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ORIGINAL ARTICLE

Fine-tuning control on CNT diameter distribution, length and density using thermal CVD growth at atmospheric pressure: an in-depth analysis on the role of flow rate and flow duration of acetylene (C_2H_2) gas

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Abstract An optimization control has been demonstrated to obtain carbon nanotubes having specific diameter distribution, length, homogeneity, and yield during its growth by thermal chemical vapor deposition technique under atmospheric pressure. Carbon nanotubes (CNTs) were grown on silicon wafer where a predeposition of iron catalyst of 2 nm thickness was made by sputtering. The growth was conducted under two variable parameters, i.e., flow rate and flow duration. Argon and hydrogen mixture was used for pretreatment of catalyst and as etching gas, and acetylene as a carbon precursor. In-depth analysis shows that increase in flow rate from 10 to 50 sccm resulted in increase in the concentration of amorphous carbon, CNTs diameter range and decrease in length, we found best result at 20 sccm flow rate of acetylene gas. On the other hand, as we varied flow duration from 6 to 14 min, with keeping flow rate of acetylene 20 sccm constant, dense homogeneous growth of horizontal CNTs network plus an increase in length and diameter range were observed. An optimization of flow rate and flow duration is presented here to obtain a selective diameter distribution and length as expected by this growth technique. Atomic force microscopy, field emission scanning electron microscopy and Raman spectroscopy were used to investigate the samples' morphologies in support of the observations made.

Keywords Carbon nanotubes · Chemical vapor deposition · FESEM · Raman spectroscopy · AFM

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Introduction

Since the discovery of carbon nanotubes (CNTs) (Iijima et al. 1991), a huge interest has been generated all over the world because of its high mobility, large current-carrying capacities, very high mechanical strength and host of other potential applications in the area of electronics, medical sciences and many other fields. Due to these facts, CNTs have been considered as one of the best candidates for future applications in nanotechnology (Ghosh et al. 2009; Lee et al. 2006; Cheng 2012; Vashist et al. 2011; Durrer et al. 2008). This has motivated the researchers to look into the growth aspect of CNTs and its characterization with an aim to have tailor made properties. A fine-tuning control over its physical and electronic aspects is in very much demand for commercialization of CNT-based devices.

Over the years, the researchers have tried to grow CNTs by various techniques including arc discharge, chemical vapor deposition (CVD), laser ablation techniques and others. In the beginning, arc discharge (Zhao et al. 2005; Cadek et al. 2002; Bethune et al. 1993; Keidar and MWaas 2004; Tamir and Drezner 2006) and laser vaporization (Terrado et al. 2006; Dai 2012) were considered as ideal methods for bulk CNTs growth process (Ebbesena and Ajayan 1992; O'Conel and Editor 2006). In both methods, condensation of hot gaseous was used and generation of carbon atoms was done by evaporation of solid carbon. But there are several shortcomings in these methods such as: (1) only bundles of CNTs in the form of powder are produced, (2) systematic growth on substrates with selective structure is not possible, and (3) need of equipments and consumption of energy is very large (Kobayashi et al. 2004). These limitations make these techniques less favorable for CNT growth. However, thermal CVD technique (LeNormand and Cojocaru 2007; Meyyappan et al.

2003; Zobir et al. 2012; Lou et al. 2004; Song et al. 2009; Dai 2002; Atthipalli et al. 2011) using carbon source gas has become a promising one for CNTs growth. It has following merits: lower preparation temperatures, better techniques for large-scale CNTs production, easy to design a CVD system, easy to control the parameter and having much chances to grow aligned, horizontal and selective growth of CNTs, these are very useful in electronic device applications. In CVD method, nanosize metal particles are predeposited on the substrate to act as nucleation sites for the initiation of CNTs growth in the presence of carbon produced by decomposition of gaseous or volatile compound.

Research activities had been done so far for controlled growth of CNTs, by varying the parameters such as temperature and pressure of CVD system. Besides, nobody has ever succeeded to grow CNTs where they have control on the diameter distribution, length and density. Even systematic study on the growth of CNTs by variation of flow rate and flow duration of source gas (C_2H_2 gas), is not yet explored.

In this work, we present a profound study about the effect of different flow rate and flow duration of the carbon precursor gas (C_2H_2) on the growth of CNTs by thermal CVD method. We have successfully achieved the capability to fine-tune the length, diameter distribution, surface density, yield, and quality of CNTs by varying flow rate and flow duration of acetylene gas. An optimization of these two parameters, i.e., flow rate and duration, has been done for CNT growth with selective physical characteristics as expected.

Experiment

Details of CVD growth technique

Growth of CNTs was carried out by thermal CVD system (see Fig. 1), which is a horizontal reactor made of a quartz tube with diameter 65 mm and length 650 mm. The reactor was inserted in electric furnace with controllable heating temperatures up to 1,000 °C over a heating zone of 230 mm in length. All the reactions were done at atmospheric pressure.

