Strain Relaxation and Resonance of Carbon Nanotube Forests under Electrostatic Loading

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Abstract

Electrostatic loading is widely used for sensing and actuation in miniaturized electromechanical systems, yet classical designs involve geometric patterning of solid materials such as silicon and metal films. Conductive nanoporous materials for electrostatics may enable engineering of new functionalities arising from their compliance, internal surface forces, and high surface area. Toward this end, we investigate the response of vertically aligned carbon nanotube (CNT) "forests" to DC and AC electrostatic loads. First,

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the tensile strain-stress characteristics of patterned CNT forests was determined in a non-contact manner by cyclic DC electrostatic loading, revealing an increase of the effective Young's modulus with sequential load cycling. Next, we observed resonance can be excited by AC electrostatic loading, and that the resonance frequency increases with sequential sweeps of the AC load frequency. Both the DC and AC measurements indicate that release of residual strain arising during CNT growth is relaxed upon electrostatic loading, causing stiffening of the structure. This study shows for the first time that CNT forests can function as bulk electrostatic elements, and their intrinsic low stiffness and quality factor may be suitable for development of wide bandwidth micro-resonators and adsorption-based sensors.

Keywords:

Actuators ,carbon nanotubes, damping, electrostatic, stiffness.

1. Introduction

Electrostatically operated nano- and micro-devices based on carbon nanotubes (CNTs) have demonstrated promise as atomic-scale mass sensors [1], scanning probe tips [2] and sensitive pressure [3] and displacement [4] sensors. CNTs have also been used as high frequency resonators [1], optomechanical [5] and thermal [6] actuators, and nano-tweezers [7]. In the aforementioned applications, both the electrical and mechanical properties of the CNTs are pivotal for the device operation. For example, the piezoresistivity [3, 8] or capacitance [9] characteristics of a device determine its sensitivity to respond to small signals, while mechanically compliant materials allow relatively large deflections, enabling motion detection.

While most of these applications have involved individual CNTs, which remain challenging to integrate within microfabrication processing, vertically aligned CNTs (forests) present a lithographically definable nanostructured material [10, 11, 12], which is mechanically robust, electrically conductive, and porous. Importantly, the properties of CNT forests depend exquisitely on the hierarchical organization of the CNTs as well as their individual dimensions and geometry. Many studies have quantified the thermal [13, 14, 15] electrical [16] and mechanical [17, 18] properties of CNT forests. Building on these properties, CNT forests have been studied as building blocks of strong mechanical fibers [19] durable electrical contacts [12] micro-scale membrane sensors [20, 21] capacitance-based energy storage devices [22], electrical

switches [23], comb-drive fingers [11], structural elements of micromechanical devices [6, 24], and in other applications [25, 26].

Further, the electrical conductivity and mechanical compliance of CNT forests, along with their low mass density [27], make CNT forests a candidate material for use in RF devices. For RF applications, it is necessary to obtain a comprehensive understanding of the constituent materials to electrostatic loads. Additionally, due to their high sensitivity to growth parameters [28, 29], the mechanical properties of CNT forests vary from one structure to another. For example, the Young’s modulus was reported to span between sub-MPa [30, 31] to tens [32, 33] or hundreds [34] of MPa. Therefore, tools for precise mechanical characterization of CNT forests are very useful. In most previous studies, the mechanical properties of as-grown CNT forests were analyzed in compression tests [17, 18, 33, 35]; these generally result in large deformations and are limited to relatively low frequencies (tens of Hz). Intergration of CNT forests into microfabricated devices has also been used to examine mechanical properties. These include bending CNT varactors by means of an electrical field [36] and bending CNT forests mounted on micro-cantilever beams [34].

In this study, we use electrostatic loading to study the properties of CNT forests under static and oscillatory tensile forces. We find that strain relaxation occurs upon tensile loading, and identify that CNT microstructures exhibit a bulk electromechanical resonance upon AC excitation. The resonance is broad due to the low stiffness and extremely low mass density of the CNT forest ($\sim 15 \text{ kg}\cdot\text{m}^{-3}$ comparing to $\sim 2330 \text{ kg}\cdot\text{m}^{-3}$ for silicon), along with the high internal damping due to the large number of CNT-CNT contacts. Therefore, we demonstrate that CNT forests can function as bulk electrostatic elements integrated with microfabrication processing.

