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Horizontally aligned single-walled carbon nanotubes can bridge wide trenches and climb high steps

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ABSTRACT

The direct growth of horizontally aligned single-walled carbon nanotubes (HA-SWNTs) on the two basic structures of future SWNT-based nanodevices, *i.e.*, trenches and steps, were demonstrated. The HA-SWNTs were found to be able to bridge trenches of varying widths (up to 1000 μm across) and to climb steps of varying heights (up to ∼1500 μm high). Observations of HA-SWNTs with periodical "beeline-curve-beeline" morphologies and HA-SWNTs which grew along step walls when climbing steps indicate that the popular "tip-floating" growth mechanism for HA-SWNTs should be modified because it is not always suitable for our HA-SWNTs. The consequence of HA-SWNTs being able to bridge wide trenches and to climb high steps suggests a promising way to fabricate nanodevices by directly integrating HA-SWNTs into three-dimensional device structures.

1. Introduction

The synthesis, characterization and applications of single-walled carbon nanotubes (SWNTs) have been widely studied due to their outstanding properties and promising applications in various fields [1–5]. Many prototypes of SWNT-based devices have been prepared using a fabrication methodology involving the assembly of the desired structures onto the pre-synthesized SWNTs [6–10]. Although this construction method was relatively easily accomplished and was shown to be effective, the device fabrication process itself can induce unwanted damage to the SWNT structure and so change its surface states. So this method of building device structures onto pre-synthesized SWNTs was, to some extent, disadvantageous in achieving the intrinsic performances of SWNTs. Moreover, this method introduces highly undesirable contaminants to the fabrication equipment, in particular metals, which are due to the residual metallic catalysts in the SWNTs [1,11].

An alternative approach to fabricating SWNT-based nanodevices is to directly integrate the SWNTs into the prefabricated device structures. This method would reduce the damage or changes to the SWNT structure or surface states, should decrease the chances of SWNTs contaminating the fabrication equipment and consequently be more advantageous for obtaining the required intrinsic properties of the SWNTs. In comparison to randomly aligned SWNTs or vertically aligned SWNTs [12], the use of horizontally aligned SWNTs (HA-SWNTs) on substrates would make it easier to isolate a SWNT with the desired diameter or chirality, make contacts on SWNTs, and improve device performance [13]. Although the growth of HA-SWNTs on various flat substrates and/or their performance on spanning gaps have been reported by several groups [14–23], their abilities to bridge trenches and, especially, to climb steps have not been specially investigated and are not fully understood. Because trenches and steps are the two basic structures for a variety of devices [24,25], information regarding the ability to bridge trenches or climb steps would be important for the design and manufacture of SWNT-based nanodevices via the direct integration method discussed above. Moreover, the growth mechanism of HA-SWNTs is still not fully understood, although several mechanisms have been suggested [16,26–29].

This paper carefully studies the ability of HA-SWNTs to bridge trenches and climb steps. The results show that HA-SWNTs can grow across trenches as wide as 1000 μm and climb up steps as high as about 1500 μm and continuously grow, while still maintaining their alignments, after having bridged the trenches or climbed the steps. Based on our observations, we suggested that the "tip-floating" growth mechanism of HA-SWNTs should be modified. A "tip-floating, falling-uprising" growth mechanism was proposed for our HA-SWNTs. Our results will provide more opportunities for the design and fabrication of SWNT-

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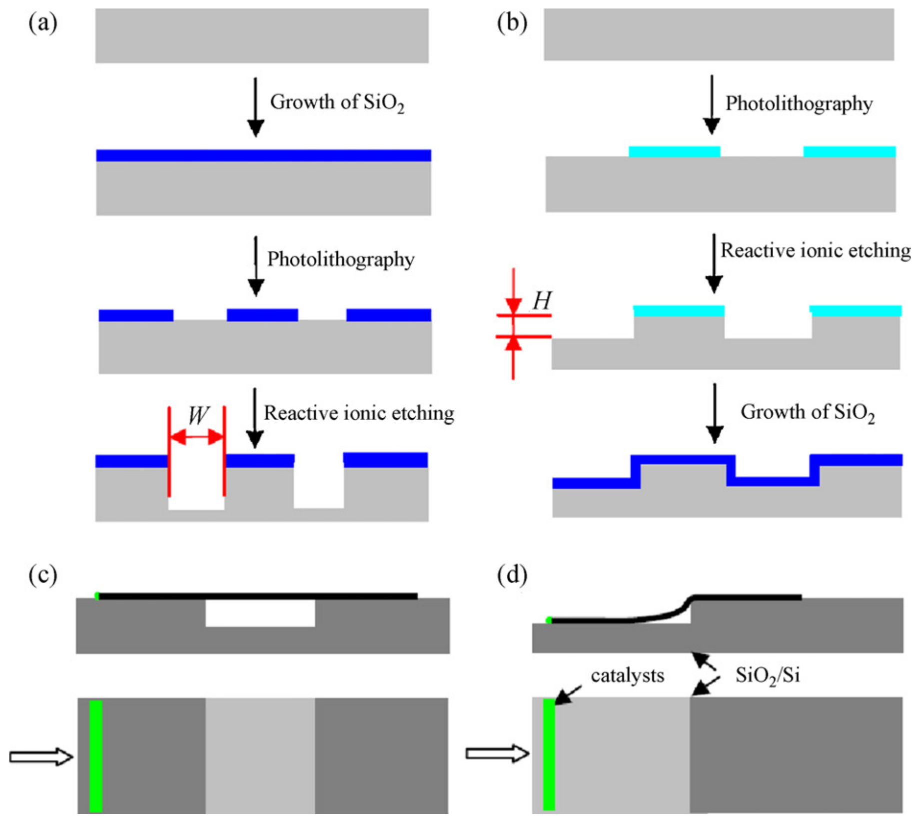

