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ROLE OF SYNTHESIS TIME IN SHAPING THE MORPHOLOGY AND STRUCTURE OF CARBON NANOWALLS

Carbon nanowalls (CNWs), a unique class of vertically oriented graphene-based nanostructures, have attracted significant attention due to their promising applications in energy storage, electronics, and sensing technologies. These materials combine high surface area, excellent electrical conductivity, and tunable morphology, making them ideal for a range of functional devices. However, controlling their structural evolution during synthesis remains a critical challenge. This study aims to systematically investigate the effect of synthesis duration on the morphological, structural, and optical properties of CNWs grown on silicon substrates using capacitively coupled plasma-enhanced chemical vapor deposition (CCP-PECVD). The research focuses on understanding how variations in growth time influence nanowall formation, defect density, and surface characteristics, which are essential for tailoring material performance. The CNWs were synthesized at four different durations—30, 60, 90, and 120 minutes—and characterized using SEM, AFM, Raman spectroscopy, and UV–Vis reflectance analysis. The results demonstrate a clear time-dependent evolution in CNW morphology and structure: from sparse and disordered features at 30 minutes to dense, petal-like graphitic walls at 120 minutes. Raman analysis showed increasing defect density with time, followed by partial graphitization at longer durations. Optical measurements revealed reduced reflectance with increasing synthesis time, indicating enhanced light trapping and surface roughness.

Key words: carbon nanowalls, deposition, substrate, growth time.

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Көміртекті наноқабырғалардың морфологиясы мен құрылымын қалыптастырудағы синтез уақытының рөлі

Көміртекті наноқабырғалар (КНҚ) – графенге негізделген тік бағытталған наноқұрылымдардың ерекше класы – энергияны сақтау, электроника және сенсорлық технологиялар салаларында кең перспективаларға ие болғандықтан, ғылыми қауымдастықтың үлкен қызығушылығын тудырып отыр. Бұл материалдар үлкен меншікті беткі ауданға, жоғары электр өткізгіштікке және реттелетін морфологияға ие, бұл оларды функционалдық құрылғылардың кең ауқымы үшін перспективалы етеді. Алайда, КНҚ-ның синтез кезіндегі құрылымдық эволюциясын басқару әлі де маңызды ғылыми мәселе болып табылады. Осы зерттеудің мақсаты – кремний негізіндегі субстраттарда сыйымдылықпен байланысқан плазмалы-химиялық бу фазасында тұндыру (CCP-PECVD) әдісімен алынған КНҚ-ның морфологиялық, құрылымдық және оптикалық қасиеттеріне синтез ұзақтығының әсерін жүйелі түрде зерттеу. Зерттеу барысында өсу уақытының өзгеруі КНҚ түзілуіне, ақаулар тығыздығына және беткі сипаттамаларына қалай әсер ететіні талданды. КНҚ төрт түрлі ұзақтықта — 30, 60, 90 және 120 минут — синтезделіп, СЭМ, АСМ, Раман

спектроскопиясы және УФ-көрінетін диапазондағы шағылу спектрлерімен сипатталды. Алынған нәтижелер КНҚ-ның морфологиясы мен құрылымының уақытқа байланысты айқын эволюциясын көрсетті: 30 минутта сирек және ретсіз құрылымдардан бастап, 120 минутта тығыз әрі жапырақ тәрізді графитті қабырғаларға дейінгі өзгеріс байқалды. Раман спектроскопиясы синтез уақыты артқан сайын ақаулар тығыздығының өскенін, ал одан кейінгі кезеңде ішінара графитизацияның орын алғанын көрсетті. Оптикалық өлшеулер синтез ұзақтығы артқан сайын шағылудың төмендейтінін көрсетті, бұл өз кезегінде жарықты тиімді тұзақтауға және беткі кедір-бұдырлықтың артуына байланысты екенін білдіреді.

Түйін сөздер: көміртекті наноқабырғалар, тұндыру, субстрат, өсу уақыты.

