and EUVL; and (ii) explore in the out-of-band soft X-ray range the leading photo-absorption edges of the central atoms compounding our freestanding pellicles: C K -edge (at ~ 284 eV), O K -edge (near ~ 543 eV), and Fe $L_{2,3}$ -edge (near ~ 706 –721 eV), with energy values taken from Henke et al. and the Center for X-ray Optics (CXRO) online database [38]. We show the EUV transmittance comparison in Section 3.1, and the results from the X-ray transmittance measurements and analysis will be part of the chemical atomic fraction results in Section 3.2 and model-fits results in Appendix B.

We model each pellicle as a thin, laterally uniform mixture of elements j (here C, O, Fe) with areal densities $\Sigma_j = \rho_j t_j$ (mass per unit area, e.g. g cm⁻²), the product of density and thickness of each sample

¹ Photon energy (eV) and wavelength (nm) are converted via $E = hc/\lambda \simeq (1239.84 \text{ eV} \cdot \text{nm})/\lambda$; Planck's and speed of light constants from CODATA [37].

layer. The optical depth to use, which follows Beer–Lambert additivity, is

$$\tau(E) = \sum_j \left(\frac{\mu}{\rho} \right)_j(E) \Sigma_j, \quad (1)$$

where $(\mu/\rho)_j(E)$ are the mass attenuation coefficients, depending on the atomic species and the related tabulated atomic scattering factors, retrieved for this work by the Henke/CXRO source [38,39]. This strategy is commonly employed for the study of multilayered thin films, as shown in research [2,17,28]. The model transmittance is

$$T_{\text{model}}(E) = \exp[-\tau(E)]. \quad (2)$$

Away from absorption edges, Eqs. (1)–(2) describe the spectra with a smooth-looking curve alone, which also correctly models the photo absorption jumps using tabulated Henke-based mass attenuation coefficients. Spectral regions dominated by near-edge fine structure [40] were masked in the XAS analysis and fitting routine. Fits are performed in the areas around each edge using the baseline $(\mu/\rho)_j(E)$, and the step-like edge term is used to infer the areal density, i.e., free parameter Σ_j , for each absorbing element. Moreover, even though hydrogen is potentially part of our structure (in a hydrogen-per-carbon fraction between 0 and 1, considering limiting cases of graphene and benzene), this cannot be probed with our XAS analyses as H, as opposed to C, O, and Fe with their X-ray photo-absorption edges, is highly transparent and lacks notable experimental features in the EUV and X-ray energy range [39].

From a modeling perspective, the *total* areal density associated to a CNT sample is $\Sigma_{\text{tot}} = \Sigma_C + \Sigma_O + \Sigma_{Fe}$. When needed, we convert mass areal density to atomic areal density (atoms' count per cm^{-2}) as

$$n_j = \frac{\Sigma_j}{A_j} N_A, \quad (3)$$

with A_j the (abridged) standard atomic weight [41] and N_A Avogadro's number. We refer to $(j : C)$ as the (dimensionless) atomic ratio of element j to carbon, given by the following

$$(j : C) \equiv \frac{n_j}{n_C} = \frac{\Sigma_j}{\Sigma_C} \frac{A_C}{A_j}, \quad (4)$$

which is what we report for O:C and Fe:C in Section 3.2. These definitions also set up the later normalization of broadband data to an effective per-carbon atom effective extinction cross section (Section 3.3).

Best-fit values for Σ_C , Σ_O , and Σ_{Fe} are retrieved via the Levenberg–Marquardt least-squares routine implemented in SciPy [42,43]. We report best-fit parameter uncertainties at 3σ confidence level, as resulting from the covariance matrix provided by the fitting procedure and confirmed via a Bayesian inference approach using Chi-squared contour plots. Comprehensive treatments of these procedures can be found in Press et al. [44].

2.3. UV–Vis–IR spectroscopy

We measured broadband direct transmission of CNT pellicles in their TF110 frames (Table 1) from 190 nm to 25000 nm using two instruments available at the INAF–OAPA laboratory. For the UV–Vis–IR wavelength range (190–1500 nm) we used a PerkinElmer Lambda 1050+ double-beam spectrophotometer: both instruments operated in air under normal incidence conditions in a standard direct transmission geometry. Spectral resolution was 2 nm (UV/Vis) and 20 nm (NIR), with a rectangular beam roughly vertical by horizontal, $10 \times 2 \text{ mm}^2$ on the sample. We extended the coverage to the mid and far-infrared range (1.28–25 μm), hereafter IR range in this work, with a PerkinElmer Spectrum 3 Fourier-transform infrared (FTIR) spectrometer with constant resolution in energy of 4 cm^{-1} (i.e. $\sim 5 \times 10^{-4} \text{ eV}$). In all cases, we measured multiple positions on each $\sim 1 \text{ cm}$ aperture to assess spatial uniformity within a sample, in terms

of transmittance uniformity: the standard deviation across positions and instrument repeatability define the reported uncertainty in the transmittance at a given wavelength, $T(\lambda)$. The partial overlap between the two instruments' ranges allowed for stitching the spectra.

Napierian apparent absorbance $\mathcal{A}_{\text{app}}(\lambda) = -\ln(T(\lambda))$ is retrieved from direct transmittance [45]. The optical and infrared spectra were acquired in standard direct-transmission geometry. For a porous thin film material (such as the CNT bundle networks in this work), $\mathcal{A}_{\text{app}}$ should not be interpreted as a pure absorbance, since it can include true absorption together with residual light scattered outside the collection angle of the detector.

For normalization purposes and to compare samples with slightly different thicknesses, we use a Beer–Lambert-like extinction formalism, $\mathcal{A}_{\text{app}}(\lambda) = n_C \sigma_C^{\text{eff}}(\lambda) t$, where n_C is the carbon number density with cm^{-3} unit, t is the effective film thickness and σ_C^{eff} is the effective extinction cross section per carbon atom. Absorbance is normalized to the number of carbon atoms per unit area using the areal-density framework of Section 2.2. As $n_C t = \Sigma_C / (A_C m_u)$, with Σ_C the carbon mass areal density from the XAS edge fits, A_C the standard atomic weight of carbon, and m_u the atomic mass unit, the carbon-normalized effective extinction cross section is

$$\sigma_C^{\text{eff}}(\lambda) = \mathcal{A}_{\text{app}}(\lambda) \frac{A_C m_u}{\Sigma_C}. \quad (5)$$

Throughout the manuscript, σ_C^{eff} should be interpreted as an effective extinction cross section obtained from direct transmittance, including absorption together with possible residual reflection and scattering losses outside the detector collection angle and thus not only considering pure absorption of light. Clearly, product $A_C m_u$ is just the mass per C atom. Eq. (5) removes the explicit dependence on the film thickness, using only quantities obtained either from optical/infrared spectroscopy or from the XAS fits described in Section 2.2, thus the effective extinction cross section σ_C^{eff} allows to compare different pellicles on a common “per carbon atom” basis across UV–Vis–IR.

We use Eq. (5) to compare SW and MW pellicles on the same footing in the 0.05–6.5 eV range (i.e., 25 μm –190 nm), reporting both the raw $T(\lambda)$ and the derived $\sigma_C^{\text{eff}}(\lambda)$ curves in Section 3.3. This normalization lets us compare different samples with different thicknesses by focusing on the per-carbon treatment, and thus allows us to compare MWs to SWs. Moreover, we present cross-section values to compare with literature results, either as peak values or as integrated cross sections, to compare different allotropes of carbon. The (dimensionless) oscillator strength f over an absorption band in the energy interval (E_1, E_2) – described in Supplementary Information 1 – is

$$f = \frac{1}{\mathcal{K}} \int_{E_1}^{E_2} \sigma_C^{\text{eff}}(E) dE, \quad \mathcal{K} = \frac{\alpha \lambda_C^2 m_e c^2}{2} \approx 109.761 \text{ Mb eV} \quad (6)$$

where α is the fine structure constant, λ_C is the electron Compton wavelength, m_e is the electron mass, and c is the speed of light in vacuum.[37,46] We use Mb = 10^{-18} cm^2 , i.e. megabarn, as the unit of cross section. Because $\sigma_C^{\text{eff}}(E)$ is obtained from direct-transmission effective extinction, the oscillator strength quantities reported in this work should be interpreted as an effective oscillator-strength proxy for comparing CNT pellicles and also for comparison with literature values.

2.4. Mechanical testing

We investigate the mechanical properties of pellicles made with both SW-CNT and MW-CNT using the spherical indentation technique. This technique was chosen explicitly as opposed to pull-tests described in industry standards such as ASTM D882² because such standards are not tailored to the study thin and ultra-thin pellicles with thicknesses $t \lesssim 100 \text{ nm}$ – comparable to the thicknesses used in our work and detailed in Table 1. Specialized techniques can indeed allow the pull-tests

² Related website: <http://www.astm.org/Standards/D882>.

