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IMPACT OF MECHANICAL DEFORMATION ON THE ELECTRICAL PROPERTIES OF CARBON NANOWALLS

Abstract

Carbon nanowalls (CNWs) are promising carbon nanomaterials for flexible and wearable electronic applications due to their unique vertically oriented architecture and high electrical conductivity. In this work, the influence of mechanical deformation on the electrical properties of flexible CNW films was systematically investigated. CNWs were synthesized by inductively coupled plasma-enhanced chemical vapor deposition and transferred onto polymer substrates for electromechanical testing. Hall effect measurements revealed that increasing bending strain and cyclic mechanical loading result in a gradual increase in sheet resistance accompanied by a decrease in electrical conductivity and charge carrier mobility, while the carrier concentration remains nearly unchanged. Scanning electron microscopy showed the formation of deformation-induced microcracks and partial disruption of conductive pathways after repeated bending, whereas the overall nanowall morphology was largely preserved. Raman spectroscopy confirmed the stability of the sp² carbon framework, with an increased defect-related signal after deformation. The strong correlation between electrical, morphological, and spectroscopic results demonstrates that defect accumulation governs the electromechanical response of CNWs. These findings highlight the mechanical robustness of CNWs and their suitability for flexible electronic and sensing devices.

Keywords: carbon nanowalls, mechanical deformation, electrical properties, flexible electronics, Raman spectroscopy.

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Introduction

Carbon allotropes have attracted significant attention in recent years for the development of advanced electronic devices, including tactile sensors and strain sensors [1–3]. Their relatively low cost, natural abundance, and high chemical and thermal stability make carbon-based materials among the most promising candidates for next-generation electronic and sensing technologies [4, 5]. In layered carbon materials, van der Waals forces govern interlayer interactions, while precise control of the interlayer spacing enables accelerated charge transport and enhanced sensitivity in sensor applications [6, 7]. Moreover, tuning the electronic structure – such as the bandgap and band-edge positions – can be achieved by modifying the material thickness, crystal structure, atomic arrangement, surface functionalization, or by forming hybrid systems with other materials.

Carbon nanowalls (CNWs) represent a unique class of carbon nanostructures composed of self-assembled multilayer graphene sheets, where individual graphene layers serve as fundamental building blocks [8–10]. CNWs exhibit a distinctive morphology in which vertically oriented graphene sheets grow perpendicular to the substrate surface with a high degree of preferential alignment. These interconnected sheets form a self-supporting three-dimensional network with wall thicknesses ranging

from several to several tens of nanometers [11]. Owing to their unique architecture, CNWs possess an exceptionally high specific surface area of up to $\sim 1000 \text{ m}^2 \text{ g}^{-1}$, a highly porous microstructure, and excellent electrical conductivity. These properties make CNWs highly attractive for a broad range of applications, including energy storage systems, catalytic supports, sensors, and various electronic devices, particularly flexible and wearable electronics [12–16].

Recent studies on the mechanical properties of CNWs, led by Prof. Hori and co-workers, have demonstrated that CNWs exhibit a Young's modulus of approximately 28 GPa and a compressive strength of about 50 MPa [17]. These values surpass those reported for many other graphene-based materials and indicate pronounced elastoplastic behavior under mechanical deformation, highlighting the suitability of CNWs for tactile sensing applications. Furthermore, Zhou et al. reported the development of flexible and highly sensitive pressure sensors based on microstructured CNW electrodes [18]. The fabricated sensors exhibited ultrahigh sensitivities of 6.64 kPa^{-1} at pressures below 0.2 kPa, 1.26 kPa^{-1} in the range of 0.2–1 kPa, and 0.15 kPa^{-1} at 10 kPa. In addition, the sensors demonstrated an ultralow detection limit of 0.6 Pa, achieved through surface modification of polydimethylsiloxane (PDMS) with CNWs, as well as a fast response time of only 30 ms. These results indicate the superior overall performance of CNW-based pressure sensors compared to other microstructured sensing platforms.

Despite the rapid progress in CNW-based flexible and tactile devices, a fundamental understanding of how mechanical deformation influences the morphological, structural, and electrical properties of CNWs remains limited. Such knowledge is crucial for the rational design of mechanically robust and reliable flexible electronic devices. Therefore, systematic investigation of the effects of mechanical deformation on the electrical behavior of CNWs is of significant scientific and practical importance.