Fig. 1. Schematic show of the processes for fabricating (a) trenches and (b) steps on SiO_2/Si substrates and the relationships among trench (or step), catalyst strip and gas flow for investigating the ability of HA-SWNTs in (c) bridging trenches and (d) climbing steps. The trench width, *W*, was defined by the mask for photolithography. The depth of the trench is 200 μm. The step height, *H*, was dependent on the reactive time of RIE. The arrows show the gas flow directions.

based devices, such as nanoresonators, using the direct integration method.

2. Experimental methods

The trenches/steps with varied widths/heights were preformed on SiO_2/Si substrates using micromachining technologies, prior to SWNT growth. For producing trenches, a 500–1000 nm thick SiO_2 layer was thermally grown on a 4 in. Si wafer. The trenches were then formed with photolithography technologies followed by the reactive ionic etching (RIE) using the SiO_2 layer as the etching mask (Fig. 1a). The width of the trench, *W*, was varied from 5 to 1000 μm. The trench depth which depends on the RIE etching time was set to be 200 μm to avoid the HA-SWNT sticking to the bottom of the trenches during the growth of HA-SWNTs.

Fig. 1b shows schematically the processes for producing steps. A 4 in. Si wafer was patterned with photolithography followed by etched with RIE to form steps using photoresist as the etching mask layer. After removing the photoresist, a 200 nm thick SiO_2 were thermally grown on the wafer. The step height, H, which depends on the reactive time of RIE, was ranged from 1 to 120 μm. After those processes, the wafers with trenches or steps were diced to chips as substrates for growth of HA-SWNTs.

HA-SWNTs were grown by chemical vapor deposition (CVD), using ferritins as the catalyst source, employing similar processes described in our previous paper [30]. Several drops of 93 μg/ml ferritin aqueous solution were locally deposited on one side of a substrate with a trench, or on the bottom surface of a substrate with a step, as is schematically shown in Fig. 1c and d, respectively. Then the substrate was placed at the center of the quartz tube, with the catalysts at the windward site and the trench/step being perpendicular to the gas flow direction. The catalysts were first calcined in pure air at 800 °C for 10 min. Then HA-SWNTs were grown at 900 °C, with gas flows of 50 sccm CH_4 and 100 sccm H_2, for a typical growth time of 90 min. Subsequently, the samples were cooled and characterized by scanning electron microscopy (SEM, Hitachi, S-4700).

3. Results and discussions

3.1. HA-SWNTs bridge trenches

Fig. 2 shows the typical SEM images of the HA-SWNTs grown on SiO_2/Si substrates with trenches of 5, 10, 20, 40, and 63 μm widths, respectively. It can be seen that the HA-SWNTs, which are well aligned along the gas flow direction, have grown across the trenches. Following this, they grew continuously while still maintaining their alignment in the same direction as they did prior to meeting the trenches. The obvious differences in the widths of the trenches did not significantly influence the growth of the HA-SWNTs. Closer observations of the HA-SWNTs grown across the 10 and 40 μm wide trenches having rectangular and reversed-trapezium cross sections respectively, are shown in the insets of Fig. 2. These clearly show that the HA-SWNTs were suspended over the trenches, despite the trench cross section shape, proving that HA-SWNTs are able to bridge trenches of varied widths. All of the HA-SWNTs which had grown across the trenches were confirmed, by SEM observation, to have bridged those trenches.

To further investigate the ability of HA-SWNTs to bridge trenches, we again increased the width of the trench for several similar experiments. Fig. 3 shows the SEM images of the HA-SWNTs grown on the SiO_2/Si substrates with trench widths varied from 200 to 1000 μm. From these images, we can see that the HA-SWNTs, which have grown across the trenches, were suspended

