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Effective Supergrowth of Vertical Aligned Carbon Nanotubes at Low Temperature and Pressure

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We present the generalised supergrowth of single and double walled carbon nanotubes like “water assisted supergrowth” at low temperature (630 °C) and pressure (1 Torr) by chemical vapour deposition using various thicknesses of iron supported alumina substrate as a catalyst. Reduced temperature and low pressure synthesis of single walled (SW) and double walled (DW) carbon nanotubes is of interest for their efficient growth into device architectures. Pure SW with 2.5 nm diameter are obtained with 0.37 nm Fe catalyst at 630 °C. We demonstrated the decrease of the density versus temperatures and also obtained high density materials ($1.4 \times 10^{12} \text{ cm}^{-2}$) at low temperature (580 °C). Scanning and transmission electron microscope studies provided information on the height, density of the carpets and the structure and diameter of SW and DW carbon nanotubes, respectively.

Keywords: Single Walled Carbon Nanotubes, Double Walled Carbon Nanotubes, Vertical Aligned Growth, Supergrowth, Radio Frequency Chemical Vapour Deposition.

1. INTRODUCTION

Low temperature growth of CNTs is one of the most fundamental issues, to be determined for growth of CNTs based interconnects technology compatible with LSI manufacturing processes. CNTs are a promising material for numerous applications due to their unique physical and scale-dependent physical properties.^{1,2} Dense aligned arrays of CNTs (sometimes called forests or carpets) are especially promising for a number of applications. CNTs within the forest possess excellent properties such as alignment, long length, good electrical conductivity, high carbon purity and high surface area. SWCNTs and DWCNTs have high structural stability, which may be suitable for many industrial applications.^{3,4} To utilize them for electrical devices, it is often desired to synthesize vertically aligned (VA) SWCNTs and DWCNTs directly on a substrate. Furthermore, low temperature growth is often important for avoiding damage to materials used in electronic devices. The ability to tune the functional interface of SWCNTs in a versatile manner is key to the success of deploying them as an active material in chemical and biological sensors.^{5–7}

Up to now, three methods are familiar to synthesize CNTs such as arc discharge,⁸ laser ablation⁹ and chemical vapour deposition (CVD).¹⁰ Though nanotubes synthesized by arc-discharge and laser ablation processes are high on purity, scaling up this process to produce nanotubes on a larger scale is not very effective. Although many efforts have been made in the development of synthesis methods, CVD and Plasma Enhanced (PE) CVD have become more popular and are considered at the moment to be the best approach for low-cost and large-scale synthesis of high-quality entangled or aligned (single-walled or multi-walled) CNT materials in the temperature range of 700–1200 °C. Among them, CVD appears to be a simple and economic technique to synthesize CNTs. CVD method is based on the thermal decomposition of hydrocarbon compounds (acetylene, ethylene, benzene, naphthalene, toluene, etc.) over transition metal catalyst particles.^{11,12} However, one of the fundamental limitations toward profound technological advances in these applications is the ability to have intricate control of the growth process.

Most reported SWCNTs and DWCNTs were grown in a random form at high temperatures (1000 °C).^{13,14} Murakami et al.¹⁵ first demonstrated the growth of VASWCNTs at a growth temperature of 800 °C by using ethanol as a carbon source. After this, Hata et al.¹⁶ demonstrated a lower temperature (750 °C) growth of vertically

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aligned SWCNTs. Cantoro and co-workers¹⁷ reported the growth of vertically aligned SWCNTs but the growth condition and ratio for SWCNTs are not clear. The optimal parameters for oxide assisted growth of vertical aligned single walled carbon nanotubes were reported by Pint et al.¹⁸ Very recently, Futuba et al.¹⁹ reported highly efficient growth of carbon nanotubes using various growth enhancers. Although vertically aligned SWCNT growth has been reported at relatively low temperatures, there are no reports regarding the growth of aligned SWCNTs at temperatures below 650 °C without the addition of growth enhancer.

