

Humidity sensing properties of carbon nanowall films synthesized by CCP-PECVD

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Abstract. Carbon nanowall (CNW) films were synthesized by capacitively coupled plasma-enhanced chemical vapor deposition and evaluated as active layers for resistive humidity sensing. SEM observations confirmed the formation of vertically oriented CNW sheets, while Raman analysis showed typical defect-related and graphitic carbon bands, confirming the formation of a petal-like carbon nanowall network. Humidity-sensing performance was studied over a relative humidity range of 10–100% at 27, 50, and 75 °C. The fabricated sensors exhibited repeatable electrical responses over short cycling tests and a clear temperature dependence. At 27 °C, the sensor showed the highest sensitivity, reaching 2.27% at 100% RH, with response and recovery times of 87 and 97 s, respectively. Increasing the operating temperature reduced the sensitivity to 0.49% at 50 °C and 0.22% at 75 °C, which can be attributed to enhanced desorption of water molecules from the CNW surface. Good short-term repeatability was observed at 27 and 50 °C over five consecutive humidity cycles, whereas a gradual signal drift appeared at 75 °C. The sensor response was approximately linear over the investigated RH range at 27 °C, while increasing nonlinearity at higher humidity levels was observed at elevated temperatures. These findings indicate that transferred CNW films can be considered as simple and inexpensive platforms for humidity monitoring.

Keywords: carbon nanowalls, humidity sensors, CCP-PECVD, relative humidity, resistive sensing.

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Introduction

Accurate control of relative humidity is important for environmental monitoring, food storage, agricultural production, and industrial processes [1]. Humidity sensors operate by converting the interaction between water vapor and a sensitive material into an electrical or optical signal [2]. In resistive devices, this interaction usually changes the charge transport properties of the sensing layer. Conventional humidity sensors are commonly fabricated using polymeric, oxide-based, or porous materials. Their response is usually governed by water adsorption, which changes resistance, capacitance, or other electrical parameters [3,4]. For practical use, a humidity sensor should combine sufficient sensitivity with stable operation, reproducible response, and acceptable response/recovery times. To meet these performance requirements, sensors based on various nanomaterials have been proposed for use as highly efficient humidity sensors [5]. Among these, sensors based on nanotubes and related carbon composites have attracted attention due to their low cost, high sensitivity over a wide range of relative humidity (RH), and fast response or recovery times [6]. For decades, carbon nanomaterials have been investigated as ideal candidates for humidity sensors. Graphene and related carbon nanomaterials are considered promising candidates for sensor fabrication because of their high surface area, electrical conductivity, and adsorption-sensitive surface properties [7].

Carbon nanowalls represent a three-dimensional graphene-like architecture composed of vertically standing nanosheets [8]. Such an open structure provides exposed edges and accessible surface sites, which may facilitate the adsorption of water molecules and influence the resistance of the film.

This study investigates the potential of CNW films synthesized by the CCP-PECVD method as active layers for resistive humidity sensing. The morphological and structural characteristics of the resulting CNW films were investigated using scanning electron microscopy (SEM) and Raman spectroscopy. The humidity sensitivity of the CNW-based sensor was studied over a wide range of relative humidity from 10% to 100% at various temperatures (27 °C, 50 °C, and 75 °C). A maximum sensitivity of 2.27% was achieved at 100% relative humidity and a device temperature of 27°C. The sensor showed good short-term repeatability at 27 and 50 °C over five consecutive humidity cycles, whereas a gradual drift in the response was observed at 75 °C.

