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Fabrication and Characterization of Suspended Carbon Nanotube Devices in Liquid

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ABSTRACT

Suspended carbon nanotube devices are a promising platform for future bio-electronic applications. Suspended carbon nanotube transistors have been previously fabricated in air; however all previous attempts to bring them into liquid failed. We analyze forces acting on the suspended nanotube devices during immersion into liquids and during device operation and show that surface tension forces acting on the suspended nanotubes during transfer into the liquid phase are responsible for the nanotube damage. We have developed a new strategy that circumvents these limitations by coating suspended nanotubes with a rigid inorganic shell in the gas phase. The coating reinforces the nanotubes and allows them to survive transfer through the interface. Subsequent removal of the coating in the solution phase restores pristine suspended nanotubes. We demonstrate that devices fabricated using this technique preserve their original electrical characteristics.

Introduction. Single-wall carbon nanotubes (SWNTs) represent the ultimate example of a new class of one-dimensional nanomaterials. Their unique electronic properties have not only led to discovery of new physical phenomena at the nanoscale but also show a great promise for a variety of technological

applications (Baughman et al., 2002). While the majority of the carbon nanotube electronic devices reported in the literature consist of nanotubes grown or deposited on the surface, it is often advantageous to have suspended nanotubes to avoid nanotube-substrate interactions. (In the context of this paper suspended nanotube always refers to a nanotube double-clamped to the substrate only at its ends and hanging freely above the substrate.) The easiest and most widely used approach to fabricate suspended nanotube devices with long ($> 1 \mu\text{m}$) suspended regions is chemical vapor deposition (CVD) growth across preformed channels (Franklin et al., 2002). This method produces straight suspended nanotubes $10 \mu\text{m}$ and longer. Researchers have previously synthesized carbon nanotubes suspended over trenches and studied their mechanical and electronic properties in air and vacuum (Cao et al., 2005, Franklin et al., 2002, Han et al., 2001, Kim et al., 2002, Lee et al., 2004, Minot et al., 2003, Nygard and Cobden, 2001, Peng et al., 2003, Son et al., 2004, Walters et al., 1999). However, very little information exists about the behavior of these suspended devices in liquids. One of the main reason why previous attempts to study suspended carbon nanotube devices in liquid have been unsuccessful is that suspended nanotubes bridges break upon transfer into liquid phase (Franklin et al., 2002, Nygard and Cobden, 2001).

We estimate forces acting on a suspended carbon nanotube and conclude that it is surface tension that leads to nanotube fracture while viscosity effects play a minor role. To circumvent this limitation we describe a method to preserve suspended carbon nanotubes during immersion in liquid. We achieve it by temporarily coating carbon nanotubes with an inorganic shell in the gas phase, and subsequently removing it after the structure is transferred into liquid.

Forces on suspended nanotubes. During the transfer from the gas phase to the liquid phase a suspended carbon nanotube experiences a variety of stresses caused by the surface tension and the viscosity of the liquid. The failure of the carbon nanotube in response to these forces can involve two possible mechanisms: (1) detachment of the intact nanotube from the substrate at one of the ends and (2) nanotube rupture at some point along the suspended portion of the nanotube while the ends of the nanotube remain clamped to the surface. To determine relative probability of these events we compare strength of the nanotube and its adhesion energy to the substrate. The force required to break a nanotube, F_{break} , is given by

$$F_{break} = \sigma \cdot S = \sigma \cdot \pi \cdot d \cdot t \quad \text{Eq. 1}$$

where σ is the tensile strength of the nanotube, S is its cross-sectional area, d is the nanotube diameter and t is the thickness of the nanotube wall (Yu et al., 2000). Using a literature value of $\sigma \approx 10$ GPa (Li et al., 2000) and assuming that a typical SWNT has the diameter of 2 nm and wall thickness of 3.4 Å, we can estimate $F_{break} = 20$ nN. Adhesion of a nanotube to the surface is mostly governed by van der Waals interactions. The van der Waals force per unit length, F_{vdw} , between a cylinder of radius $d/2$ and a flat surface separated by distance x is given by (Israelachvili, 1992)

$$F_{vdw} = \frac{A \cdot \sqrt{d/2}}{12 \cdot \sqrt{2} \cdot x^{1.5}} \quad \text{Eq. 2}$$

where A is the Hamaker constant, which is on average equal to $(0.5-2) \cdot 10^{-19}$ J (Israelachvili, 1992). We can use this expression to estimate that for a 2-nm