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Роль времени синтеза в формировании морфологии и структуры углеродных наностен

Углеродные наностены (УНС), уникальный класс вертикально ориентированных наноструктур на основе графена, привлекли значительное внимание из-за их многообещающих применений в технологиях хранения энергии, электронике и сенсорных технологиях. Эти материалы сочетают в себе большую площадь поверхности, превосходную электропроводность и настраиваемую морфологию, что делает их идеальными для ряда функциональных устройств. Однако контроль их структурной эволюции во время синтеза остается критической проблемой. Целью данного исследования является систематическое изучение влияния продолжительности синтеза на морфологические, структурные и оптические свойства УНС, выращенных на кремниевых подложках с использованием емкостно-связанного плазменно-химического осаждения из паровой фазы (CCP-PECVD). Исследование сосредоточено на понимании того, как изменения во времени роста влияют на формирование УНС, плотность дефектов и характеристики поверхности, которые необходимы для адаптации характеристик материала. УНС были синтезированы в течение четырех различных продолжительностей — 30, 60, 90 и 120 минут — и охарактеризованы с помощью СЭМ, АСМ, КРС и анализа отражения в УФ-видимом диапазоне. Результаты демонстрируют четкую эволюцию морфологии и структуры УНС со временем: от редких и неупорядоченных особенностей через 30 минут до плотных, лепесткообразных графитовых стенок через 120 минут. Анализ Рамана показал увеличение плотности дефектов со временем, за которым следует частичная графитизация при более длительных длительностях. Оптические измерения выявили снижение отражательной способности с увеличением времени синтеза, что указывает на улучшение улавливания света и шероховатости поверхности.

Ключевые слова: углеродные наностены, осаждение, подложка, время роста.

Introduction

Carbon nanomaterials – including zero-dimensional (0D) fullerenes and carbon dots, one-dimensional (1D) carbon nanotubes (CNTs) and carbon nanofibers (CNFs), two-dimensional (2D) materials such as graphene and its derivatives, as well as three-dimensional (3D) forms like nanodiamonds – have attracted significant attention since their discovery [1–3]. Unlike conventional carbon materials such as graphite, carbon black, and diamond, carbon nanostructures exhibit unique mechanical and transport properties due to their distinct atomic arrangements [4]. Despite variations in geometry and dimensionality, these materials share a common structural motif: a hexagonal (honeycomb-like) lattice composed of sp - hybridized carbon atoms [2]. This structural feature endows them with

exceptional electrical conductivity, chemical stability, and a range of other outstanding physical properties.

For the practical implementation of carbon nanomaterials in various devices, precise control over their morphology and chemical characteristics during deposition onto substrates is essential. In particular, the ability to synthesize these materials at low temperatures is crucial for applications involving thermally sensitive substrates.

Graphitic carbon is of particular interest for use in battery electrodes and thermal management systems in energy and electronics due to its high electrical and thermal conductivity, chemical inertness, and mechanical strength [5–9]. It also finds applications in biotechnology, benefiting from its chemical stability. Graphite flakes, with their high specific surface area, promote uniform substrate coverage and facilitate the vertical growth of graphene sheets. Nanowall structures, due to their porosity and interplanar spacing, provide a high surface-to-volume ratio, which is especially advantageous for sensor and electrochemical applications [10,11].

To date, a variety of carbon nanostructures – such as carbon nanowalls (CNWs), vertical graphene (VG), and three-dimensional graphene (3DG) – have been synthesized using plasma-enhanced chemical vapor deposition (PECVD) on different types of substrates and with various plasma sources [10,12–14]. However, the detailed mechanisms governing the deposition process remain insufficiently understood. Further investigation into the deposition kinetics is necessary, with particular emphasis on the interactions between ions and radicals generated by the plasma, as well as the effects of deposition duration and plasma exposure time on the resulting material's morphology and structure.

This study focuses on investigating the effect of synthesis time on the morphological and structural properties of carbon nanowalls. CNWs were synthesized on silicon substrates using the PECVD method, with synthesis durations ranging from 30 to 120 minutes. The morphological and structural characteristics of the resulting samples were examined to determine how synthesis time influences the formation and properties of CNWs.

Methodology

CNWs were synthesized via capacitively coupled plasma-enhanced chemical vapor deposition (CCP-PECVD) [8,15] on silicon substrates (Si (100), 500 μm thickness, ST orientation, University Wafer, USA). Prior to deposition, all substrates were sequentially cleaned in an ultrasonic bath using acetone and isopropanol (Sigma-Aldrich), followed by drying with nitrogen gas. Following substrate preparation, CNWs were synthesized according to the procedure described in [8] for different durations (30, 60, 90, and 120 minutes) to systematically investigate the effect of deposition time on the morphology and structural characteristics of the CNWs. The resulting samples were characterized using various analytical techniques. The morphology of the CNWs was examined using a scanning electron microscope (SEM, ZEISS Crossbeam 540). Surface topography and roughness were analyzed using atomic force microscopy (AFM, Solver Spectrum NT-MDT) operated in semi-contact mode with silicon probes (NSG01, tip radius ~10 nm, resonance frequency ~170 kHz). Raman spectroscopy (Solver Spectrum NT-MDT, 473 nm laser) was employed to study the structural and vibrational properties of the samples [16]. UV–Vis reflectance spectra were recorded in diffuse reflectance mode over the 200–800 nm range using an Agilent Cary 5000 spectrophotometer equipped with a DRA-2500 integrating sphere. All spectra were acquired in dual-beam mode with a spectral bandwidth (SBW) of 2.0 nm.

