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INVESTIGATION OF THE EFFECT OF SHORT-PULSED ION IRRADIATION ON THE STABILITY OF CARBON NANOWALLS

Carbon nanowalls (CNWs) are promising carbon-based nanomaterials for radiation-resistant electronic and optoelectronic applications due to their unique three-dimensional graphene-like architecture and outstanding physicochemical properties. In this work, the effect of short-pulsed high-current ion irradiation on the stability of carbon nanowalls was systematically investigated. CNWs were synthesized on quartz substrates by inductively coupled plasma–enhanced chemical vapor deposition and subsequently irradiated using the high-current pulsed ion accelerator INURA at current densities of 4, 7, and 10 A/cm^2. The radiation-induced changes in morphology, structure, optical transparency, and electrical properties were analyzed using atomic force microscopy, Raman spectroscopy, UV–Vis spectrophotometry, and four-point probe measurements. Atomic force microscopy revealed only moderate surface rearrangement and slight variations in roughness, while the characteristic vertically oriented nanowall morphology was preserved even at the highest irradiation density. Raman analysis confirmed the retention of the graphene-like sp^2 carbon structure, with minimal changes in the I_D/I_G ratio, indicating limited defect formation. Optical measurements showed moderate variations in transmittance correlated with surface restructuring, without spectral degradation. Electrical characterization demonstrated a stable or slightly reduced sheet resistance after irradiation, suggesting improved interwall electrical contact and structural stabilization. Overall, the results demonstrate the high resistance of carbon nanowalls to short-pulsed ion irradiation and confirm their suitability as functional materials for radiation-resistant electronic, optoelectronic, and sensor devices.

Keywords: carbon nanowalls, ion irradiation, radiation resistance, short-pulsed ion beam, surface morphology, Raman spectroscopy, electrical properties.

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Көміртекті наноқабырғалардың тұрақтығына қысқа импульсты ионды сәулелендірудің әсерін зерттеу

Көміртекті наноқабырғалар (CNWs) бірегей үшөлшемді графенге ұқсас архитектурасы мен жоғары физика-химиялық қасиеттерінің арқасында радиацияға төзімді электрондық және оптоэлектрондық құрылғыларда қолдануға перспективалы көміртекті наноматериалдар класына жатады. Бұл жұмыста қысқа импульсті жоғары токты иондық сәулеленудің көміртекті наноқабырғалардың тұрақтылығына әсері жүйелі түрде зерттелді. CNWs кварцты төсеніштерде индуктивті байланысқан плазма қолданылатын химиялық бу фазасында тұндыру әдісімен синтезделіп, кейін INURA үдеткішінде 4, 7 және 10 A/cm^2 ток тығыздықтарында иондық сәулеленуге ұшыратылды. Сәулеленудің морфологиялық, құрылымдық, оптикалық және электрлік қасиеттерге әсері атомдық-күштік микроскопия, рамандық спектроскопия, ультракүлгін-көрінетін аймақ спектрофотометриясы және төртзондты әдіс арқылы беттік кедергіні өлшеу көмегімен зерттелді. АСМ талдауы сәулеленуден кейін бет бедерінің тек шамалы қайта құрылуын және кедір-бұдырлық параметрлерінің аз ғана өзгеруін көрсетіп, ең жоғары сәулелену тығыздығында да наноқабырғалардың тік бағытталған морфологиясының сақталғанын дәлелдеді. Рамандық спектрлер көміртектің графенге ұқсас sp^2-құрылымының сақталғанын және I_D/I_G қатынасының мардымсыз өзгерісін көрсетті, бұл дефектілердің шектеулі түзілуін білдіреді. Оптикалық өлшеулер бет құрылымының қайта ұйымдасуымен байланысты мөлдірліктің орташа өзгерістерін көрсетті, алайда спектралдық сипаттамалардың деградациясы байқалмады. Электрлік өлшеулер сәулеленуден кейін беттік кедергінің тұрақты немесе аздап төмендегенін көрсетті, бұл наноқабырғалар арасындағы электрлік байланыстардың жақсаруымен түсіндірілуі мүмкін. Алынған нәтижелер көміртекті наноқабырғалардың қысқа импульсті иондық сәулеленуге жоғары төзімділігін көрсетіп, оларды радиацияға төзімді электрондық, оптоэлектрондық және сенсорлық құрылғылар үшін функционалдық материалдар ретінде қолданудың жоғары әлеуетін растайды.

