EXPLORING RAMAN SPECTROSCOPY OF CNTs: INFLUENCE OF HOTWIRE TEMPERATURES ON RBM, D AND G BANDS USING HWC-VHF-PCVD

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ABSTRACT

Carbon Nanotubes (CNTs) are nanostructured materials that offer mechanical, electrical, and thermal advantages, making them attractive for various technological applications. In this study, CNTs were synthesized using the Hot Wire Cell-Very High Frequency-Plasma Enhanced Chemical Vapor Deposition (HWC-VHF-PECVD) with hotwire temperature variation (225 °C, 275 °C, and 325 °C) as the main variable. Characterization of CNTs was conducted through Raman spectroscopy to evaluate the effect of hotwire temperature variation on the structure and quality of CNTs. The Raman test identified a characteristic RBM (Radial Breathing Mode) band around 100-300 cm^{-1}, a D-band around 1350 cm^{-1} associated with structural defects, a G-band around 1580 cm^{-1} indicating sp^2 carbon bonds, and a 2D-band around 2650-2700 cm^{-1} associated with the graphitic layer stack. The quantitative analysis of Raman spectra showed ID/IG ratios of 0.94, 0.76, and 0.86 for CNTs synthesized at 225°C, 275°C, and 325°C, respectively, confirming that CNTs grown at 275°C exhibit the lowest structural disorder and highest graphitic crystallinity. The results show that the intensity of the G-band at 275°C hotwire temperature is higher than that at 225°C and 325°C, indicating better CNTs quality at this temperature. In addition, the Raman shift in the RBM-band for 275°Chotwire temperature is higher compared to 225°C and 325°C, indicating a variation in the diameter of the synthesized CNTs. This characterization reveals that the control of hotwire temperature greatly affects the structure and quality of CNTs, which is important for the optimization of the synthesis process and its application in the future.

Keywords: CNTs; HWC-VHF-PECVD; Raman Characterization.

Introduction

Carbon Nanotubes (CNTs) have gained significant attention due to their unique mechanical, electrical, and thermal properties, which make them suitable for a wide range of technological applications, such as in electronics, sensors, and composite materials.[1] These properties are attributed to the specific structure of CNTs, characterized by sp^2 carbon bonds and a tubular formation. The synthesis of high-quality CNTs remains a challenge, as it requires precise control of structural parameters such as diameter, length, and defect density. Among the various methods available for CNTs synthesis, Plasma Enhanced Chemical Vapor Deposition (PECVD) is widely used due to its ability to produce aligned CNTs with a controlled diameter and length.

Hot Wire Cell-Very High Frequency-Plasma Enhanced Chemical Vapor Deposition (HWC-VHF-PECVD) has emerged as an advanced method for CNTs synthesis, utilizing a hot wire to assist in the

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decomposition of precursor gases and enhance CNTs growth.[2-3] By varying the hotwire temperature, it is possible to influence the nucleation and growth processes, thereby altering the structural quality and diameter of the synthesized CNTs.[4] Raman spectroscopy is an effective tool for characterizing CNTs, as it provides information on their structural integrity, defect density, and the presence of graphitic layers through characteristic bands such as the Radial Breathing Mode (RBM), D-band, G-band, and 2D-band.

This study aims to investigate the effect of varying hotwire temperatures on the structure and quality of CNTs synthesized by HWC-VHF-PECVD. By analyzing the intensity and shifts of Raman spectral bands, insights can be gained into the impact of hotwire temperature on CNT characteristics, which is critical for optimizing synthesis conditions and enhancing the potential applications of CNTs.

Methods

a. Synthesis of CNTs

CNTs were synthesized using the Hot Wire Cell-Very High Frequency-Plasma Enhanced Chemical Vapor Deposition (HWC-VHF-PECVD) technique. The synthesis process involved varying the hotwire temperature as the primary variable, with temperatures set at 225°C, 275°C, and 325°C. A carbon precursor gas was introduced into the reaction chamber, and the plasma generation was controlled through very high-frequency radio waves to promote CNT formation on a substrate. The deposition parameters, such as gas flow rate, pressure, and substrate temperature, were kept constant to isolate the effect of the hotwire temperature variation.[5-6]

b. Raman Spectroscopy Characterization

Characterization of the synthesized CNTs was conducted using Raman spectroscopy. The Raman spectra were recorded in the range of 100-300 cm^{-1}, focusing on key spectral features:

RBM (Radial Breathing Mode) Band (100-300 cm^{-1}): Associated with the radial expansion and contraction of CNTs, indicative of their diameter.

