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Effect of the ratio of catalyst to carbon source on the growth of vertically aligned carbon nanotubes on nanostructured porous silicon templates

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Abstract

Background: The effect of the ratio of catalyst to carbon source on the growth of vertically aligned carbon nanotubes (VACNTs) has been studied.

Results: Dense VACNTs were successfully synthesised on optimised nanostructured porous silicon templates using modified floated carbon source-catalyst in a two-stage hot filament thermal chemical vapour deposition system with different amounts of ferrocene as the catalyst at 800°C. The surface morphologies of the VACNTs were analysed using field emission scanning electron microscopy, and the crystallinity of the nanotubes was observed using micro-Raman spectroscopy.

Conclusions: These data revealed that the amount of catalyst used significantly affected the diameter, crystallinity and growth rate of the synthesised nanotubes. The average diameter of the nanotubes ranged from ≈ 9 to 30 nm with lengths of $\approx 110 \mu\text{m}$ when 0.5 g ferrocene was used.

Keywords: Porous silicon, Carbon nanotubes, Catalyst ratio, Botanical hydrocarbon, Diameter.

Background

Carbon nanotubes (CNTs) are tube-shaped structures made of carbon atoms and can have diameters on the nanometre and micrometre scales [1]. CNTs have attracted much attention since their discovery 20 years ago [2] due to their properties. They can be used as components in light-emitting diodes [3], electron field emitters for optical displays and electrode materials for fuel cells and lithium ion batteries due to their extraordinary properties [4]. CNTs have superior mechanical, chemical, electronic and optoelectronic properties [3].

Camphor ($\text{C}_{10}\text{H}_{16}\text{O}$) is a valued material in biotechnology and nanotechnology research, and it was first

reported that CNTs could be synthesised using camphor in 2001. This environmentally friendly carbon source for the synthesis of CNTs can be used to produce multiwall carbon nanotubes, single-wall carbon nanotubes (SWCNT) and vertically aligned nanotubes. In this context, the striking feature of camphor as a precursor is that a small amount of catalyst is needed compared to what is needed in other similar kinds of CNT syntheses that employ hydrocarbons [5].

Carbon nanotubes are commonly synthesised using one of three techniques: arc discharge, laser ablation and chemical vapour deposition (CVD) [6]. CVD is the most popular and extensively used method because it is easy to scale-up, can be performed at a lower temperature [6], has a lower set-up cost and higher production yield than other approaches [1]. Most of the CNT literature claims that the additional template synthesis used in the CVD technique is a promising way to synthesise well-aligned CNT structures with control over diameter, size

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uniformity and density [6-9]. The pore diameter of the template can be controlled which consequently enables tube diameter and orientation to be dictated [10]. For example, anodic aluminium oxide (AAO) has been used as a template to grow aligned carbon nanotubes (ACNTs). ACNTs are defined as quasioone-dimensional carbon cylinders oriented in a parallel fashion perpendicular to a substrate [11]. Thess et al. [12] first reported the synthesis of ACNTs in bundle form, where 70% of the volume produced consisted of nanotubes. Ali et al. [13] reported that the pore sizes of the substrates used played a role in controlling the growth directions and diameters of the carbon nanotubes. The literature also reports the use of porous silicon templates with pore sizes ranging from 10 to 100 nm [14].

By using an AAO template, ACNTs were successfully grown, as reported by Sui [15]; however, fabrication of AAO is a time-intensive and expensive process that takes approximately 10 h to complete [10]. In a different approach to synthesising well-aligned CNTs, Cao et al. [16] reported the use of a gold-patterned quartz substrate. According to Li et al. [6], porous silicon templates have been loaded with catalytic particles by sputtering iron (Fe) as a route to the synthesis of ACNTs. In addition, Kordás et al. [17] reported that carbon nanotubes have been synthesised on oxidised porous silicon. Catalysts play an important role in the synthesis of CNTs; common catalysts that are widely used for the preparation of CNTs include nickel (Ni), iron (Fe), cobalt (Co) and combinations thereof [18].

This paper will report the use of a porous silicon template and an Fe catalyst to produce vertically aligned carbon nanotubes (VACNTs) using a novel method that involves modifying the catalyst-nanotemplate interaction in the CVD technique. In this study, we focused on understanding how the ratio of the catalyst to carbon source affected the nanotube growth rate and size, and the carbon and ferrocene distributions.

