Dielectrophoretic Assembly of Carbon Nanofiber Nanoelectromechanical Devices

Stephane Evoy, Michael A. Riegelman, Nevin Naguib, Haihui Ye, Papot Jaroenapibal, David E. Luzzi, and Yury Gogotsi

***Abstract*—We report a technique for the assembly of bottom-up nanomechanical devices. This technique employs the dielectrophoretic manipulation of nanostructures within a multiple layer lithography process. Mechanical resonators were specifically produced by assembling and clamping tubular carbon fibers onto prefabricated pads. Our preliminary results showed that an assembled cantilevered fiber with length $L = 5$ μm and width of $W = 180$ nm possessed a resonant frequency of $f = 1.17$ MHz. A shorter $L = 3$-μm-long singly clamped resonator of similar width showed a resonance of $f = 3.12$ MHz. This frequency range is in agreement with the low gigapascal bending moduli previously reported for carbon structures showing extensive volume defects. This technology would allow the integration of bottom-up nanostructures with other more established fabrication processes, thus allowing the deployment of engineered nanodevices in integrated systems.**

***Index Terms*—Detectors, materials processing, microelectromechanical systems, microresonators, nanotechnology.**

I. Introduction

THE LAST several decades have seen a phenomenal growth in the availability of computational power and communications capacity. A second microelectronics revolution is underway, defined by the integration of transistor-based electronics with micromechanical and nanomechanical actuators, micropumps, and valves, as well as physical, biological, and chemical sensors. These integrated systems are poised to receive, process, and distribute large quantities of data concerning the physical world, and offer great interest for the development of distributed sensor networks in a wide range of commercial and military applications. Nanometer scale research and development provide control over materials at the level of individual atoms and molecules. At these lengths lie the basic phenomena that determine health versus disease, govern chemical reactions, control electronic behavior, and determine materials strength. Such nanometer scale engineering offers new approaches for the implementation of materials and devices that would offer key attributes of low-cost, low power consumption, and high sensitivity in integrated systems.

The manipulation of individual nanostructures is necessary for the characterization of their electrical and mechanical behavior, the analysis of their response to outside agents and stimuli, and, more importantly, their eventual integration into single-chip systems. Following the pioneering work of Pohl [1], we and others have used dielectrophoretic (DEP) forces to manipulate and assemble nanostructures from liquid suspensions. Dielectrophoresis is defined as the motion of uncharged polarizable particles in a nonuniform electric field. These forces were used to trap biomaterials such as single-virus particles, capsids, and latex spheres [2]. More recently, dielectrophoeretic manipulation enabled transport measurements across individual DNA strands trapped within nanometer-scale electrode gaps [3]. The DEP assembly of single-walled carbon nanotubes (SWNTs) has also been reported, demonstrating its viability for the assembly of such materials [4]–[6].

While dielectrophoresis presents demonstrated potential for the manipulation of nanostructures, it also offers the important advantage of being compatible with standard microelectronics foundry technologies, eventually allowing the on-chip assembly and integration of nanodevices with transduction, readout, signal processing, and communications circuitry. We have proposed a novel integrated systems technology based on the DEP assembly of nanostructures (Fig. 1). We have obtained a preliminary proof of this concept by successfully positioning one-dimensional metallic nanowires onto prefabricated sites of silicon circuitry, and have reported the preliminary integration of such structures with CMOS chips [7]. Cantilever-based devices specifically represent promising platforms for high-performance sensing devices. Sensors based on a mechanical resonating element enable the frequency modulation of the output, thus greatly improving the stability/noise-immunity of the reading [8]. This report, therefore, describes the fabrication and characterization of mechanical resonators produced through the DEP assembly of tubular carbon nanofibers.