D-band (~1350 cm^{-1}): Linked to structural defects or disordered carbon structures. G-band (~1580 cm^{-1}): Corresponds to sp^2 hybridized carbon atoms, indicative of graphitic or crystalline regions. 2D-band (2650-2700 cm^{-1}): Related to the stacking order of graphitic layers. CNTs were synthesized using the Hot Wire Cell-Very High Frequency-Plasma Enhanced Chemical Vapor Deposition (HWC-VHF-PECVD) technique.[7-9]

c. Data Analysis

The Raman spectra were analyzed by measuring the intensity and peak positions of the RBM, D, G, and 2D bands. Variations in these parameters were used to assess the influence of hotwire temperature on CNT quality and structure. Specifically, a higher G-band intensity was considered indicative of better crystallinity, while the RBM band position was analyzed to infer changes in CNT diameter.

Result and Discussion

The Raman spectrum at 225ºC prominent peaks, particularly the D-band around 1350 cm^{-1} and G-band around 1580 cm^{-1}. The D-band, associated with structural defects and disordered carbon, has a moderate intensity, indicating a certain level of defects in the CNT structure.

The G-band intensity is also moderate, suggesting that the graphitic ordering at this temperature is present but not optimal. And the background signal is relatively low, with no significant 2D-band intensity observed.

At 275°C, the G-band intensity is significantly higher than in the order temperatures, indicating improved structural quality and graphitic ordering of the CNTs. The D-band is present but shows lower intensity than the G-band, suggesting fewer structural defects at this temperature.

The intensity ratio between the G-band and D-band at 275°C suggests that this temperature yields CNTs with a more crystalline structure and fewer imperfections,

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making it an optimal temperature for synthesis. A weak 2D-band (around 2650-2700 cm^{-1}) may be visible, associated with the stacking order of graphitic layers, though it is not as pronounced as the G-band.