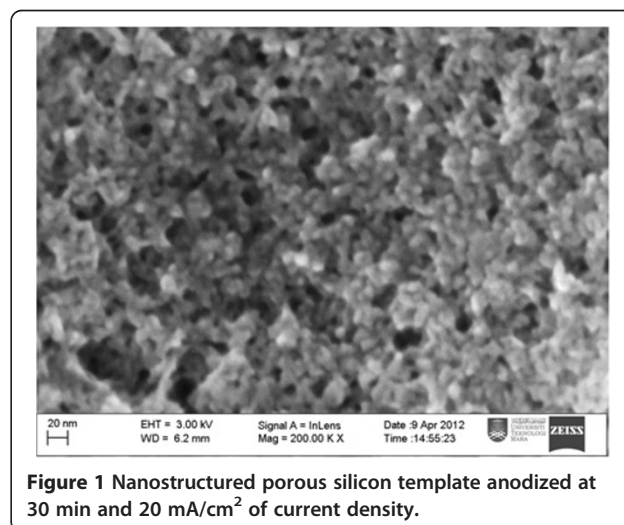
Methods

NPSiT were prepared as outlined by Asli et al. [19]. Briefly, boron-doped, p-type (100) Si wafers that were 525 ± 25 - μm thick were subjected to a photo-electrochemical anodisation process with 30-min etching time and 20 mA/cm^2 of current density. These Si wafers were placed in Teflon cells with an electrolyte consisting of a 1:1 ratio of ethanol and HF (48%) and illuminated with a halogen lamp. Then, the resulting templates were annealed in ambient air at 300°C overnight. A quartz tube with a length of 80 cm and an inner diameter of 3 cm served as a thermal CVD reactor and was kept horizontal in a two-stage zone furnace. The samples were prepared by varying the weight of ferrocene (obtained from Sigma-Aldrich, MO, USA) from 0.3 to

0.8 g, while holding the weight of camphor oil used at 5 g. The catalyst and carbon source were then placed in separate alumina boats and positioned side-by-side in furnace zone 1. NPSiT was placed in the centre of furnace zone 2. Zone 2 was heated to 800°C and was left to stabilise for 10 min after it was purged with nitrogen gas (0.05 to 1.00 L min^{-1}). Then, zone 1 was heated to 180°C for the precursor vapourisation process. Once it reached the required temperature, the synthesis process continued for 60 min. The nitrogen gas was continuously flowed before, during and after synthesis to prepare the ambient nitrogen environment. Zone 1 was turned off after the synthesis process was complete, and zone 2 was left for 30 min after the annealing process. The samples were taken out for characterisation after the furnaces cooled down to room temperature.

Results and discussion

Figure 1 is a typical field emission scanning electron microscope (FESEM) top-view image of a nanostructured porous silicon template (NPSiT) fabricated by the photo-electrochemical anodisation technique that was described in a previous study [19]. In the top-view image, the dark areas are pores that have an average diameter of approximately 20 nm. Figure 2a,b,c,d,e,f shows the morphology of CNTs grown on the optimised NPSiT using different amounts of catalyst. FESEM images at low ($\times 100$) and high magnifications ($\times 100,000$, insets) are shown for CNTs prepared with varying amounts of ferrocene (from 0.3 to 0.8 g) and a constant amount of camphor oil (5 g). The low magnification image in Figure 2a shows that a low yield of randomly entangled CNTs was obtained when 0.3-g ferrocene was used. The high magnification FESEM image in the inset of Figure 2a shows randomly entangled nanotubes with an abundance of amorphous carbon (a-C) content.



