## **CARBON NANOTUBE ARRAYS**

This colorized, perspective SEM image shows aligned arrays of single-walled carbon nanotubes grown on a quartz substrate in two sequential cycles of chemical vapor deposition (red and blue), as reported by John Rogers and co-workers. The gold line depicts an electrode contact to the array. The SEM image in the background shows regions of dual and single growth on the left and right, respectively.

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# Improved Density in Aligned Arrays of Single-Walled Carbon Nanotubes by Sequential Chemical Vapor Deposition on Quartz

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Guided chemical vapor deposition (CVD) growth of single-walled carbon nanotubes (SWNTs) on certain crystalline substrates, such as sapphire and quartz, provides an attractive route to linear, horizontally aligned arrays of individual SWNTs.<sup>[1-2]</sup> Such configurations, particularly those formed on quartz, are of interest because of their demonstrated ability for wafer-scale integration into devices, including transistors,<sup>[3–4]</sup> light-emitting diodes,<sup>[5]</sup> sensors, photodetectors, and fully integrated systems, such as transistor radios,<sup>[6]</sup> logic gates,<sup>[7]</sup> and radio-frequency oscillators<sup>[8]</sup> and devices.<sup>[9]</sup> The most well-developed strategies use CVD with narrow, patterned lines of catalyst on singlecrystalline quartz substrates. Detailed experimental and theoretical studies reveal that orientation-dependent van der Waals interactions between the SWNTs and the quartz lattice can explain all observations in this system, including growth results for X, Y, Z, AT, and ST cuts of crystalline quartz and amorphous quartz.<sup>[10]</sup> For ST and AT cuts, the process yields a single, predominantly preferred alignment direction in the SWNTs.<sup>[2,11–15]</sup> When the catalyst lines are oriented perpendicular to this direction, CVD yields aligned arrays, with exceptionally high levels of perfection in alignment and linearity.[16-17]

In many applications, increasing the density of such SWNT arrays, as measured by the number of tubes per unit length perpendicular to the alignment direction, improves the properties of the devices that are formed with them. One means to increase the SWNT density involves simply increasing the density (i.e., the

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latter effect arises partly from a decrease in the alignment forces with increasing diameters of the SWNTs (or bundles of SWNTs). Misaligned SWNTs that grow on the surface can physically block the emergence of aligned SWNTs from the catalyst lines, thereby further frustrating the realization of high densities. Similar phenomena can limit the effectiveness of approaches that optimize the growth conditions to improve the yield of SWNTs from catalyst particles at a fixed density. A different strategy that also involves fixed density relies on reducing the spacing between the lines. However, experiments show clearly that this method is ineffective because SWNTs that originate from one catalyst line cannot pass through another catalyst line (see the Supporting Information). This behavior results from the near-surface growth mode observed in these systems, in which even very slight relief, associated with either with misaligned SWNTs or with catalyst particles, can terminate the growth.<sup>[17,18–19]</sup> As a result, the SWNT density does not depend strongly on catalyst line separation, provided that this separation is substantially less than the average lengths of the grown SWNTs (e.g., between 200 and 500 µm for typical cases). Given these constraints, although high densities of SWNTs can be obtained over small areas in certain circumstances, they cannot generally be achieved with reproducible, area-averaged values much larger than  $10 \text{ SWNTs } \mu \text{m}^{-1}$ ; typical results are often in the range of 5 SWNTs  $\mu m^{-1}$ 

