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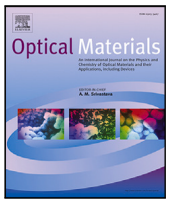

Research article

Single-walled carbon nanotube pristine films: Thickness determination, antireflective properties and two-photon 3D lithography demonstration

Denis M. Zhigunov [a,*], Margarita I. Sharipova [b], Yury G. Gladush [a], Dmitry V. Krasnikov [a], Vladimir O. Bessonov [b,c], Andrey A. Fedyanin [b], Albert G. Nasibulin [a]

[a] Skolkovo Institute of Science and Technology, Bolshoy Boulevard 30, bld. 1, 121205 Moscow, Russia
[b] Faculty of Physics, M. V. Lomonosov Moscow State University, Leninskie Gory 1, Moscow, 119991, Russia
[c] Frumkin Institute of Physical Chemistry and Electrochemistry, Russian Academy of Sciences, Leninsky prospect 31, bld. 4, Moscow, 119071, Russia

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ABSTRACT

We present a straightforward approach for measuring the thickness of highly porous and rough single-walled carbon nanotube (SWCNT) pristine thin films. Using the developed approach, the linear dependence of the film thickness on its absorbance at 550 nm was obtained with a slope coefficient of 404 nm, which can be used as a calibration curve for an optical determination of SWCNT pristine film thickness on the basis of spectrophotometry measurements. Transmittance and reflectance of free-standing SWCNT pristine films with thicknesses in the range of 20–120 nm are studied, and ultra-low specular reflectance below 0.05 % is obtained for the thinnest membrane. Perspectives of SWCNT-based antireflective coatings are explored using Si wafer as a reference. Finally, femtosecond laser 3D lithography is performed using free-standing SWCNT film as a substrate, which opens up new directions for the application of SWCNTs, for instance, in X-ray optics.

1. Introduction

Due to the unique combination of properties, single-walled carbon nanotube (SWCNT) thin films show exceptional performance for diverse applications [1,2]. Being carbon nanotube random networks, SWCNT films are chemically stable, conductive, flexible, mechanically robust and transparent, making them highly desirable for the new generation of devices [3–8]. Most of the properties of SWCNT films can be tuned by adjusting their thickness, typically in the range from a few to hundreds of nanometers. In particular, a transmittance greater than 90% in the visible can be achieved for macroscale free-standing SWCNT films with a thickness as low as 8 ± 3 nm [9], which is noteworthy for transparent electronics applications. Hence, knowledge of SWCNT film thickness is one of the key points for the precise prediction and control of its mechanical, electrical, and optical response.

The challenge of thickness determination for such a highly porous medium as SWCNT film arises from its structural softness, surface roughness, and blurred film-to-air interface. Standard direct methods such as atomic force microscopy (AFM) or profilometry suffer from large errors in thickness determination since the device tip can easily penetrate the film surface. Scanning electron microscopy (SEM) is typically also not applicable for this purpose due to lack of accuracy and not well-defined interfaces. In turn, among indirect optical methods ellipsometry technique is the classical tool for film thickness calculation based on experimental data modeling. Standard spectroscopic ellipsometry gives thicknesses comparable with those obtained by AFM measurements for *ethanol-densified* SWCNT films [9], however, this technique is also powerless for the thickness determination of SWCNT *pristine* films owing to the suppressed film reflection to less than 1% [10]. Transmission electron microscopy (TEM) was shown to be useful for measuring the thickness of pristine SWCNT films [11]. Nevertheless, the last method is sophisticated and not easily accessible, hence search for a more simple measurement approach is still of current importance.

Aerosol synthesized SWCNT pristine films collected on a nitrocellulose filter without any contact with a liquid [12], possess uniquely small specular (≤ 0.1%) and diffuse (≤ 1%) reflection, which makes them ideal substrates for nanophotonic applications [13]. However, the thickness dependence of reflectance of SWCNT pristine membranes has not been studied so far, while our research fills this gap. Owing to the inherently small reflectance, it has been also proposed to use SWCNT films as antireflective coatings, which provides up to several times reduction of reflection from test Si wafer [14]. To verify this result, we prepared similar samples and measured corresponding reflection spectra.

* Corresponding author.
E-mail address: d.zhigunov@skoltech.ru (D.M. Zhigunov).