SWNT separated from the surface by 3.4 \AA , the van der Waals force is equal to $140 \text{ nN}/\mu\text{m}$ assuming a Hamaker constant of $1 \cdot 10^{-19} \text{ J}$. A quick comparison of this value with the calculated value of the nanotube rupture force shows that $F_{VdW} = F_{break}$ when the nanotube is touching the surface over the length of 140 nm . Therefore, if the contact length is shorter than 140 nm the nanotube will slip and detach from the surface when enough force is applied. On the other hand, if the overlap is more than 140 nm , the nanotube will likely break before its ends start slipping and detaching. In typical nanotube devices the nanotube usually overlaps the surface by $1 \mu\text{m}$ or more (see also Figure 2 for an example of such device); therefore we conclude that the prevalent mechanism of carbon nanotube device failure involves nanotube breakage along a suspended region rather than detachment from the substrate.

Now we consider forces acting on the nanotube during immersion into liquid. The first force that the nanotube will experience is the surface tension of liquid-air interface, $F_{interface}$, which ranges from 15 to $70 \text{ mN}/\text{m}$ ($\text{nN}/\mu\text{m}$) for most liquids. Therefore, we can estimate that a $1\text{-}\mu\text{m}$ long nanotube will experience forces as high as $15\text{-}70 \text{ nN}$ depending on the liquid. Note that these forces are equal or greater than a typical 20 nN force required to break a nanotube. Thus we estimate that surface tension of a typical liquid would cause rupture of suspended nanotubes that are longer than $1 \mu\text{m}$ (and high surface tension liquids will cause rupture of even shorter nanotubes!). This observation agrees well with the experimental observations that long nanotubes suspended over TEM grids do not survive unprotected transfer into aqueous solutions (Artyukhin et al., 2004).

If the nanotube survives the transfer process and becomes immersed in liquid it will experience viscous drag forces. For an infinite cylinder drag force per unit length, F_{flow} , can be approximated as

$$F_{flow} = 4 \cdot \pi \cdot \mu \cdot \varepsilon \cdot U \quad \text{Eq. 3}$$

where μ is the viscosity of the fluid, U is the flow velocity, and ε is the shape factor, equal to natural logarithm of the cylinder aspect ratio, $\ln\left(\frac{2 \cdot L}{d}\right)$

(Batchelor, 1970). For a nanotube with an aspect ratio of 1000 immersed in water flowing with velocity of 1 m/s (higher than typical values for microfluidic devices) we can estimate that the drag force is 0.2 nN/ μm . Note that this number is 2 orders of magnitude smaller than surface tension values. This estimate also shows that for a typical carbon nanotube, which is several micrometers long, the drag force is much smaller than the typical force required to break the nanotube. This comparison strongly suggests that if we can avoid nanotube breakage during transfer through the gas-liquid interface, the viscous drag forces should not damage the nanotube and thus would not pose a problem for device operation.

Protecting the carbon nanotube during transfer into liquid. The prevalent approach for fabricating single-wall carbon nanotube devices involves growing the nanotubes directly in the prefabricated device from catalyst particles using high-temperature CVD process. This method tends to produce predominantly straight high-quality suspended carbon nanotubes that presumably have very low density of defects. Unfortunately, high temperatures necessary for the CVD process require researchers to transfer the devices in liquid *after* the growth stage. There are two ways to protect suspended

nanotubes during transfer through the interface: (1) reduce or eliminate surface tension to reduce force acting on the nanotube or (2) reinforce the nanotube to make it stronger. The first approach (reducing surface tension) is limited by the values of surface tension. These values range from 72 mN/m for water (Lide, 1992) and 30-40 mN/m for aqueous surfactant solutions (Lyklema, 2000) to 22 mN/m for hexane (Lide, 1992) and 13 mN/m for tetramethylsilane (TMS) (Mills and Mackenzie, 1954). TMS has the lowest surface tension among commonly available liquids. We tested immersion of suspended carbon nanotubes supported on a TEM grid (Artyukhin et al., 2004) or across a channel in Si (see fabrication below) in a variety of organic liquids and aqueous surfactant solutions. Subsequent SEM observations revealed that less than 1 % of nanotubes with lengths ranging from 2 to 10 μm survived the immersion. In agreement with our estimates, these experiments show that long suspended nanotubes still break when dipped in methanol, hexane, or TMS, so we conclude that simple reduction of surface tension is not enough to ensure reasonable device yield after the transfer operation. It is possible to completely eliminate the liquid-gas interface by traversing from gas to liquid through the supercritical region on the phase diagram. This method is in fact a powerful solution for the transfer of the devices from the liquid phase to the gas phase (we discuss the applications for the critical point drying technique in the next section). However, using this technique for transferring devices into the solution phase (i.e. “critical point wetting”) is practically impossible. Thus we conclude that the most practical approach for overcoming this interfacial transport problem should rely on reinforcement of nanotubes with a removable protective layer of material. We propose to coat