Here, we report a simple route to improve the density of SWNTs that avoids limitations described above. The approach relies on multiple, separate CVD growth cycles on a single substrate, as illustrated schematically in Figure 1. Briefly, the first step involves CVD growth on quartz, using optimized densities of catalyst particles and separations between catalyst lines, as per the discussion above and as reported previously [2,11,16,17] Next, an additional set of catalyst lines is deposited in regions next to the original ones. Another cycle of CVD growth yields SWNTs predominantly from the new catalyst lines, thereby increasing the density of the SWNTs in the arrays. The details of each growth process builds on previously reported procedures. In particular, the substrates consist of ST-cut quartz wafers (Hoffman Materials Inc.) preannealed for 8 h at 900 °C in air. Photolithography and liftoff define catalyst lines (typical widths are 10 µm) that consist of a  $\sim$ 1-Å-thick film of iron deposited by electron-beam evaporation (Fig. 1a). Annealing followed by reduction in

surface-area coverage) of the catalyst particles in each of the

catalyst lines. This approach is effective, but only up to some level

beyond which the growth tends to yield bundles of SWNTs and

multiwalled tubes that involve substantial growth out of the plane

of the substrate and in otherwise unaligned configurations. The

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**Figure 1.** Schematic illustration of the procedures for multiple CVD growth of aligned arrays of SWNTs on quartz. a) The process begins with lithographically patterned iron catalyst lines on annealed quartz. b) CVD growth yields SWNTs that are horizontally aligned on the quartz surface, in a direction orthogonal to the catalyst lines. c–d) Patterned removal of SWNTs in narrow lines parallel and next to the iron catalyst regions followed by deposition and annealing of iron forms a new collection of catalyst particles. e) CVD growth of SWNTs from these new iron catalyst lines creates additional aligned SWNTs, thereby increasing the density. For clarity of illustration, the diagrams here show the SWNTs that result from the first and second growth steps in blue and red, respectively. Multiple cycles of this process are possible.

hydrogen gas forms catalytic iron particles. The CVD process uses ethanol as a carbon feedstock, with growth in a quartz reaction tube (1-in. diameter) at 925 °C under argon and hydrogen gas flow (Fig. 1b). Next, selective exposure of segments of the SWNT grown in this manner to an oxygen plasma creates lines (typical widths are 10 µm) of bare quartz next to the original set of catalyst particles (Fig. 1c). Depositing fresh catalysts in this region (Fig. 1d), followed by a second CVD growth yields a new set of aligned SWNTs, thereby increasing the density by roughly 1.5 times for the case illustrated here (Fig. 1e). For the second growth, the conditions for annealing and reducing the catalyst must be carefully selected to avoid damaging the pre-existing SWNTs. In particular, flushing the reaction chamber with argon gas helps to eliminate oxygen that would otherwise consume the SWNTs during the annealing step. Likewise, diluted (with argon) levels of hydrogen gas avoid degradation during reduction.

Scanning electron microscopy (SEM) and atomic force microscopy (AFM) images presented in Figure 2a show typical results from the scheme of Figure 1. The samples in these cases involve some regions of single growth (i.e., Fig. 1b) and other adjacent regions of double growth (i.e., Fig. 1e). The SEM images are brighter in the double-growth areas (dashed box in yellow, including both first and second catalyst lines), resulting from the increased density of SWNTs compared to the single-grown areas (dashed box in blue, including first catalyst only). We found that the yields in the second growth were highest when the second catalyst lines did not overlap the first. Improved processes for removing material (i.e., unreacted catalyst and tubes) from the first lines might avoid this limitation. AFM images (insets:  $5\,\mu\text{m} \times 5\,\mu\text{m}$ ) confirm that the densities of SWNTs in the double-grown areas are approximately 1.5-2 times higher than in the single-grown areas, as expected based on the simple



**Figure 2.** SEM and AFM images of single and double growths of SWNTs on a quartz substrate. a) The second catalyst lines are adjacent and parallel to the first catalyst lines. The brighter contrast in the double-growth areas (dashed box in yellow) results from a higher density of aligned SWNTs compared to the single-grown area (dashed box in blue). AFM images (insets:  $5\,\mu\text{m}\times5\,\mu\text{m}$ ) confirm that the density of the SWNTs in the double-growth areas is between 1.5 and 2 times that in the single-grown area. The images shown here represent a typical case. b) Images of a substrate similar to that shown in (a) but with SWNTs at densities that are better than typical. Here, the densities are 20–30 SWNTs  $\mu\text{m}^{-1}$ , uniformly over the entire double-growth area, with peak values that approach  $\sim$ 45 SWNTs  $\mu\text{m}^{-1}$ .

arguments presented previously and on additional information in the Supporting Information. Figure 2b shows a case in which the second growth yielded substantially more SWNTs than the first growth due to slight variability in the process. The densities are, as a result, somewhat better than a typical case, reaching values of 20–30 SWNTs  $\mu$ m<sup>-1</sup> uniformly over the entire double-growth area, with peaks of ~45 SWNTs  $\mu$ m<sup>-1</sup> in certain regions (inset: AFM image).