Realizing the unique advantages offered by CVD a systematic parametric study of this nature, such as performed for DWCNTs grown by thermal CVD,²⁰ provides insights that could greatly enhance the quality of SWCNTs and enable the control of their properties (e.g., density, alignment, length and diameter). Here, we demonstrate the highly efficient growth can be achieved without using water or any other growth enhancer. In this investigation, we have discussed and summarized the comprehensive parametric study of the synthesis parameters affecting the growth of SWCNTs and DWCNTs using CVD method through presenting some outstanding work in the area of surface synthesis. These parameters include the synthesis temperature, growth time, catalyst thickness and pressure. We also demonstrated a novel technique to densify CNT grown. Samples obtained after reaction are characterized using field emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM). The results indicate that SWCNTs and DWCNTs selectivity can potentially be controlled by selection of synthesis parameters.

2. EXPERIMENTAL DETAILS

The CVD apparatus utilised for the growth of VACNTs array (carpets) were described in our previous reports.^{21,22} In this process, an iron alumina bi layer was deposited on a silicon substrate with a native oxide layer as catalyst substrate. The substrate were first treated with plasma air 70 W (10 min) and 30 W (20 min) at room temperature (RT). Before growth, the substrate was preheated with gas mixtures from room temperature to reaction temperature for 15 min. Then the feedstock flow of gas mixtures acetylene, hydrogen and helium (10, 30 and 50 sccm, respectively) were introduced into the chamber at various reaction temperatures and pressure for a growth period of 10 and 60 min. Once the reaction process is completed, the process chamber is cooled to room temperature under helium flow.

A field emission scanning electron microscope (FESEM) LEO 1530 is used to observe the morphology of the grown CNT arrays. Transmission electron microscopy is performed using a field-emission Jeol JEM 2010FEF operating at an accelerating voltage of 200 kV and

equipped with an in column Omega filter to characterize the catalyst-substrate interface and the atomic structure of CNTs. Cross-sectional samples are prepared using a conventional precision ion polishing system (PIPSTM). The CNTs removed from substrate are dispersed in ethanol and dropped on a holey carbon coated copper grid.

In order to measure the tube density on the grown carpet, the densification was done by immersing the sample into a beaker of isopropyl alcohol (IPA) for 24 hrs. It was then dried naturally in air at room temperature until IPA completely evaporates. The tubes compressed and the initial filling factor F of the material is determined by measuring the ratio between the compressed tubes area and the total area. To do that SEM images are used after compaction and the areas are determined using Analysis Software (soft imaging system).

3. RESULTS AND DISCUSSION

3.1. Effect of Low Pressure During the Growth

In this study, we describe the effect of low pressure process for the growth of vertical aligned single walled carbon nanotubes at fixed temperature (730 °C) and time (60 min), we identify in the pressure from 0.3 to 1 Torr (T), there is gradually increase in the height of the carpet which is clearly shown in Figure 1 and the results are also tabulated in Table I.

One of the key features of growth of carpet is the pressure of the growth gas in the reaction zone, the rate of C_2H_2 collisions to the catalyst. To increase the reaction pressure, the flow velocity of gases in the reactor are adjusted which means that the number of collisions between gas molecules and the catalyst surface is increased proportionally. C_2H_2 is known for its ability to decompose in a single collision reactions leaving it as the most reactive of all growth gases for catalytic carbon nanotube growth.

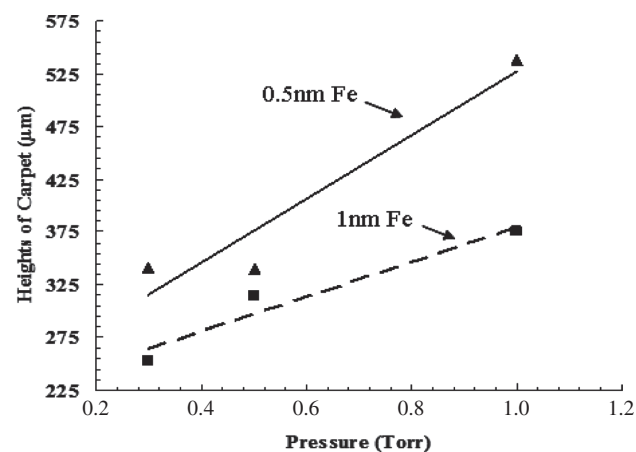


Figure 1. Graphical representation for pressure vs height of carpets grown on (a) 0.5 nm Fe and (b) 1 nm Fe at 730 °C for 60 min.

Table I. Represent the height of carpets grown at various pressures under constant temperature (730 °C), time (60 min) and flow rate gas mixtures (C₂H₂/H₂/He—10/30/50 sccm) for two different thicknesses of catalysts.