In this work, we investigate the impact of mechanical deformation on the electrical properties of CNWs. The obtained results provide fundamental insights into the electromechanical response of CNWs and establish a solid foundation for their future application in flexible electronics and tactile sensing systems.

Materials and methods

Synthesis of Carbon Nanowalls and Sample Preparation

Carbon nanowalls (CNWs) were synthesized on copper foil substrates ($2 \times 2 \text{ cm}^2$, thickness 50 μm) using inductively coupled plasma-enhanced chemical vapor deposition (ICP-PECVD) in a split-tube furnace PECVD system (OTF-1200X-PEC4LV, MTI Corporation). A detailed description of the synthesis setup and procedure has been reported in [19]. During growth, an Ar/CH₄ gas mixture was introduced at a total flow rate of 20 sccm, while H₂ was supplied at 5 sccm. The deposition process was carried out for 50 min.

The electrical properties of the samples under mechanical stress were evaluated on polymer substrates. For this purpose, CNWs initially synthesized on copper foils were transferred onto polymer substrates. The transfer process was carried out by immersing the CNW-coated copper foils in an aqueous ferric chloride (FeCl₃) solution to selectively etch the copper substrate [20, 21]. After complete dissolution of the copper, the freestanding CNW films were thoroughly rinsed with deionized water to remove residual etchant. Subsequently, the cleaned CNW films were carefully transferred onto clean polymer substrates by scooping them from the water surface. Following the transfer, the electrical characteristics of the CNWs were measured under various mechanical deformation modes to evaluate their flexibility and mechanical robustness.

Materials Characterization

The morphology of the synthesized CNW films was characterized using a scanning electron microscope (SEM, ZEISS Crossbeam 540). The structural properties of the samples were investigated by Raman spectroscopy (LabRAM Horiba Evolution and OmegaScope) using an excitation laser wavelength of 514.5 nm. The electrical properties of the CNW films were evaluated by Hall effect measurements employing a Van der Pauw configuration (HMS-5500, Ecopia).

Results and discussion

Figure 1 presents the electrical characterization of flexible CNW films subjected to different levels of mechanical bending [22]. Photographs in Figures 1a–c illustrate the sample configurations in the undeformed state (wo) and under bending radii of 0.5 cm and 0.1 cm, respectively. The electrical measurements were performed using Hall effect analysis in a Van der Pauw configuration to systematically evaluate the influence of mechanical deformation on the key electrical parameters of the CNW films.

As shown in Figures 1d–g, increasing the bending degree leads to a gradual increase in sheet resistance (Figure 1d) accompanied by a corresponding decrease in electrical conductivity (Figure 1e). This behavior can be attributed to the formation of microcracks, disruption of inter-wall electrical contacts, and partial modification of conductive pathways within the interconnected CNW network. In addition, the charge carrier concentration (Figure 1f) decreases with increasing mechanical deformation, which is further reflected in the reduction of carrier mobility (Figure 1g).

The most pronounced variations in electrical parameters are observed at a bending radius of 0.1 cm, indicating the critical deformation regime associated with the mechanical flexibility limits of the CNW films. Notably, despite these changes, the samples maintain measurable electrical conductivity even under severe bending conditions, demonstrating the high mechanical stability and structural integrity of the CNW architecture.