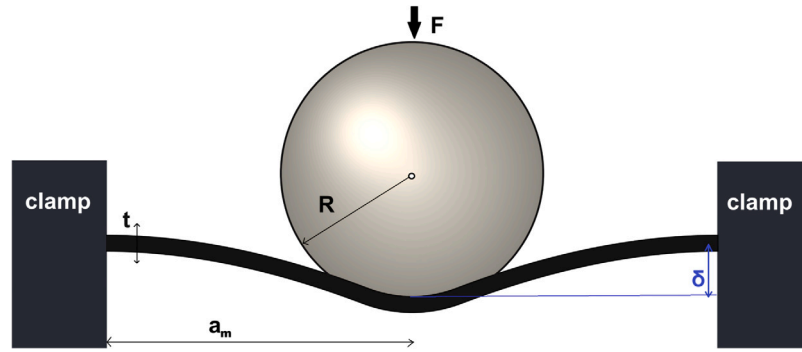


Fig. 4. Illustration of the mechanical spherical indentation geometry. A clamped circular CNT membrane of radius a_m and thickness t is indented by a sphere of radius R to a central deflection δ under force F . The central deflection δ is measured from the clamped plane to the membrane apex under the indenter, or equivalently, as the distance traveled vertically by the sphere to reach equilibrium force F .

to infer mechanical properties from ultra-thin polymers [36,47], though both the adaptability of these techniques to CNT pellicles, and the effect of the ultra-high surface area of CNTs and CNT bundles [48] on the reliability of mechanical properties inferred with these tests, are largely unexplored aspects. On the other hand, spherical indentation has been successfully employed in the past with nano-indentation atomic force microscopy (AFM) setups to study ultra-thin pellicle layers such as a single graphene layer by Lee et al. [49] obtaining values of the in-plane Young's modulus of $E_{\text{graphene}} = (1.0 \pm 0.1)$ TPa, comparable to the values found in literature for the moduli of very thin SW-CNT bundles (diameter $d \leq 5$ nm) [50,51], diamond [52] or (in-plane) graphite [53]. Alternative techniques, as bulge tests, can be the preferred choice to test thin-films for space instrumentation [2,16], but they are challenging to adapt to air-permeable materials such as the CNT films in this work. Thus, the spherical indentation technique is suitable for the study and inference of mechanical properties of thin and ultra-thin membranes such as CNT thin films, and extensive research is present on the accuracy of approximate and exact modeling of the problem [54,55], with modern approaches also interested in extending the validity of the model to include hyper-elastic materials like rubber [56].

As noted in Section 2.1, the CNT pellicles from Table 1 used for mechanical testing are mounted on circular frames with a 21 mm clear aperture (membrane radius $a_m = 10.5$ mm). We ran indentation tests on a Mark-10 F105 machine equipped with a spherical indentation head (indenter radius $R = 4.5$ mm), which measured the central out-of-plane deflection δ . The stage position's distance resolution was 0.01 mm. We used a constant approach speed of 10 mm min^{-1} and sampled force and displacement at 20 Hz. The pre-contact baseline in the force-time plot was useful for zeroing the force values, and the beginning of non-zero deflection ($\delta \gtrsim 0$) was set at the first non-zero force, thus defining $\delta = 0$ at first contact. Force-displacement (F, δ) data points were registered up to the breaking point F_b , as the samples were all brought to failure during the experiments. Fig. 4 provides a simple schematic of the geometry with membrane radius a_m , spherical indenter radius R , and center displacement δ .

To extract more than the breaking loads from the experiments, we adopt the finite-contact ("blunt sphere") formulation of Begley and Mackin [54] – original paper's Eq. 23 hereafter referred to as the Begley model – for a clamped circular membrane with uniform prestress in the elastic regime, whose range of applicability covers our case as the ratio between indenter radius and membrane radius is $R/a_m \simeq 0.4$. The Begley model approximation explicitly depends on the Young's modulus and on the membrane prestress, and it does not depend on Poisson's ratio (assuming $\nu = 0.5$).

The following models employ deflection δ - or its non-dimensional counterpart $x = \delta/R$ normalized with the indenting sphere radius- as a function of force F . Defining the two fitting parameters as

$$A = \frac{E t R}{3\pi^2}, \quad B = 4\pi^2 \epsilon_0, \quad (7)$$

with A in N and B dimensionless, E the in-plane Young's modulus, and ϵ_0 the dimensionless pre-stretch of the membrane which is proportional to the radial displacement of the membrane's diameter due to previously present stress which is independent on the indentation process as per description in Begley et al. [54], the expression of the Begley model of deflection depending on impinging force is the following.

$$\frac{\delta}{R} = \frac{2}{3\pi} \left(\frac{a_m}{R} \right)^{3/4} \frac{\left(\sqrt{(F/A)^2 + B^3} + F/A \right)^{2/3} - B}{\left(\sqrt{(F/A)^2 + B^3} + F/A \right)^{1/3}}. \quad (8)$$

We also benchmarked the exact large-deflection spherical indentation membrane model by Jin et al. [55] against representative experimental datasets. While both models reproduced the measured load-deflection curves, unconstrained Jin fits push the Poisson ratio ν to converge to large negative values, $\nu < -1$, which is not considered physical for the CNT pellicles under study; a full Begley–Jin model comparison is provided in the Supporting Information.

Begley model-derived parameters (E, ϵ_0) can be inferred from the parameters (A, B) in Eq. (7) via simple inversion, and the procedure to obtain best-fit parameters over experimental data with Levenberg–Marquardt parallels Section 2.2. Young's moduli retrieved with this procedure are comparable to those retrieved by best-fit to numerical simulations within about 13% for comparable R/a_m (Begley and Mackin's [54] original paper's Fig. 8). Since thicknesses from Table 1 are not as precise as the uncertainties in displacement and force in our experimental setup, the 2D Young's modulus $E_{2D} \equiv E \cdot t$ can be inverted from left side Eq. (7) with the highest precision: the resulting best fit values of (E_{2D}, ϵ_0) along with the pre-stretches will be listed in Appendix C. In the same framework, the membrane stress in the contact region $\bar{\sigma}$ is the following:

$$\bar{\sigma}_{\text{Begley}} = \sqrt{\frac{E F}{6\pi t R}} \quad (9)$$

Considering that pellicles are studied up until fracture load F_b , ultimate tensile stress can be inferred with Eq. (9), using the breaking force F_b , i.e. $\bar{\sigma}_{\text{Begley}}^{\text{UTS}} = \sqrt{\frac{E F_b}{6\pi t R}}$.

Standard deviation errors on the force values are taken from the pre-contact baseline (typically 4 to 6×10^{-4} N for the experiments in this work), about equal to 2 to 3 times the force sensor resolution. Effective thickness t carries $\sim 10\%$ relative uncertainty (Table 1), which propagates directly into E and $\bar{\sigma}^{\text{UTS}}$ retrieved by using Eq. (9) at $F = F_b$ and inverting the first part of Eq. (7). Best-fit uncertainties on A and B via Levenberg–Marquardt are smaller by comparison. Small errors on values for a_m and R are negligible at our tolerances.

3. Results and discussion

3.1. EUV (10–14 nm) transmittance comparison

Fig. 5 shows the experimental EUV transmittance for all TF110-mounted pellicles in the restricted 10–14 nm window (124–89 eV),