Carbon nanotube growth was performed on a 5 mm × 5 mm n-type Si (100) substrate. Cleaning process of the substrate was done by ultrasonication in acetone for 10 min. A thin film of Fe of 2 nm thickness was deposited on Si substrate as a catalyst for CNTs growth purpose by RF sputtering, followed by heating at 800 °C for 20 min to form nanosize particles from thin Fe catalyst film. Growth process was then initiated under the inert atmosphere of Ar at 50-sccm flow rate inside the CVD chamber to prevent the metal particle from oxidation during temperature rise of

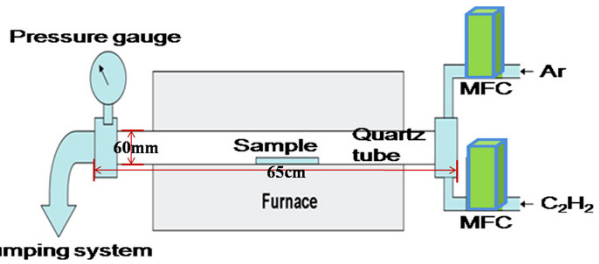

Fig. 1 Schematic diagram of the CVD system

furnace from room temperature to 800 °C. CNTs were grown using acetylene (C_2H_2) gas with 10-sccm flow rate in addition to Ar gas for 10 min. After 10 min, C_2H_2 flow and furnace were turned off while keeping Ar flow till the furnace was cooled down to room temperature. Flow of Ar during furnace cooling process is only to secure the grown CNTs from burning at such a high temperature. After cooling, the substrate was ready to be removed for analysis.

To see the effect of flow rate of C_2H_2 on CNTs growth process, the growth process was repeated with flow rate from 10 to 50 sccm of C_2H_2. We observed that sample grown under 20-sccm flow rate of C_2H_2 had better results as compared to other flow rates. Then keeping the flow rate of C_2H_2 at 20 sccm, the growth process was repeated with different flow duration of C_2H_2 gas from 6 to 14 min, where all other parameters of CVD process were kept same as previous. The effect of flow rate and flow duration of C_2H_2 gas on the growth of CNTs, samples were investigated by high resolution scanning electron microscope (FESEM, NOVA NANOSEM 450) and Micro-Raman spectrometer with 488 nm Ar^+ laser (LabRAM HR800, JY).

Result and discussion

Figure 2a shows the atomic force microscopy (AFM) image of iron catalyst particle and its height profile just after pretreatment with Ar at 800 °C. Figure 2b shows the FESEM image of Fe-sputtered Si surface after treatment at 800 °C in Ar for 15 min where it is seen that the Fe film breaks into small islands. It is evident from the AFM height profile of the substrate that Fe nano particles are distributed uniformly on the Si substrate. It is confirmed that the pretreatment with Ar plays an important role in the formation of uniformly separated nanosize iron particles, which shall act as a catalytic support for CNTs growth [ref]. All the growth conditions other than growth duration and flow rate of C_2H_2 for the samples studied here were kept constant to study only the various effects of C_2H_2 gas on the growth of CNTs. Before explaining effects of flow rate and flow duration of acetylene gas, we briefly explain the growth mechanism of CNTs from Figs. 4 and 7. When acetylene gas made contact with hot metal catalyst particle,

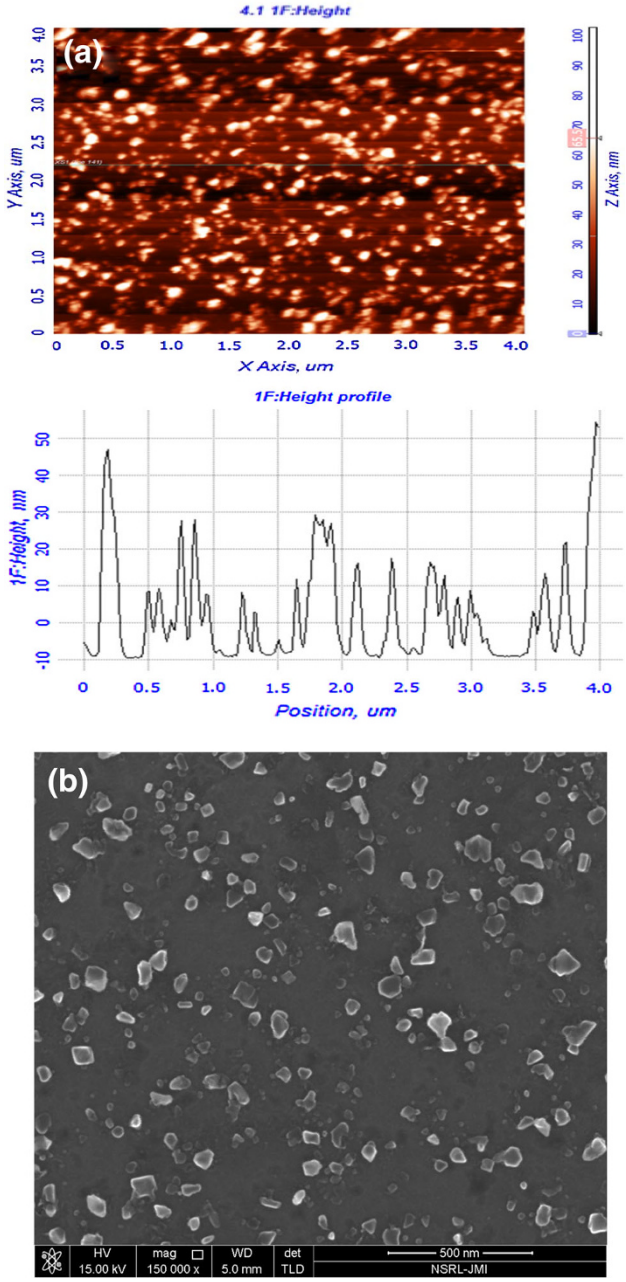

Fig. 2 **a** AFM images of nanosize iron catalyst particles and its height profile. **b** FESEM images of nanosize iron catalyst particles

acetylene gas diffused into carbon and hydrogen gas. Hydrogen flies away from chamber and carbon atoms start to dissolve into metal catalyst particles. After saturation of catalyst particles, the carbon atoms precipitates in the form of CNTs.