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SWCNT free-standing thin films (i.e., membranes) can be used as substrates not only in visible waverange, but also for X-ray optics and synchrotron radiation experiments, as proposed in our recent paper [13]. Indeed, SWCNT membranes are durable under chemical, mechanical, thermal and X-ray influence [15]. The substrates used in X-ray optics are usually thin membranes made of materials with high enough hardness and X-ray stability together with the appropriate transmission in a given wavelength region. For instance, SiN_x-based membranes in a rigid Si frame are commercially available [16]. As compared to the SiN_x, the advantages of SWCNT membranes are lower thickness and higher transmittance for X-rays (caused by the lower atomic number of carbon) [17]. Additionally, SWCNT can be more practical in terms of fast cost-effective production of substrates with a large variety of available entrance apertures. Several methods are applicable for the fabrication of elements for X-ray optics, amongst them two-photon lithography is preferable as it allows 3D printing of free-form micro-optics customized for a specific task [18]. Micro-sized elements for X-ray optics are shown to be successfully fabricated by two-photon lithography [19–21]. Realization of all-carbon X-ray optics is also possible if SWCNT substrates are combined with pyrolyzed two-photon printed X-ray optical elements [22], which will additionally lead to higher optical power and thus better resolution due to the increased material density after pyrolysis. SWCNTs have already been used for X-ray tubes and are considered promising in EUV lithography [15,23].

In this work, we present a combination of thin metal film deposition followed by mechanical profilometry as a straightforward approach for measurement of SWCNT pristine films thickness. Taking into account the profilometry data, transmission and reflection coefficients are measured for the uncoated pristine films as a function of their thickness. The resulted linear dependencies of these coefficients for a typical wavelength 550 nm provide calibration curves for a fast method of SWCNT films thickness determination based on the spectrophotometry measurements. Antireflective properties of SWCNT-based coatings are studied using Si wafer as a reference. Finally, we demonstrate, for the first time to our knowledge, that free-standing SWCNT films can be used as substrates for 3D printing of microstructures by means of two-photon polymerization.

2. Materials and methods

2.1. Sample fabrication

SWCNTs were grown by aerosol chemical vapor deposition (CVD) method using the Boudouard reaction on iron-based catalysts in the gas phase (ferrocene vapor decomposition in the atmosphere of CO). Aerosol SWCNTs were filtered from the gas phase and collected on a microporous filter forming a sparse uniform film of randomly oriented SWCNTs. These films were subsequently dry transferred either over square openings with the size of 20 × 20 mm^2 for transmission and reflection measurements, or over round openings with a diameter from 3 to 7 mm for 3D printing. We distinguish hereafter between as-transferred films, denoted as pristine films, and those compressed in the out-of-plane direction by ethanol drop-casting and subsequently dried in the air, denoted as ethanol-densified films [9,10].

2.2. Thickness measurement

The film thickness measurements are performed using D-600 stylus profilometer (KLA Tencor, USA) with sub-nanometer height resolution. For this purpose, SWCNT films were transferred onto Si substrates, followed by deposition of platinum thin films by magnetron sputtering. Transmittance and reflectance of pristine membranes are measured using Agilent Cary 5000 spectrophotometer equipped with an integrating sphere. By varying SWCNTs collection time during preparation, films with different thicknesses were obtained.

2.3. Optical characterization

As a standard initial film characterization, we have measured transmittance and absorbance at 550 nm (T_{550} and A_{550}, respectively), to be correlated later with the measured thicknesses (for details, see Supplementary Material, Fig. S1). The diffuse reflectance was measured at a normal incidence, while the total reflectance was measured at an angle of incidence of 3°20′. The specular reflectance was then calculated by subtracting the diffuse component from the total one. Scanning electron microscopy (SEM) images were obtained in the high-vacuum mode at an accelerating voltage range from 10 to 30 kV.

2.4. Printing of 3D microstructures on free-standing SWCNT membranes

A laboratory two-photon lithography printing setup was used to fabricate 3D structures on SWCNT membranes. The radiation from femtosecond fiber laser Toptica FemtoFiber Ultra 780 (80 MHz pulse rate, 780 nm central wavelength) passes through a high-speed acousto-optic modulator acting as a shutter, and then propagates through a beam expander. The beam is then deflected by a scanning galvomirror (for xy positioning in the focal plane) and directed into a 100x/NA1.3 objective (Olympus) mounted on a piezo stage, which provides z positioning in the direction of the optical axis. Commercial photoresists OrmoComp and SZ2080 were used for two-photon lithography. SZ2080 photoresist was pre-baked at 80 °C for 60 min. After exposure, the uncured resist was removed by post-development in methyl isobutyl ketone for 2 h, then the samples were rinsed with isopropyl alcohol and slowly dried at ambient conditions. Both pristine and ethanol-densified SWCNT membranes were tested as substrates for two-photon lithography with no significant difference.