Substrate	Pressure (Torr)	Height of CNTs (μm)
0.5 nm Fe on Al ₂ O ₃	0.3	341
1 nm Fe on Al ₂ O ₃		253
0.5 nm Fe on Al ₂ O ₃	0.5	340
1 nm Fe on Al ₂ O ₃		314
0.5 nm Fe on Al ₂ O ₃	1	538
1 nm Fe on Al ₂ O ₃		375

According to the previous report, CNTs were synthesised under atmospheric pressure at high temperature (800 °C) in the presence of water. Wen et al.^{23,24} also reported the growth of long double and triple walled CNTs at 1000 °C with addition of water. Pint et al.¹⁸ found that one can increase the pressure from 1.4 to 25 T which results in a carpet that grows to nearly 600 μm in the course of only 30 min. In another experiment same authors²⁵ reported that the carpet height increased linearly with growth time up to 120 min implying a long catalyst lifetime in the presence of H₂O, whereas in the absence of H₂O, the carpet height remained roughly constant (10–20 μm) suggesting the early termination of growth occurs before 15 min. According to them, the continuous growth of the carpet and the lifetime of the catalysts are depending on the presence of water in the chamber. When compared with their results, we have achieved the maximum carpet height (538 and 375 μm for 0.5 nm Fe and 1 nm Fe thickness, respectively) at temperature (730 °C) under low pressure (1 T) without water.

We performed TEM analysis for the sample grown under very low pressure (0.3 T) at 730 °C using 0.5 nm iron thickness catalyst which is shown in Figure 2(a). The TEM measurement shows that the majority of nanotubes are single wall with diameter ranging between 3–4.5 nm (Fig. 2(a)). The use of gas mixtures involving C₂H₂ in growth under low pressure results in the growth of single wall carbon nanotubes, due to a low number of collisions between C₂H₂ molecules and the catalyst. The observed catalyst lifetime longer than one hour confirm that a super growth mode is excited in agreement with previous reports²⁶ but without addition of water under low pressure.

If the gas mixtures involving C₂H₂ in growth under atmospheric or high pressures results in the growth of large diameter multiwalled carbon nanotubes in the array, due to a higher number of collisions between C₂H₂ molecules and the catalyst. When the pressure is low, we can achieve the growth of single-walled carbon nanotube arrays from C₂H₂ with smaller diameter. The focus of characterization of the nanotubes will be those grown under optimal conditions for SWNT growth in reaction pressures of 0.3 Torr.

We also examined sample obtained from 1 nm Fe thickness catalyst under similar reaction conditions in that case,

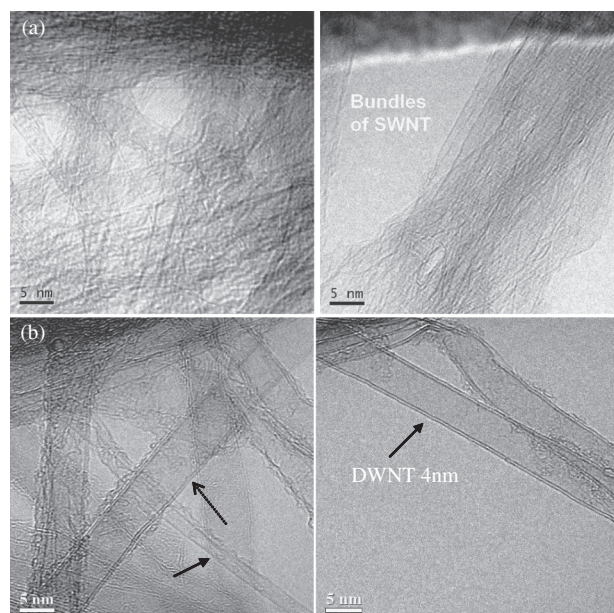


Figure 2. The TEM images of CNT grown at 730 °C On top (a): mostly SWCNT observed for 0.5 nm Fe of catalyst; diameter range between 2.8 and 4.5 nm. On bottom (b): mostly DWCNTs observed for 1 nm Fe catalyst; diameter range between 4 and 6 nm. Some SWCNTs are also observed.

most of the nanotubes observed were DWCNTs which is shown in Figure 2(b) with larger diameter (4–6 nm). The result is important and points out that the same catalyst under the same synthesis conditions (temperature, time, etc.) can have different growth rate and nature of the tubes, respectively. The efficiency of the two catalyst thicknesses can be compared by calculating S_g the surface of graphene deposited for one catalyst particle which is:

$$S_g = 2\pi r_i h N_{\text{wall}} \quad (1)$$

where h is the tube height; r_i is the tube diameter; N_{wall} is the number of walls.