Түйін сөздер: көміртекті наноқабырғалар, иондық сәулелену, радиациялық төзімділік, қысқа импульсті иондық сәулелену, бет морфологиясы, рамандық спектроскопия, электрлік қасиеттер.

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Исследование влияния короткоимпульсного ионного облучения на стабильность углеродных наностен

Углеродные наностены (CNWs) представляют собой перспективный класс углеродных наноматериалов для применения в радиационно-стойких электронных и оптоэлектронных устройствах благодаря своей уникальной трёхмерной графеноподобной архитектуре и выдающимся физико-химическим свойствам. В данной работе проведено систематическое исследование влияния короткоимпульсного сильноточного ионного облучения на стабильность углеродных наностен. CNWs были синтезированы на кварцевых подложках методом химического осаждения из паровой фазы с использованием индуктивно связанной плазмы, после чего подвергнуты ионному облучению на ускорителе INURA при плотностях тока 4, 7 и 10 А/cm^2. Радиационно-индуцированные изменения морфологии, структуры, оптических и электрических свойств были исследованы методами атомно-силовой микроскопии, рамановской спектроскопии, УФ-видимой спектрофотометрии и измерений поверхностного сопротивления по четырёхзондовой схеме. Анализ АСМ показал лишь умеренную перестройку поверхности и незначительные изменения параметров шероховатости при сохранении характерной вертикально ориентированной морфологии наностен даже при максимальной плотности облучения. Рамановские спектры подтвердили сохранение графеноподобной sp^2-структуры углерода с минимальными изменениями отношения I_D/I_G, что свидетельствует об ограниченном образовании дефектов. Оптические измерения выявили умеренные изменения прозрачности, коррелирующие с перестройкой поверхности, без деградации спектральных характеристик. Электрические измерения показали стабильность или умеренное снижение поверхностного сопротивления после облучения, что может быть связано с улучшением межстеночных электрических контактов. Полученные результаты демонстрируют высокую устойчивость углеродных наностен к короткоимпульсному ионному облучению и подтверждают их перспективность в качестве функциональных материалов для радиационно-стойких электронных, оптоэлектронных и сенсорных устройств.

Ключевые слова: углеродные наностены, ионное облучение, радиационная стойкость, короткоимпульсный ионный пучок, морфология поверхности, рамановская спектроскопия, электрические свойства.

Introduction

Prolonged exposure to ionizing radiation leads to the accumulation of radiation-induced damage in functional semiconductor materials used in electronic devices and ultimately results in their degradation and failure, significantly limiting device lifetime [1,2]. Radiation interaction with matter can cause the rupture of chemical bonds, alter morphological and structural characteristics, and initiate processes such as swelling, polymerization, corrosion, crack formation, and degradation of mechanical, optical, and electronic properties [2–4]. Achieving high material stability under intense ionizing radiation is therefore a critical challenge for a wide range of applications, from nuclear energy systems to electronic components for the rapidly expanding space industry. Spacecraft and satellites operating in near-Earth orbits are continuously exposed to both electron and proton irradiation [4,5]. As scientific and commercial space missions become increasingly complex and long-term, the requirements for radiation tolerance of functional electronic materials continue to grow. Reliable operation under extreme conditions involving multiple types of ionizing radiation is a key prerequisite for the successful realization of such missions.

In response to these challenges, extensive research efforts have been directed toward the development of radiation-resistant materials, as well as toward gaining a deeper understanding of radiation-induced processes and strategies for their mitigation [6,7]. In this context, various allotropes of carbon have attracted considerable attention due to their unique physicochemical properties and wide range of technological applications [8,9]. Carbon-based nanomaterials are increasingly regarded as promising candidates for radiation-hard electronics and optoelectronics [10–12].