2. Methods

2.1. Experimental setup

A custom built system was used for electrostatic characterization of CNT forests, as shown in Fig. 1. The experimental system includes a flat capacitance probe (Capacitec HPB-75 Button Probe) attached to a linear voice-coil actuator and a linear optical encoder (Heidenhahn REF1000E25A), enabling control and measurement of the vertical position of the probe. The probe position is controlled by a computer using LabView software and is imaged

optically using a CCD camera (Edmund NT85-871) with long working distance CMOS optics (Edmund NT63-232) (see a typical CNT forest image in Fig 1(b)). Using this optical system, the physical length and width of a pixel are $2\mu\text{m}$ on the sample. Capacitance measurements were taken using a C-V plotter (HP4280A). The electrostatic field was used for application of tension forces to CNT forests. For DC electrostatic loading, a high voltage DC power source (SRS PS310) was used directly. For AC loading, a function generator (Aim TTi TG4001) in series with a linear voltage amplifier (Tegam A400) was used.

In addition to electrostatic tension experiments, a custom built loading system was used for mechanical compression tests, and includes a stepper motor (DRV001) to actuate a flat indenter, a laser displacement sensor (Keyence LK-G152) and a load cell (Futek LSB 200) to measure the applied displacement and force, respectively.

Youngs moduli in all experiments (tension and compression) was extracted from the slope of the unloading curve [37]. Information regarding the uncertainty in calculation of Youngs moduli is given in Appendix A.

Measurements were performed on CNT forest pillars grown by atmospheric pressure thermal chemical vapor deposition (CVD) from lithographically patterned catalyst [38]. A series of samples were prepared, with dimensions of 0.25 - 1.5mm diameter and 0.20 - 0.75 mm height (Table 1). All experiments herein were performed on as-grown CNT forests, and therefore the measured properties in our study represent those of the pristine CNT forest. A scanning electron microscope (SEM) image of a typical pillar is shown in Fig. 1(c).

2.2. Electrostatic force calculation

We first directly measured the capacitance of each CNT pillar, at varying gap distances, g , between the probe and the top surface of the pillar [39]. This relationship is shown in Fig. 2(a), wherein the capacitance of the substrate surrounding the pillar was subtracted from the total measured capacitance. The capacitance is used here to evaluate the electrostatic potential energy and to calculate the total force exerted on the pillar at a specified DC voltage applied to the probe. The electrostatic potential energy is [40]

$$U_E = \frac{1}{2}C(g)V^2 \quad (1)$$

Figure 1: (a) Schematic of the experimental setup. A vertically moving probe is used to apply DC and AC electrostatic fields to the CNT forests. (b) Optical side view of the CNT forest and the probe. (c) SEM image of a typical millimeter-scale CNT pillar.

Table 1: CNT pillars dimensions in [$\mu\text{m.}$]

Pillar number	Diameter	Height
1	1456	600
2	268	222
3	962	508
4	1514	744
5	766	458
6	762	422
7	986	530

where C is the pillar capacitance, g is the gap and V is the actuation voltage. The total electrostatic force was calculated for a given gap g_0 (measured optically) and an input voltage V ,

$$F_E(g_0, V) = \left. \frac{\partial U_E}{\partial g} \right|_{g_0, V} \quad (2)$$

We emphasize that this calculation yields the electrostatic force from its capacitance, regardless to its structure [40]. The total electrostatic force as a function of the gap and voltage is shown in Fig. 2(b). The calculated electrostatic force decreases rapidly with increasing gap. The capacitance measurement (i.e., the distribution of the capacitance along the height [39]) was further used to calculate the distributed electrostatic force F_{ES} (external force) applied to the CNT forest, based on the extracted capacitance area, as shown in Fig. 2(c). By maintaining equilibrium (namely, the net electrostatic force equals the internal force inside the forest at any horizontal plane that sections the forest) and integrating the electrostatic force over the vertical coordinate, the internal force averaged over the cross section area is derived (Fig. 2(c), inset). In other words, the internal force is the mechanical force per unit length applied by the individual CNTs to balance the electrostatic force.