Experimental section

1. CNW synthesis

CNWs were grown using a CCP-PECVD system. Copper foil with a size of $2 \times 2 \text{ cm}^2$ was used as the growth substrate. Before deposition, the Cu substrates were ultrasonically cleaned for 10 min in each solvent, including deionized water, acetone, and ethanol, and then dried in air. After cleaning, the substrates were placed on the lower grounded electrode equipped with a vacuum-flange heater containing a ceramic-coated NiCr filament (Thermic Edge Ltd., United Kingdom). The electrode assembly was mounted inside a cylindrical vacuum chamber with a water-cooling system, which ensured stable operation and chamber sealing at elevated temperatures. The upper electrode, acting as the powered electrode, was connected to a 13.56 MHz radio-frequency (RF) power supply through an automatic matching unit (CESAR 1310, China) [9]. The synthesis process consisted of several stages. First, CNW nucleation was performed at a plasma power of 30 W for 10 min under an Ar flow of 25 sccm. Then, the treatment was continued in an Ar/H₂ atmosphere with flow rates of 8 and 20 sccm, respectively. The main CNW growth step was conducted at 35 W for 90 min using an Ar/CH₄ gas mixture with flow rates of 15 and 12 sccm, respectively, together with an additional H₂ flow of 5 sccm. After the growth stage, the heater was turned off, and the chamber was allowed to cool naturally under a constant Ar flow of 10 sccm. This cooling step was used to minimize thermal stress, prevent oxidation, and preserve the morphology of the synthesized CNW films.

2. Humidity sensor fabrication

The humidity sensor was fabricated using a lift-off and transfer approach, as illustrated in Figure 1. CNW films synthesized on copper foil were immersed in an aqueous ferric chloride (FeCl₃) solution prepared by dissolving 8.3 g of anhydrous FeCl₃ in 50 mL of deionized water, corresponding to an FeCl₃ concentration of approximately 1.02 M, in order to selectively etch the copper substrate. After complete dissolution of the copper foil, the remaining CNW film was thoroughly rinsed with deionized water to remove residual FeCl₃. The freestanding CNW film was then transferred onto a clean quartz substrate. Finally, silver paste was applied to the edges of the film to ensure reliable electrical contact and to reduce contact resistance during electrical measurements.

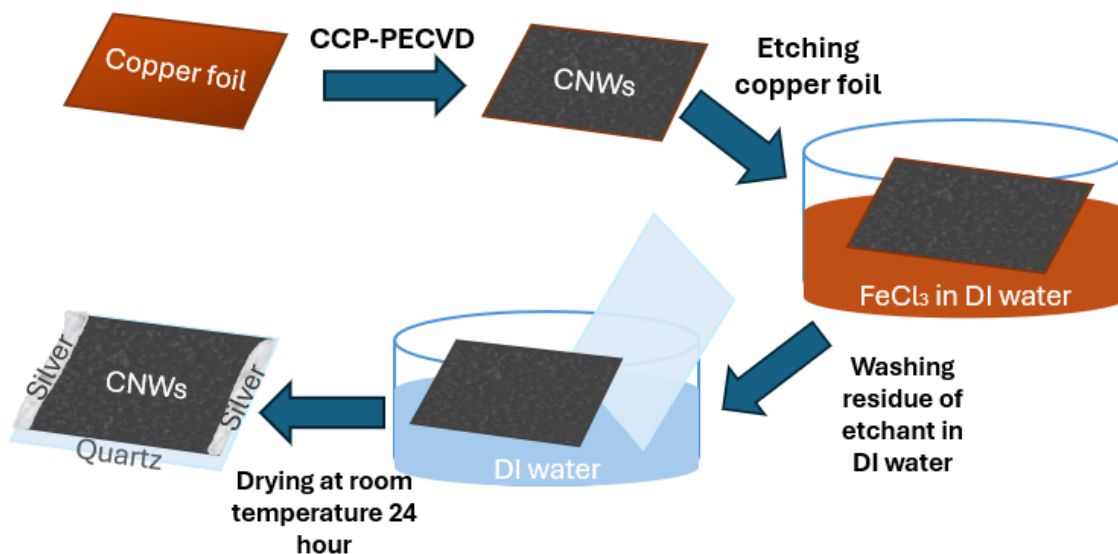


Figure 1. Schematic illustration of the fabrication process of the CNW-based humidity sensor.