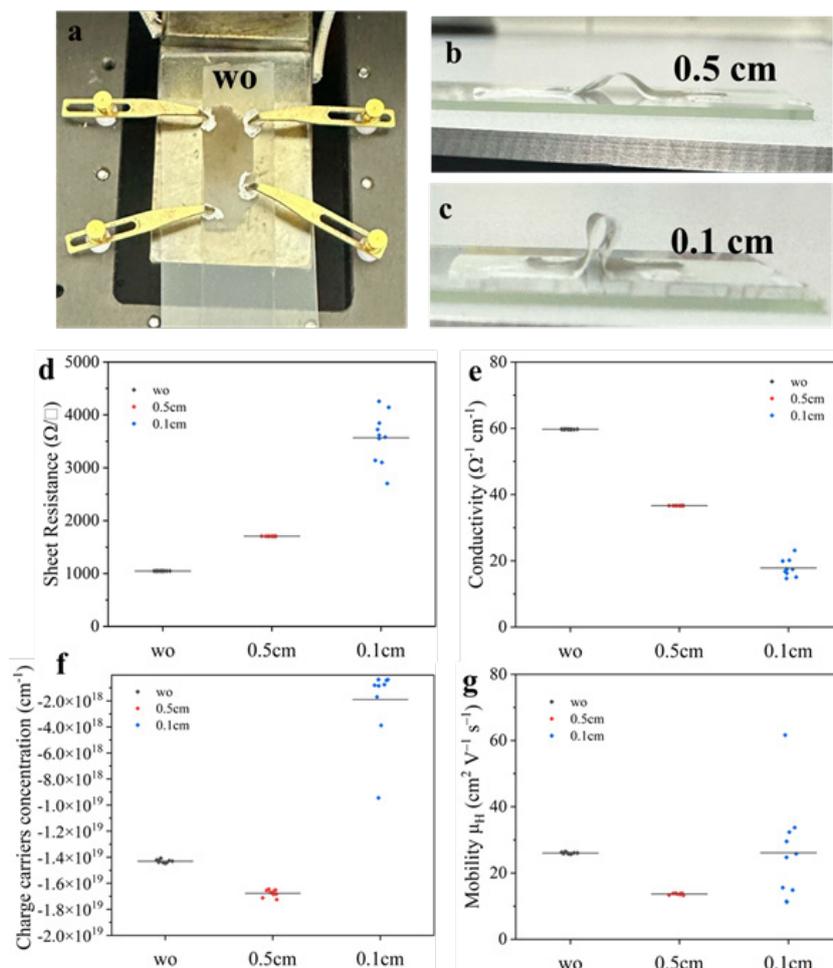


Figure 1 – Electrical properties of CNWs films at different degrees of mechanical bending: (a) non-deformation measurement configuration (wo), (b) at 0.5 cm bending radius and (c) at 0.1 cm bending radius; (d) sheet resistance, (e) electrical conductivity, (f) concentration of charge carriers, and (g) mobility of charge carriers

Figure 2 presents the electrical response of CNW films subjected to repeated mechanical bending cycles, aimed at evaluating the stability of their conductive properties under cyclic deformation – a critical requirement for flexible electronic applications. Figures 2a–d depict the evolution of sheet resistance, electrical conductivity, charge carrier concentration, and carrier mobility as a function of the number of bending cycles (10, 50, and 200 cycles).