Figure 3a and 3b present SEM images of SWNT arrays formed by first and second growths using iron catalyst patterned into continuous- and dashed-line geometries, respectively. These dashed catalyst lines (second growth) lie parallel to and equidistant between the continuous catalyst lines (first growth; Fig. 3b). This layout provides directly adjacent regions composed of areas of single and double growth, suitable for investigation using electrical test structures. These structures involved pairs of Pd electrodes (45 nm) formed by electron beam evaporation and photolithography and liftoff on both single- and double-growth areas (the inset of Fig. 3c shows the case of double growth), followed by oxygen-plasma etching to remove SWNTs not located in the space between the electrodes (Fig. 3c and 3d). The widths and separations of the Pd electrodes were 76 and 4  $\mu$ m,



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Figure 3. a-b) SEM images of SWNTs after a double-growth process, where the first and second growths involved iron catalyst patterned into continuous- and dashed-line geometries, respectively. The dashed lines are parallel to and equidistant between the continuous lines. This layout provides adjacent areas that have SWNTs at single- and double-growth densities, for ease of comparing the collective electrical properties of the arrays. c-d) Pairs of electrodes (Pd, 45 nm) were patterned on top of single-growth and double-growth areas (inset). The devices were then isolated by etching away the SWNTs not located in the space between the electrodes. The widths and spacing of the electrodes were 76 µm and 4  $\mu$ m, respectively. e) AFM image of SWNTs between two electrodes. f) *I–V* characteristics for representative devices. Linear fits to these data define effective resistances. g) Histogram of the resistance between pairs of electrodes for the single-growth and regrowth regions (20 devices in each region were measured; single growth - black; double growth - red bars).

respectively. Figure 3e shows an AFM image of SWNTs between two electrodes (top and bottom). Current-voltage (I-V) characteristics measured in ambient conditions yield responses like those presented in Figure 3f. As expected, the current levels measured from devices built with double-growth SWNTs are between 1.5 and 2 times larger than the single growth cases, for both positive and negative bias conditions. This outcome is consistent with the corresponding densities of SWNTs obtained from AFM and SEM images (Fig. 2a). Linear fits to these data define the effective resistances, R. A histogram that shows the distributions of R values measured from 20 devices in each region appear in Figure 3g (single growth - black; double growth - red bars).

To demonstrate the utility of multiple-growth strategies for devices, we built arrays of top-gated SWNT-array-based field-effect transistors. Figure 4a shows the schematic side view of such a