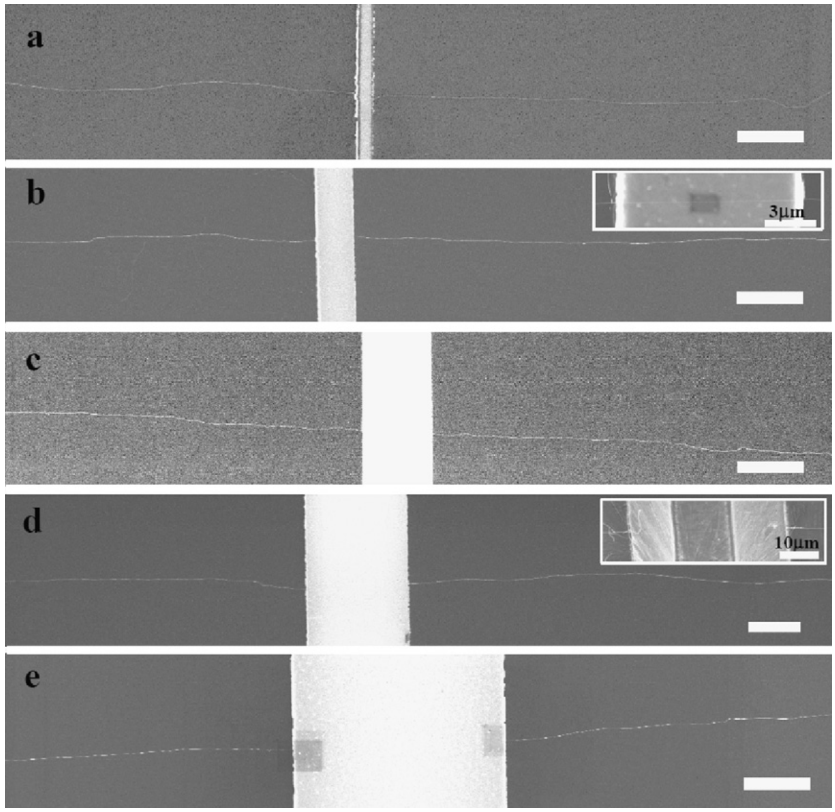

Fig. 2. SEM images of HA-SWNTs grown on SiO_2/Si substrates with different trenches. The trench width was: (a) 5 μm, (b) 10 μm, (c) 20 μm, (d) 40 μm and (e) 63 μm. Scale bars: 20 μm. Insets show local SEM images of HA-SWNTs suspended on a 10 and 40 μm trench with the rectangle and reversed-trapezium cross section, respectively.

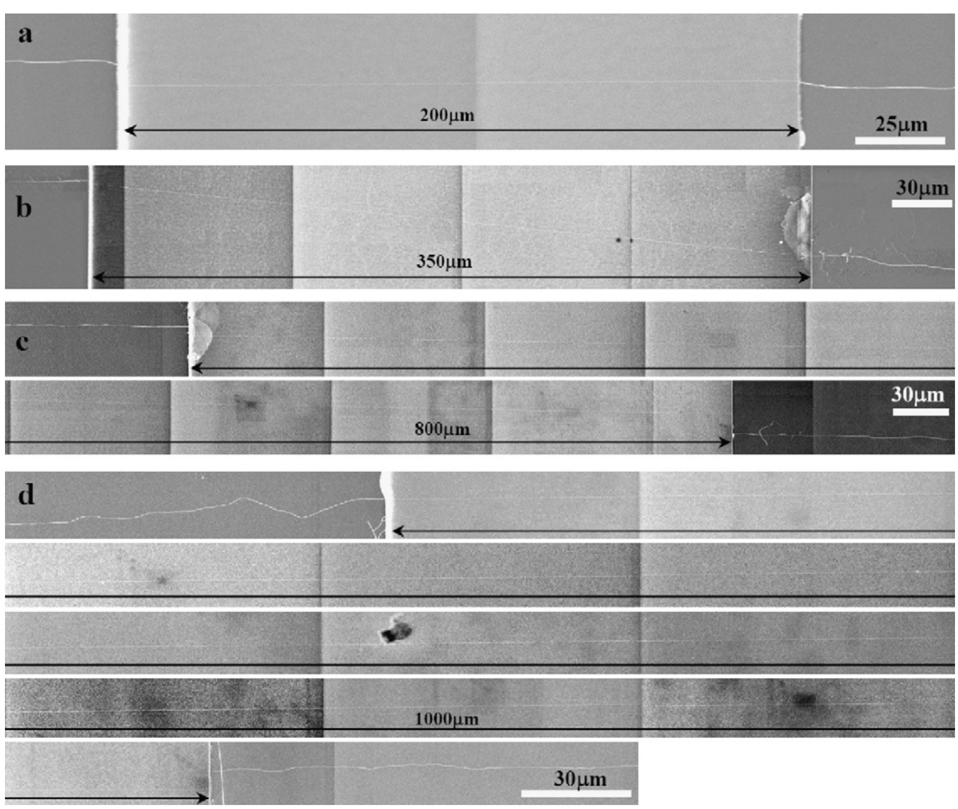

Fig. 3. SEM images of the HA-SWNTs which have bridged different wide trenches on SiO_2/Si substrates. The trench width was: (a) 200 μm, (b) 350 μm, (c) 800 μm, and (d) 1000 μm. Each panel was assembled using several SEM images taken continuously along the SWNT bridge with a higher magnification to clearly discern the SWNT bridge on the trench.

on those wide trenches without exception, even for the 1000 μm wide trench. The depth of the trenches, 200 μm, also prevents the HA-SWNTs from sticking to the bottom of the trenches even when the elastic deformations of the very long SWNT bridges are taken into consideration.