II. Experimental

Experiments were performed using commercially available Pyrograph tubular carbon nanofibers purchased from Applied Sciences Inc., Cedarville, OH. The fibers were grown by catalytic chemical vapor deposition (CVD) using methane as a

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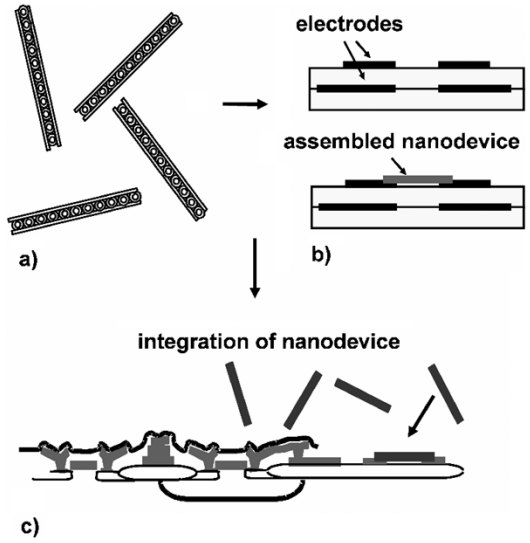

Fig. 1. DEP integration of nanodevices. (a) Functional structures are designed using "bottom-up" synthesis techniques. (b) Dielectrophoresis allows the inclusion of these structures within a multilayer fabrication process and (c) their integration with prefabricated processing circuitry.

precursor. The material was then heat treated at 3000 °C to graphitize the carbon and remove the catalyst, suspended in isopropanol at a concentration of 0.1 mg/mL, and ultrasonicated for 30 min. The solution was then filtered through a 10-μm micropore membrane (Osmonics Inc., Minnetonka, MN) to dispose of the shorter fibers. The fibers were re-suspended in isopropanol at a concentration of 0.1 μg/mL, and ultrasonicated for an additional 15 min immediately prior to assembly. Structural inspection of the material was performed using a JEOL 2010F transmission electron microscope (TEM). The end material consisted of a distribution of tubular nanofibers with diameters ranging from $D = 75$ to 200 nm, lengths ranging from $L = 5$ to 15 μm, and wall thicknesses ranging from $t = 20$ to 40 nm.

Using an approach reported by Smith *et al.* [9], individual fibers were dielectrophoretically positioned onto capacitively coupled electrode pairs fabricated on a silicon wafer (Fig. 2). First, a 300-nm electrically insulating SiO_2 layer was grown using thermal oxidation at 1000 °C. A first set of electrodes was patterned out of a 170-nm layer of Au, with an underlying 30-nm layer of Cr, using photolithography and liftoff. The electrodes were then buried under 300 nm of CVD SiO_2. Top floating electrodes with gap spacings varying from 1 to 5 μm were then fabricated in a similar metal layer [see Fig. 2(a)]. The top electrodes are capacitively driven by feeding a 45-V_{pp} 100-kHz assembly signal between the sets of buried electrodes for 5 min. This signal was provided by a Topward 8110 function generator, and amplified by a Bogen GA-6A amplifier [see Fig. 2(b)]. Following assembly, a final electron beam lithography step is performed to deposit a 200-nm-thick "clamping" layer onto the extremities of selected structures using the assembly electrodes themselves as alignment marks. The nanofibers are then released by etching down the underlying oxide layers in a 10 : 1 buffered HF for 45 s [see Fig. 2(c)].

Preliminary mechanical assaying was performed using an interferometric method originally developed for the characterization of surface machined silicon nanoelectromechanical