Among carbon nanomaterials, carbon nanowalls (CNWs) have emerged as a particularly intriguing class of structures. CNWs are three-dimensional networks of vertically oriented, self-assembled multilayer graphene sheets forming a labyrinth-like architecture [13–16]. They are also referred to as carbon nanosheets, graphene walls, vertical graphene, graphene nanoflakes, or graphene nanopetals. The thickness of individual walls typically ranges from several to several tens of nanometers, corresponding to more than ten graphene layers, while their height—controlled by the growth duration—can reach several micrometers. Owing to their distinctive morphology, CNWs exhibit a high aspect ratio and a large specific surface area of up to ~1000 m^2/g [16,17].

The architecture of CNWs is characterized by a high density of exposed edges, bends, and branching features, forming an interconnected three-dimensional network. Each wall consists of nanographitic domains with a high degree of graphitization, separated by domain boundaries associated with enhanced defect density and crystallographic distortions [13]. This structural organization underlies the outstanding electrochemical, catalytic, and sensing properties of CNWs, making them one of the key research directions in the field of functional carbon nanomaterials. Due to their unique combination of electrical, thermal, and mechanical properties, CNWs have been explored for applications in supercapacitors, sensors, field emitters, and fuel and solar cells [7,18,19]. Their high defect density and developed surface morphology further render CNWs a versatile platform for functionalization and targeted property modulation [8,18,20].

Recent studies have demonstrated that electron and proton irradiation can induce pronounced changes in the morphological and functional properties of carbon nanowalls [21,22]. In particular, irradiation with 5 MeV electrons and 1.8 MeV protons has been shown to reduce wall density, modify the electronic structure, increase surface resistance, and enhance optical transmittance, highlighting the complex interplay between morphology and functional performance after irradiation [21]. At the same time, heterostructures incorporating CNWs have exhibited remarkable radiation tolerance and, in some cases, even partial improvement of device performance following proton exposure. For instance, photodiodes based on self-adaptive CNWs/CdZnTe heterostructures retained stable photoelectric characteristics after irradiation with 1.5 MeV protons at fluences on the order of 10^{12} p cm^{-2}, underscoring the potential of CNWs for operation in radiation-harsh environments [22].

Despite this progress, the effects of short-pulsed ion irradiation on the stability of carbon nanowalls remain insufficiently explored. In particular, a systematic understanding of how transient ion beam exposure influences the morphological, structural, and functional characteristics of CNWs is still lacking. Therefore, the present work aims to investigate the impact of short-pulsed ion irradiation on the stability of carbon nanowalls and to assess their suitability for radiation-resistant electronic and optoelectronic applications.

Methodology

Synthesis of Carbon Nanowalls

CNWs were synthesized on quartz substrates using inductively coupled plasma–enhanced chemical vapor deposition (ICP-PECVD) in a PECVD Split Tube Furnace system (OTF-1200X-PEC4LV, MTI)[13]. The experimental setup consists of a horizontal CVD furnace equipped with a quartz tube with an inner diameter of 76 mm and an inductive coil connected to a radio-frequency power generator operating at 13.56 MHz with an automatic impedance-matching network. The gas delivery system is connected to one end of the quartz tube, while the opposite end is coupled to a rotary vacuum pump.

The synthesis process was carried out as follows. Quartz substrates with dimensions of 2 × 2 cm² were placed inside the quartz tube, after which the reaction chamber was sealed and evacuated to a base pressure of approximately 10^{-6} Torr. The substrates were then heated to 800 °C. At this stage, argon gas was introduced into the chamber at a flow rate of 5 sccm, and inductively coupled plasma was ignited at a power of 140 W. The substrates were exposed to these conditions for 10 min in order to remove residual surface contaminants and to generate localized high-temperature regions (“hot spots”) on the substrate surface.