3.2. HA-SWNTs climb steps

The performance of HA-SWNTs in climbing steps, which is another basic feature required in future SWNT-based nanodevices, was also investigated. Fig. 4 shows the typical SEM images of the HA-SWNTs grown on the SiO_2/Si substrate with step heights of 2, 4 and 8 μm, respectively. Before meeting a step, the HA-SWNTs initially grew on the bottom surface of the step because the catalysts were deposited there. It can be seen that steps on the substrates were no obstacle in preventing the highly "adaptable" HA-SWNTs in growing forwards. The HA-SWNTs, rather than halting their growth or growing only at the bottom surfaces of the steps, climbed all the steps regardless of their heights, leaving a section of each HA-SWNT suspended between the bottom and the upper surfaces of the steps. The lower magnification SEM images show that the HA-SWNTs also continue growing while maintaining their alignments, after having climbed the steps.

To further investigate the ability of HA-SWNTs in climbing steps, the heights of the steps were again increased by increasing the reactive ionic etching time to the SiO_2/Si substrates. The SEM images of the HA-SWNTs synthesized on the SiO_2/Si substrates with 30, 80 and 120 μm high steps, shown in Fig. 5, clearly show that the HA-SWNTs can climb up all the steps, demonstrating their strong ability in climbing over obstructions in their growth paths.

With the motivation of investigating how high a step the HA-SWNTs can actually climb, the step height was further increased by adding one or more SiO_2/Si substrates onto a flat SiO_2/Si substrate to form a step with a height equal to the total thickness of the added SiO_2/Si substrates. Then HA-SWNTs were grown from catalysts deposited on the undermost SiO_2/Si substrate. After the growth experiments, the top SiO_2/Si substrate surfaces were observed by SEM to investigate whether the HA-SWNTs could climb up such high steps. Fig. 6 shows the SEM images of the upmost SiO_2/Si substrate surfaces after the HA-SWNT growth process and with adding 1, 2 and 3 SiO_2/Si substrates onto a SiO_2/Si substrate, respectively. The thickness of each SiO_2/Si substrate was about 500 μm, so the heights of the steps were about 500, 1000 and 1500 μm, respectively. The SEM images show that HA-SWNTs can be observed on all three substrates, demonstrating that at least some HA-SWNTs, grown from the base SiO_2/Si substrates, can climb up the high steps and keep continuously growing on the top SiO_2/Si substrates even when the step was ∼1.5 mm high.

3.3. Growth mechanism of HA-SWNTs

Although the growth of HA-SWNTs, without any external forces, has been reported by several groups [14–16,26,29,31–35], the growth mechanism of such HA-SWNTs is still not fully understood. The mechanisms proposed for HA-SWNTs directed by external forces [17–21] are not applicable for our HA-SWNTs. The "tip-

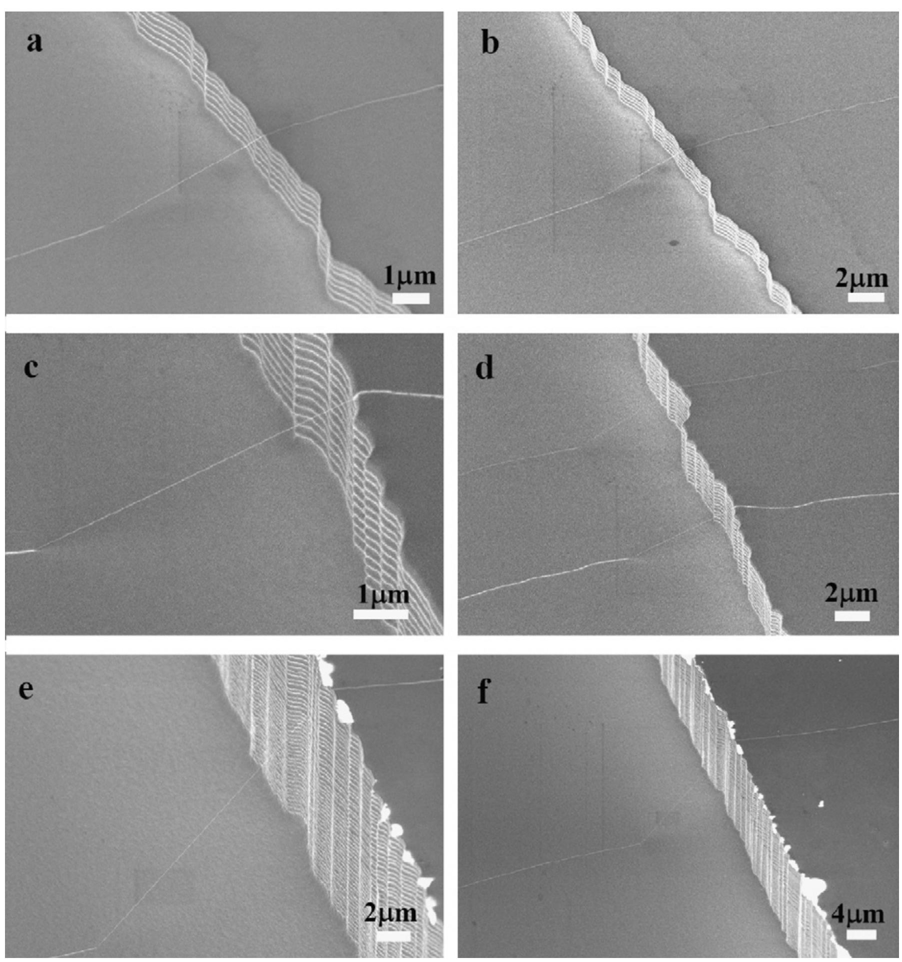

Fig. 4. SEM images of the HA-SWNTs grown on the SiO_2/Si substrates with micromachined steps. The step height was: (a, b) 2 μm, (c, d) 4 μm, (e, f) 8 μm. (b), (d), (f) are lower magnification images of the same HA-SWNTs in (a), (c), (e), respectively.