We further note that the CNT forest behaves as a Faraday cage [39], i.e., the electrostatic force acts only on its external surface, as indicated

Figure 2: Electrical measurement of CNT forest sample 1 ($D = 1456\mu\text{m}$, $H = 600\mu\text{m}$). (a) Measured capacitance versus the gap after subtracting the substrate capacitance. (b) Calculated total force (extracted from the measured capacitance, Eq. (2)) versus the gap. (c) Distributed electrostatic force (external force) for $V = 100\text{V}$. Inset: internal force applied by the individual CNTs to balance the electrostatic force. (d) Schematic description of the electrostatic force applied to CNTs on the periphery of the forest within the penetration depth of the electric field.

schematically by the red color in Fig. 2(d). As a result, the electrostatic field penetrates only several microns into the forest. The penetration depth depends on the density of the CNTs. We note that, since the magnitude of the electrostatic force is proportional to the capacitor area (extracted from the capacitance measurements [39]) and inversely proportional to the square of the length of field lines [40], the electrostatic pressure (namely, electrostatic force divided by the capacitance area) gets its maximal value at the top portion of the CNT forest.

3. Results

As a reference, compression tests were carried out for sample 5 ($D = 1456\mu m$, $H = 600\mu m$) and sample 6 ($D = 762\mu m$, $H = 422\mu m$). The measurement for sample 5 is shown in Fig. 3. Young's moduli of 1.00 ± 0.01 and 0.70 ± 0.01 were extracted for samples 5 and 6, respectively (see Table 2).

Next, to explore the response of CNT forests to DC electrostatic loads, a steady voltage was applied while maintaining a constant gap between the probe and the forest. Small displacements (tenth of a pixel or sub microns) induced by the electric field were captured using the camera system described above, and were processed using an interpolation based edge detection algorithm, previously shown to enable optical detection of nanoscale edge displacements [41]. Briefly, the algorithm includes coarse edge finding using an intensity thresholding and a fine (sub-pixel) edge detection using a gradient based interpolation between two adjacent pixels. Typical deflections as a function of the actuation voltage for three cycles are shown in Fig. 4. Note that as the voltage increased, the top edge moved upward relative to the substrate, indicating vertical extension of the CNT forest (i.e., tension). After removal of the load ($V = 0$) the CNT pillar did not relax to its initial height.

Based on the force calculation in Eq. (2) and optical height measurement, the engineering strain and stress were calculated, giving the data shown in Fig. 5. Note that the capacitance was measured before electrostatic pressure was applied and after each cycle, in order to have an accurate estimation of the force, taking into account possible changes in the CNT forest morphology, which are discussed below. An equivalent Young's modulus was calculated from the initial slope of the unloading curves. For sample 1 analyzed here, the Young's modulus increases from its initial value of 0.37 ± 0.04 MPa to 3.35 ± 0.22 MPa. Comparable increases were measured for other samples, as listed in Table 2. While the applied tensile strain (and stress) is significantly less than in a contact-based compression test using a flat punch indenter, the effective modulus measured by DC tension after initial relaxation is comparable to that obtained under large compressive strains of pillars grown under the same process, see Fig. 3. We later return to interpret the hysteresis in the CNT forest height upon initial tension loading and unloading, and conclude that after initial relaxation the electrostatic technique gives an accurate measurement of the elastic modulus of the CNT forest for small elongation strains.

Figure 3: Mechanical compression test results for sample 5 ($D = 1456\mu\text{m}$, $H = 600\mu\text{m}$). Youngs moduli was obtained from the unloading slope.

Figure 4: Measurement deflection, u , for sample 1 ($D = 1456\mu\text{m}$, $H = 600\mu\text{m}$) under DC loading. Black triangles pointing up correspond to loading (increasing the voltage) and green triangles pointing down correspond to unloading (decreasing the voltage). Solid lines are fitting curves. (a) First loading cycle. (b) Second loading cycle. (c) Third loading cycle.

Table 2: Measured initial Young’s modulus in [MPa]. Final Youngs modulus under tension was extracted after three cycles.