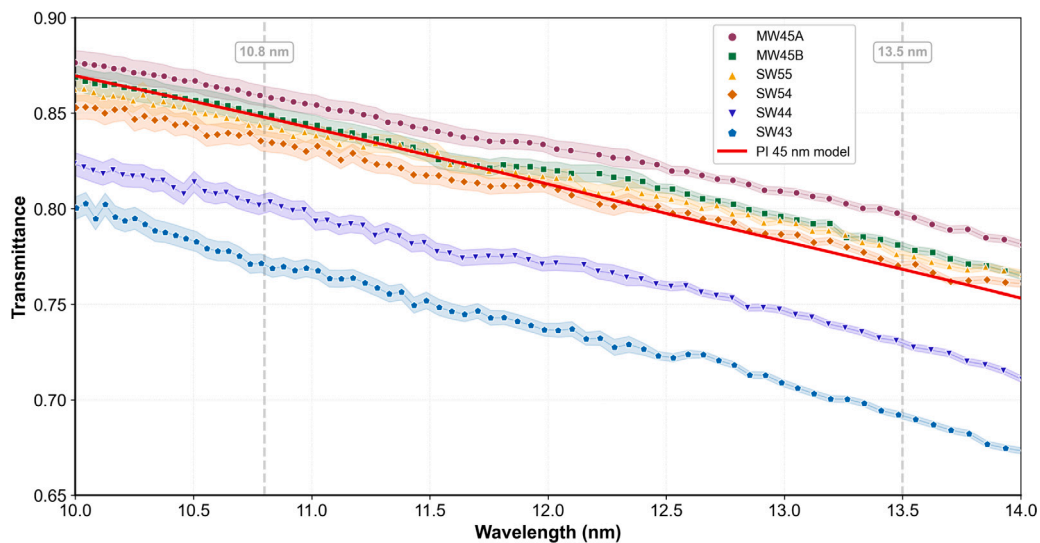


Fig. 5. Measured EUV transmission in the 10–14 nm window for all TF110-mounted pellicles in Table 1. Shaded bands show the experimental measurement uncertainties. Vertical lines are at wavelength positions 10.8 nm and 13.5 nm. The simulated transmittance of a 45 nm thick PI pellicle is also displayed for comparison purposes.

highlighting two wavelengths of interest, 10.8 nm and 13.5 nm (Section 1), while also comparing their performance to the simulated transmittance of a membrane of PI with chemical formula $C_{22}H_{10}N_2O_5$, density $\rho_{PI} = 1.43 \text{ g cm}^{-3}$, and thickness $t_{PI} = 45 \text{ nm}$, which correspond to a C-areal density for this pellicle of $\approx 44.5 \times 10^{-7} \text{ g cm}^{-2}$, comparable to the carbon amount found in the other pellicles of the work and detailed in in Appendix B, Table B.4. The chosen simulated PI pellicle is also representative of the thickness of the substrate pellicle recently studied during the design and development of Athena X-IFU thermal filters in Puccio et al. [17]. We detail corresponding transmittances at these wavelengths in Table 2, where the model is evaluated by employing the machinery described in Section 2.2. For completeness, it is relevant to state that diffuse transmittance could be relevant for the pellicles under study in the EUV range: while this particular aspect is outside the scope of this work, recent works have shown that this effect is very small in this EUV range for highly transmissive pellicles. Pollentier et al. [57] found less than 0.5% scattered light for CNT pellicles more transmissive than 85% at 13.5 nm, whereas Kim et al. [58] have shown also an almost negligible impact of scattering and reflectance in the EUV range of interest for pellicles of similar transmittance to the ones investigated in this work.

A clear trend emerges: the MW pellicles (MW45A, MW45B) transmit systematically more than the SW set (SW55, SW54, SW44, SW43) over the whole 10–14 nm EUV band. In particular, MW45A/MW45B outperform SW55/SW54 with a $\sim 1\text{--}3\%$ absolute transmittance improvement at both wavelengths of interest, despite being less transparent in the visible band at 550 nm. For example, at 13.5 nm, the MW pellicles transmit $T \approx 80\%$ whereas SW samples with comparable visible transmittance (SW43/SW44) at the same EUV wavelength transmit $\sim 69\text{--}73\%$ (Table 2).

One possible explanation for this behavior lies in the chemical composition. As shown next (Section 3.2), and relative to C atoms present in all samples, the SW pellicles carry more residual Fe than the MW ones, with similar O:C atomic fraction content. The change in chemical composition does reflect a change in transmittance in the EUV range due to absorption from the Fe $M_{2,3}$ edge. The effect is relevant regardless of the amount of carbon in each pellicle: in fact, the MW pellicles, as detailed in Table B.4, are found to possess more carbon than the other pellicles on average, while oxygen is present in similar ways throughout the various CNT pellicles under study.

Two remarkable experimental aspects are, thus, found: (i) the MWCNT pellicles can outperform PI pellicles with similar carbon content in the EUV transmittance, which is of great potential interest to the

Table 2

EUV transmittance at the two reference wavelengths for the TF110-mounted samples in Fig. 5. Values registered as averages accompanied by their experimental measurement uncertainty. The simulated transmittance of a 45 nm PI pellicle is overlaid for comparison.

Sample	$T(10.8 \text{ nm})$	$T(13.5 \text{ nm})$
MW45A	0.859 ± 0.005	0.797 ± 0.002
MW45B	0.849 ± 0.005	0.780 ± 0.002
SW55	0.843 ± 0.005	0.776 ± 0.002
SW54	0.835 ± 0.005	0.770 ± 0.002
SW44	0.802 ± 0.005	0.730 ± 0.002
SW43	0.770 ± 0.005	0.691 ± 0.002
PI 45 nm model	0.848	0.768

NASA MUSE mission, or future solar missions, as currently the baseline adopts 75 nm PI membranes as structural support; and (ii) that the amount of carbon is not the primary driver in EUV transmittance, with scientific evidence pointing out to the iron leftover from the synthesis process as the main one.

3.2. Chemical composition analysis

In Appendix B, Table B.4, and Fig. B.11 report the full EUV/X-ray spectra along with XAS best-fit analyses and best-fit parameter values. They provide the elemental areal densities, which we will later convert to atomic fractions. The fits followed the description in Section 2.2: C K, O K, and Fe $L_{2,3}$ edge-regions are fit with the models in Eq. (1) and (2), using Henke-based mass attenuation coefficients. The fits are excellent between the models and the experimental data over most of the energy range 30–1600 eV. For $E \lesssim 100 \text{ eV}$ (i.e. $\lambda \gtrsim 6 \text{ nm}$), systematic deviations do appear, suggesting future approaches to improve the modeling in the EUV region including effects from leftover hydrogen, and tailor the carbon's tabulated scattering factor coefficients to include collective effects in CNT bundle networks and/or losses of EUV/soft X-rays due to scattering by the nanotube structures.

Fig. 6 illustrates one of the main results in terms of atomic fraction resulting from the application of Eq. (3): that SW pellicles carry a higher Fe:C atomic fraction than the MW ones. The data show that SW pellicles possess an Fe atomic fraction of $\sim 3\text{--}4\%$ compared to $\sim 0.5\text{--}1\%$ in MW pellicles.

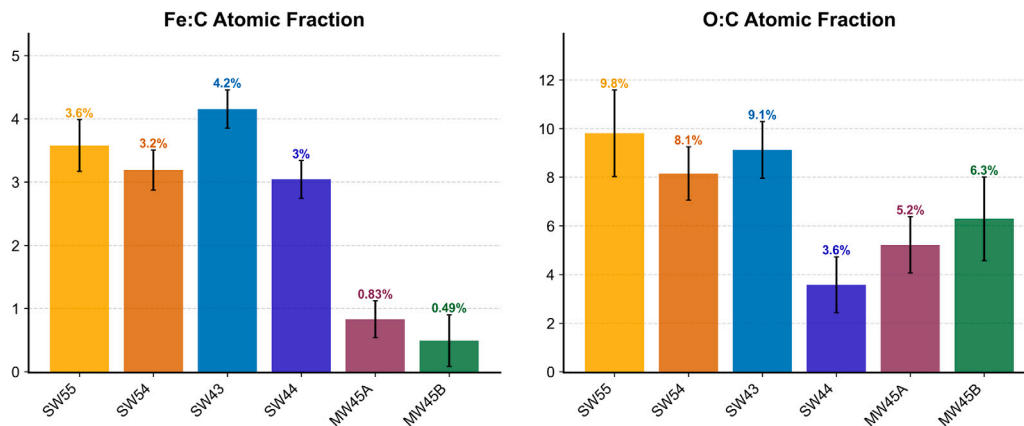


Fig. 6. Atomic fraction in % based on the XAS data analysis of all of the TF110-mounted pellicles in Table 1, i.e., the amount of Fe atoms (left) or O atoms (right) expected per C atom present in the CNT network. Values reported in the figures represent the average atomic fraction, and vertical black error bars show the propagated uncertainties (3σ).