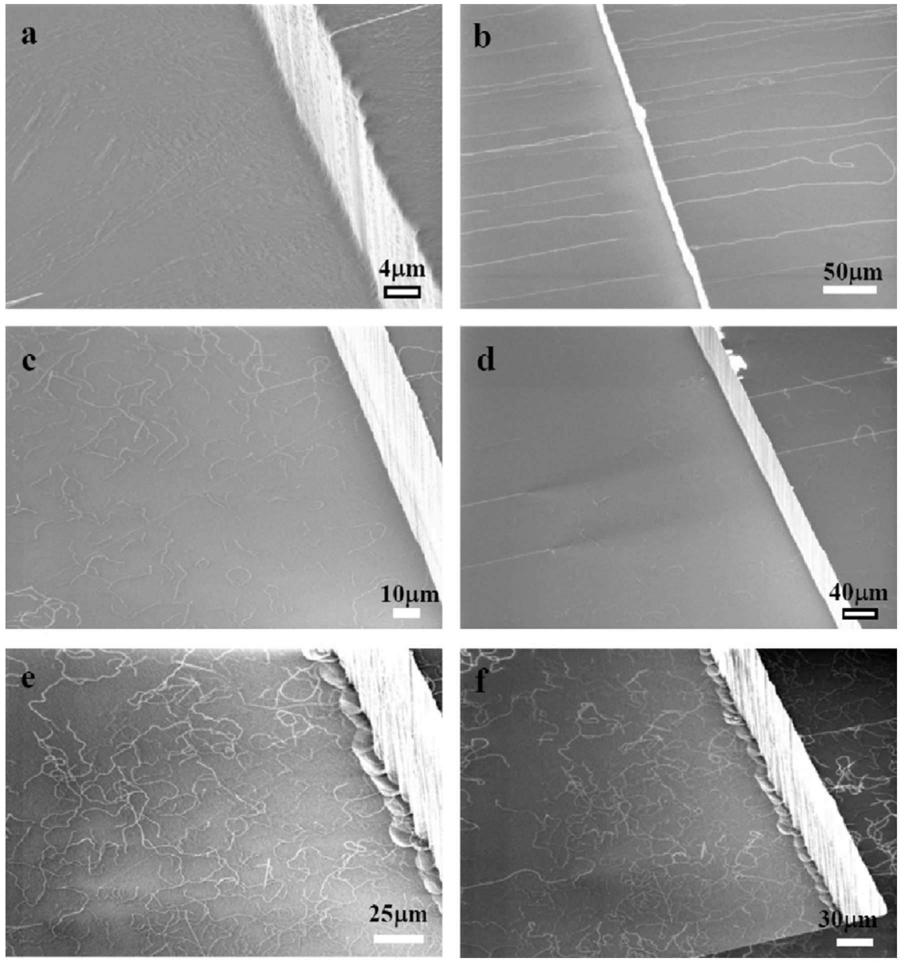

Fig. 5. SEM images of HA-SWNTs grown on the SiO_2/Si substrate with a step of (a, b) 30 μm, (c, d) 80 μm and (e, f) 120 μm in height, respectively. (b), (d), (f) are lower magnification images of the HA-SWNTs in (a), (c), (e), respectively.

floating" growth mechanism suggested by several groups was popular in interpreting the production of HA-SWNTs directed by only gas flows with "fast-heating", "ultralow flow speed" and "laminar flow" growth techniques. Huang et al. [26] suggested that the catalysts of the HA-SWNTs can be elevated by a high speed CO gas flow when they were fast heated to 900 °C. This mechanism is not applicable for our HA-SWNTs because the "fast-heating" technique was not adopted in our experiments. Li and co-workers [16] suggested that both the catalyst in the front tip of a HA-SWNT and the downstream section of the HA-SWNT were floating in the gas environment during the growth of HA-SWNTs, when using very low speed gas flows. The force required to lift the catalyst and the section of HA-SWNT was attributed to the "buoyant effect" which is believed to be generated due to the existence of a temperature gradient between the quartz tube inner wall and the substrate. Unfortunately, this mechanism was also shown to be unsuitable for our HA-SWNTs after we performed simulations in the gas environment of our growth conditions (CH_4 50 sccm and H_2 100 sccm). The results given in Fig. 7 show that the temperature difference between the quartz tube inner wall and the substrate at the center of the quartz tube was less than 0.01 K. This very small temperature gradient may not generate enough of a "buoyant effect" to lift