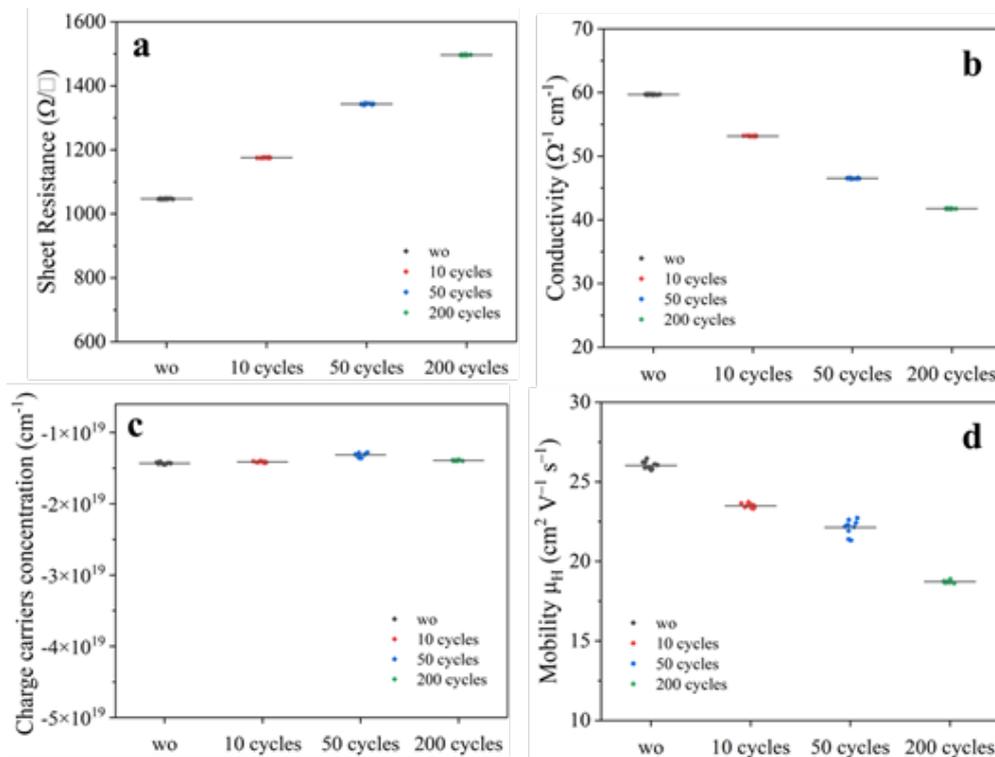


Figure 2 – Changes in the electrical properties of CNWs and N-CNWs films during cyclic mechanical bending: (a) sheet resistance, (b) electrical conductivity, (c) concentration of charge carriers, and (d) mobility of charge carriers after 10, 50, and 200 bending cycles

As shown in Figures 2a and 2b, increasing the number of bending cycles results in a gradual rise in sheet resistance accompanied by a corresponding decrease in electrical conductivity. These trends can be attributed to the progressive accumulation of microdefects and partial disruption of conductive pathways within the nanowall network, leading to reduced electrical percolation and inter-wall connectivity.

Notably, the charge carrier concentration (Figure 2c) remains nearly constant throughout the bending cycles, indicating the preservation of the chemical composition and the absence of significant degradation at the crystallographic or electronic structure level. In contrast, the carrier mobility (Figure 2d) exhibits a monotonic decrease with increasing cycle number, which can be associated with enhanced carrier scattering at deformation-induced defects and interfaces.

To gain deeper insight into the mechanisms underlying the observed changes in electrical characteristics, CNW samples were examined by scanning electron microscopy (SEM) before and after 200 cycles of mechanical deformation. Figure 3 presents SEM images of the CNW surface in the pristine state (Figure 3a) and after cyclic bending (Figure 3b).

Prior to deformation, the surface exhibits a homogeneous distribution of vertically aligned nanowalls, forming a dense, well-interconnected conductive network without any visible structural damage. After 200 bending cycles (Figure 3b), the emergence of microcracks and localized regions of partial structural rupture can be observed, particularly along the directions of applied mechanical strain.

Despite the presence of these local defects, the overall morphology and vertical architecture of the nanowall network remain largely preserved, indicating high mechanical robustness and structural resilience of the CNWs. This morphological stability explains the retention of electrical conductivity even under repeated bending.

These observations are in good agreement with the electrical measurements presented in Figure 2, confirming that the gradual increase in sheet resistance and the reduction in charge carrier mobility arise from the accumulation of deformation-induced microstructural defects under cyclic mechanical loading.

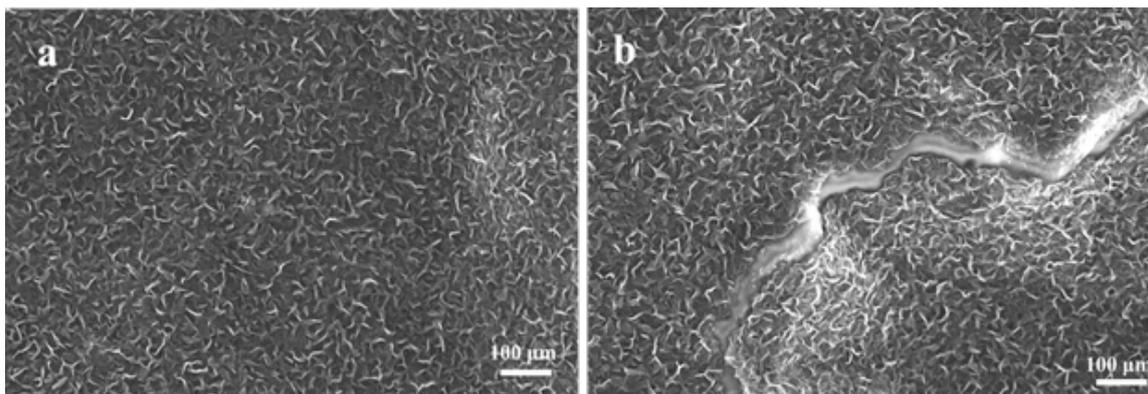


Figure 3 – SEM images of the surface of N-CNWs films:
(a) before and (b) after 200 cycles of mechanical deformation