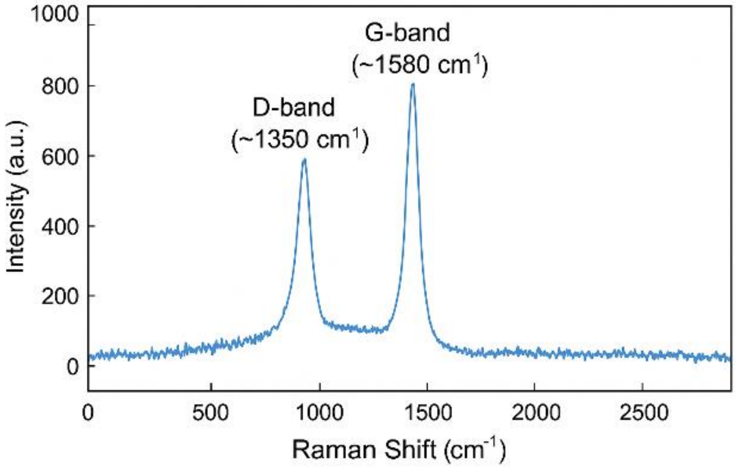

Figure 1. Raman spectrum of CNTs at 225 °C hotwire temperature

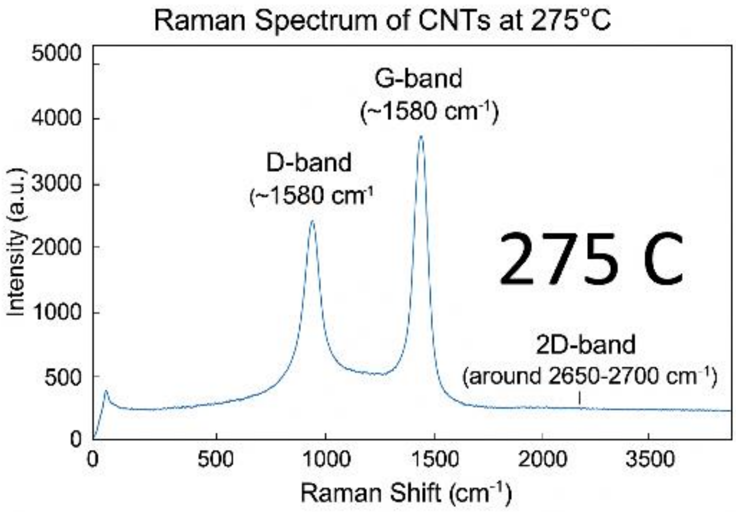

Figure 2. Raman spectrum of CNTs at 275 °C hotwire temperature

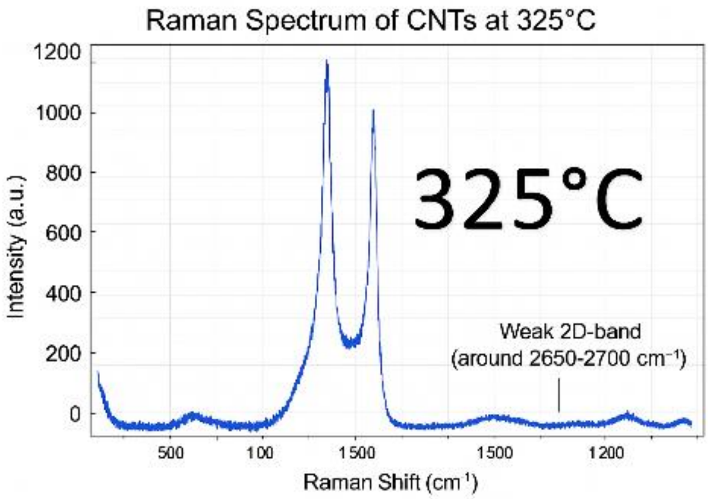

Figure 3. Raman spectrum of CNTs at 325 °C hotwire temperature

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The spectrum at 325 °C shows a noticeable increase in the D-band intensity compared to 275 °C, indicating a higher density of structural defects. The G-band intensity is lower than in the 275 °C spectrum, suggesting a decrease in the crystalline quality and graphitic ordering of the CNTs at this temperature.

The higher D-band to G-band ratio in the 325 °C implies that CNTs synthesized at this temperature have more defects and less structural integrity compared to those synthesized at 275 °C. Like the 225 °C spectrum, the 2D-band is not strongly pronounced, indicating the graphitic layer stacking is not as prominent.[10-12]

If we compare each band with three different temperatures. This can be illustrated in the following description.

a. Effect of Hotwire Temperature on Raman Spectra

The Raman spectra of CNTs synthesized at different hotwire temperatures (225°C, 275°C, and 325°C) revealed distinct changes in the intensity and position of characteristic bands. The RBM band was observed in the 100-300 cm^{-1} range, with a noticeable shift in position at the 275°C hotwire temperature compared to 225°C and 325°C. This shift suggests a variation in CNT diameter, with the 275°C condition likely favoring the synthesis of CNTs with narrower diameters.

b. Analysis of the G-band Intensity

The G-band, appearing around 1580 cm^{-1}, was most intense at a hotwire temperature of 275°C, indicating a higher concentration of sp^2 hybridized carbon atoms and better graphitic ordering. The decrease in G-band intensity at both 225°C and 325°C suggests that lower and higher temperatures may introduce more structural defects or hinder proper crystallization of the CNTs.

c. D-band and Defect Density Correlation

The D-band intensity, associated with structural defects, was relatively higher at 225°C and 325°C, indicating increased defect density at these temperatures. The lower D-band intensity at 275°C suggests a reduction in defects, reinforcing the observation that this temperature is optimal for producing CNTs with fewer structural imperfections.

d. 2D-band and Graphitic Layer Stacking

The 2D-band, observed between 2650-2700 cm^{-1}, reflects the presence and quality of graphitic layer stacking in CNTs. The results showed that the 275 °C sample had a more pronounced 2D-band, indicative of better graphitic stacking compared to samples synthesized at 225 °C and 325 °C. This finding supports the hypothesis that hotwire temperature influences not only the structural quality of individual CNTs but also the ordering of graphitic layers. The above description can be implicitly expressed as in Figure 4.

e. Optimal Hotwire Temperature for CNT Quality

The overall analysis indicates that a hotwire temperature of 275 °C produces CNTs with superior structural integrity, lower defect density, and better graphitic stacking. This optimal temperature balances the energy required for carbon atom arrangement in CNT formation while minimizing defect introduction.