Effect of flow rate of C_2H_2

Figure 3 shows the FESEM images for the CNTs grown on a Fe-deposited Si substrate at 800 °C with different flow rates of 10, 20, 30, 40 and 50 sccm, respectively, of C_2H_2 for 10 min and one common thing observed on all samples that CNTs' growth equally distributed on full substrate; and Fig. 4 shows the growth mechanism for different flow rates. The Raman spectra of CNTs synthesized at different flow rates are shown in Fig. 5. The horizontal network of CNTs is homogenously grown on all substrates. From FESEM investigations, it is observed that one tip growth (metal particle peeled off on the tip of CNT) is common in grown CNTs on all substrates.

Figure 3a shows the uniformly grown network of CNTs at 10-sccm flow for 10 min, having diameter distribution range of grown CNTs from 12 to 30 nm. In addition, the length of CNTs is very small and many Fe particles are seen on the surface. The reason behind such type of growth is that the produced carbon from decomposition is insufficient vis-à-vis the requirement for CNT growth; as a result, some of the Fe particles are left out, plus carbon deficiency limits the CNT length for further growth. Growth mechanism for this work is demonstrated in Fig. 4b; while Fig. 4a shows the position of catalyst after pretreatment, and the substrate as reference for comparing growth mechanism in all cases. Raman analysis on the characteristic peak positions of D- and G-band and their ratio ($I_G/I_D = 1.238$) confirms that the grown CNTs are multiwall, and surface is less defective due to the absence of less amorphous carbons.

When the flow rate was increased to 20 sccm, relative effects were seen in the FESEM investigation (Fig. 3b). Comparatively long and much straight CNTs were observed as compared to the 10-sccm flow rate, and diameter of CNTs lies in the range of 12–38 nm which is almost same as growth at 10-sccm flow rate. It was also observed that almost on every Fe particle, CNTs has been grown, leaving very few iron particles visible on surface and consequently the number of CNTs per unit area has been increased. Therefore, it can be said that at 20-sccm flow rate, the decomposition rate and diffusion rate of carbon to make CNTs were almost same; hence the tip of CNTs was free from amorphous carbon, which is deposited when number of carbon particle exceeds the limit as needed for CNT growth, and this is the reason that catalyst particle did not lose its catalytic activity throughout the growth process leading to the growth of long and straight CNTs. Growth mechanism for this is shown in Fig. 4c, where we can see that during complete growth process iron catalyst particles were free from carbonaceous particles or amorphous carbon. I_G/I_D ratio of Raman spectra for this like in previous experiment shows that grown CNTs are almost same in quality as obtained with 10-sccm flow rate of C_2H_2 (Fig. 5).

Figure 3c, shows the FESEM image of grown CNTs at 30 sccm. Here the length and yield of grown CNTs decreased, while diameter increased as compared to the