Figure 4 shows the Raman spectra of CNW films measured before and after mechanical bending. Both spectra exhibit the characteristic Raman features of graphitic carbon materials, including the D band ($\sim 1350\text{ cm}^{-1}$), G band ($\sim 1580\text{ cm}^{-1}$), D' band ($\sim 1620\text{ cm}^{-1}$), and the second-order 2D band ($\sim 2700\text{ cm}^{-1}$), confirming the graphitic nature and multilayer structure of the carbon nanowalls [23, 24].

Before bending, the spectrum is dominated by a pronounced G band accompanied by a well-defined D band, which is typical for vertically oriented CNWs with a high density of edge sites and structural defects inherent to the nanowall architecture. The presence of the 2D band further indicates the multilayer graphene character of the nanowalls.

After mechanical bending, no significant shifts in the positions of the main Raman peaks are observed, indicating that the overall sp^2 carbon framework and crystallographic structure of the CNWs remain preserved under mechanical deformation. However, a noticeable increase in the relative intensity of the D and D' bands compared to the G band is observed after bending, suggesting an increase in defect density. This effect can be attributed to the formation of deformation-induced microdefects, such as microcracks, edge disruptions, and local distortions within the nanowall network.

These Raman results are in good agreement with the SEM observations (Figure 3), which revealed the appearance of microcracks and partial structural rupture after cyclic bending, as well as with the electrical measurements (Figures 1 and 2), which showed an increase in sheet resistance and a reduction in charge carrier mobility. Together, these findings confirm that the degradation of electrical performance under mechanical deformation is primarily associated with defect-induced carrier scattering and disruption of conductive pathways, rather than with chemical degradation or destruction of the graphitic backbone.

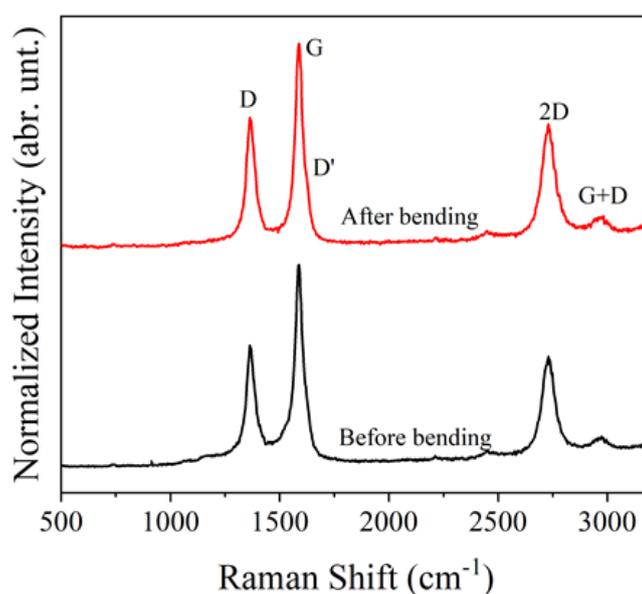


Figure 4 – Raman spectra of CNW films before and after mechanical bending

Conclusions

In this work, the effect of mechanical deformation on the electrical properties of CNW films was systematically investigated. Electrical measurements revealed that increasing bending strain and cyclic mechanical loading lead to a gradual increase in sheet resistance and a corresponding decrease in electrical conductivity and charge carrier mobility, while the carrier concentration remained largely unchanged. These results indicate that mechanical deformation primarily affects charge transport rather than the intrinsic electronic structure of the material.

SEM analysis demonstrated that cyclic bending induces microcracks and partial disruption of conductive pathways within the CNW network, whereas the overall vertical architecture and structural integrity of the nanowalls are largely preserved. Raman spectroscopy further confirmed the stability of the sp^2 carbon framework, showing no significant peak shifts after bending, while an increase in defect-related Raman modes indicated deformation-induced microstructural damage.