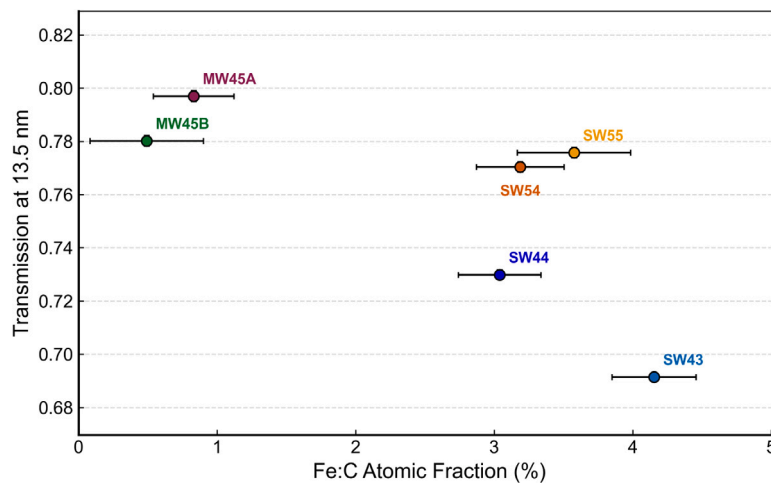


Fig. 7. Transmission at 13.5 nm as a function of Fe:C atomic fraction for the SWCNT and MWCNT pellicles under study. Average EUV transmission at 13.5 nm is shown, and horizontal error bars indicate the uncertainty in the Fe:C atomic fraction extracted from XAS fits. MW pellicles (MW45A, MW45B) have lower Fe:C fractions and show systematically higher transmission than SW pellicles (SW43, SW44, SW54, SW55), demonstrating a superior EUV performance primarily linked to their reduced Fe content, and lessened photo-absorption by the Fe $M_{2,3}$ edge.

Discussing further, the reported O:C atomic fraction does not follow peculiar trends with respect to the different chemical structures in the various pellicles: one pellicle in particular, SW44, stands out in having many fewer O atoms than all of the other pellicles, but we could not find an explanation for this in terms of macroscopical or microscopical properties. Atomic fraction estimations from the XAS analyses tend to be remarkably imprecise when there is a low amount of absorbers for that particular species of atom (i.e., in the case of Fe for our MW pellicles). The ratio $(\text{Fe}:\text{C})_{\text{SW}}/(\text{Fe}:\text{C})_{\text{MW}}$ can be interpreted in two ways: assuming one iron atom one finds how many more carbon atoms are found in the MW pellicles' average tube per Fe atom in the catalyst, while considering one carbon atom one sees how much more iron per C atom is present in the SW pellicles. SW's Fe:C atomic fraction comes up to a midrange of 3.6% ($\pm 0.6\%$ as half-range), while the same for the MW pellicles comes up to 0.66% ($\pm 0.17\%$). The ratio is about 5.5, which is qualitatively consistent with nanotubes up to 5 walls, as in the TEM images of Fig. 2. To further exemplify the correlation between EUV transmittance decreasing with higher Fe:C atomic fraction, a relevant $T(13.5 \text{ nm})$ vs Fe:C fraction scatterplot is shown in Fig. 7.

Thus, reducing Fe:C from $\sim 3\%$ – 4% (typical of our SW pellicles) to $\lesssim 1\%$ (MW pellicles) yields an $\sim 5\%$ – 10% absolute gain in $T(13.5 \text{ nm})$ at fixed visible transparency. We infer a similar conclusion for the 10.8 nm line. Furthermore, MW pellicles can be more transmissive alternatives in the EUV to the PI pellicles in general, showing that they can be alternative substrates to the more commonly chosen and safer option of PI for EUV thin film filters in solar missions where EUV observations can be relevant, allowing to increase the photon counts of observation of flares, or fingerprint of coronal loops in the 10.8 nm lines [6,59], simply by tuning the kind of pellicle and the number of catalyst particles during the pellicle growing process.

3.3. Broadband optical properties

Fig. 8 shows the room-temperature UV–Vis–IR transmittance and effective per-carbon extinction cross section, as described in Section 2.3, of three representative membranes from Table 1 (SW55, SW43, MW45A). The left panel shows the absolute transmittance $T(\lambda)$ —which

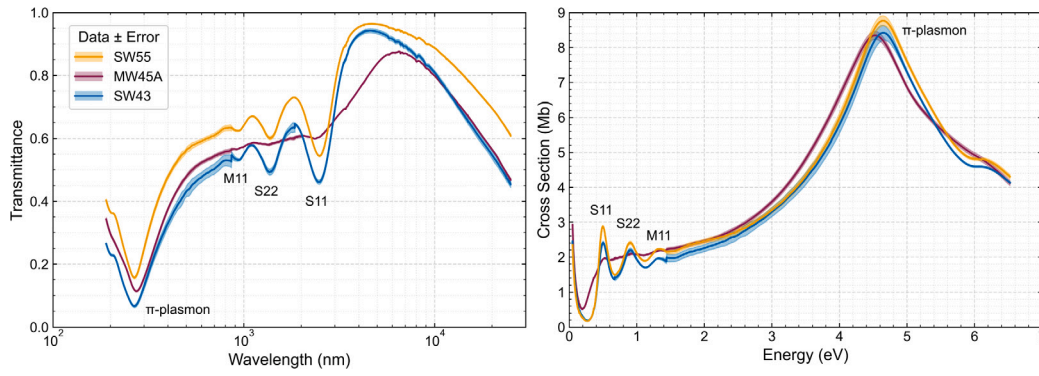


Fig. 8. Broadband optical response figure for pellicles (SW55, SW43, MW45A). The same legend applies to both plots. **Left:** absolute transmittance $T(\lambda)$ from 190 to 25000 nm. **Right:** effective per-carbon atom extinction cross section $\sigma_C^{\text{eff}}(E)$ obtained employing Eq. (5). Written labels indicate the π -plasmon and the CNT-characteristic van Hove features (S_{11} , S_{22} , M_{11}). Shaded ribbons show experimental uncertainties for $T(\lambda)$, and propagated experimental uncertainties for the $\sigma_C^{\text{eff}}(E)$.

depends on sample thickness— while the right panel reports the effective per-carbon extinction cross sections $\sigma_C^{\text{eff}}(E)$ obtained via Eq. (5) which uses the Σ_C results from the XAS analyses of Section 3.2 and Appendix B to normalize absorbance and place different samples on the same per-carbon atom standpoint. Plotted as $\sigma_C^{\text{eff}}(E)$, the three datasets nearly overlap above ~ 2 eV and diverge mainly in the IR. Two features are most informative from the figure: the strong UV π -plasmon band and the weaker Vis–IR interband transitions, where present.

As a caveat, $\sigma_C^{\text{eff}}(\lambda)$ is an effective extinction cross section derived from direct-transmission measurements. For a porous CNT network, this quantity may include absorption together with residual reflection and scattering outside the detector collection angle. It is therefore used here as a comparative carbon-normalized optical descriptor of the SWCNT and MWCNT pellicles measured under the same experimental geometry, rather than as a complete separation of absorption, reflection, and diffuse scattering. Furthermore, the quantity is in principle a broadband effective extinction cross section averaged over the random in-plane orientations of the single nanotubes and their distribution in bundles, as seen in the SEM images of Fig. 3. Unpolarized (thermal) illumination is employed in the instrumentation used for the optical and infrared spectroscopy (Section 2.3). Since it is known that for isolated, aligned SW-CNTs the absorption is strongly polarization-dependent [60], the ensemble averaging over random polarization directions and the network of nanotubes is expected to reduce the apparent cross section of our findings relative to a perfectly aligned case with polarized light. We state the polarization dependency of the optical properties of carbon nanotubes for completeness: the results remain relevant to the extent of comparative results between the pellicles studied in the work and in all applications where incoherent sources of light are employed.

The strongest band—an absolute minimum in $T(\lambda)$ and a maximum peak in $\sigma_C^{\text{eff}}(E)$ —marks the π -plasmon, at 4.65 eV for MW45A and 4.53 eV for the SW pellicles (± 0.05 eV). The π -plasmon absorption band is common to CNTs and to carbon allotropes with sp^2 hybridization, as seen in studies of optical properties of SW pellicles, [35] graphene, [61] and graphite, [62,63] with tabled refractive index values³ available in the refractiveindex.info database maintained by Polyan-skiy [64]. At the π -plasmon, peak effective per-carbon extinction cross sections $\sigma_C^{\text{eff},\pi} \approx 8\text{--}9$ Mb, of the same order of magnitude as values in the literature for graphene (≈ 14 Mb) [61] and graphite $\approx 10\text{--}12$ Mb [62,63]. By fitting the peak with a pseudo-Voigt to assess the integral of cross section in energy of the π -plasmon absorption band,

and making use of Eq. (6), oscillator strengths are found to be $f_{\text{SW43}}^{\pi} \approx 0.26$ and $f_{\text{SW55}}^{\pi} \approx f_{\text{MW45A}}^{\pi} \approx 0.28$. Applying a similar strategy to the broad UV-peaks of graphene in Tikušis et al. [61] yields an oscillator strength of $f_{\text{graphene}}^{\pi} \approx 0.27$, which means that in all of these cases, the transitions are highly allowed. The integrated cross sections are comparable, even if the peak values for SWs are lower. Since the π -plasmon energy has been shown to depend on the CNT diameters [65], lower peaks are likely caused by Gaussian broadening due to the CNT diameter distribution within the network. This broadening lowers the peak amplitude without altering the integrated oscillator strength, which remains $f \sim 0.26\text{--}0.28$ for all SW, MW pellicles, and graphene cases.