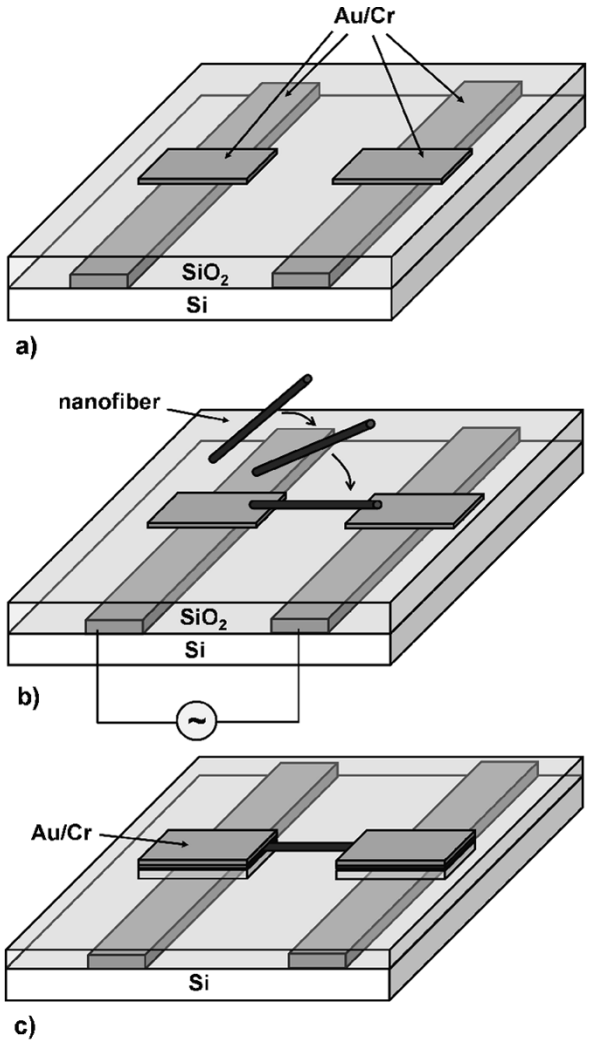

Fig. 2. DEP assembly of carbon nanotube resonators. (a) Assembly is performed on microfabricated pads capacitively coupled to buried electrodes. (b) A 100-kHz signal is applied between the buried electrodes to induce the assembly of the tubes across the top pads. (c) Structures are clamped using post-assembly lithography and released by partial etching of the oxide layer.

systems (NEMS) resonators [10], [11]. The chip is mounted onto a small piezoelectric element (Radio Shack 273-059), which is inserted in a small vacuum chamber pumped down to the 10^{-3}–torr range [see Fig. 3(a)]. The piezoelectric element is then actuated by the tracking output of a Hewlett-Packard ESA-L1500A spectrum analyzer amplified by an ENI UA-400A power amplifier. An He–Ne laser ($\lambda = 633$ nm) was focused onto the nanofiber device using a 0.35-NA microscope objective. When actuated at resonance, relative motion of the structure with respect to the underlying substrate modulates the reflected signal through interferometric effects [see Fig. 3(b)]. The modulated signal is reflected back through the microscope objective. A beam splitter is then employed to divert the reflected signal toward a New Focus 1601 ac coupled photodetector, whose output is fed to the input of the spectrum analyzer. While the diameter of the resonating structures is substantially smaller than the laser wavelength, their surfaces induce sufficient amount of optical scattering and interference with an underlying substrate to generate a reflected optical signal that is discernible by the photodetector. The same technique

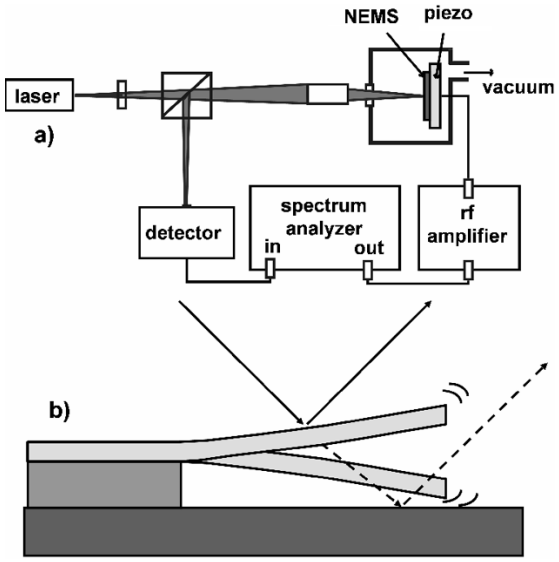

Fig. 3. Schematic diagram of interferometric setup employed for nanomechanical characterization of assembled resonators. (b) Resonant cantilever induces modulation of reflected signal through interference with underlying substrate.

has indeed previously been demonstrated for the assaying of surface machined resonators with lateral dimensions as small as 50 nm [11].