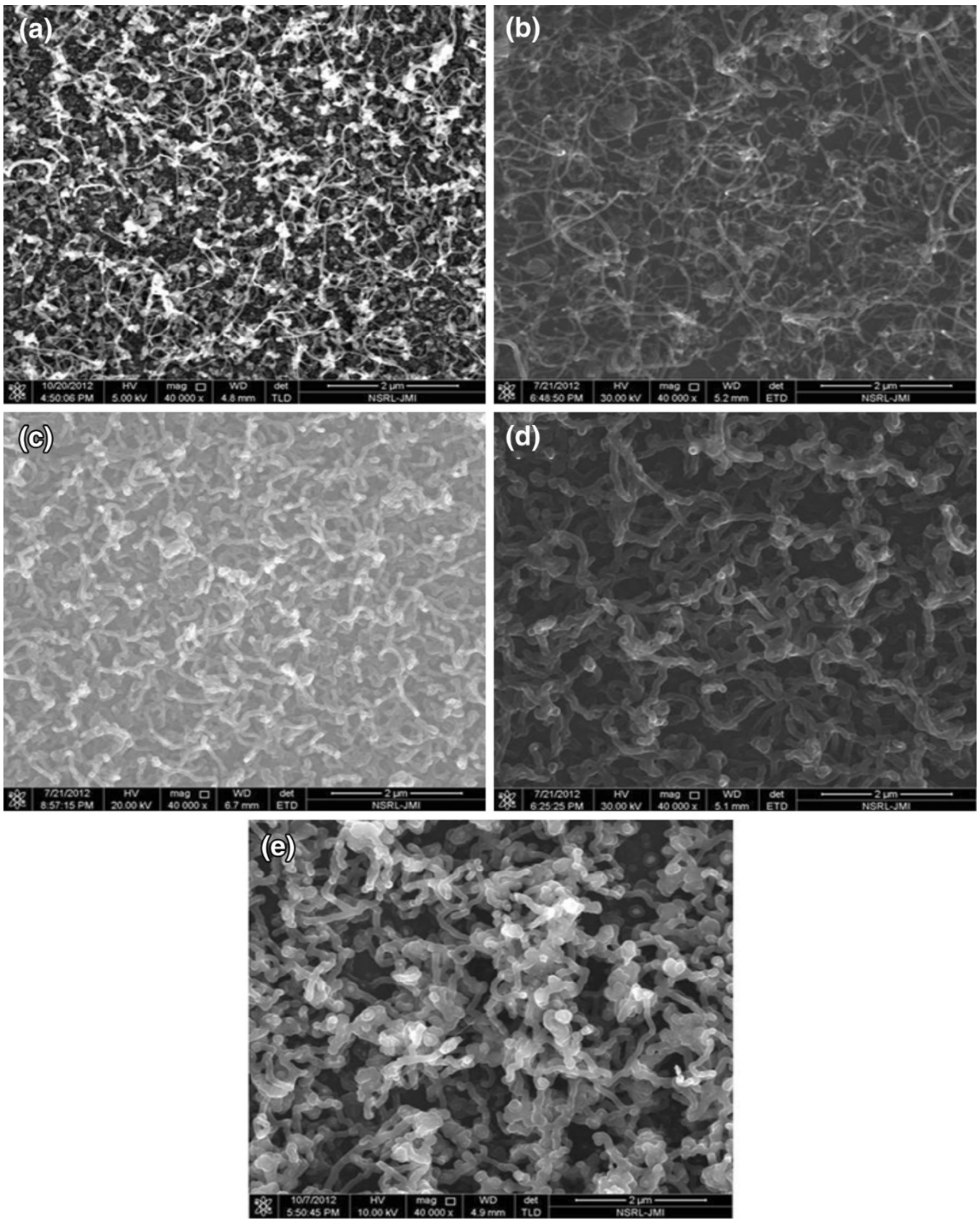

Fig. 3 Morphologies of carbon nanotubes grown at 800 °C in 10 min for flow rate of C_2H_2 **a** 10 sccm, **b** 20 sccm, **c** 30 sccm, **d** 40 sccm, **e** 50 sccm

case of 20-sccm flow rate of C_2H_2. Diameter range in the present case is 45–60 nm. At 30 sccm, decomposition rate of C_2H_2 became higher than diffusion rate of carbon to make CNTs because of the availability of higher quantity of C_2H_2. Remaining part of carbon, which was not used to make C–C bond in CNT making process, was deposited on tip as well as through the surface of CNTs in the form of amorphous carbon. Due to presence of large amount of carbon, the catalyst activity starts to decrease and speed of CNT growth reduces. Once the catalytic property of catalyst saturates completely the growth process also stops. This is the reason behind short length of CNTs in 30-sccm

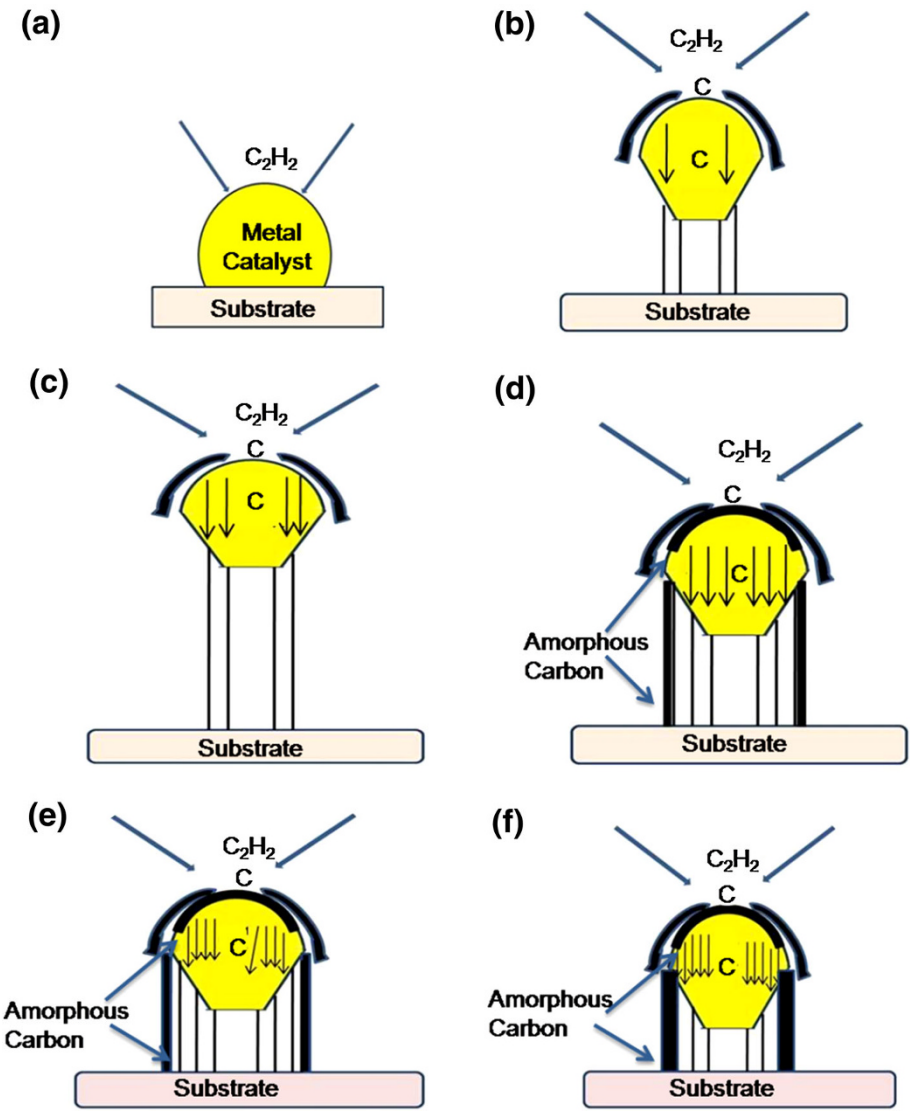

Fig. 4 A CNT growth mechanism model. **a** Position of catalyst before starting growth process. **b–e** and 3f represent the growth mechanism step for 10, 20, 30, 40 and 50 sccm, respectively