Results and discussion

Figure 1 presents SEM images of carbon nanowalls (CNWs) synthesized on silicon substrates at different deposition times. The results clearly demonstrate the strong influence of synthesis duration on the surface morphology of CNWs. At 30 minutes (Figure 1a), the silicon surface appears mostly smooth, with virtually no visible formation of CNWs or vertical graphene sheets. By 60 minutes (Figure 1b), initial growth of CNWs is observed, characterized by a labyrinth-like morphology with emerging vertically aligned graphene nanosheets. Extending the synthesis time to 90 minutes (Figure 1c) leads to a denser and more developed network of nanowalls with increased wall thickness. At 120 minutes (Figure 1d), the CNWs exhibit a transition from labyrinthine to petal-like morphology. Additionally, individual graphene sheets become more prominent, and thicker wall structures are observed. Occasional graphite-like deposits also begin to appear on the surface, indicating the onset of secondary carbon aggregation processes. These morphological changes indicate that synthesis time

plays a critical role in the evolution and structural characteristics of CNW films.

The SEM series in Figure 1 clearly demonstrates how CNW morphology evolves with synthesis time – from negligible growth at 30 min, through labyrinthine walls at 60-90 min, to petal-like, edge-rich structures at 120 min. These findings are consistent with existing literature, providing strong evidence that deposition time critically controls wall thickness, surface area, and nanostructure edge characteristics – key factors for optimizing CNWs in functional devices.

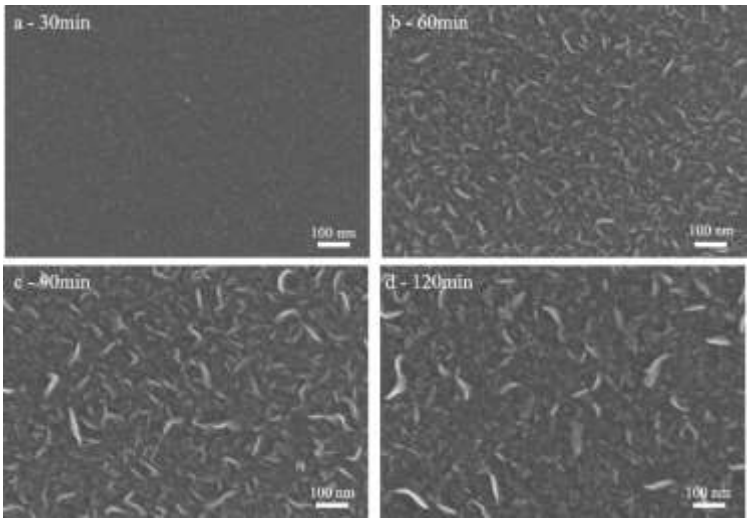

Figure 1 – SEM images showing the evolution of carbon nanowall (CNW) morphology synthesized on silicon substrates via CCP-PECVD at different deposition times:
(a) 30 min, (b) 60 min, (c) 90 min, and (d) 120 min.

The AFM analysis clearly demonstrates the time-dependent evolution of the surface morphology of CNWs (see Figure 2). At 30 minutes (Figure 2a), the substrate remains relatively smooth, with only a few isolated nucleation sites and a maximum height of ~28 nm, indicating minimal CNW growth. After 60 minutes (Figure 2b), the surface becomes more uniformly covered with early-stage CNWs exhibiting a granular texture and a height increase to ~49 nm. At 90 minutes (Figure 2c), the CNWs begin forming vertically oriented structures with noticeable roughness and a further increase in height to ~74 nm. By 120 minutes (Figure 2d), a well-developed, densely packed CNW network is observed, with a distinct petal-like morphology and a maximum height reaching ~119 nm. This trend highlights the progressive thickening and vertical alignment of CNWs over time, consistent with the SEM observations in Figure 2.