Following the plasma pretreatment, CNW growth was initiated by introducing a gas mixture of argon (Ar) and methane (CH_4) with concentrations of 89.1% Ar and 9.9% CH_4, along with hydrogen (H_2). The flow rates were set to 20 sccm for the Ar/CH_4 mixture and 5 sccm for H_2. Hydrogen facilitates the dissociation of methane and serves as a source of reactive species that promote nucleation. Carbon atoms and hydrocarbon radicals generated during CH_4 decomposition adsorb preferentially at the surface hot spots, leading to the formation of graphitic nano-islands that act as nucleation centers for the vertical growth of multilayer graphene sheets. The CNW films were grown for 40 min under these conditions.

Irradiation of Samples Using the High-Current Pulsed Ion Accelerator INURA

Experiments on ionization irradiation were carried out at the INURA high-current pulsed ion accelerator located at Nazarbayev University [23]. The INURA accelerator operates on the basis of a high-voltage pulse generator that delivers pulses with a duration of ~100 ns to an accelerator diode with a voltage slew rate of more than 10^{12} V/s. For irradiation, CNWs samples were installed inside a vacuum chamber on a rotating holder located at a distance of 35 cm from the diode. The vacuum system was pumped out to a pressure of about $5 \cdot 10^{-6}$ mTorr, which ensured the minimization of impurity effects and the stability of the experimental conditions.

All irradiation procedures were performed in a single vacuum cycle to prevent possible changes in the properties of the samples between exposures. The ion beam was directed to the surface of the carbon nanowalls from the side of active layer and passed through the substrate. Beam parameters were controlled using a collimated Faraday cylinder equipped with a magnetic cut-off, which made it possible to accurately measure the current density and energy of the particles. The stability of the accelerator was checked by providing at least ten test pulses before the main series of irradiation. Samples of CNWs were exposed to an ion beam with different current densities – 4, 7, and 10 A/cm² – to assess dose-dependent effects.

Materials Characterization

The synthesized samples were characterized using a range of analytical techniques. Surface topography and roughness were examined by atomic force microscopy (AFM, Solver Spectrum NT-MDT) operated in semicontact mode using silicon cantilevers (NSG01) with a tip radius of approximately 10 nm and a resonance frequency of ~170 kHz. Raman spectroscopy (Solver Spectrum NT-MDT) with a 473 nm excitation laser was employed to investigate the structural and vibrational properties of the samples. Ultraviolet–visible (UV–Vis) transmittance spectra were recorded in the wavelength range of 200–1200 nm using an Agilent Cary 5000 spectrophotometer. All spectra were acquired in a double-beam configuration with a spectral bandwidth (SBW) of 2.0 nm. The sheet resistance of the samples was measured using a four-point probe system (RM3000, Jandel).

Results and discussion

Figure 1 shows the AFM results of CNWs samples before and after ion irradiation with an ion beam of different current densities (4, 7 and 10 A/cm²). The images (Figures 1a–h) show changes in surface morphology as a function of radiation dose. The initial sample (Figures 1a, b) is characterized by a uniform and dense structure of vertically oriented carbon nanowalls with a well-defined relief and element height of about 150–180 nm.

After exposure to an ion beam with a current density of 4 A/cm^2 (Figures 1c, d), a slight smoothing of the surface and partial enlargement of the nanowall formations are observed, which is associated with the initial stages of radiation-induced surface rearrangement. With an increase in the current density to 7 A/cm^2 (Figures 1e, f), the structure remains mostly homogeneous, but a slight decrease in the height of the nanowalls and the density of their distribution is recorded, which may be due to the local destruction of thin areas under the influence of the ion flux. At a maximum current density of 10 A/cm^2 (Figures 1g,h), the morphology retains the characteristic nanowall structure, although there are some flattening of the relief and a decrease in the amplitude of roughness, which indicates partial relaxation of the surface and possible compaction of the top layer.

In general, AFM analysis shows that the structure of CNWs demonstrates high resistance to ionization radiation: even at a maximum current density of 10 A/cm^2, the typical morphology of nanowall formations is preserved without significant destruction or degradation. This indicates the high radiation resistance of carbon nanowalls and their potential for use in conditions of exposure to ionizing radiation.