3. Humidity-sensing measurements

Humidity measurements were performed in a closed test chamber equipped with temperature and humidity control. The CNW/quartz sensor was connected to the Nextron-based electrical measurement system, and the current signal was recorded at a constant bias of 0.5 V using a Keithley 2401 source meter. The operating temperature was regulated by the MPS controller, while the relative humidity was adjusted using the HCS-2M humidity control system. The sensing performance of the fabricated device was evaluated at RH values ranging from 10% to 100% and at operating temperatures from room temperature (27 °C) to 75 °C. The selected temperature range provides a systematic assessment of the sensor response under near-ambient and elevated-temperature conditions and facilitates evaluation of the role of thermally enhanced desorption in determining the overall sensing performance. To evaluate device-to-device reproducibility, four independently fabricated CNW-based humidity sensors were tested under identical experimental conditions. The reported sensitivity and resistance values represent the mean values obtained from four devices ($n = 4$), and the error bars indicate the corresponding standard deviation.

4. Material characterization

The surface morphology of the transferred CNW films was examined by scanning electron microscopy (SEM, Quanta 200i 3D (FEI Company, USA)). Raman spectroscopy (NT-MDT Solver Spectrum) was used to evaluate the graphitic structure and defect-related features of the CNW films. The Raman spectra were recorded at a laser excitation wavelength of 473 nm.

Results and discussion

Morphological and structural characterization

Figure 2(a) shows SEM images of the CNW films transferred onto quartz substrates. The SEM image shows that the transferred film consists of densely distributed vertical carbon nanosheets. This confirms the preservation of the CNW morphology after the wet-transfer process [10]. Such a morphology provides a large accessible surface area and numerous exposed edge sites, both of which are beneficial for adsorption-based sensing applications.

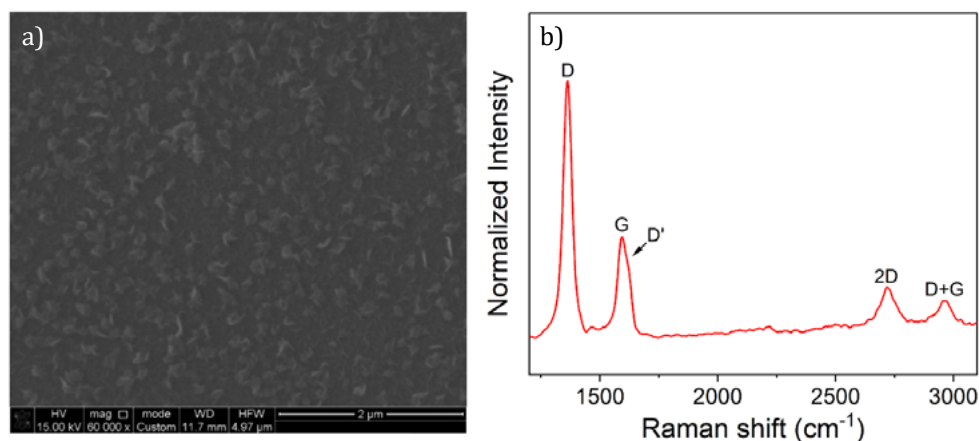


Figure 2. SEM image of the CNW film (a). Raman spectra of the CNW film (b).

The Raman spectrum of the CNW film is presented in Figure 2(b). The Raman spectrum contains the typical carbon-related D, G, D', 2D, and D+G bands. The presence of an intense D band indicates a considerable amount of disorder and edge defects, while the G band confirms the graphitic sp^2 carbon framework [11]. The 2D and D+G features further support the formation of graphene-like carbon sheets in the CNW film [12]. Overall, the Raman results confirm the formation of a graphitic nanocarbon structure with a noticeable level of structural disorder, which may enhance interaction with water molecules during humidity sensing.