3. Results and discussion

Fig. 1 shows SEM images of SWCNT thin films before and after 25 nm of Pt deposition, which were transferred to the Si substrate. This coating strengthens the top layer of the SWCNT film in order to make it hard enough for contact profilometry measurements. Indeed, intact SWCNT films are too soft, so profilometer stylus can easily penetrate the surface. At the same time, we expect that such Pt coating does not increase significantly the total film thickness due to its high porosity and relatively large surface roughness. This assumption is validated below by the comparison of our thickness estimations with those found in the literature. In turn, no visible traces of Pt were observed after removing SWCNT films from the substrate, which argues for the primarily top film layer modification (see Supplementary Material, Fig. S2).

The results of profilometry measurements are summarized in Table 1. The height difference between Pt coated SWCNT film (excluding the very edge) and adjacent Si substrate is taken as a film thickness (see Supplementary Material, Fig. S3). The attempts to measure SWCNT thickness prior to top layer strengthening resulted in unexpected thickness values and excessive measurement errors (see Supplementary Material, Fig. S4), moreover, each time the film was damaged by the stylus. Using metal coating, we succeeded to measure the thicknesses for the films with T_{550} = 50-80%: several measurements were made at different points of each film, and the resulting thickness was obtained by data averaging together with the calculation of the standard deviation in order to estimate the measurement error (also shown in Table 1). In turn, the thickness of the thinnest film under study with T_{550} = 90% could not be measured by our approach seemingly due to insufficient mechanical strength and/or weaker adhesion to the substrate. Thus, to successfully determine the thickness of a thin porous film using the proposed method, a combination of film parameters such as sufficient strength (also after metal deposition), as well as strong enough adhesion to the substrate is required.

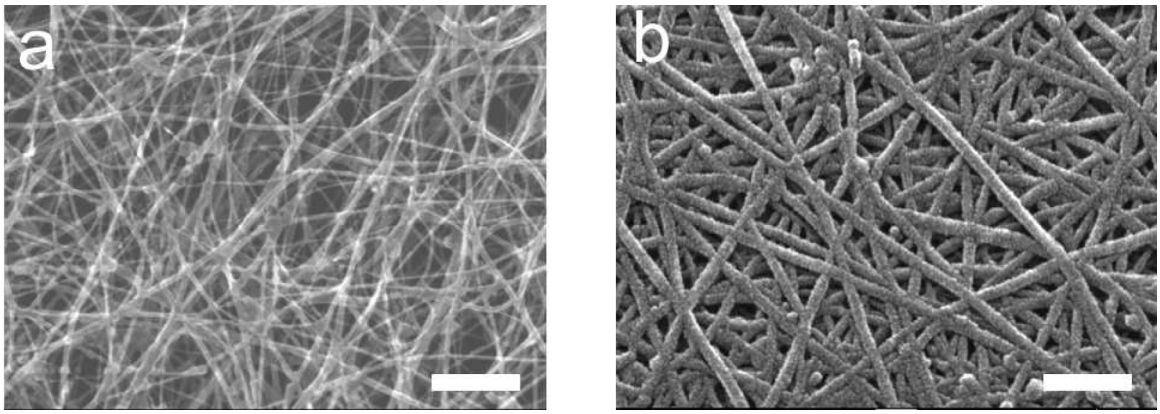

Fig. 1. SEM images of (a) pristine and (b) Pt coated SWCNT thin films on Si substrate. Scale bars: 200 nm.

Table 1
SWCNT pristine films thicknesses measured by stylus profilometry.