For 0.5 nm catalyst $S_g = 5.9 \mu\text{m}^2$ and for 1 nm catalyst $S_g = 11.8 \mu\text{m}^2$ which is proportional to the catalyst thickness. Thus while the growth rate is twice for the 0.5 nm catalyst its efficiency is half which is consistent with the smaller amount of carbon species decomposed on the surface of the smaller catalyst particle. This results show the formation of high quality SWCNTs and DWCNTs for two different thicknesses of catalysts at fixed temperature (730 °C) and low pressure (0.3 T). Hence in order to achieve the generalised super growth at low temperature, we have kept the pressure (1 T) constant which is able to achieve the maximum height of the carpet.

3.2. Effect of Temperatures for Carpet Growth

An important parameter in the growth of carpets is the temperature. It is generally accepted that temperature affects the structure of CNTs since catalytic activity and

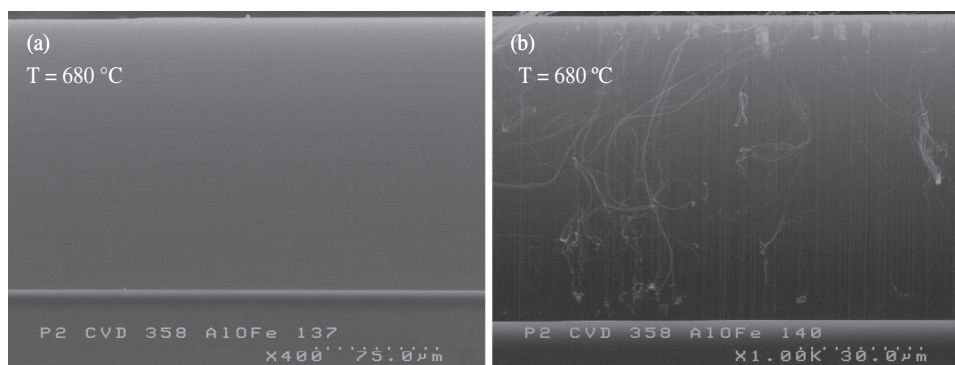


Figure 3. SEM images of carpet grown at temperature 680 °C (a) 0.5 nm Fe $h = 160 \mu\text{m}$, (b) 1 nm Fe $h = 80 \mu\text{m}$.

rate of decomposition of carbon containing molecules are significantly influenced by temperature. SEM image which is shown in Figure 3 clearly represent the height of carpets and the results were also tabulated in Table II. The height of the carpets grown at 680 °C within short reaction period of time (10 min) under constant pressure (1 Torr) using 0.5 nm Fe and 1 nm Fe, respectively were 165 and 75 μm .

In this study, we have reduced the temperature upto 530 °C. We find that there is no carpet growth at 530 °C even though the surface of the growth substrate is blackened by carbon due to the early termination of the catalyst, probably due to the overwhelming amount of carbon that is being introduced to a catalyst particle that can not support such a rapid growth rate. Growth of carpets start at the temperature in the neighbourhood of 580 °C at which there is enough mobility of the catalyst atoms to enable the diffusion of carbon through the particle and to initiate the growth of well aligned nanotubes. The mobility of carbon in iron increases with temperature since the diffusion coefficient is highly dependent on temperature. However at 580 °C and above, the carpet height increases in temperature up in anticipation of a crucial temperature is reached at 680 °C (Fig. 4), where a further increase in temperature leads to a sudden decrease in carpet height which coincide with reaction modelling.²⁷ This decay in

carpet height has been attributed to the chemical reaction between catalyst and substrate, which caused the catalyst being poisoned as a consequence, reduced the catalyst life time during CNT growth. Thus when compare with previous reports,^{15, 18, 24, 25} we have obtained maximum carpet height at 680 °C under lower pressure.