suspended nanotubes with such a layer in the gas phase, bring these coated, protected nanotubes into liquid, and then remove the coating by wet etching.

Fabrication of suspended SWNT devices. We have demonstrated the procedure for nanotube protection by fabricating suspended SWNT field effect transistors (FETs) capable of chemical sensing in aqueous solutions. Our device fabrication sequence (Figure 1) starts with patterning source and drain metal electrodes (5 nm Cr / 50 nm Pt) on top of a thick layer (2.5-3 μm) of thermally grown oxide on a Si wafer using standard photolithographic techniques and e-beam evaporation (Artyukhin et al., 2006). The separation between electrodes, which defines the nanotube channel length, is 2-10 μm . We then use this metal layer as a mask for wet etching a SiO_2 layer in buffered HF to create a 2- μm deep channel between the electrodes (Figure 1, step 2). Next we pattern thin-layer metal catalyst islands (10 nm Al / 3 \AA Mo / 5 \AA Fe) (Christen et al., 2004, Seidel et al., 2003) for nanotube growth near the edges of metal electrodes (Artyukhin et al., 2006). We grow nanotubes by ethylene-based CVD (Cheung et al., 2000, Hafner et al., 1999) at 850 $^\circ\text{C}$ across preformed channels in SiO_2 (Figure 1, step 4). By adjusting separation between source and drain electrodes, size of catalyst islands and distance between a catalyst island and the front edge of the electrode we can obtain up to 20% of devices consisting of an individual SWNT suspended between the two electrodes. Figure 2a shows an SEM image of a typical suspended device. We then coat entire devices with 2 nm Ti / 100 nm Au or SiO_2 (Figure 1, step 5) by e-beam deposition. Titanium is used as an adhesion layer to ensure uniform coating of nanotubes (Zhang et al., 2000). After this step our nanotubes are encased in a strong protective metal layer (Figure 2b) and we can

safely transfer our devices in water and back without losing suspended structures (Figure 1, step 6). To recover pristine suspended nanotubes in liquid we remove the protective coating by wet etching (KI/I₂ gold etchant and H₂O₂/HF-based titanium etchant (Williams et al., 2003)) (Figure 1, step 7). Note that it is crucial that during and after etching of the coating, the samples are not exposed to the liquid-air interface; therefore we perform all rinses and processing by carefully exchanging the solutions in the process beaker. To bring suspended nanotubes back from liquid to air for subsequent SEM examination we have used critical point drying (CPD). This method, widely used for drying biological samples (Bozzola and Russell, 1998) where surface tension can also damage delicate structures, relies on going from liquid carbon dioxide to the gas phase through the supercritical region that avoids direct crossing of an interface. Carbon dioxide is a commonly used solvent for CPD due to its low critical temperature and pressure. Since CO₂ is immiscible with water we exchange water with methanol before placing samples in the CPD chamber. Figure 2c demonstrates that suspended carbon nanotube devices successfully survive protected immersion in water, removal of the coating, and subsequent CPD. Note that we also can successfully use this procedure to recover suspended nanotube devices for the SEM inspection after electrical measurements and multiple solution exchanges in the flow cell. These data support our conclusion that viscous drag forces are not strong enough to break suspended nanotubes and the major obstacle to obtain these devices in fluid is the liquid-air interface.