III. Results and Discussion

Fig. 4 shows TEM micrographs of typical fibers that have been directly deposited from solution onto a sample grid. The tubular fibers show diameters ranging from $D = 75$ nm to 200 nm, and wall thicknesses from $t = 20$ nm to 40 nm. Some fibers also show bamboo-shaped features [see Fig. 4(a)], which have previously been observed in large-diameter tubes grown by CVD [12]–[14], and can be regarded as stacking of truncated conical graphene sheets [13]. A higher resolution TEM image [see Fig. 4(b)] reveals the finer detail of this conical scroll structure, which is similar to the one described in [15]. Finally, the same image shows that tube surface also possesses a "herringbone" wall structure and chemically stable arched edges [16] that were induced by the 3000 °C heat treatment employed to graphitize the carbon and remove the catalyst.

Fig. 5 shows a typical nanofiber dielectrophoretically assembled across a 2-μm gap. This particular micrograph was taken prior to the clamping and release of the resonator. At its extremity, the nanofiber shows an outer diameter $D = 200$ nm. However, the tube cross section shows partial compression that rather results in ribbon-shaped geometry. Additional inspection by scanning electron microscopy (SEM) confirmed the dominance of such ribbon-shaped geometry both prior and after assembly. Such deformation is common for large-diameter nanotubes having thin walls [17].

While fibers are successfully assembled across a majority of the gaps, the reliability of this approach is first limited by our ability to trap a single structure free from obstruction arising from the assembly of additional fibers in a same gap. Nonetheless, roughly 10% of the assembly gaps indeed contained a single well-positioned fiber with significant amount

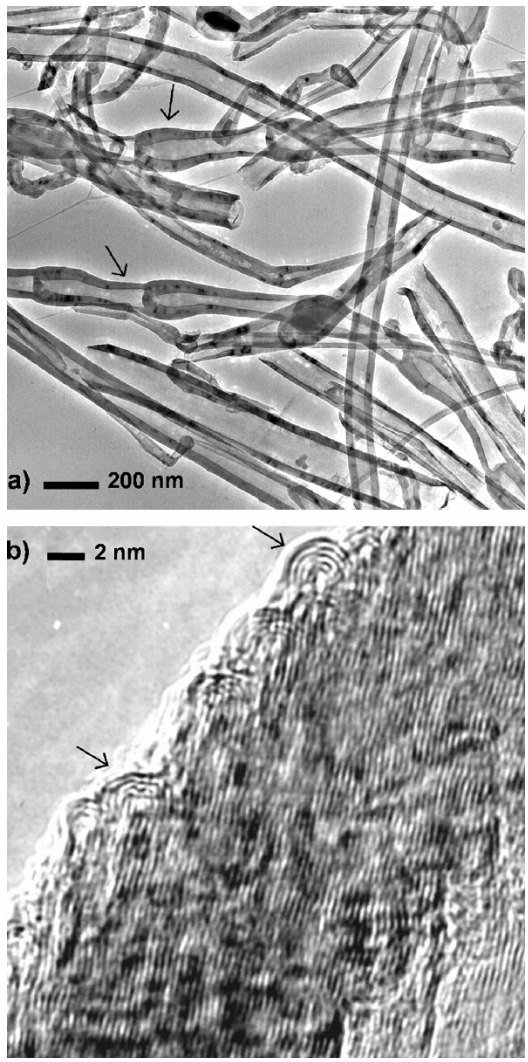

Fig. 4. Transmission electron micrographs of heat-treated carbon tubes. (a) Tubes nominally possess an outer diameter ranging from $D = 100$ nm to 200 nm, and a wall thickness ranging from $t = 25$ to 50 nm. Some tubes show bamboo-shaped structural features (arrows). (b) The tube outer surfaces also show chemically stable arched "herringbone" edges (arrows).