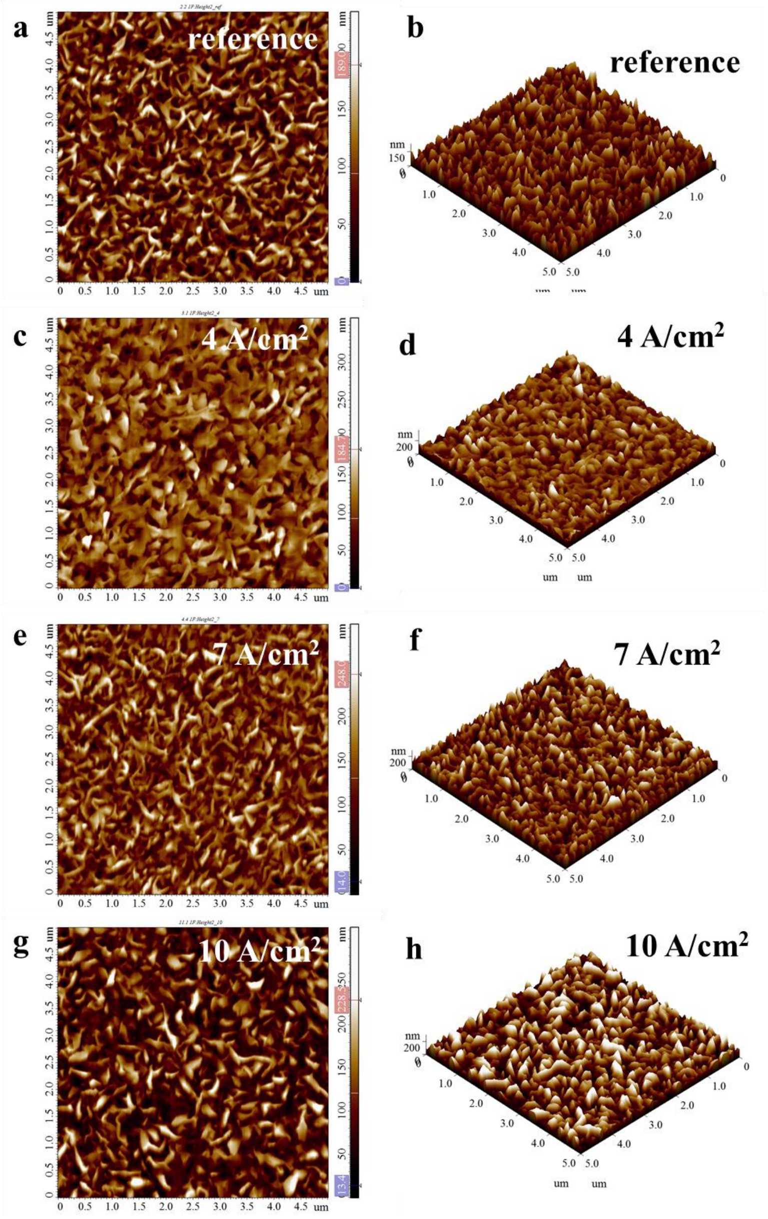

Figure 1 – AFM images of the surface of the CNWs before and after irradiation with an ion beam with different current densities: (a,b) initial sample, (c,d) 4 A/cm^2, (e,f) 7 A/cm^2, (g,h) 10 A/cm^2.

Figure 2 presents the results of the analysis of the distribution of surface heights of CNWs obtained on the basis of AFM data before and after ion irradiation of different current densities. The graph (Figure 2a) shows elevation profiles showing the change in surface morphology with increasing radiation dose. The original sample is characterized by a pronounced relief with an amplitude of oscillations up to 150 nm, which corresponds to a dense vertically oriented nanowall structure. When irradiated with a beam with a density of 4 A/cm^2, a partial smoothing of the surface and a decrease in the amplitude of altitude fluctuations are observed, which indicates the initial stage of radiation-induced modification. With a further increase in the current density to 7 and 10 A/cm^2, the shape of the profile becomes more orderly, while maintaining the characteristic nanowall relief, which confirms the structural stability of the material. The graph (Figure 2b) shows the average values of roughness parameters — arithmetic mean (R_a) and quadratic (RMS). It can be seen that with an increase in irradiation density, a slight increase in these parameters is observed, which can be associated with the formation of local microirregularities and changes in surface stress under the influence of ion flux.