The current-voltage (I-V) characteristics of the CNW-based humidity sensor are shown in Figure 3. Within the voltage range under study (± 0.5 V), the current-voltage relationship is linear, indicating the formation of ohmic contacts between the silver conductive paste and the CNW film [13]. Analysis of the I-V curve shows that the on-resistance of the CNW-based humidity sensor is approximately 2.2 k Ω .

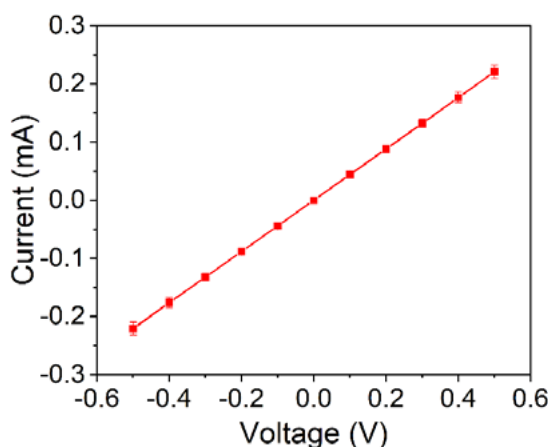


Figure 3. I-V characteristic of the CNWs humidity sensor.

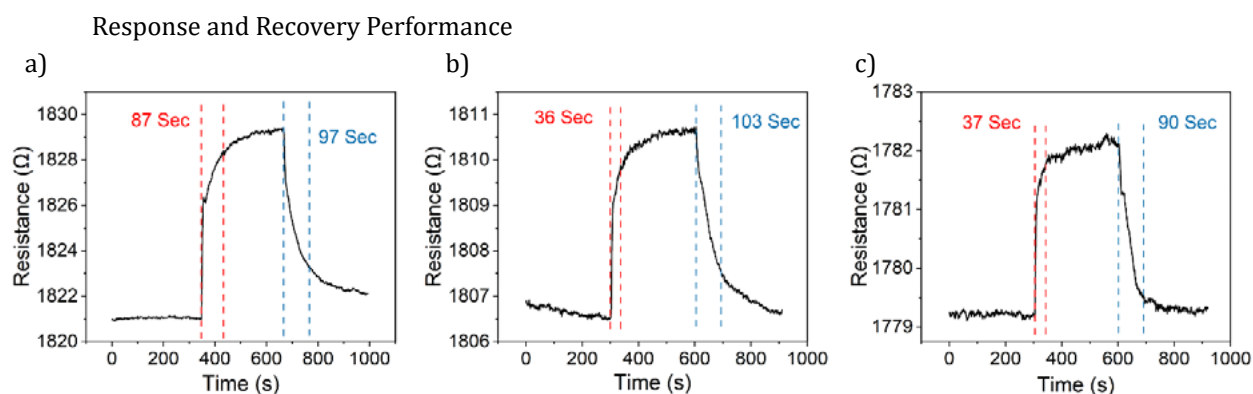


Figure 4. Dynamic response and recovery curves of the CNW-based humidity sensor at 50% RH measured at (a) 27 °C, (b) 50 °C, and (c) 75 °C.

The dynamic characteristics of the sensor were evaluated from its response and recovery behavior under alternating dry and humid air conditions. Figure 4 presents the dynamic response of the CNW sensor at room temperature, 50 °C, and 75 °C at 50% RH. In these experiments, the sensor was initially exposed to dry air for 5 min, then to humid air at 50% RH for 5 min, and finally again to dry air for 5 min.

At room temperature, the response and recovery times were 87 and 97 s, respectively. When the operating temperature was increased to 50 and 75 °C, the response time decreased to 36 and 37 s, respectively, while the recovery time was 103 and 90 s. The shorter response time at elevated temperatures may be associated with faster adsorption/desorption dynamics and faster equilibration of water molecules on the sensor surface. In contrast, the recovery process remained relatively slow, which is typical for humidity sensors and is usually associated with the time required for desorption of adsorbed water molecules [14].