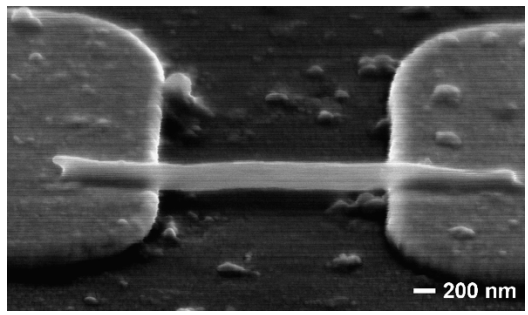

Fig. 5. SEM of assembled carbon nanofiber.

of clamping length on either or both electrodes. In addition, throughput of the technique is further restricted by the inclination of the assembled structures to stick to the underlying surface upon release. Such issue could be greatly alleviated by using a critical point drying process in order to avoid the structures from being pulled down to the substrate during drying.

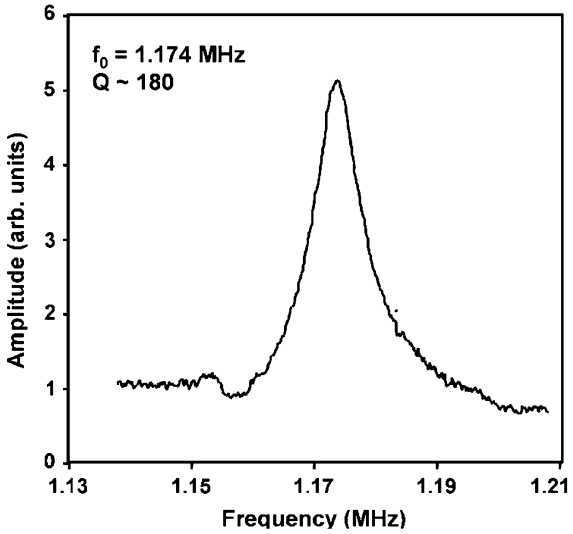

Fig. 6. Frequency response or assembled resonator of length $L = 5$ μm and width $W = 180$ nm.

Nonetheless, a number of the straighter and thicker fibers were successfully released, thus allowing a preliminary assessment of mechanical properties.

Fig. 6 shows the resonant spectrum of a released single-clamped nanofiber. The 7-μm-long and 180-nm-wide fiber was assembled and clamped on one extremity with a free length $L = 5$ μm. The structure showed a resonant frequency of $f = 1.17$ MHz. A similar structure of similar width showing a free length of 3 μm possessed a fundamental resonance of $f = 3.12$ MHz. Resonance qualities of $Q \sim 150$–180 were determined from the width of the Lorentzian frequency response. These qualities are not necessarily intrinsic to the fibers, but could be affected by combination of clamping-point dissipation and viscous damping due to the moderate vacuum employed.

The following relationship between resonant frequency and dimensions is derived from the Euler–Bernoulli analysis of a prismatic cantilevered beam [18]:

$$f_i = \frac{B_i^2}{2\pi L^2}\sqrt{\frac{E^* I}{\rho A}} \tag{1}$$

with

$$I = \frac{\pi\left(D_o^4 - D_i^4\right)}{64} \tag{2}$$

and

$$A = \frac{\pi\left(D_o^2 - D_i^2\right)}{4} \tag{3}$$

where f_i is the resonant frequency, L is the length, I is the cross-sectional moment of inertia of a cylindrical tube, A is its area, D_o is its external diameter, D_i is its internal diameter, E^* is the effective bending modulus, ρ is the density of graphite ($\rho = 2.22$ g/cm^3) [19], and B_i is a constant for the ith harmonic oscillation $B_1 = 1.875, B_2 = 4.694$.