Characterization of suspended SWNT devices. To demonstrate that deposition and removal of the coating do not destroy electronic properties of the

nanotube we record transfer characteristics of an as-grown suspended SWNT device in air and of the same device in liquid after removing the Ti/Au coating (Figure 3). To gate the device in air we apply voltage to the back of the Si substrate. For gating in liquid we mounted our device chips in a custom made flow cell and inserted an Au or a Pt wire in the flow cell (Rosenblatt et al., 2002). Consistent with previous reports (Cao et al., 2005) our suspended devices (Figure 3) do not show hysteresis effects typical for planar nanotube transistors in which the nanotube sits directly on the silicon oxide surface (Fuhrer et al., 2002). (Note that the blue curves on Figure 3 lay exactly on top of each other!) The gating curve of the device in water (red curve on Figure 3) clearly shows the same p-type semiconducting behavior that we observed in air and efficient switching with on/off ratio 10^3 . Thus we conclude that our procedure results in successful transfer of the nanotube devices into liquid phase without any noticeable degradation in the device properties.

We have fabricated suspended SWNT devices in liquid by first reinforcing them in the gas phase with an inorganic shell and then transferring in liquid with subsequent etching the coating. Both theoretical predictions and experiments indicate that surface tension is the major cause for rupture of unprotected suspended nanotubes upon transfer in liquid. We demonstrated that suspended devices in water preserve the electrical characteristics and high efficiency typical for carbon nanotube transistors. We believe that the fabrication procedure that we have described opens up a way for reliable, simple and cost-effective fabrication of high-quality suspended carbon nanotube transistors in liquids that could be used in a variety of applications.

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Biosketches:

Alexander Artyukhin was born in Moscow, Russia, in 1975. He received his M.S. degree in chemistry from Moscow State University (Russia) in 1997 and is currently working on his Ph.D. degree in materials science and engineering at UC Davis and Lawrence Livermore National Laboratory under the direction of P. Stroeve and A. Noy.

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Olgica Bakajin was born in Novi Sad, Yugoslavia, in 1972. She received her BA in Physics with satisfied requirements for a BA in Chemistry from the University of Chicago in 1996 and her Ph.D. in Physics from Princeton University in 2000. After receiving her doctorate she spent a year at the National Institute of Health as a Postdoctoral Researcher and then moved to the Lawrence Livermore National Laboratory as an E.O. Lawrence Fellow. She is currently a Staff Scientist and a Team Leader at the Chemistry, Materials, and Life Sciences Directorate at LLNL. Her research interests span the fields of microfluidics, nanoscience and nano technology, especially as applied to molecular confinement, molecular separations and kinetics of protein folding.

Aleksandr Noy was born in Moscow, Russia, in 1971. He received his BSc in Physical Chemistry from Moscow State University (Russia) in 1992, and his MS and PhD in Chemistry from Harvard University in 1998. After receiving his doctorate he was awarded the inaugural E.O. Lawrence Fellowship at the Lawrence Livermore National Laboratory, where he is currently a Staff Scientist and a Theme Leader at the Chemistry, Materials, and Live Sciences Directorate. He also serves as an adjunct associate Professor at the School of Natural Sciences at the UC Merced. His research interests range from single molecule biophysics to bio-nanoscience and applications of nanotubes and nanowires to molecular transport and sensing.

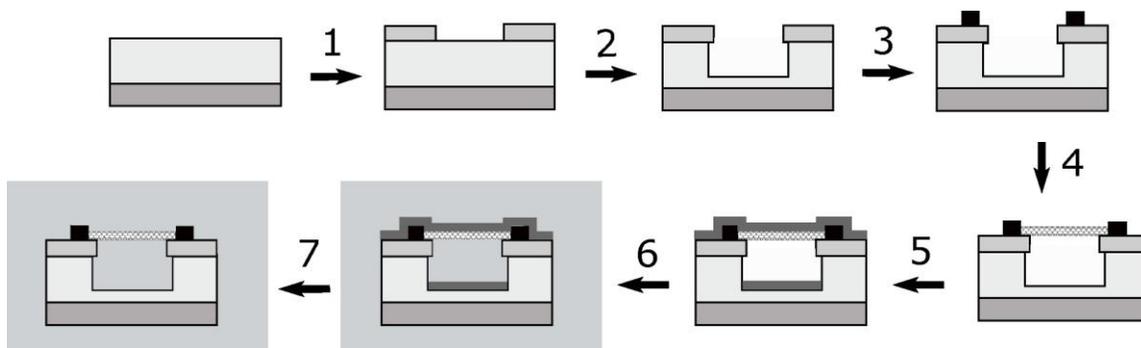


Figure 1. Schematics of the procedure for fabricating suspended carbon nanotube devices and their transfer in liquid. Step1: electrode patterning and deposition, Step 2: channel etching, Step 3: nanotube catalyst patterning and deposition, Step 4: nanotube CVD growth, Step 5: nanotube protection by metal deposition, Step 6: immersion in liquid, Step 7: etching of metal coating. See text for more details.