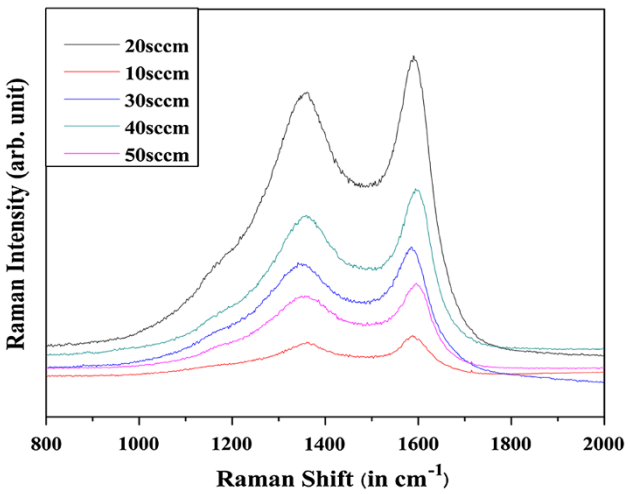

Fig. 5 Raman spectra for grown CNTs; different flow rate of C_2H_2

flow rate as compared to 20-sccm flow rate. This can be understood from growth mechanism shown in Fig. 4d. In Fig. 4d it is observed that Fe catalyst has been completely covered by amorphous carbon and a thin layer of amorphous carbon also covered the outer surface of CNTs. In this case, two reasons have been proposed for increment in diameter of grown CNTs: first, in the beginning of growth process, yield of carbon particle is very high and hence the number of walls as compared to last experiment for 20 sccm is increased; second, deposition of amorphous carbon on CNTs outer wall surface. Reason behind reduction in the yield is the cloud formation of carbonaceous particle, which covers few Fe catalyst particles before the starting of the growth and hence, these particles become unable to participate in growth process. This, in turn, led to decrease the number of CNTs per unit area.