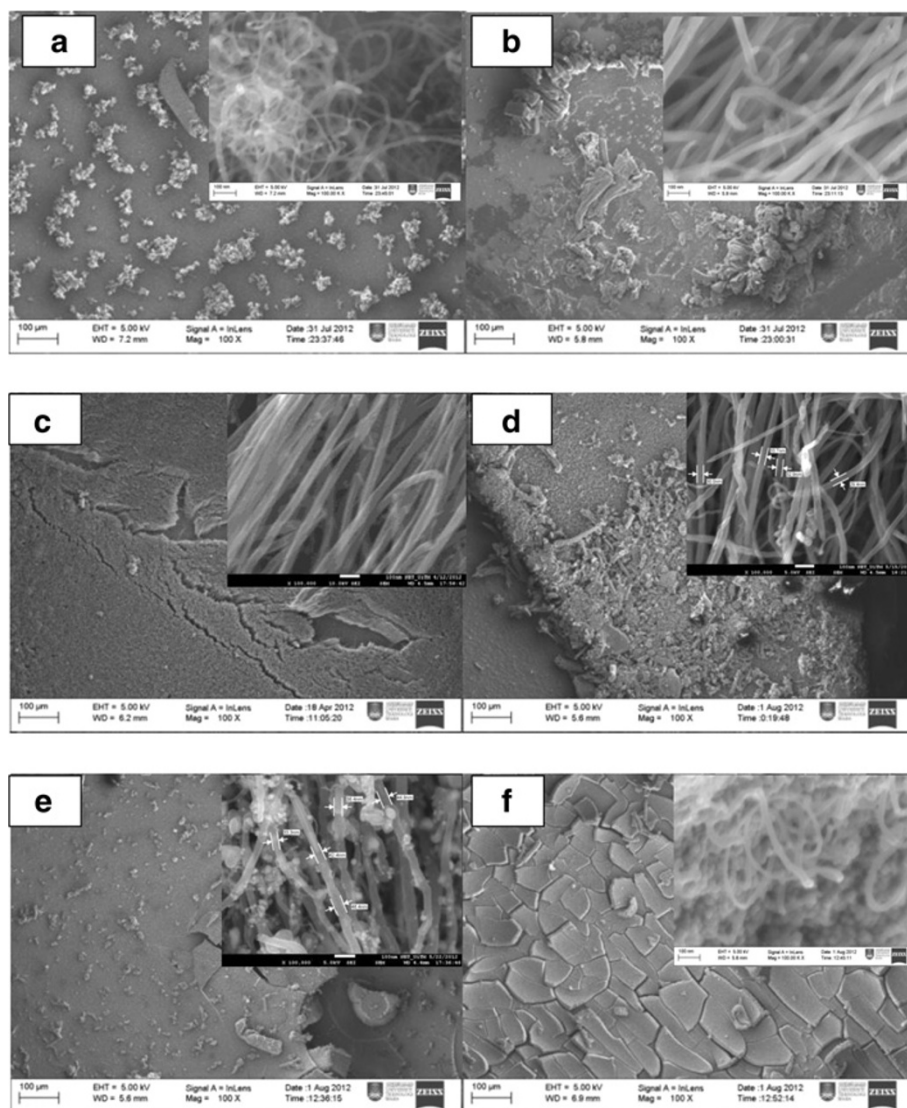


Figure 2 FESEM images of CNT at different Fe ratio for 5 g camphor oil. (a) 0.3 g, (b) 0.4 g, (c) 0.5 g, (d) 0.6 g, (e) 0.7 g, (f) 0.8 g.

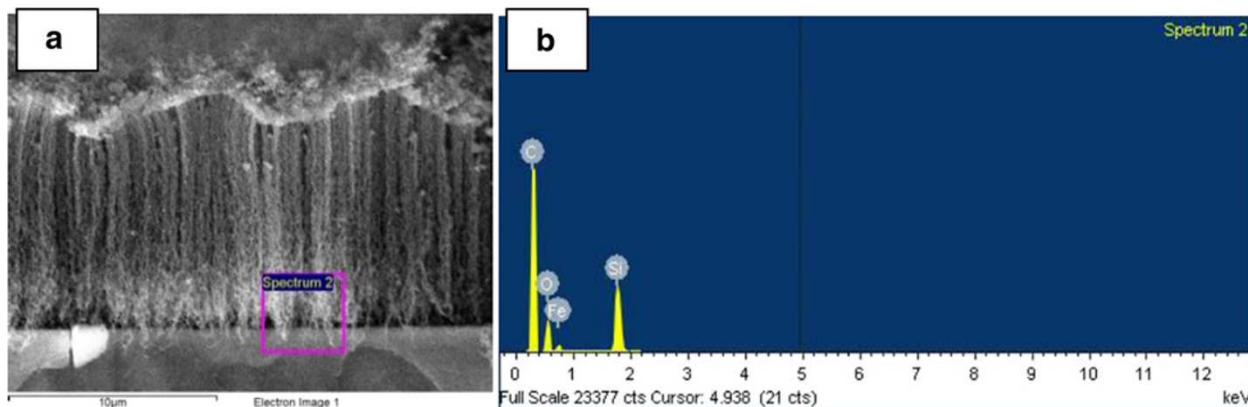


Figure 3 Carbon signals both inside and outside the pores. (a) FESEM cross-section image and (b) EDX analysis.