Sensor Responses Repeatability

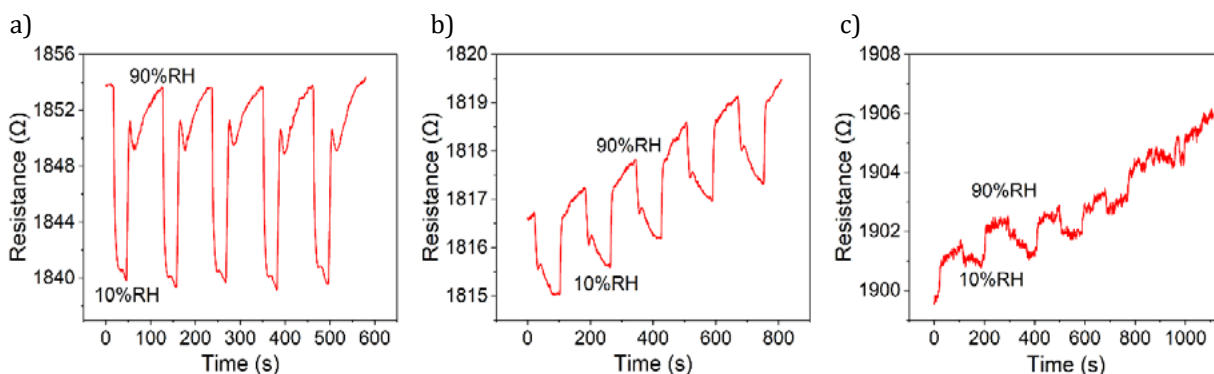


Figure 5. Repeatability of the CNW-based humidity sensor during five cycles between 10% and 90% RH at (a) 27 °C, (b) 50 °C, and (c) 75 °C.

The repeatability of the sensor response is shown in Figure 5. To verify the repeatability of the devices, the resistance responses of the CNW sensors were measured at room temperature (27 °C), as well as at 50 °C and 75 °C, over 5 cycles while exposed to 10% and 90% humidity. At room temperature, 10% humidity was applied for 30 seconds at the start, followed by 90% humidity for 80 seconds. At 50 °C, 10% humidity was applied for 80 seconds at the start, followed by 90% humidity for 80 seconds. At 75 °C, the duration of each humidity level was 100 seconds, which was necessary to achieve signal stabilization. More time was required for stabilization.

The results obtained show that at 27 °C and 50 °C, the resistance response curves exhibit good repeatability and stable cycling behavior over the five measured cycles. In the 75 °C graph, a gradual decrease in resistance level

and a reduction in the severity of the cyclic response are observed. This may be due to the intensification of water desorption processes [15].

Figure 6 shows the relationship between sensitivity and relative humidity (10–100%) at three different temperatures (RT, 50 °C, and 75 °C). As can be seen from the graphs, the sensor's sensitivity increases as relative humidity rises. At room temperature, the sensor sensitivity shows an approximately linear dependence on relative humidity over the investigated range. At 50 °C, the response remains close to linear up to about 90% RH, whereas at 75 °C, noticeable deviation from linearity appears above approximately 60% RH, indicating a stronger contribution of thermally activated desorption at elevated temperature. At elevated temperatures, the enhanced competition between adsorption and desorption becomes more pronounced, which may limit the accumulation of adsorbed water molecules on the CNW surface at high relative humidity and consequently result in a deviation from the linear sensitivity response[16].