	SWCNT film	Measured thickness (nm)	Calculated thickness (nm)
T_{550}(%)	A_{550}	Pt coated pristine film on Si substrate	Thickness = 404 × A_{550}
50	0.301	123 ± 5	121.6
70	0.155	62 ± 5	62.6
80	0.097	42 ± 5	39.2
90	0.046	–	18.6

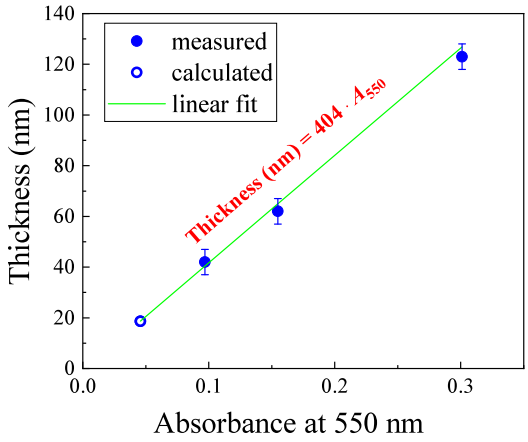

Fig. 2. Absorbance dependence of thickness for SWCNT pristine films on Si substrate.

The linear dependence of film thickness on its absorbance at 550 nm with 239 nm slope coefficient was already established for ethanol-densified SWCNTs, providing an express technique for thickness estimation [9]. In present study, we also obtained such a linear dependence for pristine SWCNT films with the slope coefficient equal to 404 nm (see Fig. 2). This result correlates well with the previously shown 417 nm slope of the similar linear dependence of thickness on absorbance for as-deposited SWCNT films [11], which in turn serves as an independent validation of our measurement approach. TEM was used by the authors of Ref. [11] for SWCNT film thickness determination, which is much more complicated and cost ineffective method as compared to the contact profilometry. According to the comparison of our data and above mentioned 239 nm slope coefficient, we can conclude that densification decreases SWCNT film thickness by approximately 1.7 times.

The results of thickness calculation performed using the obtained slope coefficient are also presented in Table 1, showing agreement with the measurements within the respective standard deviations, and allowing estimation of the thickness ~ 18.6 nm for the thinnest film with T_{550} = 90%. The obtained value can be compared with the experimentally measured thickness of the corresponding ethanol-densified film (8 ±3 nm) [9]: using 1.7 times ratio, our approach gives the thickness estimation of ~ 11 nm after densification, which matches well the experimental result for such SWCNT film. In fact, the linear dependence shown in Fig. 2 allows similar fast thickness determination method for pristine SWCNT films, as in case of their ethanol-densified counterparts. More generally, our approach can be applied for the thickness measurements of other soft and/or porous thin films.

Antireflective properties of free-standing pristine SWCNT film with some unknown thickness were studied previously [13]. In this work, we have measured diffuse and specular reflectance spectra of pristine membranes under study to demonstrate respective thickness dependence. Fig. 3 shows the experimental spectra together with the thickness dependence of reflectance at characteristic wavelength of 550 nm. As can be seen, both diffuse and specular reflectance decrease as membrane transmittance increases, i.e., thickness decreases. For the thinnest membrane under study, ultralow specular reflectance not exceeding 0.05% is detected, while the corresponding diffuse reflectance is about an order of magnitude higher. Hence, the total film reflectance is almost completely defined by its diffuse component, which is typical for so-called Lambertian reflectors. A clear linear dependence of reflectance specular component measured at 550 nm on membrane thickness is obtained (see Fig. 3c). Hence, this dependence can be used as an alternative calibration curve for a fast thickness determination of pristine SWCNT membranes.

The intrinsic low reflectance together with highly porous structure (which guarantees low refractive index [10]) both might be attractive for the fabrication of SWCNT-based antireflective coatings. Thus, a suppression of optical reflection from Si wafer was demonstrated by means of SWCNT thin films deposition [14]. Namely, about two-fold reduction of total reflectance (from 27%–70% to 14%–33%) was achieved, when 32 ± 5 nm thick SWCNT film, fabricated by vacuum filtration of SWCNT suspension, was deposited by means of dry-transfer printing method onto Si wafer. To explain this effect, authors suggested an effective medium model including graded refractive index profile for SWCNT random network film. Indeed, specifically designed graded-index coatings are known to suppress drastically the optical reflection from the underneath material [24,25]. In order to verify the effectiveness of SWCNTs as an antireflective coating for Si, we have transferred our films onto Si (100) wafer using ethanol wetting for a