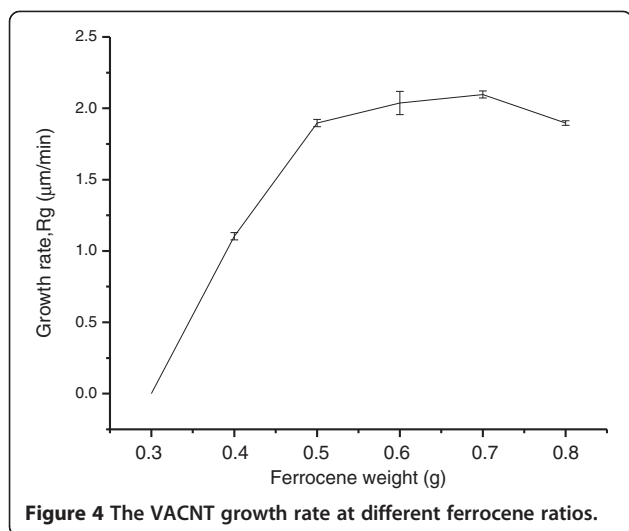


Figure 4 The VACNT growth rate at different ferrocene ratios.

When a low amount of ferrocene was used, the nanotubes form in low yield, bend and grow in random directions. The nanotubes grow without support from neighbouring nanotubes, resulting in the growth of randomly oriented carbon nanotubes. When the amount of ferrocene used is less than 0.4 g, aligned CNTs are not produced. By increasing the ferrocene amount, the CNT yield increases substantially, as seen in Figure 2b,c,d,e,f. Nevertheless, carbonaceous crusts were present and covered the top part of the vertically aligned nanotubes, as seen in Figure 2d,e,f. The formation of this crust might be due to the higher amount of ferrocene present relative to the amount of carbon source and related to the non-uniformity in the evaporation processes of ferrocene and camphor oil during synthesis [8]. The CNTs produced using 0.5 g ferrocene and 5 g camphor oil are shown in Figure 2c. In this image, the nanotubes are densely packed uniform vertical columns; CNTs, as long as 110 to 113 µm are evident. In this case, the growing carbon nanotubes are densely packed on the NPSiT in vertical columns and support each other to grow in the same direction (perpendicular to the template surface). Therefore, a low amount of catalyst resulted in less dense CNTs with a-C content and poor alignment, as seen in Figure 2a,b. Disproportionate catalyst addition

results in the agglomeration of the catalyst, leaving unwanted catalyst impurities in the final product (Figure 2d,e,f).

The diameters of the CNTs were also significantly affected by the amount of ferrocene used. In addition, the size of catalyst particles determined the diameter of the tubes. The carbon atoms diffused into the iron nanoparticles during the CVD process and formed FeC. Fe catalyst particles with various dimensions were found encapsulated within the tubes. Therefore, when catalyst particles become supersaturated with carbon atoms, the diameters of the tubes become bigger, and precipitate carbon atoms were found attached to the nanotube bundles [20].

Nanotube sizes in the range of 14.0 to 25.0 nm were observed when a low amount of ferrocene (0.3 g) was used. By increasing the ferrocene amount to 0.4 g, the tube size increased greatly to between 30.1 to 39.9 nm. Interestingly, the smallest diameter (9.9 nm) was observed when 0.5 g ferrocene was used. However, when this amount of ferrocene was used, the tube diameters ranged from 9.9 to 30.5 nm. The inconsistency in the size of the tubes is due to their growth from inside the pores and on the top surface of the NPSiTs. It was clearly observed that CNTs grew from both sites, as verified by examining the cross-sectional FESEM images and energy dispersive X-ray spectrometer (EDX) analysis showing carbon signals both inside and outside the pores (Figure 3). In contrast, the nanotube diameters continue to increase when the amount of ferrocene used was greater than 0.5 g. In summary, ferrocene amounts greater or less than 0.5 g are not capable of producing nanotubes with small diameters when 5 g camphor oil is used.