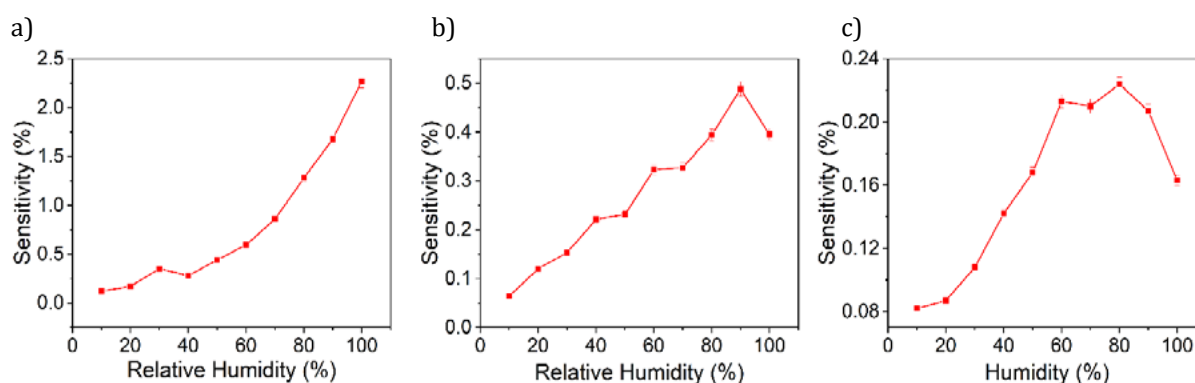


Figure 6. Sensitivity of the CNW-based humidity sensor as a function of relative humidity at (a) 27 °C, (b) 50 °C, and (c) 75 °C.

The sensitivity (S) of the fabricated humidity sensors was calculated as the relative change in sensor resistance when transitioning from dry air (R_0) to humid air (R_{RH}) using the equation (1) [17]:

$$(1)$$

The maximum sensitivity (2.27%) is achieved at room temperature, indicating that the adsorption mechanism plays a dominant role in the moisture detection process [18]. When the temperature is raised to 50°C, the sensitivity decreases to 0.49%, and at 75°C, to 0.22%. This decrease in sensitivity at elevated temperatures may be associated with an intensification of water molecule desorption processes.

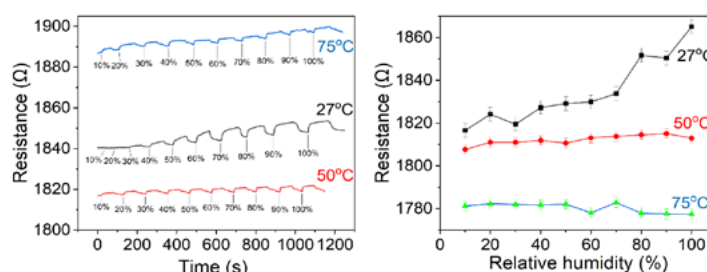


Figure 7. Dynamic resistance response of the CNW-based humidity sensor at relative humidity levels from 10% to 100% RH at different operating temperatures (a). Dependence of sensor resistance on relative humidity at different operating temperatures (b).

Figure 7(a) presents the resistance behavior of the CNW-based sensor under different relative humidity levels and operating temperatures. During the measurements, the sensor was exposed to each relative humidity level for 80 seconds at a fixed temperature. After each humidity exposure step, the test chamber was purged with dry air for 50 seconds to restore the baseline signal. At room temperature, only slight changes in resistance were observed when the relative humidity was increased up to 30%. A more distinguishable response appeared when the relative humidity reached 40%. With further increase in relative humidity, the resistance variation became more pronounced. This trend can be explained by the increased adsorption of water molecules on the CNW surface at higher humidity levels. As more water molecules interact with the sensing layer, the charge transport through the CNW network is affected more strongly, resulting in an improved sensor response.

Figure 7(b) presents the variation of sensor resistance with relative humidity at different operating temperatures. The solid curves correspond to the humidification stage, during which water molecules are adsorbed on the CNW surface. At room temperature and 50 °C, the sensor resistance changes almost linearly as the relative humidity increases. This indicates that, under these conditions, water adsorption on the CNW surface produces a relatively uniform change in charge transport through the sensing layer. However, at 75 °C, the resistance–humidity dependence deviates from linear behavior, especially at higher RH values. This nonlinearity can be attributed to enhanced thermal desorption of water molecules and a shorter residence time of adsorbed species on the CNW surface. As a result, thermally activated desorption increasingly competes with adsorption, limiting the surface coverage of water molecules and producing a weaker and nonlinear resistance response.