In general, the results of the analysis confirm that ion irradiation does not cause significant damage to the surface of the CNWs, but only leads to minor changes in topography. The preservation of roughness parameters in a narrow range of values indicates a high radiation stability of the morphology of carbon nanowalls.

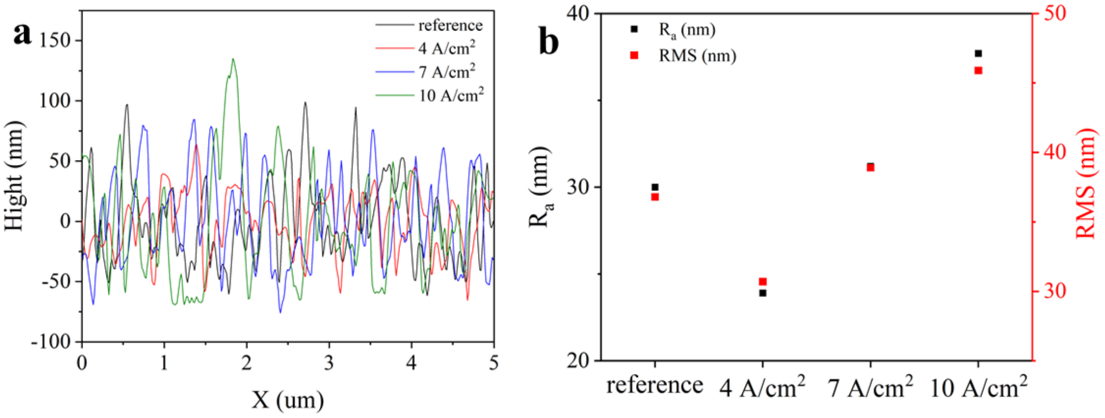

Figure 2 – Height profile and surface roughness parameters of CNWs before and after irradiation with an ion beam of different current densities: (a) altitude profiles obtained from AFM data; (b) arithmetic mean (Ra) and quadratic (RMS) surface roughness.

Figure 3 shows the results of Raman analysis of CNWs samples before and after ion irradiation with different current densities. The spectra (Figure 3a) show characteristic peaks of carbon nanostructures — D (~1350 cm^{-1}), G (~1580 cm^{-1}), D′ (~1620 cm^{-1}) and 2D (~2700 cm^{-1}), which confirms the preservation of the graphene-like sp^2 structure after exposure to ionizing radiation [24]. Regardless of the density of the irradiation current, the main peaks retain their position and shape, which indicates that there is no destruction of the crystal lattice and no degradation of the structure. The observed slight change in the relative intensity of the D and G peaks with an increase in the irradiation density may be due to the partial formation of radiation-induced defects that occur near the surface of the nanowall. However, the I_D/I_G ratio remains virtually unchanged, indicating minimal structural damage. An increase in the intensity of the 2D peak at 7 and 10 A/cm^2 (Figure 3b) may indicate local ordering or recrystallization of surface regions under the influence of energetic particles. Thus, the results of Raman spectroscopy show that even at high ion irradiation densities, the structure of CNWs retains a graphene-like nature and does not undergo significant degradation. This confirms the high radiation resistance of the material, which makes it promising for use in radiation-resistant optoelectronic and sensor devices.