Using the data collected from the FESEM images, the growth rate of the nanotubes can be calculated. The relationship between the growth rate of the VACNTs and the amount of ferrocene used (ranging from 0.3 to 0.8 g for 5 g camphor oil) is shown in Figure 4. The growth rate can be calculated as $\alpha = \beta (\mu\text{m}) / \gamma (\text{min})$ [15], where β is the distribution length of the tubes, and γ is the deposition time. The lengths, diameters and growth rates of CNTs synthesised using at 0.3 to 0.8 g of ferrocene and 5 g of camphor oil are shown in Table 1. As

Table 1 Lengths, diameters and growth rates of CNTs

Camphor oil (g):ferrocene (g)	Length (µm)	Diameter (nm)	Growth rate (µm/min)
5.0:0.3	No side view	14.0 to 25.0	-
5.0:0.4	60 to 72	30.1 to 38.0	1.1
5.0:0.5	110 to 113	9.9 to 30.5	1.9
5.0:0.6	115 to 120	20.7 to 33.0	2.0
5.0:0.7	125 to 129	22.3 to 37.1	2.1
5.0:0.8	100 to 125	35.0 to 39.9	1.9

The CNTs were synthesised using 0.3 to 0.8 g ferrocene catalyst and 5.0 g camphor oil.

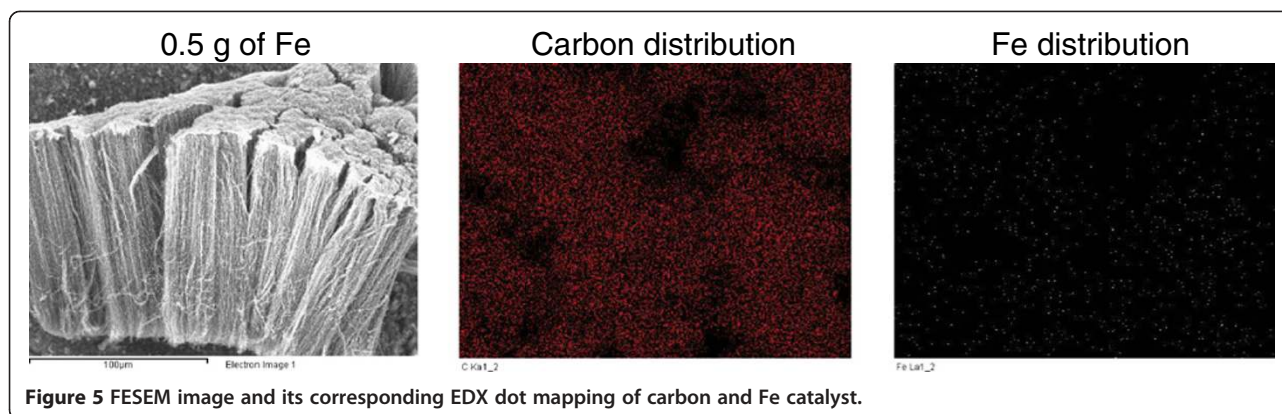


Figure 5 FESEM image and its corresponding EDX dot mapping of carbon and Fe catalyst.

the amount of ferrocene used increased from 0.3 to 0.5 g, the growth rate of the VACNTs increased rapidly from 0 to 1.9 µm/min. The growth rates continued to increase gradually, and a maximum rate of 2.1 µm/min was found for a sample catalysed with 0.7 g ferrocene. Then, the growth rate decreased to 1.9 µm/min when 0.8 g of ferrocene was used. Figure 2a shows entangled tubes with short length and poor alignment which resulted from an insufficient number of Fe particles to catalyse the growth of VACNTs. However, the reduced growth rates for samples synthesised with 0.8 g ferrocene were due to low catalytic activity that resulted from an increment of Fe particle size. Figure 2e,f shows that the CNTs produced contain impurities that stick to the nanotubes and result in nanotube with larger diameters. This phenomenon happens at high ferrocene concentrations when ferrocene decomposes to an abundance of Fe

particles, which tend to agglomerate into a larger cluster. The catalytic effect is reduced when the catalyst size increases, resulting in larger nanotube diameters as discussed before. According to Bai et al. [21], the catalyst acts as a nucleation site and dictates the tube diameter.