Table 1. Comparison of humidity-sensing performance of carbon-based sensors reported in the literature.

Materials	Operating temperature	Measurement range	Sensitivity	Response time (s)	Recovery time (s)	References
CNT@CPM	RT	30–100% RH	56.7–111.1%	10–40	10–40	[19]
PVA/graphene	60°C 30°C	10– 80% RH	66.4% 38.2%	11 22	35 50	[20]
rGO/PU	RT	10-70% RH	9%	3.5	7	[21]
SDC	RT	0-90% RH	50%	0.14	1.7	[22]
GQDs prepared from waste	25°C	40-90% RH	N/A	15	55	[23]
CNWs	RT	10-100% RH	2.27%	87	97	This work

The performance characteristics of the CNW-based humidity sensor were compared with data obtained by other researchers, as shown in Table 1. Compared with other carbon-based humidity sensors reported in the literature, the CNW-based sensor developed in this work demonstrates a measurable humidity response, although its sensitivity and response/recovery characteristics remain lower than those of several optimized carbon-based humidity sensors. It should also be noted that humidity-sensing studies based specifically on carbon nanowall films are still very limited. Therefore, the present work provides additional experimental data and contributes to a better understanding of the potential of CNWs as active materials for humidity sensor applications.

Conclusion

A resistive humidity sensor based on transferred CNW films was fabricated and evaluated under different RH and temperature conditions. The CNW film exhibited a petal-like morphology and Raman features consistent with graphitic nanocarbon containing structural disorder. The fabricated sensor showed humidity-dependent electrical response over the range of 10–100% RH. The highest sensitivity of 2.27% was obtained at 27 °C, whereas the sensitivity decreased to 0.49% and 0.22% at 50 °C and 75 °C, respectively, which is consistent with an increasing contribution of water desorption at elevated temperatures. The response time was 87 s at 27 °C and decreased to

36–37 s at higher temperatures, while the recovery time ranged from 90 to 103 s. Good short-term repeatability was observed at 27 and 50 °C, whereas gradual drift became apparent at 75 °C during the five-cycle test. At 27 °C, the response was close to linear over the studied RH range, while at higher temperatures, increasing nonlinearity was observed at high RH. The obtained results show that CNW films can be used as simple conductive layers for humidity detection, although further optimization is required to improve sensitivity and long-term stability.

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Author Contributions

B.N. Dembergenov - Investigation, Visualization, Writing - original draft.

B.A. Kyrykbay - Investigation, Visualization, Writing - original draft.

R.Ye. Zhumadilov - Investigation, Formal analysis, Methodology, Visualization, Validation, Writing - original draft, Writing - review & editing.

Yerassyl Yerlanuly - Investigation, Formal analysis, Methodology, Visualization, Validation, Writing - original draft, Writing - review & editing.

Maratbek T. Gabdullin - Project administration, Resources, Methodology, Funding acquisition.

Conflict of Interest

The authors declare that they have no conflict of interest related to the publication of this article.

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**ССР-PECVD әдісімен синтезделген көміртекті наноқабырға қабықшаларының
ылғалдылықты сезу қасиеттері**