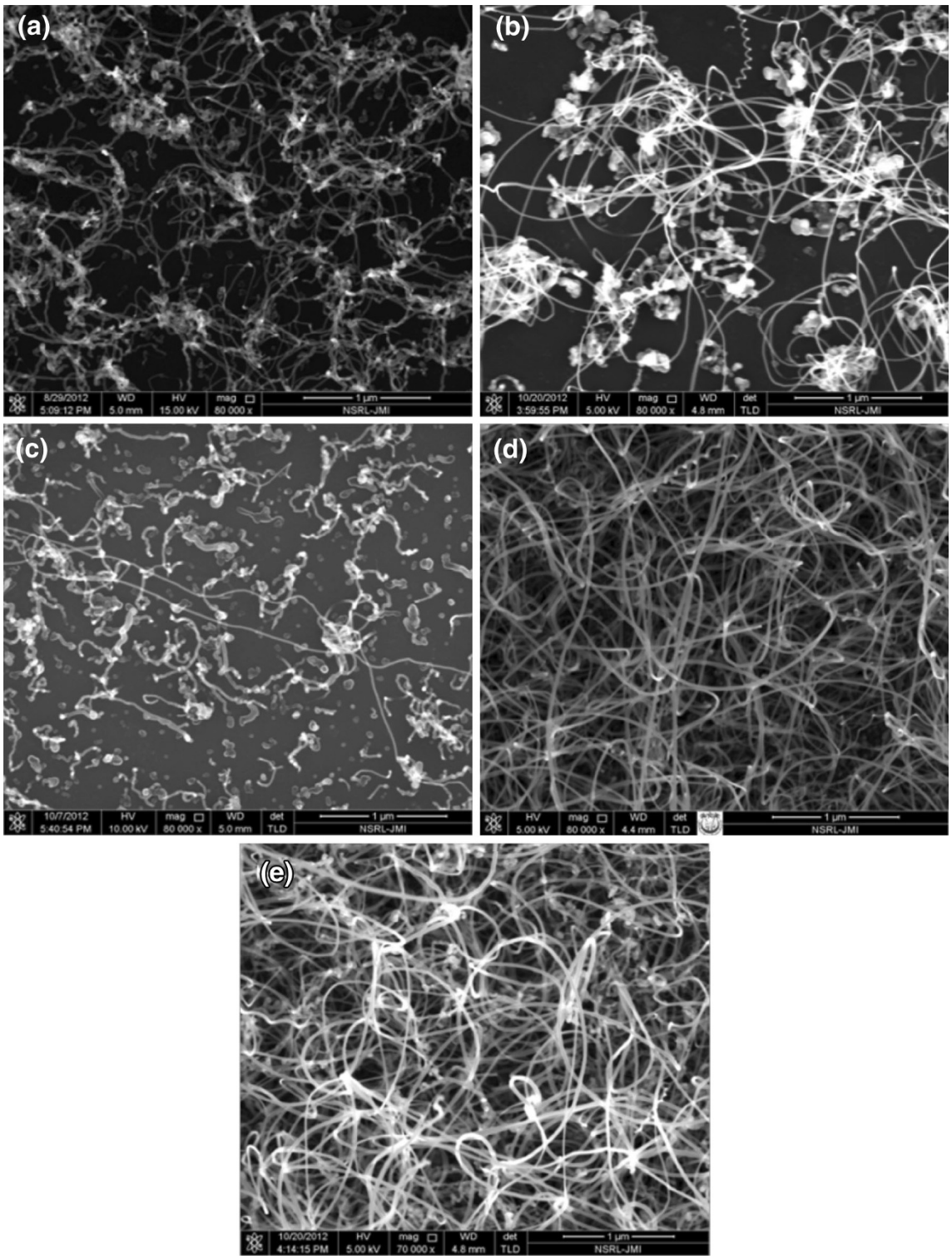

Fig. 6 Morphology of carbon nanotubes grown at 800 °C with 20-sccm constant flow rate in C_2H_2 in duration **a** 10 min, **b** 20 min, **c** 30 min, **d** 40 min, **e** 50 min

Decreased I_G/I_D ratio (from 1.239 in case of 20-sccm flow rate to 1.162 in 30 sccm) in Raman spectroscopy analysis also indicates the increment in impurity in the form of amorphous carbon or carbonaceous particles (Fig. 5).

On further increase of the flow rate to 40 sccm, we observed that amorphous carbon covered the catalyst surface in comparatively lesser time, much shorter length of grown CNTs, and thicker layer of amorphous carbon on the

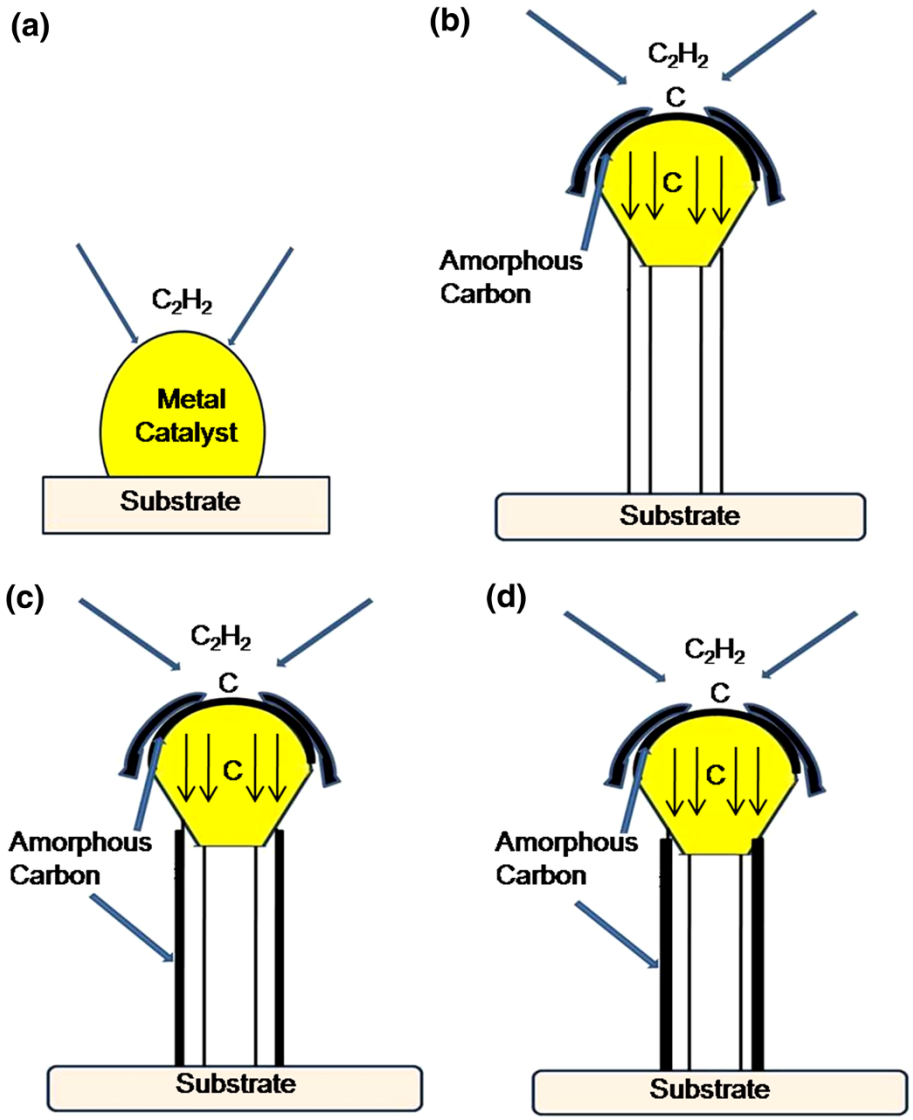

Fig. 7 A CNT growth mechanism model. **a** Position of catalyst before starting growth process. **b** Growth mechanism for 6, 8 and 10 min. **c** Growth mechanism for 12 min. **d** Growth mechanism for 14 min

outer wall of CNTs as compare to 30 sccm flow rate (see Fig. 4e). That is why diameter of grown CNTs is also more. We can see all these effects in FESEM image of Fig. 3d and in Raman spectra of Fig. 5. I_G/I_D ratio of Raman spectra is decreased further to 1.151, and hence more impurity deposited on surface of CNTs in the form of amorphous carbon. Further increase of the flow rate to 50 sccm did not add qualitatively anything good. Rather, we observed the same effects (Fig. 4f) as seen in case of 40 sccm with lesser CNT length, larger in diameter, and smaller I_G/I_D ratio (1.139). FESEM image (Fig. 3e) shows the diameter of grown CNTs is around 90 nm, large enough to make the CNTs touch each other leading to bundling. Large diameter CNTs cover larger surface and occupied more than one catalyst particle. Experiments for high flow rates (40 sccm and 50 sccm) showed that cloud of carbonaceous particle covers the more catalyst particles and due to larger diameter of CNTs, leading to number of CNTs per unit area is very low.