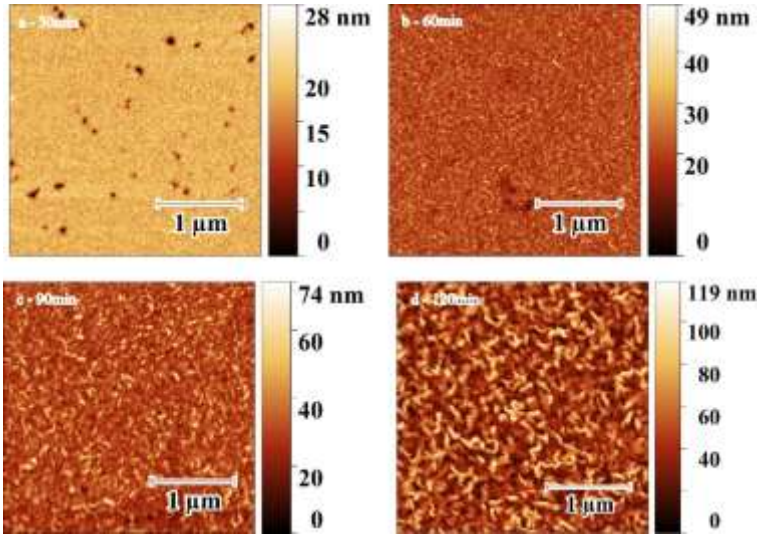

Figure 2 – AFM images showing the surface topography of CNWs synthesized on silicon substrates via CCP-PECVD at different deposition times: (a) 30 min, (b) 60 min, (c) 90 min, and (d) 120 min.

Raman analysis results are presented in Figure 3. All obtained spectra (Figure 3a) exhibit a peak at approximately ~520 cm^{-1}, corresponding to the silicon substrate [17]. As the deposition time increases, the intensity of this peak decreases, which is attributed to the increasing thickness of the CNW films that attenuate the silicon signal.In addition, all spectra in Figure 3a reveal the key structural features of CNWs, including the D, G, D′, and 2D bands, which indicate the presence of sp^2-hybridized carbon networks with varying degrees of disorder [12,18,19]. At the shortest deposition time (30 min), the intensities of the D and G bands are relatively low, consistent with minimal CNW growth observed in SEM (Figure 1a) and low surface roughness seen in AFM (Figure 2a). As the deposition time increases to 60 and 90 minutes, the intensities of the D and G bands rise, and the I_D/I_G ratio (Figure 3b) increases, indicating a higher density of structural defects and wall formation. This observation correlates well with the labyrinth-like structures observed in SEM (Figure 1b–c) and the increased surface topography seen in AFM (Figure 2b–c). Notably, the I_D/I_G ratio peaks at 60 minutes, suggesting a maximum in structural disorder or edge density. Beyond this point, at 90 and 120 minutes, a slight decline or plateau in the I_D/I_G ratio is observed, which may indicate partial graphitization or increased wall thickness and stacking of CNWs. This is in agreement with the petal-like morphology and thick walls observed in SEM (Figure 1d) and the increased height in AFM (Figure 2d). The inset in Figure 3b shows the evolution of the G_f parameter, which reflects the relative contribution of the G band to the total spectrum and can be interpreted as the degree of graphitization. The trend further supports the observed structural transition. In Figure 3c, the $I_{D'}/I_G$ ratio, which is sensitive to edge defects, gradually decreases with increasing synthesis time, indicating a reduction in edge defect density and the lateral growth or coalescence of CNW walls at longer deposition durations. These spectroscopic results strongly correlate with the morphological evolution observed in both SEM and AFM analyses, providing a comprehensive understanding of CNW growth dynamics as a function of deposition time.