In the near-IR and in the right panel (cross section), the SW pellicles show the expected van Hove singularities-related transitions: S_{11} at $\sim 0.55\text{--}0.75$ eV and S_{22} at $\sim 0.9\text{--}1.1$ eV, characteristic of *semiconducting* CNTs, and M_{11} at $\sim 1.2\text{--}1.4$ eV, characteristic of *metallic* CNTs. These assignments follow the standard Kataura/empirical maps for CNT ensemble spectra [66–68]. The MW sample shows only weak van Hove structure and a smooth continuum, consistent with metallic behavior and/or strong broadening of absorption bands, which is consistent with reports on MW-CNT optical/IR fingerprints [69].

Thus, by normalizing to the number density of carbon absorbers, we interpret the optical/infrared apparent-attenuation behavior of SW and MW pellicles in terms of chemical structure and electronic states, and, since the cross section is comparable between the MWCNT and SWCNT pellicles in the π -plasmon region within error, we conclude that the lesser quantity of iron found in the MW pellicles does not impact the UV–VIS–IR interaction properties of the pellicles in a way similar to what was found in the EUV range.

3.4. Mechanical performance

The experiments of spherical indentation of the samples with 21 mm clear aperture (Table 1) are shown in the load–deflection curves in Fig. 9. The clear picture is that MW pellicles rupture at almost twice the applied force as SW pellicles: an aspect highlighted by the overlaid shaded bands and red crosses in the figure.

The spherical indentation model fits the elastic portion of the curves well—this is reported in Appendix C (Fig. C.12), together with the numerical results in Table C.5. Comparing the performance shown in Fig. 9 with the expected performance of a 45 nm thick PI pellicle, assuming a similar UTS, the expected breaking point for the polyimide assuming $E_{\text{PI}} = 10$ GPa and $\bar{\sigma}_{\text{PI}} = 310$ MPa of the latter would be: $F_{\text{PI},b} \approx 6\pi t R \bar{\sigma}_{\text{PI}}^2 / E_{\text{PI}} = 0.037N$, thus a factor $4 \div 5$ worse in terms of raw performance with respect to the registered best values for the MW pellicles under study. Therefore, there is a large margin to explore thicknesses during the design phase of thin film filters for space

³ Cross section σ and complex refractive index k are related by $\sigma = 4\pi k / (\lambda n)$, with the number density of absorbers n which, in known allotropes (i.e. graphite/graphene), can be evaluated from the density ($\rho \approx 2.267 \text{ g cm}^{-3}$).

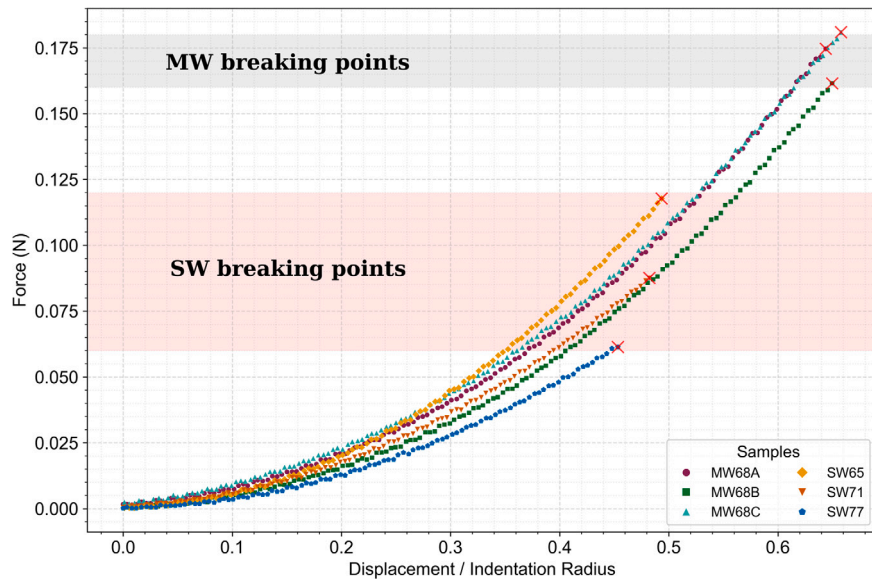


Fig. 9. Load–deflection curves for samples prepared in push-pin frames (Table 1). The x-axis shows the normalized deflection by the indenter radius, δ/R , while the y-axis shows the force F measured in N. Red crosses indicate the breaking point for each sample, shaded bands indicate the breaking-force ranges for SW (light red) and MW (light gray) pellicles.

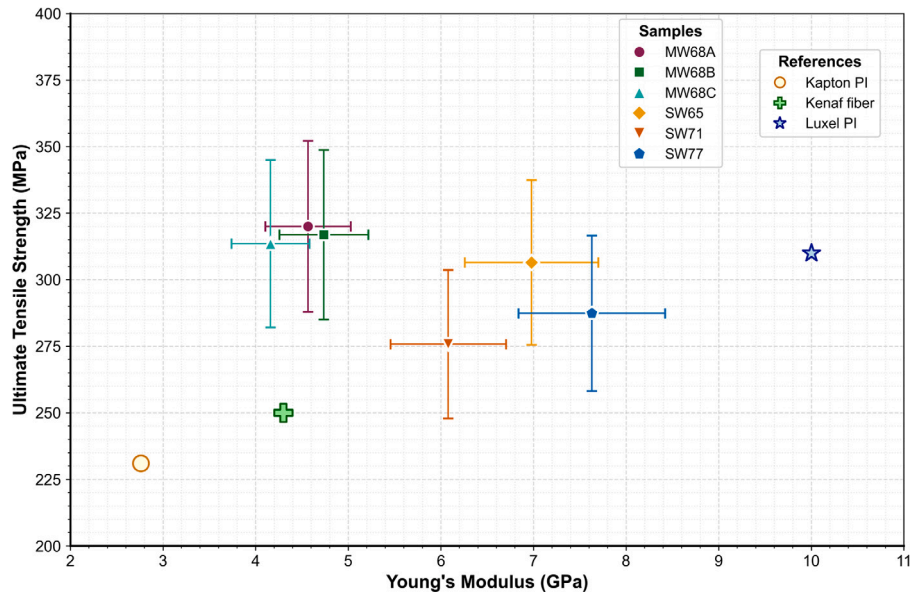


Fig. 10. Young's modulus vs. UTS Ashby map for all CNT pellicles tested by spherical indentation (push-pin samples in Table 1) along with a few references. Error bars for the parameters retrieved for SWs/MWs combine thickness and fitting parameters uncertainties (3σ). Reference markers are: Kapton[®] PI thin films (orange open circle) [71], Kenaf fibers (green plus symbol) [73], and Luxel-produced PI thin films (blue star) [16,72].

missions, as even a 200 nm thick MW pellicle is more transmissive in the EUV as previously discussed, and the larger the thickness, the sturdier the pellicle under space environment and vibroacoustic loads.

Fig. 10 summarizes the spherical-indentation best-fit results in a map of Young's modulus versus UTS, a kind of Ashby map [70]. The $(E, \bar{\sigma}^{\text{UTS}})$ pairs are shown against indicative values for the reference points for Kapton[®] films [71], Luxel-produced PI films [16,72] and Kenaf fibers [73]. Uncertainties for reference points are not always available. They are therefore omitted, while, for our pellicles, parameter uncertainties are $\sim 10\%$ (Section 2.4), dominated by samples' effective thickness errors – Table 1 – and not including the accuracy of the Begley-model inferred quantities, which would quadratically add $\sim 13\%$ more in relative error.

The SW pellicles appear stiffer than MW pellicles in our set ($E_{\text{SW}} = 6.0\text{--}7.6$ GPa compared to $E_{\text{MW}} = 4.2\text{--}4.7$ GPa), with comparable UTS between the sets: $\bar{\sigma}_{\text{SW}}^{\text{UTS}} = 276\text{--}306$ MPa, compared to $\bar{\sigma}_{\text{MW}}^{\text{UTS}} = 314\text{--}320$ MPa which, given the relative uncertainties, makes all of the found UTS values compatible within error. The magnitudes for mechanical properties of the CNT pellicles are broadly comparable with the references materials with densities of $\rho_{\text{PI/Kenaf}} \approx 1.4 \text{ g cm}^{-3}$, whereas the CNT pellicles in this work are much lighter as shown combining the XAS analyses performed the thicknesses estimations obtaining $\rho_{\text{MW}} = 0.31 \pm 0.03 \text{ g cm}^{-3}$ and $\rho_{\text{SW}} = 0.41 \pm 0.04 \text{ g cm}^{-3}$ (Appendix B, Table B.4). The density can be thought as a proxy for the porosity of the pellicles under study in this work, evident also from voids shown in the SEM images in Fig. 3: comparing these densities with the theoretical limit of the

density for ideal CNT buckypapers of $\rho_{\text{LIM}} = 1.39 \text{ g cm}^{-3}$ [74], rough porosity estimations of the porosity come to about $1 - \frac{\rho_{\text{SW/MW}}}{\rho_{\text{LIM}}} \approx 71\%$ for SW and 78% for MW.