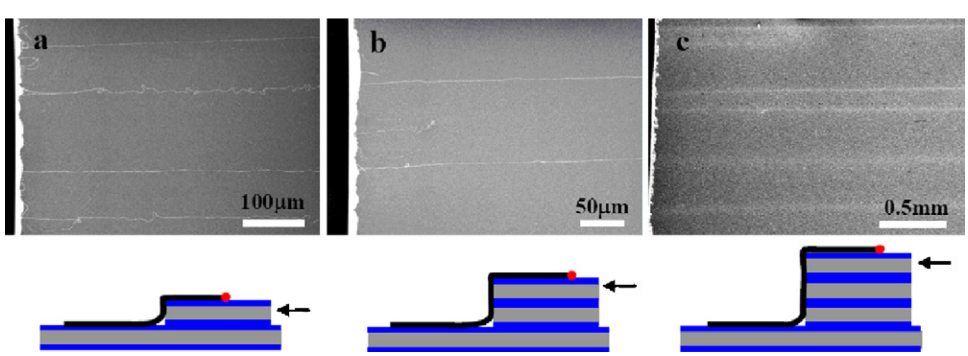

Fig. 6. SEM images of the HA-SWNTs on the upmost SiO_2/Si substrates after the growths of HA-SWNTs with adding (a) 1, (b) 2, (c) 3 substrates onto a substrate, respectively. The schematic diagrams beneath each SEM image schematically show the substrate configuration for each growth experiment. HA-SWNTs were grown from the catalysts deposited on the base substrates. The arrows point the substrates observed with SEM.

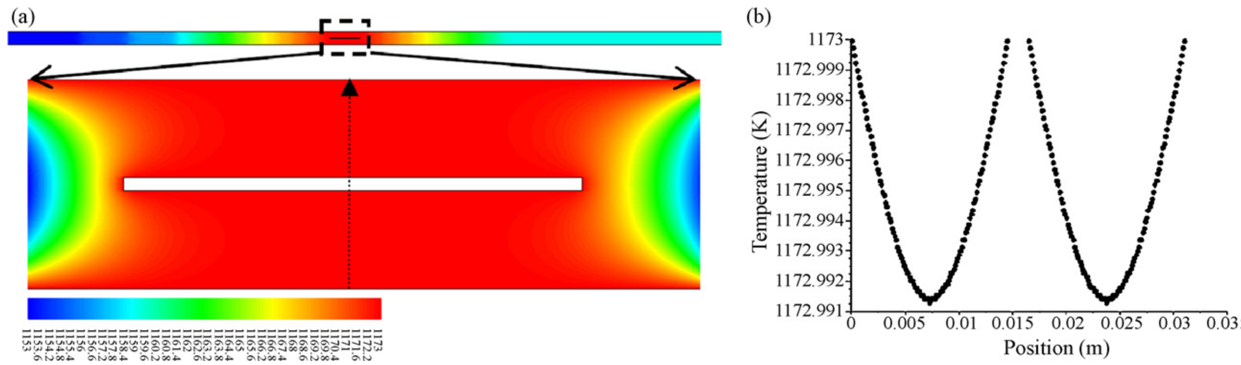

Fig. 7. Simulation results of (a) temperature distribution in the entire quartz tube. The difference in colors displays the difference of temperatures, as shown in the temperature bar, and (b) temperature distribution along the arrow shown in (a). The arrow direction also shows the increasing direction of the "Position" axis. The simulation was carried out with a computational fluid dynamics software using our growth conditions.

a section of a HA-SWNT together with a catalyst. Huang et al. [29] suggested that a HA-SWNT was first lifted above the substrate and was then caught entirely by a laminar flow of feeding gas. However, the reason why the HA-SWNTs would be lifted was not proposed.

We suggest that, at the early growth stage of a HA-SWNT, its preferential growth direction would be vertical to the substrate surface, in an environment of a laminar flow with low speed [29]. According to the most accepted vapor–liquid–solid (VLS) growth mechanism of carbon nanotubes, carbon species should go through an adsorption–diffusion–precipitation process, before forming new SWNT structures. Because the bottom surface of a catalyst is in contact with the substrate, it has relatively less probability of absorbing a carbon species, compared with the upper surface. The carbon concentration in the base of the catalyst would always be the lowest of the whole catalyst and the carbon atoms would not precipitate from the catalyst until the under surface of the catalyst was saturated with carbon. According to the "tip-growth" mode, which is the most reasonable growth mode for HA-SWNTs [16,24,25], the carbon atoms will preferentially precipitate from the undersurface of the catalyst when the whole particle was saturated with carbon. Therefore, a HA-SWNT will tend to grow in a direction vertical to the substrate in its initial growth stage in a growth environment with little or no disturbance from the reactive gas flows. That is also one of the reasons why the vertical aligned SWNTs tend to be synthesized in a growth environment with a low pressure and low speed gas flow [36–38].