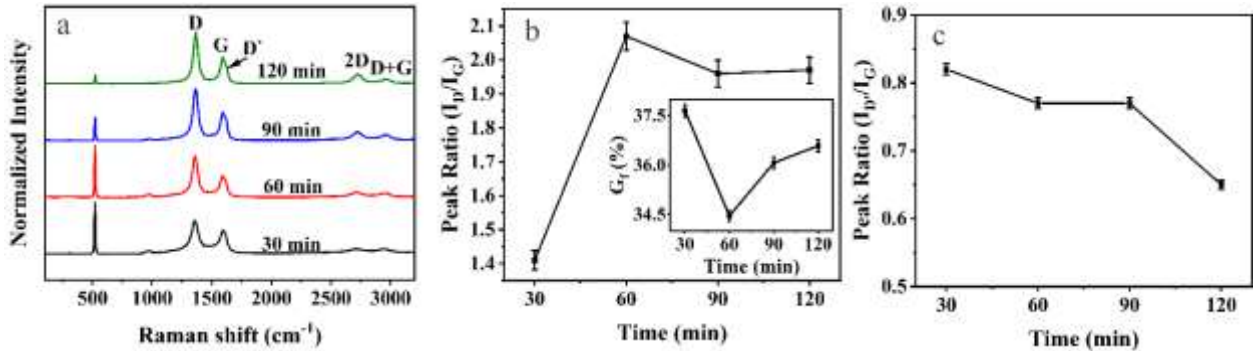

Figure 3 – Raman spectroscopy analysis of CNWs synthesized at different deposition durations. (a) Raman spectra of CNWs obtained at 30, 60, 90, and 120 minutes. (b) Evolution of the I_D/I_G peak ratio with deposition time and corresponding G_f content (inset). (c) Variation of the $I_{D'}/I_G$ peak ratio as a function of deposition time.

Figure 4 shows the UV–visible reflection spectra of CNWs synthesized for 30, 60, 90, and 120 minutes, with pristine silicon serving as a reference. All CNW samples display characteristic reflection features in the 300–500 nm range.

The sample synthesized for 30 minutes exhibits the highest reflectance, while reflection intensity progressively decreases with longer synthesis durations.

The 120-minute sample demonstrates the lowest reflectance, suggesting that prolonged growth leads to significant morphological modifications – such as increased surface roughness, wall thickness, and possibly defect density – that enhance light trapping and reduce optical reflection.

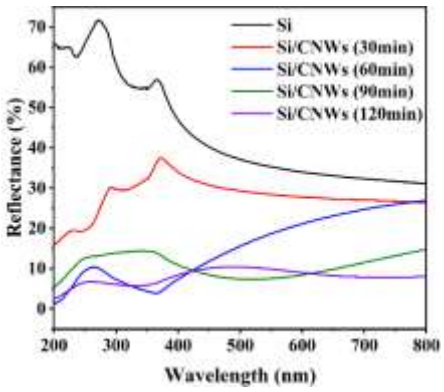

Figure 4 – UV–vis reflectance spectra of CNW films synthesized at different growth durations.

This trend is consistent with the morphological evolution observed in the SEM and AFM analyses (Figures 1 and 2), where extended synthesis time led to a transition from sparse or labyrinth-like CNW structures to dense, petal-like morphologies with thicker walls and greater vertical height. Furthermore, Raman spectroscopy (Figure 3) confirmed increased structural disorder and partial graphitization at longer synthesis durations, which may contribute to increased light absorption and scattering. Together, these results indicate that the optical reflectance of CNWs can be effectively tuned through controlled growth time, enabling their application in optoelectronic devices, anti-reflective coatings, and photovoltaics.

Conclusion

In this study, carbon nanowalls (CNWs) were synthesized on silicon substrates using the CCP-PECVD method for varying durations (30, 60, 90, and 120 minutes) to systematically investigate the effect of deposition time on their morphological, structural, and optical properties. SEM analysis revealed a clear evolution of CNW morphology with increasing synthesis time: from minimal or absent nanostructures at 30 minutes to densely packed, vertically oriented graphene-like walls at 60–90 minutes, and ultimately to thicker, petal-like structures with partial graphitization and aggregated carbon deposits at 120 minutes. AFM data supported this trend, showing increasing surface roughness and nanostructure height over time, indicating continuous vertical and lateral growth. Raman spectroscopy further confirmed these morphological transformations. The increasing D and G band intensities and rising I_D/I_G ratios up to 60 minutes reflect a rise in structural disorder and edge density during the early growth stages. At longer durations (90–120 minutes), a slight decline in I_D/I_G along with reduced I I_D/I_G suggests partial graphitization, wall merging, and a decline in edge defects – consistent with thicker, more stacked CNW walls. Optical reflectance measurements showed a steady decrease in UV–visible reflectivity with increasing synthesis time, suggesting enhanced light absorption due to morphological changes such as increased roughness, thickness, and structural disorder. This indicates that CNW films with longer growth durations may be advantageous for applications requiring light-trapping or anti-reflective properties, such as photovoltaics or sensing platforms.

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