Notably, the in-plane Young's modulus found in previous research by Gubarev et al. [32] for a 40 nm thick SWCNT film specimen was $\approx 24.5 \text{ GPa}$, about 3 to 4 times stiffer than the values found for SW pellicles in our work: the difference found could be attributed to differences in the synthesis, and/or in the physical and chemical properties of the specimens, or to the different experimental setups/environments employed, as our samples were slightly pre-stressed for spherical indentation, while their sample was slack in a vacuum bulge test setup.

In short, MW pellicles trade a slightly higher ultimate strength for lower stiffness, and this can explain how the MW pellicles' breaking forces are higher than the SW ones': as Eq. (9) models, given similar UTS, thickness, the higher the Young's modulus, the lower the breaking force. MW pellicles are thus expected to be less stiff and more tolerant to out-of-plane spherical indentation. Furthermore, the pellicles appear to have comparable mechanical properties to those of polyimide at a fraction of the density, allowing for margins during the design phase, providing improved EUV transmittance and resistance to out-of-plane loading even at doubled thicknesses.

4. Conclusions

We have compared freestanding single-walled (SW) and multi-walled (MW) carbon nanotube (CNT) based-pellicles produced by floating-catalyst CVD in terms of atomic fraction composition (Fe, C, O), optical and EUV/soft X-ray response, and mechanical performance, and compared them to simulations of the behavior of a 45 nm polyimide (PI) pellicle.

EUV/soft X-ray transmittance measurements combined with XAS-edge analyses show that there is a Fe:C atomic fraction in the SW pellicles about 5.5 times higher than in MW pellicles. At comparable carbon areal density, MW pellicles are shown to be systematically more EUV transparent in the 10–14 nm EUV band than SW pellicles, including key wavelengths 10.8 nm and 13.5 nm of relevance respectively to flaring coronas in solar physics and usual benchmarking for EUV pellicles in EUV lithography. The trend between $T(13.5 \text{ nm})$ and Fe:C atomic fraction indicates that, within our FC-CVD process window, iron content is a dominant parameter controlling the pellicles' EUV throughput; the carbon backbone and the oxygen to carbon ratios presence behave similarly in SW and MW networks. Furthermore, MW pellicles with thickness 200 nm are convincingly more transparent than the modeled performance of a 45 nm PI film, suggesting that MW pellicles in general can be a relevant substrate of choice for future optimization of thin film designs for missions such as NASA's MUSE, and ESA's NewAthena.

Broadband UV–VIS–IR transmission spectroscopy was also performed and normalized by the XAS-derived carbon areal density, yielding effective per-carbon extinction cross sections for both SW and MW pellicles. Both pellicle families of cross-section spectra behave similarly in the VIS–UV range and share a strong π -plasmon near 4.5 eV with similar peak cross sections ($\sim 8\text{--}9 \text{ Mb}$) and integrated oscillator strengths ($f \approx 0.26\text{--}0.28$) comparable to graphene in literature, i.e. confirming a common sp^2 electronic backbone. In the near infrared, SW pellicles retain pronounced van Hove features (S_{11} , S_{22} , M_{11}), whereas MW pellicles present a smoother continuum, as expected for thicker and more strongly coupled carbon nanotubes. Since the UV response per carbon atom is essentially the same in the two materials, this implies that no change in chemistry or optical interactions is driven by the difference in iron content, which mainly determines the EUV throughput due to atomic physics interactions.

Spherical indentation of $\sim 60\text{--}100 \text{ nm}$ -thick pellicles shows that SW films are stiffer ($E_{\text{SW}} = 6.0\text{--}7.6 \text{ GPa}$) than MW ones ($E_{\text{MW}} = 4.2\text{--}4.7 \text{ GPa}$), while both kinds of pellicles achieve similar ultimate tensile stresses, with $\bar{\sigma}^{\text{UTS}} = 280\text{--}320 \text{ MPa}$. Considering SW/MWCNT

pellicles with similar thicknesses and similar ultimate membrane stress, the lower Young's modulus of MW networks translates into higher rupture loads under out-of-plane loading at similar UTS, in agreement with the experimental data. XAS data-based estimations provide working values for bulk densities of $\rho_{\text{MW}} = 0.31 \pm 0.03 \text{ g cm}^{-3}$ and $\rho_{\text{SW}} = 0.41 \pm 0.04 \text{ g cm}^{-3}$, placing CNT pellicles in the same E –UTS region of strong polymeric films used in space such as polyimide, although at roughly a quarter of the density. Furthermore, experimental values of breaking force due to a 4.5 mm radius spherical indentation reach values of $\approx 0.175 \text{ N}$, about 5 times the expected breaking force of a 45 nm PI pellicle.

In summary, FCCVD-grown MWCNT pellicles offer clear advantages over SWCNT pellicles for the thickness range relevant to space filters, while also being a promising technology overall for EUVL: they have higher raw EUV transmittance at 10.8 nm and 13.5 nm, and, mechanically, are slightly less stiff and resist slightly more membrane radial stress before rupture. Furthermore, MW pellicles with thickness 100–200 nm outperform both in EUV transmittance, and in the expected breaking force under spherical indentation, the behavior of a modeled 45 nm-thick PI freestanding membrane. More broadly, the joint EUV/X-ray, UV–VIS–IR, and mechanical characterization framework demonstrated here provides quantitative estimations and analyses to (i) assess and quantify EUV throughput and iron/oxygen atomic fraction contents in grown pellicles, establishing composition control and correlations with observations, (ii) aid future designs of multilayer thin film filters for high-energy astrophysics, with a novel ultralight thin pellicle candidate (MWCNT) for high-throughput EUV/soft X-ray windows, and (iii) benchmark SWCNT and MWCNT pellicles against conventional polyimide, to aid the technology readiness level assessment of the former pellicles with a highly regarded and trusted material in space applications.

CRedit authorship contribution statement

Edoardo Alaimo: Writing – review & editing, Writing – original draft, Visualization, Software, Methodology, Investigation, Formal analysis, Conceptualization. **Michela Todaro:** Writing – review & editing, Validation, Resources, Investigation, Formal analysis, Data curation. **Luisa Sciortino:** Writing – review & editing, Validation, Resources, Investigation, Formal analysis, Data curation. **Ugo Lo Cicero:** Writing – review & editing, Validation, Software, Resources, Investigation, Formal analysis. **Federico Fiorentino:** Writing – review & editing, Visualization, Resources, Investigation, Data curation, Conceptualization. **Fabio D'Anca:** Writing – review & editing, Visualization, Validation, Resources, Project administration, Investigation. **Fabio Reale:** Writing – review & editing, Supervision, Project administration, Funding acquisition. **Gabriele Cozzo:** Writing – review & editing, Visualization. **Bjørn F. Mikkladal:** Writing – review & editing, Validation, Resources, Methodology, Investigation, Data curation. **Mika Moisanen:** Writing – review & editing, Validation, Resources, Data curation. **Bahawal Haq:** Writing – review & editing, Validation, Resources, Data curation. **Pekka T. Törmä:** Writing – review & editing, Project administration, Funding acquisition. **Marco Barbera:** Writing – review & editing, Validation, Supervision, Project administration, Investigation, Funding acquisition, Data curation, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Pekka T. Torma, Mika Moisanen, Bahawal Haq, Bjorn F. Mikkladal, Marco Barbera, Edoardo Alaimo, Fabio D'Anca, Michela Todaro, Ugo Lo Cicero, Luisa Sciortino, Federico Fiorentino reports equipment, drugs, or supplies was provided by European Space Agency. Edoardo Alaimo, Federico Fiorentino, Luisa Sciortino, Michela Todaro reports equipment, drugs, or supplies was provided by SOLEIL. Co-authors Bjorn F.