In this study, we noticed that the height of the carpet is doubled for 0.5 nm Fe when compared with 1 nm Fe thickness catalyst as already discussed in previous section. This ratio is independent on the growth temperature as described in Figure 4. An increase in the thickness of the catalyst layer causes a decrease in the CNT growth rate, because the CNT growth rate is inversely proportional to the catalyst thickness.

3.3. Effect of Catalysts Thickness

We have carried out experiments exploring how the catalyst thickness and the low growth temperature (630 °C) affect the nanotubes under constant pressure (1 T) for a reaction period of 10 min. The heights of CNTs grown at 630 °C for 0.37, 0.5 and 1 nm Fe thickness catalysts are 15, 35 and 18 μm , respectively. For the same growth temperature, pressure and time a variation in the height of CNTs was observed for different catalyst thicknesses, which indicates that the efficiency of the CNT growth process depends on the thickness of the Fe layer.^{28–30}

Table II. Represent the height of carpets grown at various temperatures under constant pressure (1 T), time (10 min) and flow rate of gas mixtures ($\text{C}_2\text{H}_2/\text{H}_2/\text{He} = 10/30/50$ sccm) for two different thicknesses of catalysts.

Substrate	Reaction temperature (°C)	Height of CNTs (μm)	Nature and average diameter of CNTs	Density of CNTs (cm^{-2})
0.5 nm Fe on Al_2O_3	580	7		
1 nm Fe on Al_2O_3		5	DW-TW (5 nm)	1.35×10^{12}
0.5 nm Fe on Al_2O_3	630	35	SW (3 nm)	1.0×10^{12}
1 nm Fe on Al_2O_3		18	DW, TW (4.5 nm)	1.17×10^{12}
0.5 nm Fe on Al_2O_3	680	165	SW (3.5 nm)	$\sim 1.0 \times 10^{12}$
1 nm Fe on Al_2O_3		75	DW, TW (5 nm)	$\sim 9.0 \times 10^{11}$
0.5 nm Fe on Al_2O_3	730	100	SW (4.5 nm)	$\sim 8.5 \times 10^{11}$
1 nm Fe on Al_2O_3		53	DW (5 nm)	7.8×10^{11}
0.5 nm Fe on Al_2O_3	780	82	DW-TW (5 nm)	6.9×10^{11}
1 nm Fe on Al_2O_3		53	DW-TW (5 nm)	6×10^{11}

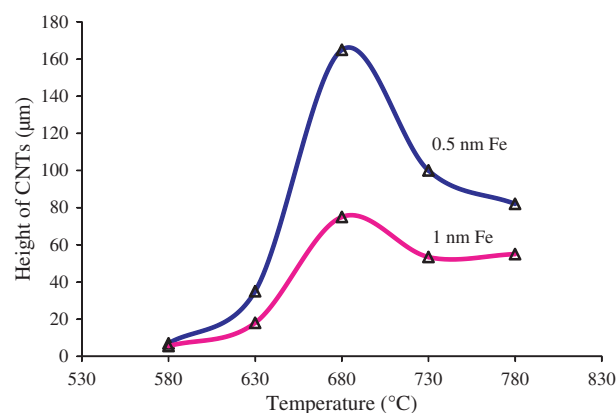


Figure 4. The heights of the carpet as a function of temperature for two different thickness of the catalysts (growth time: 10 min).

Figure 5 shows the TEM observation of CNTs for three different thicknesses of Fe 0.37 nm, 0.5 nm and 1 nm catalyst layer. With 0.37 nm, we found that most of the nanotubes are single walled nanotubes with diameters 2.5 nm Figure 5(a). The TEM images of CNTs grown from a 0.5 nm Fe catalyst layer are shown in Figure 5(b), which indicates that the formed nanotubes are mostly DWCNTs (dia ~ 4 nm) with low ratio of SWCNTs (dia ~ 3 nm).