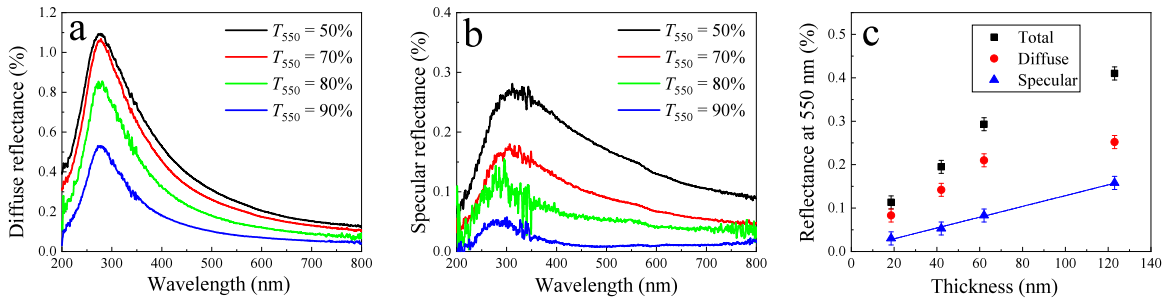

Fig. 3. Experimental spectra of (a) diffuse and (b) specular components of reflectance for SWCNT pristine membranes with different thicknesses; (c) thickness dependence of reflectance components measured at 550 nm.

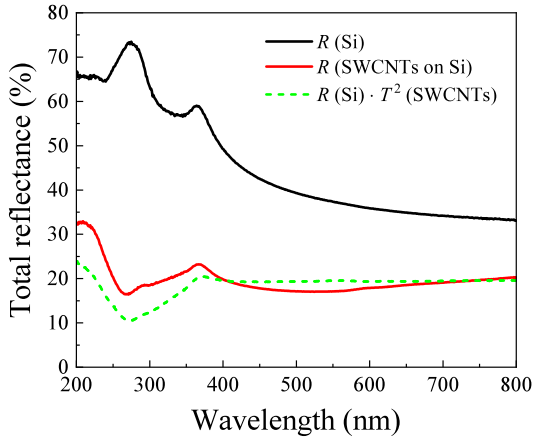

Fig. 4. Total reflectance (R) of Si wafer before and after coating with SWCNT film. Dashed green curve represents a product of Si wafer reflectance R and squared transmittance (T) of SWCNT film.

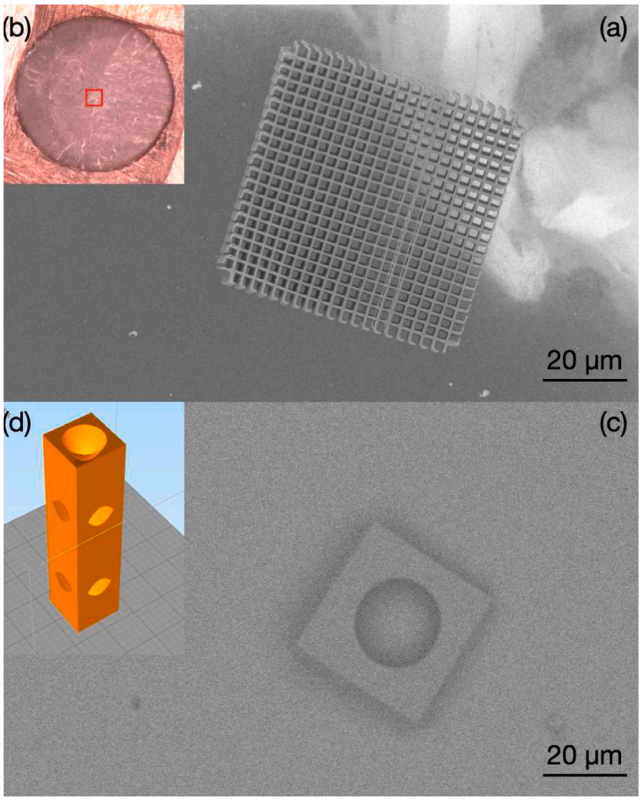

Fig. 5. (a) SEM image of a test microstructure printed by two-photon polymerization technique on a SWCNT membrane; (b) overview image of square SWCNT membrane placed onto a round hole in metal plate; (c) SEM image of an X-ray lens printed by two-photon polymerization technique on a SWCNT membrane; (d) X-ray compound refractive lens model.

better film adhesion. The results of reflectance measurements before and after the deposition of SWCNT film with T_{550} = 70% are shown in Fig. 4. One can observe a reduction of reflectance from 33%–73% to 16%–33% as a result of SWCNT coating, i.e., the effect is of the same order as mentioned above. In addition, a product of Si wafer reflectance and squared transmittance of SWCNT film is also plotted in Fig. 4 (dashed line). From the comparison of the last spectrum with the reflectance of SWCNTs on Si, it is clear that the effect of Si wafer reflectance suppression after SWCNT film deposition can be explained mainly by absorption of light by SWCNTs, which occurs twice along the path of light to and from the substrate. SWCNT films with other thicknesses have also been deposited onto Si substrates, and the resulted reflectance spectra show the similar trend (see Supplementary Material, Fig. S5). Hence, although the total reflectance of Si wafer can be indeed significantly reduced by SWCNT thin layer deposition, the mechanism of this reduction is not necessarily rely on the concept of graded-index profile of SWCNT coating.