The bending modulus of carbon tubes has been experimentally assessed by several techniques such as tensile loading tests [20], [21], observation of thermally induced vibrations [22], and *in situ* actuation in the TEM [23]. While the reported Young's moduli of SWNTs are typically around a terapascal, such values tend to be significantly lower in carbonaceous structures of increasing diameters due to increasing dominance of point defects and structural imperfections. More specifically, Poncharal *et al.* observed effective bending moduli in the $E^* \sim 200$ GPa range for ~20–40-nm-diameter multiwall carbon nanotubes (MWNTs) grown by an arc discharge method [23]. However, the same group then observed effective bending moduli as low as $E^* \sim 2$–3 GPa in structurally imperfect MWNTs produced by precursor pyrolisis, and concluded that point and volume defects played a dominant role in the reduction of the overall bending modulus [24]. Deformation of the tube cross section is also expected to lower the position of resonance in these fibers. Indeed, resonant frequency is a strong function of the transverse dimension of the beam through the cross-sectional moment of inertia. Departure from a cylindrical geometry to a more elliptical shape would significantly reduce the moment of inertia of the fiber along the bending axis. For instance, a cylindrical nanotube with outer diameter of $D_0 = 180$ nm and wall thickness of $t = 30$ nm subject to a partial collapse that would reduce its transverse dimension 20% while keeping same cross-sectional area through similar lateral extension would see its bending moment of inertia reduced by as much as 40%. Using an effective bending modulus of $E^* = 2$–3 GPa, the expected resonances of such fibers would range between 0.8–1.5 MHz for a 5-μm-long structure, and between 2.3–3.7 MHz for a 3-μm-long fiber, which is in rough agreement with the observed quantities.

In summary, the range of resonant frequencies of the dielectrophoretically assembled nanofibers resonators is in agreement with the low gigapascal bending moduli previously reported for carbonaceous tubes showing extensive volume defects [24]. Such characteristics could, therefore, be intrinsic to the conical scrolls or bamboo-shaped fibers being assembled. In addition, the partial structural collapse observed in these tubes would also reduce their resonant frequencies by decreasing their bending moment of inertia. A more systematic assessment of these properties would, however, require structures showing better uniformity and of simpler cross-sectional geometry.

IV. Conclusion

We have reported a novel technique for the assembly of "bottom-up" nanoelectromechanical devices. DEP manipulation allowed the positioning of nanostructures at specific sites of a prefabricated pattern, enabling the integration of the structures within a multiple layer lithography process. Specifically, released devices were produced by assembling tubular carbon fibers onto prefabricated pads, followed by their clamping through a post-assembly electron beam lithography step. Preliminary mechanical resonances in the low megahertz range were observed in singly clamped 3–5-μm-long cantilevered fibers. A more conclusive assessment of mechanical properties would, however, require structures showing better uniformity and of simpler cross-sectional geometry. This technology would allow the integration of bottom-up nanostructures with other more established fabrication processes, thus allowing the deployment of nanostructured devices in integrated systems.

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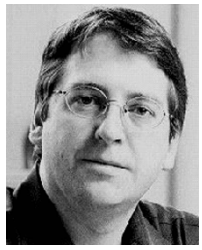

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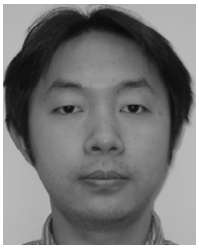

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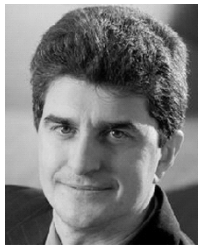

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