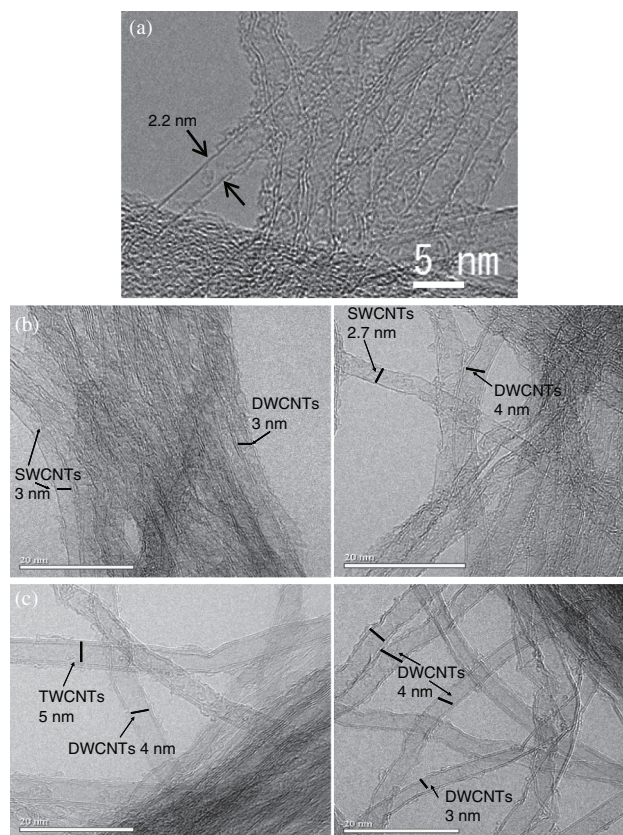


Figure 5. The TEM images of SWCNTs and DWCNTs observed for the three thickness of catalyst layer at 630 °C (a) 0.37 nm Fe, (b) 0.5 nm Fe and (c) 1 nm Fe.

The TEM image of CNTs grown from a 1 nm Fe catalyst layer is shown in Figure 5(c), which indicate that most of the nanotubes are DWCNTs (dia ~ 4 nm) and some isolated TWCNTs with diameter in the range ~ 5 nm are also obtained. If we have thicker catalyst layers (1 nm Fe) will lead to larger catalyst particles on the surface which initiates the growth of larger diameter of carbon nanotubes. Since catalyst size directly affects the nanotube diameters^{31–33} as a result, there is a mixture of double and triple walled carbon nanotubes present in the carpet. We have confirmed from this study, when the catalyst layer thickness increases from 0.37 to 1 nm, the number of walls of the synthesized nanotubes increases from 1 to 4, which confirms the direct relationship between the number of walls and catalyst thickness. To obtain pure SWCNT material we need thinner catalyst layer (0.37 instead of 0.5 nm) at low temperature when compared with high temperature (730 °C) growth. Without addition of growth enhancer, rapid growth of SWCNTs using the thinner layer of Fe catalysts (around 0.37 nm) have been successfully done under optimized conditions, i.e., low pressure and the low growth temperature.

3.4. Effect of Temperatures on the Carpet Density

A method similar to that of previous reports^{34–37} was utilized in the present study. The densification was done by immersing the substrate with aligned CNTs into a beaker of isopropyl alcohol (IPA) for 24 hrs. It was then dried naturally in air at room temperature until IPA completely evaporates. The CNT after the densification process was observed under a SEM. The SEM image which clearly illustrate in Figure 6, which represents the compacted CNT for density measurement. The density of as grown CNTs (ρ) is determined by using the formula below:

$$\rho = \frac{2F}{\sqrt{3}(d + \delta)^2} \quad (2)$$

Where F is the measured filling factor using isopropanol technique;³¹ d is the average tube diameter measured by TEM; δ is the tube distance after compaction (taken to be 0.91 nm).^{32,33}

We observed for 1 nm catalyst thickness that the density decreases monotonically when growth temperature increases as presented in Figure 7. The result presented for the density measurement is accurate and well defined. At low temperature (580 °C) very good density $1.4 \times 10^{12} \text{ cm}^{-2}$ are obtained. The observed decrease in density with temperature is not related to the tube length so as expected there is no correlation between CNT nucleation which probably govern the density and the life time of the catalyst which govern the length. The same densities are obtained at 630 °C with 1 nm and 0.5 nm catalyst thicknesses taking into account the tube diameter shrinkage (see Table II). For 1 nm Fe catalyst, the nucleation