Figure 5 shows the FESEM images and corresponding EDX maps for CNTs synthesised using 0.5 g ferrocene and 5 g camphor oil. EDX mapping was utilised to pinpoint catalyst location and distribution, and relate it to carbon production. The optimised parameters were found to be 0.5 g ferrocene and 5 g carbon precursor because this synthesis showed tolerable ferrocene catalyst in the final product. This conclusion is supported by the top-view image shown in Figure 2c and the Raman spectra that will be discussed later.

Figure 6 shows the Raman spectra of VACNTs synthesised using different amounts of ferrocene. The

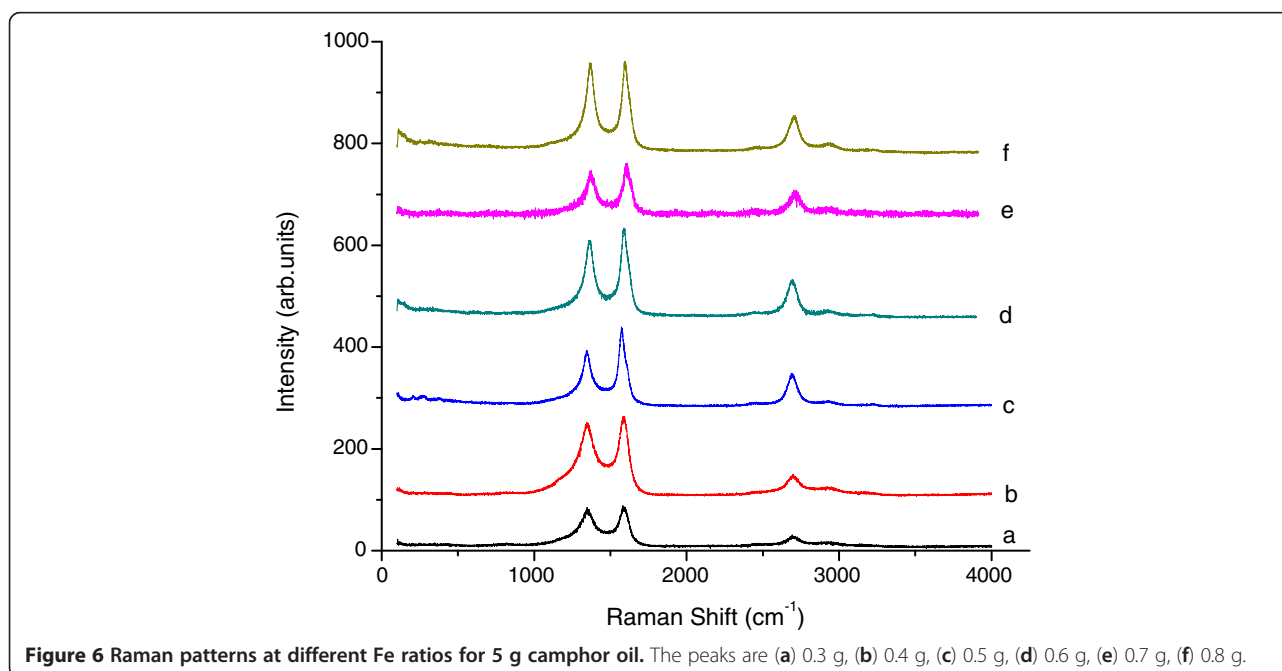


Figure 6 Raman patterns at different Fe ratios for 5 g camphor oil. The peaks are (a) 0.3 g, (b) 0.4 g, (c) 0.5 g, (d) 0.6 g, (e) 0.7 g, (f) 0.8 g.