Hence, finally it is resolved that the results obtained with 20-sccm flow rate are best in all experiments.

Effect of flow duration

A series of experiments were conducted by varying flow duration of C_2H_2 only at constant 20-sccm flow rate to see its effect on the CNT growth. Figure 6a–e shows the FE-SEM images for different flow duration of 6, 8, 10, 12, and 14 min of C_2H_2 gas. From FESEM image of grown CNTs, we can observe that the growth of CNTs occurred on whole substrate. Growth mechanism of CNTs with various flow duration of C_2H_2 is shown in Fig. 7.

Growth of CNTs with 20-sccm flow rate for 10 min is already shown in Fig. 3b and its higher magnification

image can be seen in Fig. 6a. When the flow duration of C_2H_2 was decreased from 10 to 8 min it is found that average length and yield of CNTs per unit area decreased (see Fig. 6b), while the diameter range of grown CNTs remained almost same as for 10-min duration (Fig. 6a). As explained earlier, for a constant 20-sccm flow rate of C_2H_2, the decomposition rate and diffusion rate of carbon particle to grow CNTs are in equilibrium condition. Hence, when we changed the flow duration to 8 min, the diameter of grown CNTs was almost same but due to short duration, a little decrement in the length was observed. We also observed that the length of grown CNTs was not homogeneous, and some nanosize iron particles could be seen localized on the surface with no grown CNTs. The reason for such kind of growth is that each and every iron catalyst particle did not get activated at the same time; some catalyst particles got activated in the very beginning of growth process, while others were activated with a time delay. As a result, the length of CNTs was modified due to varying activation time of Fe catalyst particle, even some iron particles were activated almost at the end of growth. Very short length or no CNTs grow on such particles, which lead to reduced yield.

Decreasing the flow duration from 8 to 6 min, a relative change in FESEM image can be seen in Fig. 6c. Length and number of CNTs per unit area decreased drastically as compared to longer durations. More nanosize Fe particles were left without CNTs growth. Reason behind all such effects is because of short flow duration of C_2H_2, which allows only few particles to be activated and rest were either inactivated or activated late. That is why only few long CNTs were found. Due to same flow rate, as in last experiment, diameter of CNTs is unaffected by decrement in flow duration.

On further increase in the flow duration of C_2H_2 from 10 to 12 min (see Fig. 6d), we observed that yield and length of grown CNTs increased. Because of the long growth duration, almost all nanosize Fe particles were activated and number of CNTs per unit area increased.

Due to the long growth duration, most of the catalyst particles get deactivated before the end of growth process, which disturbed the equilibrium condition of diffusion rate and deposition rate of carbon; and hence some carbon particles were deposited on the surface of nanotubes. As a result, a very small increment was observed on the diameter of grown CNTs as compared to Fig. 6a. Similarly, when we increase the flow duration of C_2H_2 from 12 to 14 min, we observe that yield (number CNTs per unit area) and length of CNTs were almost same, but the diameter increases (see FESEM image of Fig. 6e).

In summary, the growth mechanism for all experiments relative to flow duration of C_2H_2 gas on CNTs growth process is shown in Fig. 7. Figure 7a shows the position of catalyst particle with respect to Si substrate before growth process. Growth mechanism for 6-, 8- and 10-min flow duration is almost same and shown in a common diagram. A thin layer of amorphous carbon on outer wall of CNT is clearly shown in Fig. 7c for 12-min flow duration, and this is responsible for the increment in diameter vis-à-vis 10-min duration; this trend continued further up to 14-min duration (Fig. 7d). Raman analysis (Fig. 8) provided the complimentary data in support of the FESEM observations. It is observed that I_G/I_D ratio ($\sim$1.238) and the crystal structure of CNTs remain same for the flow duration for 6–10 min. For 12- and 14-min durations, the I_G/I_D ratios were found as 1.159 and 1.151, respectively, and these values are less as compared to lower flow durations. This means crystallinity of CNTs reduced in the growth process for 12- and 14-min durations. FESEM investigations support these facts.