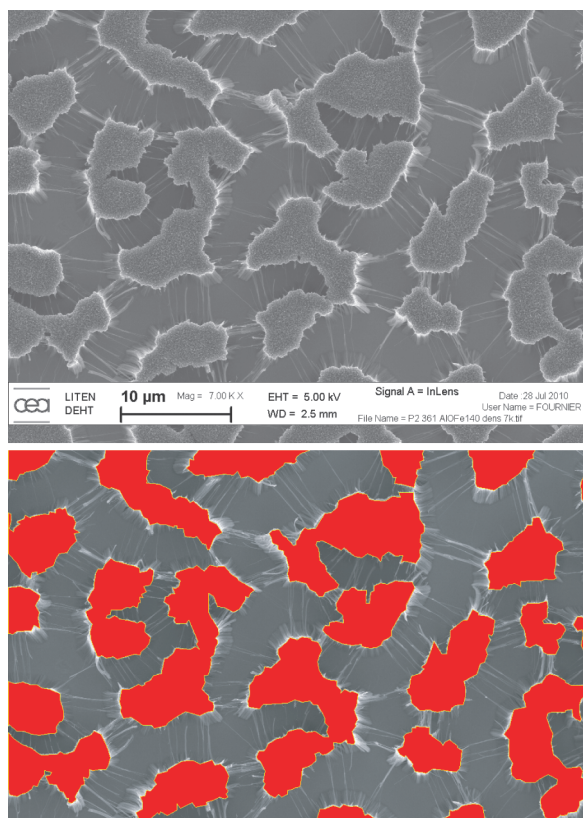


Figure 6. Compacted CNT for density measurement. The illustrated image is for 1 nm Fe catalyst at $T = 580$ °C, growth time: 10 min. On top: carpet after isopropanol. On bottom: The filled red surface is the surface of the densified tube. The filling factor is the ratio between the filled and the total surface $T = 580$ °C 1 nm Fe.

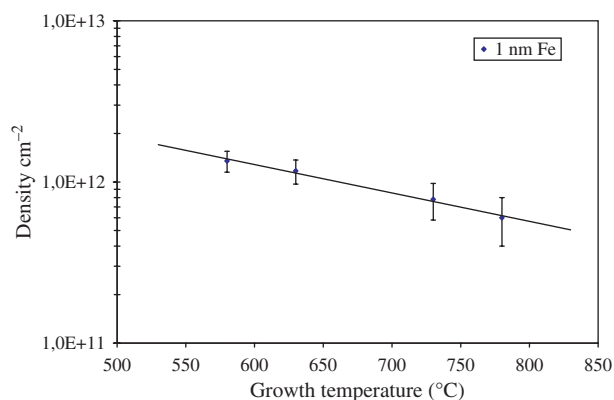


Figure 7. Tube density evolution versus growth temperature for 1 nm Fe catalyst.

density seems to be decreased while temperature increased and also the temperature is not influenced on tube diameter.

4. CONCLUSIONS

We have demonstrated a simple and effective low temperature process for the growth of highly efficient CNT on

a various thickness of catalyst on substrate and also how the growth parameters affect the synthesis of SW and DW using the CVD method. In the first case, we emphasize that the gas pressure and growth temperature dictate the quality and height of SWCNTs and DWCNTs formed as the final structure of the product. Here, without the addition of H_2O or any other growth enhancer we have greatly synthesised SWCNTs and DWCNTs (dia ~ 3 – 4 nm) at 730 °C and at very low pressure (0.3 T) which have similar growth rate as the H_2O -assisted process. Second, we obtained an interesting result at low temperature 630 °C for the catalyst (0.37 nm Fe thickness) gives mostly SWCNTs with smaller diameter (2.5 nm). We have confirmed from this study, when the catalyst layer thickness increased from 0.37 to 1 nm, the number of walls of CNTs also increased.

It is clear from these results we have obtained “generalised supergrowth” of SWCNTs and DWCNTs that to within short period (10 min) at low temperature. We believe that these methods produce the CNTs without the requirement of water or any other growth enhancer. Whereas, supergrowth is not limited to ethylene and water, but the concept can be expanded to a “generalised supergrowth” (without any growth enhancer). Consequently, we believe that the generalised growth of CNTs without any growth enhancer may act as guide to inspire the challenge for future research, which is to improve the growth effectiveness and specifically control the location and orientation of these long nanotubes.

For the first time we reliably evaluated the density versus different temperature. From these result we confirm that high density materials need low temperature growth process and that our materials are at the state of the art concerning the achieved density.

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