Аңдатпа. Көміртекті наноқабырғалардан тұратын қабықшалар сыйымдылықты байланысқан плазмалық-химиялық бу фазасынан түндыру әдісімен синтезделіп, резистивті ылғалдылық сенсорларының белсенді қабаты ретінде зерттелді. Сканерлеуші электрондық микроскопия нәтижелері тігінен бағытталған көміртекті наноқабырға қабаттарының түзілгенін көрсетті. Раман спектроскопиясы ақауларға байланысты және графиттік көміртекке тән жолақтарды анықтап, жапырақша тәрізді көміртекті наноқабырғалар торының қалыптасқанын растады. Ылғалдылықты сезу қасиеттері 10–100% салыстырмалы ылғалдылық аралығында, 27, 50 және 75 °C температураларда зерттелді. Дайындалған сенсорлар қысқа циклдік сынақтар кезінде қайталанатын электрлік жауап көрсетті және температураға айқын тәуелділік байқалды. 27 °C температурада сенсор ең жоғары сезімталдық көрсетті, 100% салыстырмалы ылғалдылықта ол 2,27%-ға жетті. Жауап беру және қалпына келу уақыттары сәйкесінше 87 және 97 секундты құрады. Жұмыс температурасының жоғарылауы сезімталдықтың 50 °C-та 0,49%-ға және 75 °C-та 0,22%-ға дейін төмендеуіне әкелді, бұл КНҚ бетінен су молекулаларының десорбциясының күшеюімен түсіндірілуі мүмкін. 27 және 50 °C температураларда ылғалдылықтың бес дәйекті циклі кезінде жақсы қысқа мерзімді қайталанымдылық байқалды, ал 75 °C-та сигналдың біртіндеп дрейфі байқалды. 27 °C температурада сенсор жауабы зерттелген салыстырмалы ылғалдылық диапазонында шамамен сызықтық сипатта болды, ал жоғары температураларда ылғалдылықтың жоғары мәндері аймағында бейсызықтықтың күшеюі байқалды. Алынған нәтижелер тасымалданған көміртекті наноқабырға қабықшаларын ылғалдылықты бақылауға арналған қарапайым әрі арзан платформа ретінде қолдануға болатынын көрсетеді.

Түйін сөздер: көміртекті наноқабырғалар, ылғалдылық сенсорлары, ССР-PECVD, салыстырмалы ылғалдылық, резистивті сенсорика.

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Сенсорные свойства плёнок углеродных наностенок, синтезированных методом ССР-PECVD, при детектировании влажности

Аннотация. Плёнки углеродных наностенок (УНС) были синтезированы методом плазмохимического осаждения из газовой фазы в ёмкостно-связанной плазме и исследованы в качестве активных слоёв для резистивного детектирования влажности. Наблюдения с помощью сканирующей электронной микроскопии подтвердили формирование вертикально ориентированных листов УНС, тогда как Раман-анализ показал характерные полосы, связанные с дефектами и графитизированным углеродом, что подтверждает образование лепесткоподобной сети углеродных наностенок. Сенсорные свойства по отношению к влажности изучались в диапазоне относительной влажности 10–100% при температурах 27, 50 и 75 °C. Изготовленные сенсоры продемонстрировали воспроизводимый электрический отклик при кратковременных циклических испытаниях, а также выраженную температурную зависимость. При 27 °C сенсор показал наивысшую чувствительность, достигавшую 2,27% при 100% относительной влажности, со временем отклика и восстановления 87 и 97 с соответственно. Повышение рабочей температуры приводило к снижению чувствительности до 0,49% при 50 °C и 0,22% при 75 °C, что можно объяснить усиленной десорбцией молекул воды с поверхности УНС. Хорошая кратковременная воспроизводимость наблюдалась при 27 и 50 °C в течение пяти последовательных циклов изменения влажности, тогда как при 75 °C отмечался постепенный дрейф сигнала. При 27 °C отклик сенсора был приблизительно линейным во всём исследованном диапазоне относительной влажности, тогда

как при повышенных температурах наблюдалось усиление нелинейности в области высоких значений влажности. Полученные результаты показывают, что перенесённые плёнки углеродных наностен могут рассматриваться как простые и недорогие платформы для мониторинга влажности.

Ключевые слова: углеродные наностены, датчики влажности, CCP-PECVD, относительная влажность, резистивное детектирование.

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