Figure 4 shows the optical transmission spectra of CNWs films before and after ion irradiation with different current densities (4, 7 and 10 A/cm^2) in the wavelength range from 200 to 1200 nm. For the original (reference) sample, there is a smooth increase in transmittance from the ultraviolet region (about 250 nm) to the near-infrared, reaching a maximum value of about 55%. After exposure to the ion beam, a decrease in optical transparency is observed in all cases, indicating a change in surface morphology and a partial increase in the concentration of defects and scattering centers in the material. The results obtained are in good agreement with the AFM data (Figures 1 and 2), according to which, with an increase in irradiation density, there is a smoothing of the surface relief and a decrease in the amplitude of roughness, accompanied by a partial restructuring of the nanowall structure. The greatest reduction in transmittance is recorded at a current density of 4 A/cm^2, which correlates with the results of AFM analysis, which show local integrity violation and enlargement of nanowall formations at an early stage of radiation exposure. With a further increase in the current density to 7 and 10 A/cm^2, the transmittance values increase slightly, which is consistent with the relaxation and surface ordering observed according to the AFM data due to thermally induced effects. Thus, the comparison of optical and morphological data allows us to conclude that ion irradiation has a moderate effect on the structure of CNWs without causing its degradation. The preservation of the shape of the spectrum and the smooth nature of the dependence of transmission on the wavelength, along with insignificant changes in roughness, confirms the high radiation resistance of carbon nanowalls and the stability of their optical properties in a wide range of irradiation conditions.

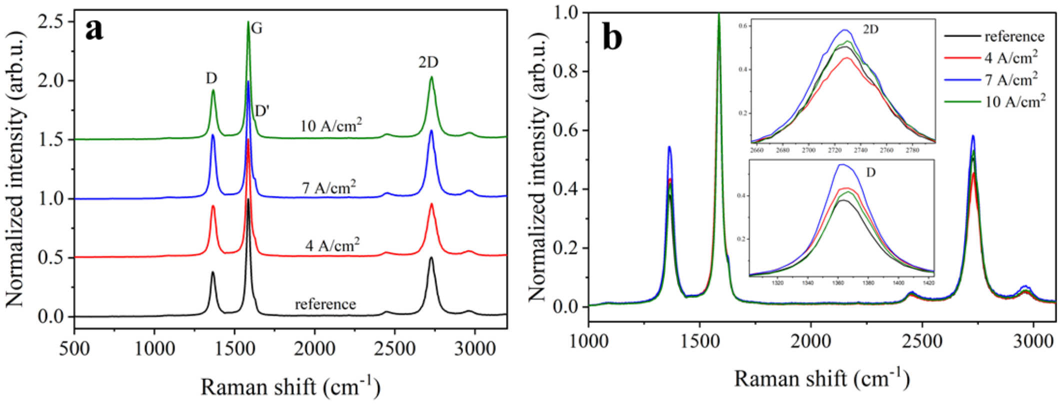

Figure 3 – Raman spectra of CNWs before and after ion irradiation: (a) comparative spectra at different current densities (4, 7 and 10 A/cm^2); (b) normalized spectra with enlarged regions of D and 2D peaks, demonstrating stability of structural properties.

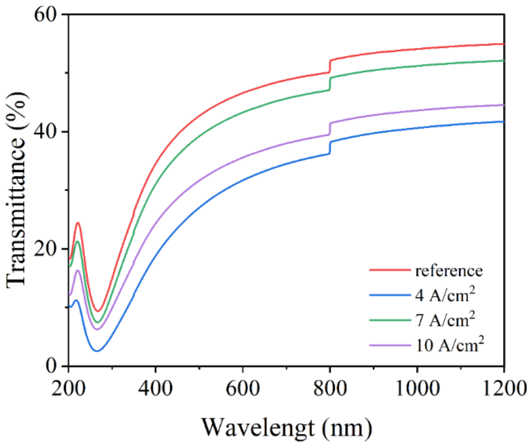

Figure 4 – Optical transmission spectra of CNWs films before and after ion irradiation at different current densities (4, 7 and 10 A/cm^2) in the wavelength range of 200–1200 nm.

Figure 5 shows the dependence of the sheet resistance of CNWs films on the ion irradiation density (4, 7 and 10 A/cm^2). For the initial sample, the value of sheet resistance is about 480 Ω/□. After irradiation, a distinct decrease in resistance is observed, especially at a current density of 4 A/cm^2, where it decreases to ~400 Ω/□, which may be due to a partial modification of the surface layers and an improvement in the electrical contact between the nanowalls due to the local thermal effect of the beam.