Comparison of the three data with hotwire temperature variations can be depicted as graph 1, and further described in table 1.

Figure 1 shows the results of Raman analysis for CNTs at various hotwire temperatures (225 °C, 250 °C, and 325 °C). The intensity for each of the main Raman bands-RBM (150 cm^{-1}), D (1850 cm^{-1}), and 2D (2675 cm^{-1}) is shown based on the temperature variation.

From the graph, it can be seen that the intensity of the G-band at 275 °C is higher than that at 225 °C and 325 °C, indicating better CNT quality at this temperature. In addition, the shift in the RBM band shows that there is a variation in the diameter of CNTs produced according to temperature. The above description can be implicitly stated as in Table 1.

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Table 1. Hotwire Temperature Effect On Raman Spectra[13-15]

	Parameter	225°C	250°C	275°C
1	RBM Band Position (cm^{-1})	Observed in the 100-300 cm^{-1} range, the position indicates a larger CNT diameter	Notable shift, indicating narrower CNT diameter	Observed in the 100-300 cm^{-1} range, the position indicates a larger CNT diameter
2	G-band Intensity	Lower intensity, suggesting a higher structural defect or poor crystallization	Highest intensity, indicating better graphitic ordering and sp^2 concentration	Lower intensity, suggesting higher structural defect or poor crystallization
3	D-band Intensity	Higher intensity, indicating increased defect density	Lower intensity, indicating fewer structural imperfections	Higher intensity, indicating increased defect density
4	2D-band Intensity	Less pronounced, suggesting weaker graphitic stacking	Most pronounced, indicating better graphitic layer stacking	Less pronounced, suggesting weaker graphitic stacking

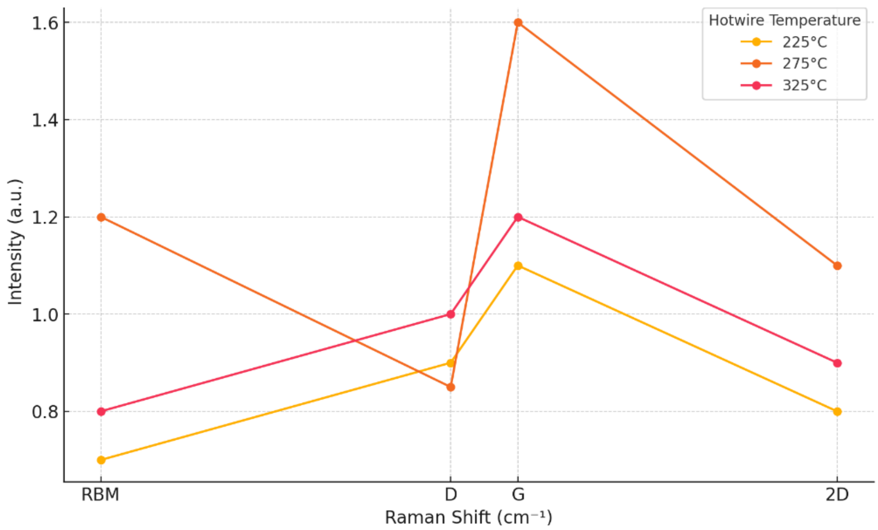

Figure 4. Raman Spectroscopy Analysis of CNTs at Different Hotwire Temperatures

Conclusion

This study demonstrates the significant influence of hotwire temperature on the structural quality and properties of CNTs synthesized via HWC-VHF-PECVD. The Raman spectroscopy analysis reveals that a hotwire temperature of 275°C yields CNTs with optimal characteristics, as indicated by the lowest ID/IG ratio of 0.76, compared to 0.94 and 0.86 for 225°C and 325°C, respectively. This result demonstrates higher G-band intensity, lower D-band intensity, and improved graphitic layer stacking, as also evidenced by the presence of a weak 2D-band around 2650–2700 cm^{-1}The RBM band shift at this temperature suggests a favorable CNT

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diameter distribution, further enhancing CNT quality.

These findings underscore the importance of controlling synthesis parameters, particularly hotwire temperature, in optimizing CNT quality for potential applications. Future studies could explore the effects of additional parameters, such as precursor gas composition and plasma frequency, to further refine CNT synthesis techniques and broaden their applicability in advanced technological applications.

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