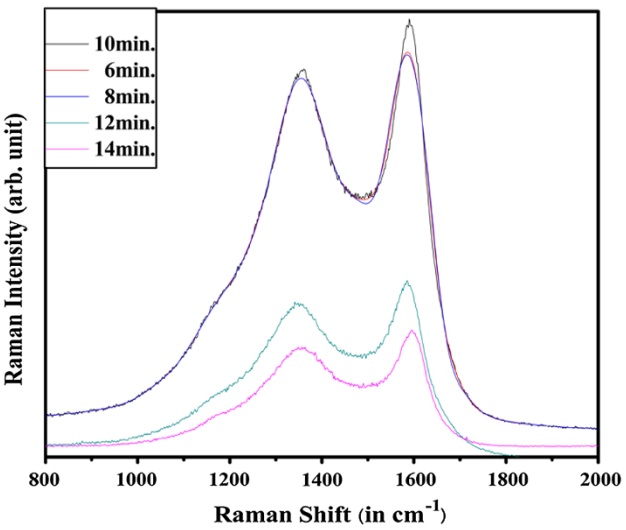

Fig. 8 Raman spectra for grown CNTs; different flow duration of C_2H_2

Optimization control on selective growth of CNTs

A detailed comparative study regarding length, diameter distribution, and yield for flow rate of C_2H_2 in Fig. 9. It can be seen that that diameter of grown CNTs in 10- and 20-sccm flow rates is almost constant and from 20 to 50 sccm, it increases linearly (black line in Fig. 9). While red line Fig. 9 shows that the length of grown CNTs increases from 10- to 20-sccm flow rate, but as we increased the flow rate from 20 to 50 sccm it decreases almost linearly. From blue line of Fig. 9, we get an information about length of CNTs and its yield. Both increased from 10 to 20 sccm and decreased linearly in the same fashion. We observed that results found with 20-sccm flow rate are best in all experiments.

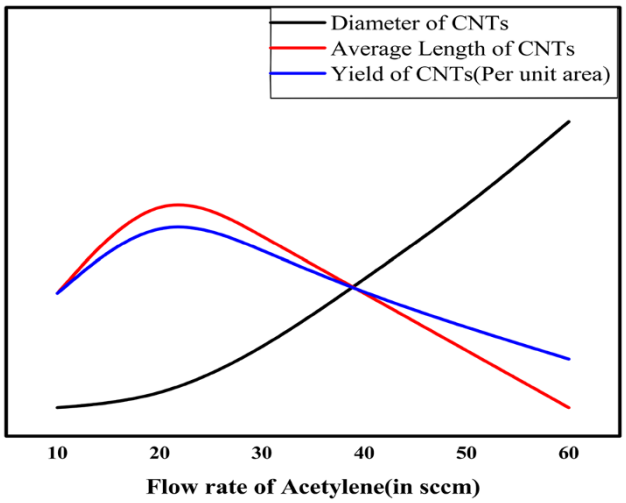

Fig. 9 Shows the relation between, diameter, yield of CNT per unit area and length of CNTs with flow duration of C_2H_2 gas

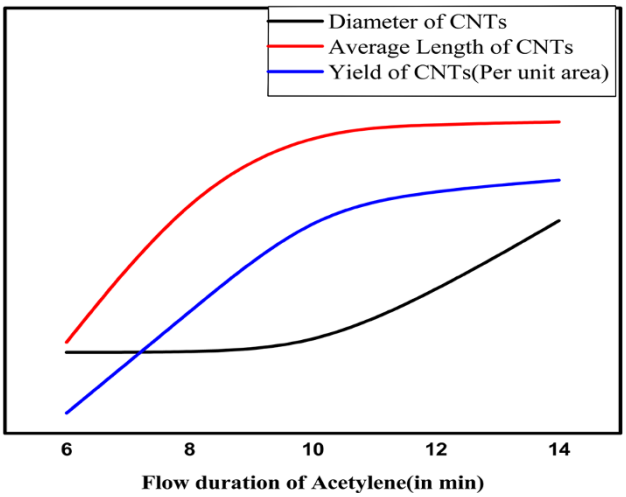

Fig. 10 Shows the relation between diameter, length of CNTs and yield of CNT per unit area with flow duration of C_2H_2 gas

A similar comparative study relative to the flow duration of C_2H_2 gas is shown in Fig. 10. From black line of Fig. 10, we can see that there is no sufficient change in diameter for C_2H_2 flow durations of 6–10 min, whereas diameter increases almost linearly for further increase in the flow duration. Red line in Fig. 10 shows that average length of CNTs increases linearly till the flow duration of C_2H_2 to 10 min. For further increase in the flow duration to 14 min, diameter of grown CNTs remains almost constant. Blue line in Fig. 10, we can see that yield of CNTs increases rapidly from 6 to 10 min and decreases with higher flow durations. In fact, yield of CNTs almost gets saturated at the 14-min flow duration of C_2H_2 gas. Therefore, the flow duration of 12 min may be considered as the best condition.

To have an optimization control over selective growth of CNTs, a simultaneous trading in between flow rate and flow duration is to be established. If we want CNTs with lesser diameter, we have to keep the flow rate and flow duration of C_2H_2 minimum, but at this condition the length and the yield of CNTs also decrease. Therefore, for achieving both smaller diameter and sufficient length and yield of grown CNTs we should optimize the growth conditions of the CVD system at 20-sccm flow rate and 12-min flow duration.

Conclusion

In this report, a systematic study has been carried out to have a better control over the growth aspects of CNTs by varying gas flow rate as well as flow duration of carbon precursor. We observed the effects of flow rate of C_2H_2 in the range of 10–50 sccm on the growth of CNTs. Effect of source gas flow duration is also analyzed in the time duration range from 6 min to 14 min with constant flow rate of acetylene, i.e., 20 sccm. We observed that the yield, diameter and length of CNTs increase with flow rate of C_2H_2 up to 20 sccm. Beyond 20 sccm, yield and length started decreasing but increments in diameter were observed. Similarly, in the case of flow duration of C_2H_2, quality, diameter and length of CNTs improve with respect to time until 10 min; further increment in time increases the yield (number of CNTs per unit area) of CNTs, but decreases the yields. Therefore, we conclude that we should optimize the growth conditions of the CVD system at 20-sccm flow rate and 12-min flow duration to get the best results vis-à-vis diameter, length, quality and yield of grown CNTs.

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