The strong correlation between electrical, morphological, and spectroscopic results confirms that the observed degradation in electrical performance originates from defect accumulation and enhanced carrier scattering under mechanical stress. Despite these effects, CNW films retained measurable conductivity even under severe and repeated bending, demonstrating high mechanical robustness. These findings highlight the potential of CNWs as reliable conductive materials for flexible and wearable electronic devices, including tactile and strain sensors.

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КӨМІРТЕКТІ НАНОҚАБЫРҒАЛАРДЫҢ ЭЛЕКТРЛІК ҚАСИЕТТЕРІНЕ МЕХАНИКАЛЫҚ ДЕФОРМАЦИЯНЫҢ ӘСЕРІ

Аңдатпа

Көміртекті наноқабырғалар (КНҚ) өздерінің бірегей тік бағытталған архитектурасы мен жоғары электрөткізгіштігінің арқасында икемді және киілетін электроника үшін перспективті көміртекті нано-материалдар. Бұл жұмыста икемді КНҚ үлдірлерінің электрлік қасиеттеріне механикалық деформацияның әсері жүйелі түрде зерттелді. КНҚ индуктивті байланысқан плазмалық-химиялық тұндыру әдісімен синтезделіп, электромеханикалық сынақтар жүргізу үшін полимерлі төсемдерге көшірілді. Холл эффектісі әдісімен жүргізілген өлшеулер иілу деформациясының және циклдік механикалық жүктеменің артуы беттік кедергінің біртіндеп өсуіне, сондай-ақ электрөткізгіштіктің және заряд тасымалдаушылар қозғалғыштығының төмендеуіне әкелетінін, ал тасымалдаушылар концентрациясының іс жүзінде өзгеріссіз қалатынын көрсетті. Сканерлеуші электрондық микроскопия көп мәрте иілу нәтижесінде микрожарықтардың түзілуін және өткізгіш арналардың ішінара бұзылуын анықтады, алайда наноқабырғалардың жалпы морфологиясы негізінен сақталды. Раман спектроскопиясы sp^2 -көміртекті қаңқаның тұрақтылығын растап, деформациядан кейін дефектілерге тән модалардың қарқындылығының артқанын көрсетті. Алынған нәтижелер КНҚ-ның жоғары механикалық тұрақтылығын және оларды икемді электрондық әрі сенсорлық құрылғыларда қолдану әлеуетін айқындайды.

Тірек сөздер: көміртекті наноқабырғалар, механикалық деформация, электрлік қасиеттер, икемді электроника, Раман спектроскопиясы.

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ВЛИЯНИЕ МЕХАНИЧЕСКОЙ ДЕФОРМАЦИИ НА ЭЛЕКТРИЧЕСКИЕ СВОЙСТВА УГЛЕРОДНЫХ НАНОСТЕН

Аннотация

Углеродные наностены (УНС) являются перспективными углеродными наноматериалами для гибкой и носимой электроники благодаря их уникальной вертикально ориентированной архитектуре и высокой электропроводности. В данной работе систематически исследовано влияние механической деформации на электрические свойства гибких пленок УНС. УНС были синтезированы методом индуктивно-связанного плазменно-химического осаждения из газовой фазы и перенесены на полимерные подложки для проведения электромеханических испытаний. Измерения методом эффекта Холла показали, что увеличение изгибной деформации и циклической механической нагрузки приводит к постепенному росту поверхностного сопротивления и снижению электропроводности и подвижности носителей заряда, при этом концентрация носителей остается практически неизменной. Сканирующая электронная микроскопия выявила образование микротрещин и частичное нарушение проводящих каналов после многократного изгиба, тогда как общая морфология наностенок в целом сохраняется. Рамановская спектроскопия подтвердила стабильность sp²-углеродного каркаса с увеличением дефектных мод после деформации. Полученные результаты демонстрируют механическую устойчивость УНС и их перспективность для применения в гибких электронных и сенсорных устройствах.

Ключевые слова: углеродные наностены, механическая деформация, электрические свойства, гибкая электроника, рамановская спектроскопия.