device. We used a layer of HfO2 (50 nm) and Au (45 nm) for the gate dielectric and the gate electrode, respectively, on source/ drain electrodes of Pd. An optical image of a set of such devices appears in Figure 4b; oval regions where the HfO2 was removed by HF provide electrical probing access points to the source and drain electrodes. Figure 4c presents plots of the drain current  $(I_d)$ as a function of gate voltage  $(V_g)$  with forward and reverse sweeps (between -3 and 3 V) for a source/drain bias ( $V_{ds}$ ) of 0.05 V for devices that use SWNT arrays formed by single (density,  $D \sim 4-7$  SWNTs  $\mu$ m<sup>-1</sup>) and double ( $D \sim 7-15$  SWNTs  $\mu$ m<sup>-1</sup>) growth. In both cases, the channel length, L, is  $4 \mu m$  and the channel width, W, is 76 µm. The histograms in Figure 4d summarize the results of similar measurements for 20 devices of each type (single growth – black; double growth – red bars). Metrics for comparison include currents measured in the p-channel "on state" (i.e.,  $V_g = -3 \text{ V}$ ), the "off state" (i.e.,  $V_g$  is set to realize the minimum  $I_d$ , typically between 0 and 0.5 V) and the n-channel "on state" (i.e.,  $V_g = 3 V$ ). Within experimental uncertainties, these results are consistent with microscopy and two-terminal-device measurements. Ignoring the effects of contacts, the resistivities (i.e., the resistances per unit length) per tube for the metallic tubes can be estimated from the resistance at the minimum current values in transfer curves such as those shown in Figure 4c. For both the single- and the double-growth areas, the average resistivity is  $\sim$ 20–50 k $\Omega$  µm<sup>-1</sup>. This number and the current in the on state can be used to estimate the resistivities for the semiconducting tubes; the result is  $\sim 60-90 \,\mathrm{k}\Omega \,\mu\mathrm{m}^{-1}$ . All of these results are consistent with previous reports of SWNT arrays grown on quartz.<sup>[21]</sup>

From the above results, the linear-regime mobilities can be calculated using the peak transconductance for single- and double-growth devices at a source/drain bias  $(V_{ds})$  of 0.05 V. Here, we define the effective field-effect mobility,  $\mu$ , as

$$\mu = \frac{L}{V_{\rm d}C_{\rm w}W} \cdot \frac{dI_{\rm d}}{dV_{\rm g}} \tag{1}$$

where C<sub>w</sub> is the specific capacitance per unit area for the electrostatic coupling of the aligned array of SWNTs to the planar gate,<sup>[16,20]</sup> according to the following expression:

$$C_{\rm w} = \frac{D}{\left[C_{\rm Q}^{-1} + \frac{1}{2\pi\varepsilon_0\varepsilon_{\rm s}} \cdot \log\left[\frac{\sinh(2\pi tD)}{\pi rD}\right]\right]}$$
(2)

where D is the density of the arrays of SWNTs per unit width,  $C_{O}$ is the quantum capacitance, *t* is the distance to the gate electrode,  $\varepsilon_0$  is the permittivity of free space,  $\varepsilon_s$  is the dielectric constant of the gate dielectric, and r is the SWNT radius. For the purpose of calculation, we consider only the semiconducting tubes in our estimate of D because the corresponding modulation by the gate originates primarily from such tubes; we assume that the ratio of metallic to semiconducting nanotubes is 1:2.<sup>[16]</sup> For the dielectric constant of the hafnium oxide and the quantum capacitance of nanotubes we used 10 and  $\sim 3.2 \times 10^{-10}$  F m<sup>-1</sup>, respectively.

The calculated effective mobilities in the double-growth areas for 20 devices of each type show values as high as  $\sim 800 \, \mathrm{cm}^2 \, \mathrm{V}^{-1} \, \mathrm{s}^{-1}$  $\sim 650 \,\mathrm{cm}^2 \,\mathrm{V}^{-1} \,\mathrm{s}^{-1}$ (median and

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**Figure 4.** a) Schematic side view of a top-gated SWNT-array transistor that uses a layer of HfO<sub>2</sub> as the gate dielectric, Au as the gate electrode, and Pd as source and drain electrodes. b) Optical image of a set of such devices. Oval regions where the HfO<sub>2</sub> was removed provide electrical contact points to the source and drain electrodes. c)  $I_d$  as a function of  $V_g$  for  $V_{ds} = 0.05$  V, for a transistor ( $L = 4 \mu$ m;  $W = 76 \mu$ m) that incorporates aligned arrays of SWNTs from single-growth ( $D \sim 4-7$  SWNTs  $\mu$ m<sup>-1</sup>) and from double growth ( $D \sim 7-15$  SWNTs  $\mu$ m<sup>-1</sup>). The open and solid circles indicate the sweep direction of gate voltage (solid – sweep up; open – sweep down). d) Histograms of current measured in the p-channel "on state" (i.e.,  $V_g = -3$  V), the "off state" (i.e.,  $V_g = 3$  V) for transistors built in regions of single growth (black bars) and double growth (red bars).