After the tips of such HA-SWNTs were lifted off the substrate surface, the HA-SWNTs grow quickly (>3 μm/s) after their nucleation. The "tip-floating" growth mechanism was also suggested to be a reasonable mechanism for our HA-SWNTs. The fact that HA-SWNTs can grow across trenches as wide as 1000 μm and grow up steps with a height of about 1.5 mm strongly indicates that the downstream part of each HA-SWNT could float in the gas flows during the CVD growth. This is because it is impossible for a HA-SWNT to bridge wide trenches and climb high steps if it only slides on the substrate surface, during its growth. In addition, our results which show the growth of well aligned HA-SWNTs on very rough SiO_2/Si substrate surfaces (Fig. 8) further excludes the slide-only growth mechanism for our HA-SWNTs. Despite suggestions by Huang et al. [29] we do not believe that the entire length of the HA-SWNT would float in the gas during its growth. It is difficult for very long HA-SWNTs (which can be more than 16 mm long after 90 min of growth) to float entirely and simultaneously in the gas during the growth and leave behind long parallel HA-SWNTs on the substrate surface when growing ends. This is because the interactions between all the two neighboring super-long HA-SWNTs, or even some local micro-disturbances of the gas flow, can easily make them attach together to form SWNT bundles.

However, we found that the "tip-floating" mechanism was not always suitable for our observations. Except the observations of very straight HA-SWNTs synthesized on the substrate surface, as shown in above figures, we always observed some HA-SWNTs which have very different periodical "beeline-curve-beeline" morphologies on the substrate surface, as typically shown in Fig. 9a. Such morphologies could not only be observed on SiO_2/Si substrates but also on sapphire substrates (Fig. 9b). These observations are difficult to explain using the "tip-floating" growth mechanism, because a HA-SWNT will always leave a straight line (or nearly so) on the substrate surface with the "tip-floating" growth mechanism.

In addition, some observations of HA-SWNTs climbing steps are also hard to explain with only the "tip-floating" mechanism. Fig. 10 shows that, when climbing steps, some HA-SWNTs seem to grow along the step walls (Fig. 10a) or initially climb a part of the step and then grow along the step wall to finish climbing the whole step (Fig. 10b). One may consider that these cases can be attributed to the adhering of a section or all of the suspended SWNT sections to the step walls due to the van der Waals force between the two sides. However, this may inevitably cause the elastic or plastic deformation of the SWNT. Because of the high Young's modulus of SWNT [39], the van der Waals force may not be high enough to make the SWNT stick to the step wall.

Those observations indicate that the "tip-floating" mechanism may need to be modified for growth of HA-SWNTs. It is suggested that, during the entire growth period of HA-SWNTs, not all HA-

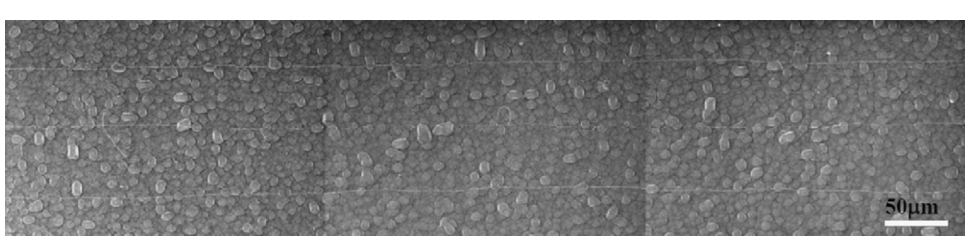

Fig. 8. SEM images of very long HA-SWNTs synthesized on a rough SiO_2/Si substrate. The figure was assembled using three continuously taken SEM images.

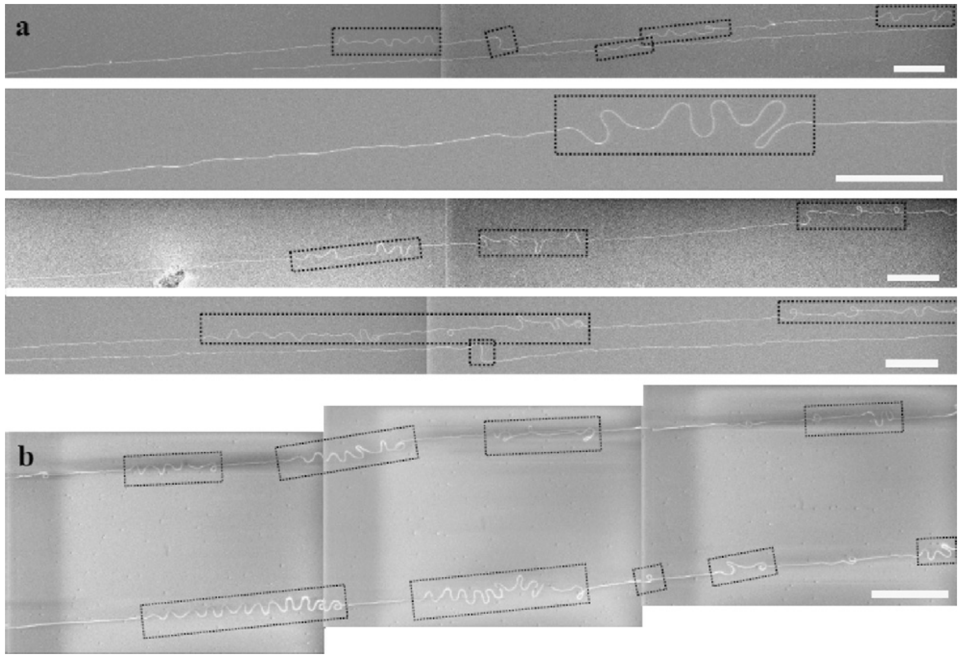

Fig. 9. SEM images of the HA-SWNTs, grown on (a) SiO_2/Si substrates and (b) a C-plane sapphire substrate, with "beeline-curve-beeline" morphologies. Scale bars: 50 μm. The rectangle boxes highlight the curved parts of HA-SWNTs.