Table A.3

For each energy range, METROLOGIE beamline soft X-ray branch best settings are detailed. These parameter settings are used for all of the measurements on CNT pellicles performed during the experimental campaigns for this work. The highest resolution in each energy range is obtained by dividing the lowest energy in the range by the respective spectral power. The monochromators used for each energy range had the following properties: 75 grooves/mm for energies lower than 300 eV and 300–1200 grooves/mm for the higher energies.

Energy Range (eV)	Band-pass Filter (Material, Angle)	Line Filter& Thickness (nm)	Photodiode Coating	Spectral Power
30–55	(Si, 3.5°)	Al/Mg/Al 500	Al	5000
45–75	(Si, 3.5°)	Al 500	Al	4000
70–105	(Si, 3.5°)	Si 500	Zr	3000
95–165	(Cr, 3.5°)	B 400	Zr	2000
160–270	(Cr, 2.8°)	C 700	Al	1500
260–305	(Cr, 2.8°)	Ti 500	Al	1000
280–325	(Cr, 2.5°)	Ti 500	Al	4000
320–445	(Cr, 2.5°)	Ti 500	Al	3000
440–766	(Si, 1.1°)	Co 500	Al	2000
760–908	(Si, 1.1°)	Cu 900	Al	5000
895–1600	None	B 400	Al	4000–2000

Mikladal, Mika Moisanen and Bahawal Haq are employed by Canatu Finland Oy which manufactured the carbon nanotube pellicles studied in the work. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Practical parameters settings employed at the METROLOGIE beamline

This appendix is devoted to the description of the experimental parameters employed in the EUV and X-ray campaigns carried out at the METROLOGIE beamline of the SOLEIL synchrotron facility. These are detailed in Table A.3.

Appendix B. X-ray absorption spectroscopy detailed plots and analyses: experimental data and fitting models

This appendix gathers the full XAS results behind Section 3.2. For each pellicle probed with XAS as discussed in Section 2.2, we present the EUV/soft X-ray transmission $T(E)$ experimental data and overlaid best-fit models from Eqs. (1)–(2). Insets focus on the C K, O K, and Fe $L_{2,3}$ edge regions, which correspond to the fundamental jumps used to obtain the best-fit for the areal densities. As stated in the main text, fine-structure (XANES/EXAFS) is not modeled, and narrow windows very close to the edge positions were masked during the fit to avoid biasing due to post- and pre-edge effects.

Table B.4 lists the retrieved mass areal densities Σ_j for C, O, and Fe (and their 3σ uncertainties). These values are those used in the main text to form atomic areal densities via Eq. (3) and element-to-carbon ratios. For completeness, the total areal density and the sample density estimates are also reported, the latter obtained by dividing the total areal density by the thickness estimates given in Table 1.

The smaller density of the CNT pellicles in general also explains how, at similar thicknesses (i.e. $t \sim 40$ – 50 nm), CNT thin pellicles can outperform PI pellicles in the EUV transmittance as stated in the experimental reports by Barbera et al. [27]. As a final note, the fits were performed under the same conditions across all samples and followed the prescriptions of Section 2.2.

Appendix C. Mechanical model load–deflection detailed plots: experimental data and fitting models

Fig. C.12 shows the force–dimensionless deflection data (F vs. δ/R , δ deflection and R indenter radius) acquired with the setup described in Fig. 4 for each push-pin sample (listed in Table 1) together with the spherical indentation best-fit curves of Eq. (8). Fits to the experimental data capture the curves' trends very well, both at the beginning — dominated by the prestressed behavior of the pellicle — and at the end, where a cubic trend forms. The discrepancy between the model and the data at the higher applied forces, right before rupture, is likely indicative of the pellicles entering a plastic regime.

At larger deflections and near the breaking force, the curves deviate from the model's predictions, which would predict shorter deflections. This is somewhat expected, as both experimental and theoretical studies on carbon nanotube structures [75,76] have shown that applied stress causes permanent strain, suggesting sliding between bundles in the network and modifications in the mechanical properties of the pellicle while enduring forces near rupture points (akin to plasticity). Since the Begley–Mackin framework models elastic behavior, it is not intended to describe local damage, which explains its failure to predict data near the rupture. This implies a significant onset of plasticity in the SW/MW pellicles at forces above about 90

Table C.5 lists the derived quantities from the best fit procedure. Although the best-fit parameters are (A , B) described in Eq. (7), we report 2D Young's moduli E_{2D} and pre-stretch ϵ_0 parameters that are immediately inferred from (A , B) without loss of relative uncertainty; a quite good accuracy reflecting the experimental precision of the mechanical indentation setup described in Section 2.4. Uncertainty in the best-fit parameters is reported at the 3σ confidence level; error propagation was used for the derived quantities. Note that $E = E_{2D}/t$ inherits the $\sim 10\%$ effective thickness uncertainty (Table 1). The same applies to the UTS: it is evaluated via Begley–Mackin stress equation (Eq. (9)) at breaking load, i.e. $\bar{\sigma}_{\text{Begley}}^{\text{UTS}} = \sqrt{\frac{E_{2D} F_b}{6\pi t^2 R}}$, thus implying that also the UTS minimum relative uncertainty is 10% from the effective thickness estimates.

Appendix D. Supplementary data

Supplementary material related to this article can be found online at <https://doi.org/10.1016/j.diamond.2026.114011>.

Data availability

The raw/processed data required to reproduce the above findings cannot be publicly shared due to contractual agreements (quoted in the acknowledgments section). Raw/processed data supporting the findings are available on reasonable request from the author at edoardo.alaimo@unipa.it, and they will be shared after ensuring no contractual agreement is violated.

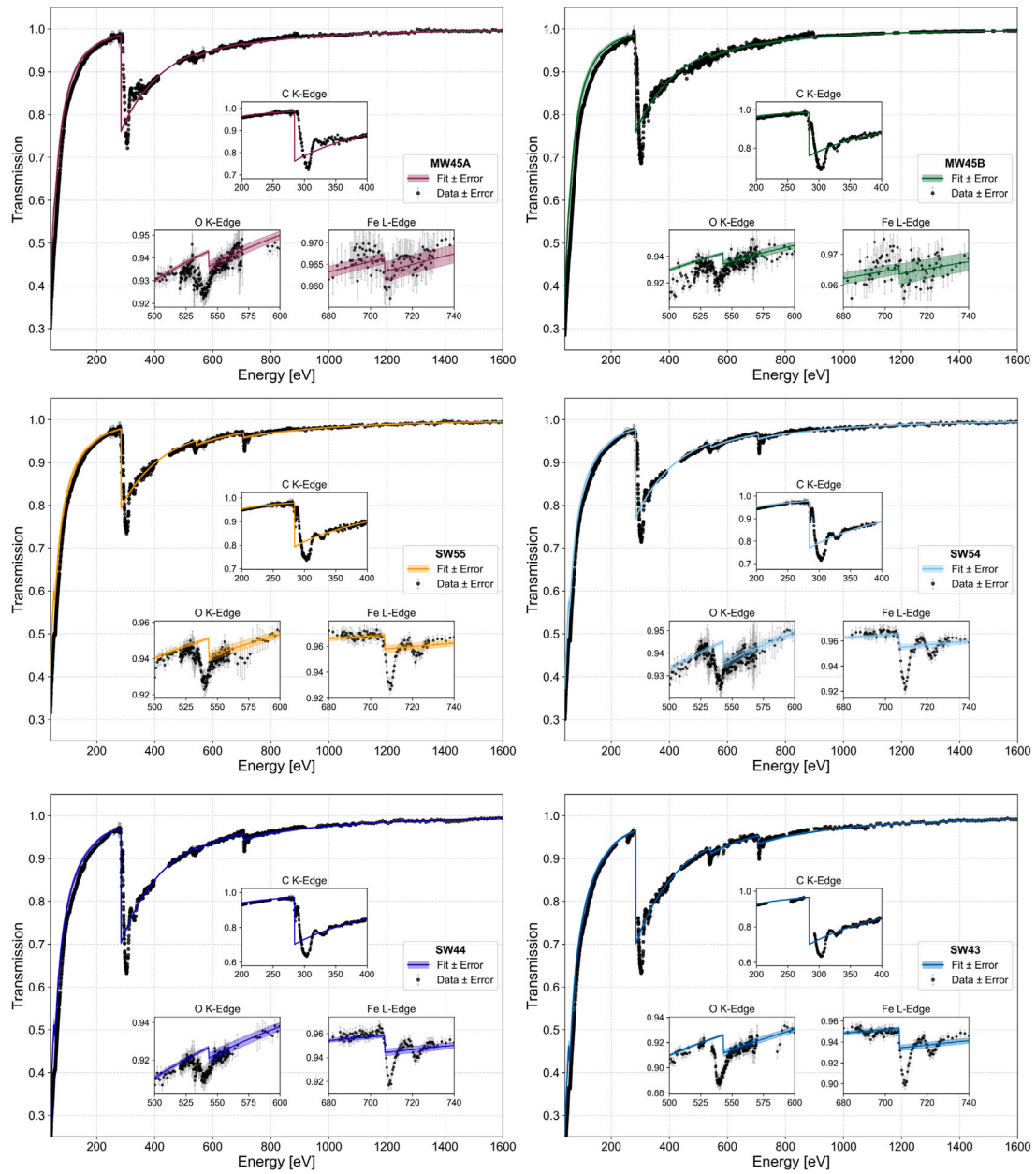


Fig. B.11. Composite view of XAS experimental data and fits for all TF110 frame samples in Table 1. Black dots with gray error bars are the measured $T(E)$ with their experimental errors. Solid-colored curves and their shaded areas are the best-fit model using Henke/CXRO mass attenuation coefficients and best-fit parameters. The insets show the behavior of the best fit models in following the trends of photo-absorption transmission jumps of relevant edges (C K, O K, Fe $L_{2,3}$).