~560 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> (median ~400 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>) for the p-channel mode and the n-channel mode, respectively. Otherwise, the values for the single-growth devices are typically ~633 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> (p-channel mode) and ~320 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> (n-channel mode). Due to the relatively large hysteresis in these devices, the extracted mobilities depend on direction of the sweep in the gate voltage. The above calculations correspond to sweeps with increasing gate voltage. Similar analysis for the opposite sweep yields values that are lower by ~20% for the p-channel mode. In addition, we do not attempt to account for effects of contact resistances between the source/drain electrodes and the semiconducting tubes. Estimates of these resistances,<sup>[21]</sup> suggest that the mobilities would be improved by ~20% by explicitly

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In conclusion, this Communication presents a simple strategy for doubling the density of horizontally aligned arrays of SWNTs on quartz substrate. Detailed characterization of the tubes by AFM, SEM, and electrical measurements on two- and three-terminal devices reveal the key characteristics of the process and the properties of the arrays. The resulting improvement has direct beneficial effect on devices made using the tubes. This strategy is compatible with various optimized single-growth techniques described recently for quartz.<sup>[7,12–14]</sup> In principle, this process can be extended to triple, quadruple, and quintuple growths and beyond. Due to the arithmetic increase in density with each growth, however, the percentage increases in the density diminish with multiple cycles. For example, double growth increases the density by up to two times compared to single growth. Triple growth only yields an increase of 1.5 times compared to double growth. Quadruple growth yields an increase of only 1.3 times compared to triple growth, and so on. One strategy around such limitation is to combine double- or triple-growth arrays with multiple transfer-printing procedures to multiply the densities.<sup>[22,23]</sup> Such approaches could realistically yield densities  $\sim$ 2–3 times higher than those associated with the usual, singlegrowth strategy, with the clear potential to realize reproducible densities in the range of 100 SWNTs  $\mu m^{-1}$ .

#### Experimental

Synthesis of Highly Densely Aligned SWNTs Arrays: ST-cut single-crystal quartz substrates (Hoffman Materials Inc.) were thermally annealed at 900 °C for 8 h. Iron catalyst lines consisted of thin films (1–5Å) deposited by electron-beam evaporation (Temescal BJD1800) and patterned by photolithography (AZ 5214) and lift-off. Heating the substrate

prepared in this fashion at 900 °C for 1 h in a quartz reaction chamber oxidized the catalyst. After cooling the substrate to room temperature, heating to 925 °C under hydrogen gas flow with 350 sccm for 10 min fully reduced the catalyst. CVD growth performed for 20 min with a flow of argon (20 sccm) and hydrogen (20 sccm) introduced through an ethanol bubbler at 925 °C, yielded aligned arrays of SWNTs. Before CVD for double growth, exposure to oxygen plasma for 5 min through a patterned layer of photoresist removed certain segments of the existing SWNTs, thereby exposing the native quartz surface in line geometries next to and parallel with the first set of catalyst lines. Next, a new layer of iron was deposited in these etched regions. Annealing was performed in this case after purging the chamber with argon gas (1500 sccm). The reduction used a flow of hydrogen (200 sccm) diluted by argon (50 sccm). The same ethanol CVD growth process was then used.







*Electrical Measurements*: Two-terminal devices and transistor measurements were carried out in air using a semiconductor parameter analyzer (Agilent 4156A). Pd contact pads were used in both cases, in layouts that involved removal of SWNTs by oxygen plasma etching in all regions except between pairs of electrodes. For the transistors, the gate-dielectric layer (~49 nm) of HfO<sub>2</sub> was deposited by electron-beam evaporation followed by atomic-layer deposition (1.5 nm) over the entire substrate. A gate electrode of Au (45 nm) was deposited on top of this dielectric. Etching away the HfO<sub>2</sub> in selected regions opened bare regions of Pd for probing the source and drain electrodes.

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