Finally, to show that a free-standing SWCNT film is suitable as a substrate for two-photon lithography, initially a test woodpile structure was fabricated atop of 50-nm-thick membrane. Fig. 5a shows a SEM micrograph of this structure made of SZ2080 photoresist with a size of 60 × 60 × 50 μm^3 and a lateral line spacing of 2 μm. The linewidth is estimated to be 0.4 μm, which corresponds to the typical value on solid substrates for given printing parameters - average laser power of 2 mW and scanning speed of 1200 μm/s. The inset in the upper left corner (Fig. 5b) shows an optical image of the membrane after the development. Residual folds and whitening can be seen in the membrane, which demonstrates some material deterioration during the printing process. The red square marks the position of the printed woodpile. The printed microctructures adhesion to membranes is somewhat lower than to the solid substrate, which can be explained by slightly loose membrane positioning in the frame and can be fixed in further experimental process. Another issue is membrane stability, as inaccurate handling can cause membrane damage, though we have not detected any difference from SWCNT original fragility compared with the membranes after two-photon lithography treatment. After the initial tests, X-ray compound refractive lens made of 5 parabolic surfaces in vertical geometry and a size of 35 × 35 × 150 μm^3 have also been successfully printed using OrmoComp photoresist on SWCNT substrate (see Figs. 5c,d). The individual plano-concave lens parameters are the following: aperture A = 34 μm, curvature radius at the optical axis R = 6 μm, space between apexes of parabolic surfaces d = 2 μm, which corresponds to the expected focal length of f = 227 cm at 8.2 keV, similar to the one experimentally tested at Diamond Light Source [20]. The expected focal length of the compound lens is f≃60 cm at 8.2 keV, relevant to focal distances of X-ray optical elements in synchrotron experiments with high image resolution [26–28]. This

result demonstrates the possibility of using SWCNT substrates for X-ray optics, which can be game-changing in terms of design freedom, cost and complexity of production. To make all-carbon stable X-ray optics, the next step would be to pyrolyze fabricated microstructures to eliminate organic components of photoresist and to achieve glassy carbon lens material on SWCNT substrate.

4. Conclusions

In conclusion, we present an approach for direct measurements of the thickness of soft and/or porous thin films by a contact profilometry technique combined with metal pre-coating. By applying this approach to the thickness measurement of SWCNT pristine films, linear dependences of thickness on film absorption and specular reflection were obtained, which paves the way for a fast thickness determination using spectrophotometry data. The obtained thicknesses of SWCNT pristine films agree with those measured in the literature by other more sophisticated methods. The thickness dependence of reflectance of SWCNT membranes revealed the lowest specular reflectance below 0.05% and total reflectance below 0.5% for a thinnest film (~ 19 nm) under study. We have shown that SWCNT pristine film that is deposited on Si substrate can be considered as an absorbing antireflective coating, since the reflectance suppression occurs mainly due to light absorption by the film. Finally, using free-standing SWCNT films as substrates, we realized 3D laser printing of woodpile and X-ray lens microstructures by two-photon lithography. The obtained results pave the way for the development of all-carbon elements of X-ray optics.

CRediT authorship contribution statement

Denis M. Zhigunov: Writing – original draft, Investigation, Conceptualization, Data curation. **Margarita I. Sharipova:** Writing – original draft, Investigation, Data curation. **Yury G. Gladush:** Writing – review & editing, Resources, Validation. **Dmitry V. Krasnikov:** Resources, Writing – review & editing. **Vladimir O. Bessonov:** Resources, Writing – review & editing, Validation. **Andrey A. Fedyanin:** Funding acquisition, Writing – review & editing, Supervision. **Albert G. Nasibulin:** Supervision, Writing – review & editing, Funding acquisition.

Declaration of competing interest

The authors 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. Supplementary data

Supplementary material related to this article can be found online at https://doi.org/10.1016/j.optmat.2025.117289.

Data availability

Data will be made available on request.

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