Table B.4

Best-fit areal densities from XAS edge fits, the model and data of which are shown in Fig. B.11. Units for Σ are in $10^{-7} \text{ g cm}^{-2} = \text{g cm}^{-3} \cdot \text{nm}$. The total areal density is $\Sigma_{\text{tot}} = \Sigma_C + \Sigma_O + \Sigma_{Fe}$. The pellicles bulk density column ρ is obtained by the estimate $\rho = \Sigma_{\text{tot}}/t$ using the thickness t in Table 1. Uncertainties are reported to 3σ confidence for the best-fit values, while thorough propagation of errors was carried out for total areal density and the bulk density.

Sample	Areal density [$10^{-7} \text{ g cm}^{-2}$]			Derived	
	Σ_C	Σ_O	Σ_{Fe}	Σ_{tot} [$10^{-7} \text{ g cm}^{-2}$]	ρ [g cm^{-3}]
MW45A	51.8 ± 0.4	3.6 ± 0.8	2.0 ± 0.7	57.4 ± 1.1	0.31 ± 0.03
MW45B	52.5 ± 0.5	4.4 ± 1.2	1.2 ± 1.0	58.1 ± 1.6	0.31 ± 0.03
SW55	42.1 ± 0.3	5.5 ± 1.0	7.0 ± 0.8	54.6 ± 1.3	0.39 ± 0.04
SW54	47.9 ± 0.5	5.2 ± 0.7	7.1 ± 0.7	60.2 ± 1.1	0.42 ± 0.04
SW43	64.2 ± 0.4	7.8 ± 1.0	12.4 ± 0.9	84.4 ± 1.4	0.42 ± 0.04
SW44	65.1 ± 0.5	3.1 ± 1.0	9.2 ± 0.9	77.4 ± 1.4	0.40 ± 0.04

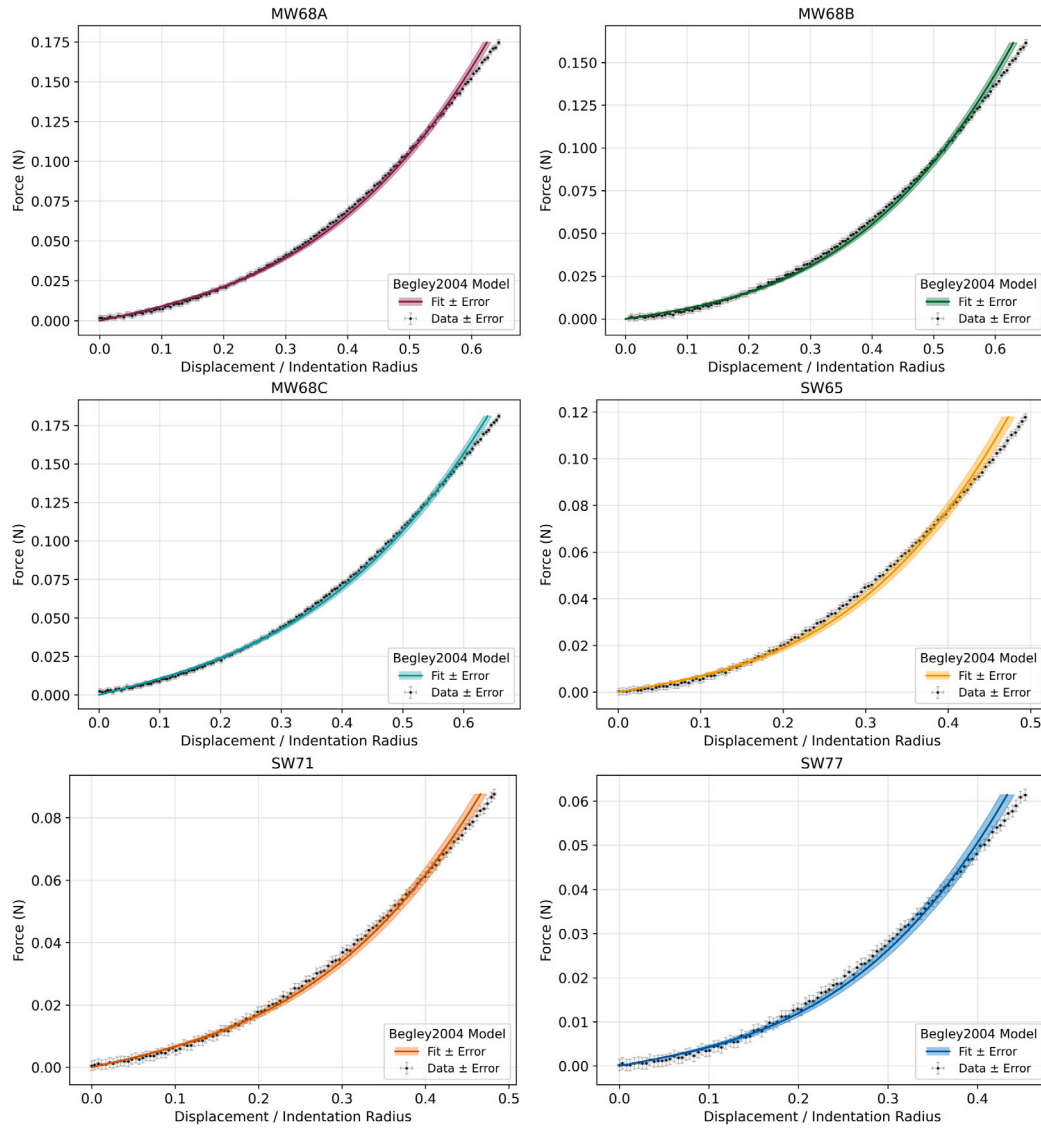


Fig. C.12. Composite view of the load-deflection experimental data and model-fits (F vs. δ/R) for all push-pin frame samples in Table 1. Black dots with gray error bars are the experimental data with their experimental uncertainties. Solid lines and shaded areas show, respectively, the spherical indentation best-fit curves and the inferred 3σ uncertainty. The model predicts elastic behavior and is expected to be less predictive near rupture, as strong plasticity sets in.

Table C.5

Spherical indentation fit results and derived quantities 2D Young's modulus and pre-stretch for the push-pin samples from Table 1. (E_{2D}, ϵ_0) are obtained directly from the models' best-fit parameters, by inversions from Eq. (7). UTS ($\bar{\sigma}^{UTS}$) is computed with the Begley finite-contact stress estimate from Eq. (9) calculated with the measured breaking load F_b , while $E = E_{2D}/t$, and both estimates employ the pellicle thickness t from Table 1. All uncertainties are 3σ , employing propagation of errors where required.

Sample	ϵ_0	E_{2D} (N m ⁻¹)	E (GPa)	$\bar{\sigma}^{UTS}$ (MPa)
MW68A	$(92.0 \pm 2.5) \times 10^{-4}$	419 ± 6	4.6 ± 0.5	320 ± 32
MW68B	$(60.1 \pm 2.3) \times 10^{-4}$	426 ± 7	4.7 ± 0.5	317 ± 32
MW68C	$(122.4 \pm 3.0) \times 10^{-4}$	373 ± 5	4.2 ± 0.4	314 ± 31
SW65	$(37.8 \pm 2.2) \times 10^{-4}$	714 ± 18	7.0 ± 0.7	306 ± 31
SW71	$(54.8 \pm 2.3) \times 10^{-4}$	495 ± 11	6.1 ± 0.6	275 ± 28
SW77	$(35.6 \pm 2.1) \times 10^{-4}$	470 ± 13	7.6 ± 0.8	287 ± 29

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