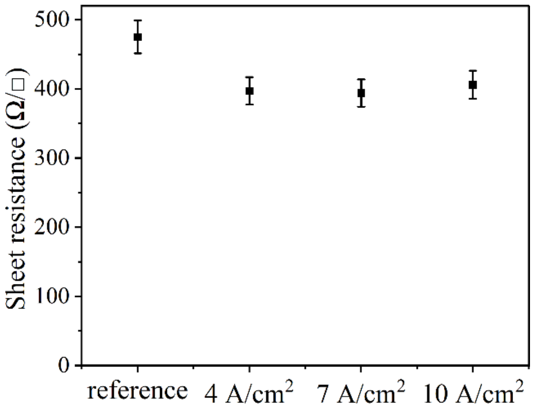

Figure 5 - Dependence of the sheet resistance of CNWs films on the density of ion irradiation (4, 7 and 10 A/cm^2).

With a further increase in current density to 7 and 10 A/cm^2, the resistance values stabilize and change slightly, which indicates the achievement of a state of structural equilibrium in which the processes of defect formation and relaxation cancel each other out. The results obtained are in good agreement with morphological analysis (Figures 1 and 2), where it was shown that the structure of the CNWs retains its integrity after irradiation and undergoes only minor changes in the surface topography.

Thus, the measurement results demonstrate that ion irradiation does not cause degradation of the electrically conductive properties of CNWs, but on the contrary, can lead to a moderate decrease in resistance due to structural stabilization and possible improvement of interwall connections. This confirms the high radiation resistance and stability of the electrical characteristics of carbon nanowalls, which makes them promising for use in radiation-resistant electronic and sensor devices.

Thus, the comprehensive analysis showed that ion irradiation does not lead to a significant degradation of the morphological, optical and electrical properties of carbon nanowalls. Slight changes in roughness and optical transmission are accompanied by stabilization of the structure and a decrease in sheet resistance at moderate doses of radiation. The results obtained indicate the high radiation resistance of CNWs and their potential for use as part of functional layers of radiation-resistant electronic and optoelectronic devices.

Conclusion

In this work, the effects of high-current pulsed ion irradiation on the morphological, structural, optical, and electrical properties of carbon nanowalls were systematically investigated. Atomic force microscopy revealed that ion irradiation with current densities of 4, 7, and 10 A/cm^2 induces only moderate modifications of surface topography. While slight smoothing of the relief, partial rearrangement of nanowall formations, and minor variations in roughness parameters were observed, the characteristic vertically oriented nanowall architecture was preserved even at the highest irradiation density. These results indicate a high morphological stability of CNWs under intense ionizing radiation.

Raman spectroscopy confirmed the preservation of the graphene-like sp^2 carbon structure after irradiation. The positions and shapes of the D, G, D′, and 2D bands remained essentially unchanged for all irradiation conditions, while only minor variations in peak intensities were detected. The nearly constant I_D/I_G ratio suggests that radiation-induced defect formation is limited and does not lead to significant lattice damage. The observed enhancement of the 2D peak intensity at higher current densities may be associated with local ordering or partial recrystallization processes induced by energetic ions.

Optical transmission measurements demonstrated that ion irradiation causes moderate changes in optical transparency, which correlate well with the AFM results. A decrease in transmittance at lower irradiation density (4 A/cm^2) is attributed to initial surface restructuring and increased scattering, whereas partial recovery of transparency at higher current densities is consistent with surface relaxation and structural stabilization. Importantly, the overall spectral shape remains unchanged, indicating the absence of severe optical degradation.

Electrical measurements showed that ion irradiation does not deteriorate the conductive properties of CNWs. On the contrary, a moderate decrease in sheet resistance was observed after irradiation, particularly at lower current density, which is likely related to improved interwall electrical contact and beam-induced thermal effects. At higher current densities, the resistance stabilizes, indicating a balance between defect generation and relaxation processes.

Overall, the comprehensive analysis demonstrates that carbon nanowalls exhibit a high degree of resistance to short-pulsed ion irradiation. The preservation of their morphology, graphene-like structure, optical response, and electrical conductivity confirms the robustness of CNWs under extreme irradiation conditions. These findings highlight the strong potential of carbon nanowalls as functional materials for radiation-resistant electronic, optoelectronic, and sensor applications.

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