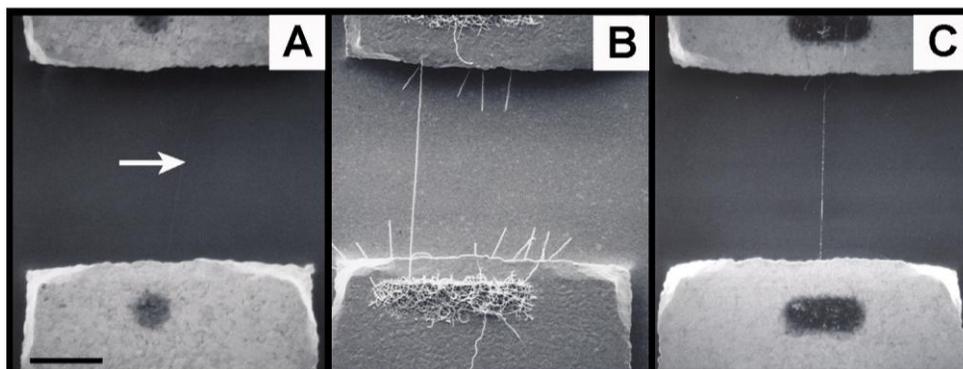


Figure 2. SEM images of suspended SWNT devices (**A**) as grown (the white arrow indicates the location of the suspended nanotube in the SEM image), (**B**) after coating with 2 nm Ti / 50 nm Au, (**C**) after transfer to water, etching Ti/Au, electrical measurements in water, and subsequent critical point drying (CPD) process. Residue on the nanotube visible on image (C) is contamination from CPD process. Scale bar, 5 μm .

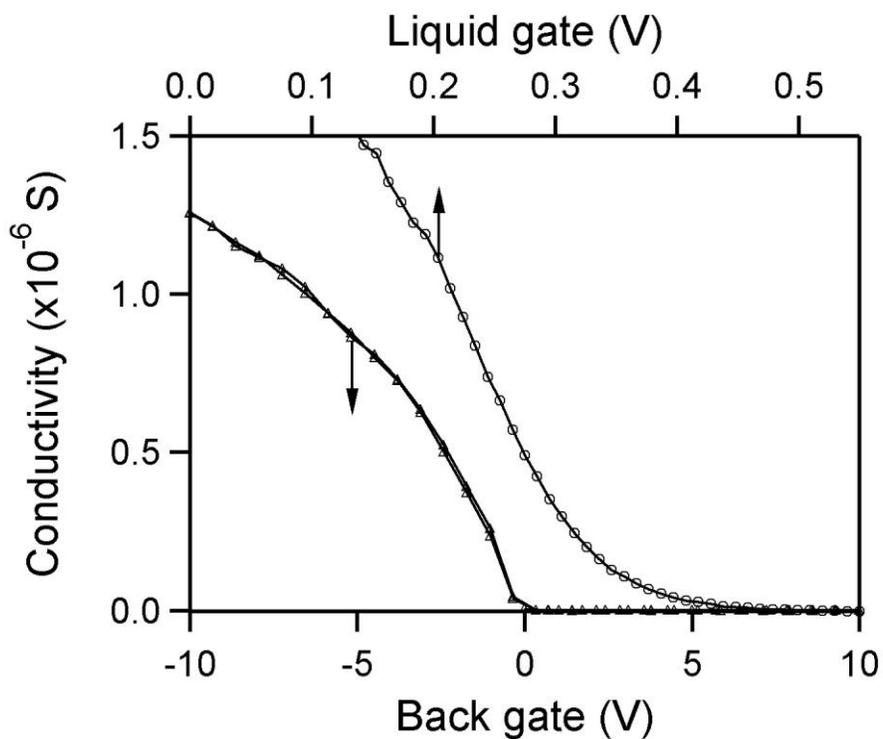


Figure 3. Transfer characteristics of a representative suspended SWNT device (measured using a source-drain voltage of 100 mV) in air (triangles) and in 100 mM NaCl in water (circles). Solid lines serve as a guide to the eye.