Table 2 Raman peak positions and D-to-G peak intensity ratios

Sample (g) (amount of ferrocene used for 5 g camphor oil)	G-peak (cm^{-1})	D-peak (cm^{-1})	I_D/I_G ratio
0.3	1,583.21	1,346.17	0.97
0.4	1,585.17	1,346.92	0.93
0.5	1,571.57	1,346.95	0.72
0.6	1,588.15	1,361.49	0.86
0.7	1,607.02	1,363.49	0.88
0.8	1,596.69	1,366.41	0.98

Raman peak positions and I_D/I_G ratios are summarised in Table 2. Significant radial breathing mode (RBM) peaks were detected in the samples synthesised using 0.5 g ferrocene and 5 g camphor oil, as shown in Figure 7. Peaks were observed at 205.8, 272.9 and 382.9 cm^{-1} , which correspond to SWCNT with diameters of 1.21, 0.91 and 0.64 nm, respectively. RBM peaks were not observed for the samples synthesised at catalyst concentrations lesser or greater than 0.5 g ferrocene. A low I_D/I_G ratio of 0.72 was detected for samples synthesised using 0.5 g ferrocene; the ratio increases to a maximum value of 0.98 for samples synthesised using 0.8 g catalyst. The poor graphite crystallinity observed for the sample prepared using 0.8 g ferrocene resulted from the low catalytic properties owing to the bigger catalyst size. A low ferrocene concentration results in an imbalance of carbon atoms compared to Fe catalyst; the huge amount of carbon is not completely decomposed by the low amount of Fe catalyst. In this situation, the catalyst is rendered inactive and low quality nanotubes are produced, as detected for samples prepared with 0.3 g ferrocene.

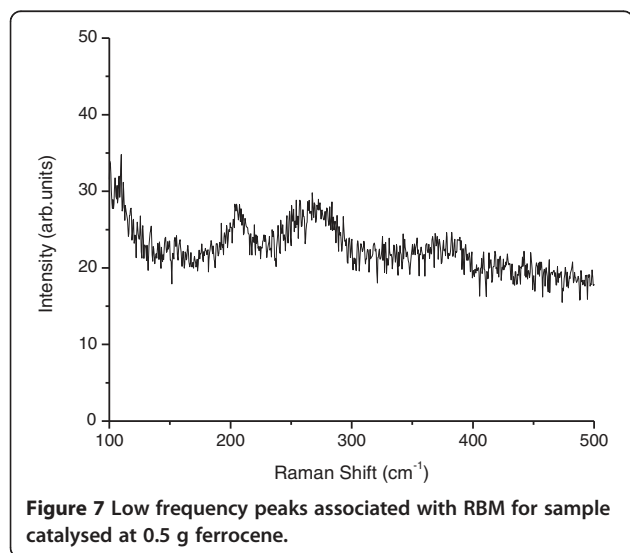


Figure 7 Low frequency peaks associated with RBM for sample catalysed at 0.5 g ferrocene.

Experimental

As-obtained VACNTs deposited on NPSiT were visualised using a FESEM (SUPRA 40 VP, Carl Zeiss SMT, Oberkochen, Germany), and an EDX was used to identify the elements constituting the specimen (qualitative analysis) and the weight concentration of the contained elements (quantitative analysis). Structural properties were observed by micro-Raman spectroscopy using argon ion laser excitation at 514.532 nm (DU420A-OE-325, Horiba Jobin Yvon, Kyoto, Japan).

Conclusion

In summary, CNT growth was detected when as little as 0.3 g ferrocene was used. However, at least 0.4 g ferrocene must be used to produce VACNTs. As the catalyst concentration increases, the density and growth rate of VACNTs were found to increase (up to 2.1 $\mu\text{m min}^{-1}$) until 0.7 g ferrocene was reached. The VACNTs produced when 0.5 g ferrocene was used have small diameters and were found to have low defect levels, as indicated by the calculated I_D/I_G ratio of 0.72. Only at this concentration, RBM peaks were clearly observed for the as-synthesised SWCNTs. The growth of vertically aligned carbon nanotubes has been successfully reported using a porous template and the appropriate amount of catalyst such that the pores can physically hold the nanotubes in the vertical direction and the bundles of nanotubes can support each other during growth. Further studies should be carried out to assess the prospective applications of these structures; for example, VACNT exhibits excellent properties for use as field electron emission cathodes.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

NAA and MSS carried out the experiments and drafted the manuscript. SAB built the experimental setup. MRM and SA reviewed the manuscript. All authors read and approved the final manuscript.

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