SWNT tips can remain floating in the gas. Except those HA-SWNTs which can always be caught by the laminar gas flow in the entire growth period and leave straight HA-SWNT lines on the substrate after the growth termination, some HA-SWNTs may not be very stable while floating in the gas and it could be easier for them to descend to the substrate from their floating status, under the influence of some local micro-disturbances. When that happens, the HA-SWNT may continuously grow with the catalyst sliding on the substrate surface, resulting in the curved track behind the catalyst (marked with box in Fig. 9). If the floating section of a SWNT fell down to the substrate surface with its final length on the surface being shorter than its initial floating length, it will also form the curved SWNT on the surface. At some time, the catalyst may be lifted again, and then the HA-SWNT would be caught again by the gas flow, leaving again a straight HA-SWNT line behind the catalyst. The repetition of such a falling-uprising process, during the growth of such HA-SWNTs, would result in the periodical "beeline-curve-beeline" morphologies on a substrate (Fig. 6 and Fig. S6). When such HA-SWNTs meet steps during their growth, the observations similar to Fig. 10 are explainable. When a HA-SWNT encounters a step, while its catalyst is sliding on the substrate surface, it will grow along the step wall, as shown in Fig. 10a. If it meets a step with a

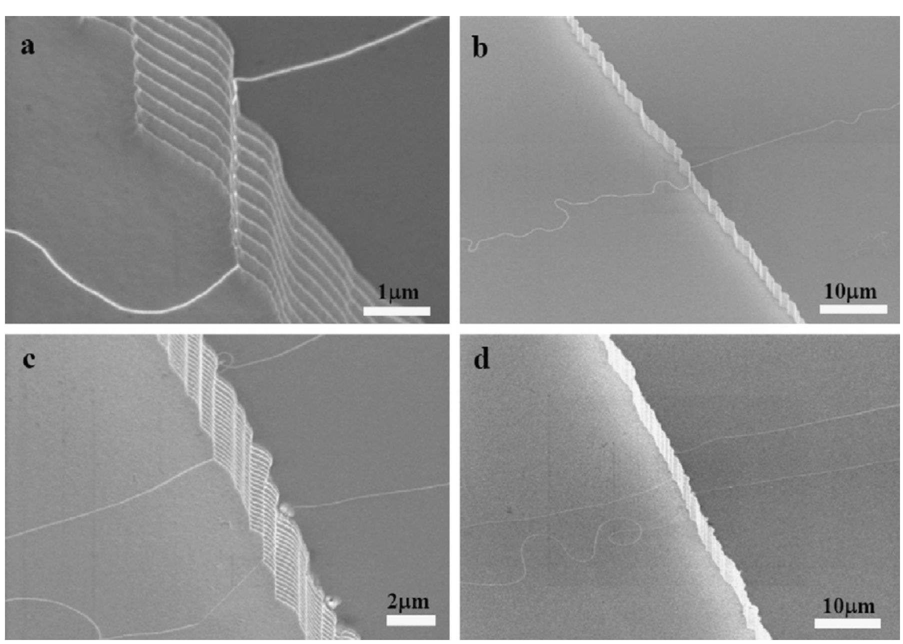

Fig. 10. SEM images of (a, b) a HA-SWNT which has climbed a 3 μm high step by growing along the step wall, and (c, d) two HA-SWNTs which have climbed a 6 μm high step with the mode of growing along the wall and the mode of first climbing part step followed by growing along the wall. (b), (d) are the lower magnification images of the HA-SWNTs in (a), (c) respectively.

height higher than the elevated height of its tip, it will first climb a part of the step and then grow along the step wall, resulting in the morphology similar to Fig. 10b. However, we think that a further investigation on the source for the possible re-lifting of the tips of such HA-SWNTs should be done.

4. Conclusions

We have demonstrated that the HA-SWNTs show impressive performances in bridging trenches and climbing steps. They are able to bridge trenches as wide as 1000 μm and climb steps with heights of ∼1.5 mm. The popular "tip-floating" mechanism was not always suitable for our observations of HA-SWNTs with "beeline-curve-beeline" morphologies and results of HA-SWNTs in climbing steps. Based on the observations we suggested that some HA-SWNTs may repeat a "falling-uprising" process during their growth. Since the trench and step are two types of basic structures used in practical devices, the attractive performance of HA-SWNTs in bridging wide trenches and climbing high steps is highly advantageous for fabricating future SWNT-based nanodevices through direct integration of HA-SWNTs onto three-dimensional structures.

Acknowledgments

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