Pillar number	Load type	Initial modulus	Final modulus
1	Tension	0.37 ± 0.04	3.35 ± 0.22
2	Tension	0.71 ± 0.04	5.46 ± 0.34
5	Compression	1.00 ± 0.01	-
6	Compression	0.70 ± 0.01	-
7	Tension	0.45 ± 0.06	1.46 ± 0.28

Next, we explored the response of CNT pillars to AC excitation, identifying for the first time the electromechanical material resonance of the CNT forest. A time-dependent voltage $V(t) = V_{AC}\sin(2\pi ft)$ (V_{AC} is the voltage amplitude, f is the actuation frequency and t is time) was applied (sample 4, $D = 1514 \mu\text{m}$, $H = 744 \mu\text{m}$). Note that since the excitation voltage has no DC component and due to the fact that the force is proportional to V^2 , the CNT forest excites at a frequency of $2f$, namely twice the actuation frequency. A peak-to-peak signal of 200 V was applied, and the frequency was swept from 0 to 1 MHz in 5 minutes. This sweep was defined as one loading cycle. The AC response is plotted in Fig. 6(a). This was defined as the peak-to-peak deflection amplitude (from optical images) of the pillar versus the excitation frequency, during the 10th, 20th and 30th cycles. For each measurement cycle, the half-resonant frequency and quality factor were extracted from the Lorentzian fit curves, as shown in Fig. 6(b). We denote the half-resonant frequency as the location of the maximum of the Lorentzian fit, and the quality factor as the parameter which relates the half power bandwidth to resonance frequency (also known as the half power method [42]).

We find that the half-resonant frequency increases significantly with each consecutive AC loading cycle. Because the electrostatic pressure is maximal on the top of the forest Fig. 2(c), we attribute the increase in half-resonant frequency to stiffening of primarily the upper portion of the pillar. This stiffening and the observed increase in quality factor are attributed to the enhanced vertical alignment of CNTs in the top section of the pillar, as discussed later.

Figure 5: Stress strain curves for sample 1 ($D = 1456\mu\text{m}$, $H = 600\mu\text{m}$) under DC loading. Solid lines correspond to loading while dashed lines correspond to unloading. Inset: Youngs modulus extracted from the unloading curves.

From the resonance response we can calculate the dissipated energy due to AC excitation. Under the assumption of steady state response, the dissipated energy in one cycle of motion is [43]

$$W_d = c\pi\omega A^2 \quad (3)$$

where $\omega = 2\pi f$ is the circular actuation frequency, $c = m\omega_n/Q$ is the dimensional damping coefficient ω_n is the natural circular actuation frequency, and A is the motion amplitude. The average excitation energy (electrostatic potential energy) can be calculated as [40]

$$U_E^{av} = \frac{1}{T} \int_0^T \frac{1}{2} CV(t)^2 dt \quad (4)$$

Here T is a cycle time, C is the measured capacitance, and $V(t)$ is the time dependent actuation voltage. The relationship between the dissipated energy and excitation frequency is shown in Fig. 6(c), and is compared to the averaged excitation energy. The dissipated energy ranges between 0-16 nJ per cycle, and is proportional to the motion amplitude and inversely proportional to the quality factor (Eq. (3)). We find that, after 10 and 20 cycles, the excitation energy, U_E^{av} , is smaller than the dissipated energy around the resonance frequency. This mismatch indicates that electrostatic excitation releases stored elastic energy within the CNT forest. After 30 cycles, the dissipated energy is lower than the excitation energy.

Figure 6: AC loading data for sample 4 ($D = 1514\mu\text{m}$, $H = 744\mu\text{m}$). (a) Measured frequency response. Markers correspond to the measurements and solid lines correspond to Lorentzian fit curves. (b) Half-resonance frequency and quality factor extracted from the resonance response. (c) Dissipated energy per loading cycle calculated using the measured quality factor and detected amplitude of motion. The black line indicates the average excitation energy due to AC loading.

4. Discussion

4.1. Mechanism of electrostatic loading

In addition to revealing the DC and AC responses of CNT forests, electrostatic loading is a non-contact mean of measuring the mechanical response of CNT forests under tension. The applied force is determined by the voltage resolution and stability of the power supply and the resolution of the optical imaging method. The force is transferred to the interior parts of the CNT forest via the surface interactions and entanglement among CNTs. As a comparison, when mechanical tests are performed using a flat indenter, the minimum force applied is limited by the need to first establish uniform contact across the top surface of the sample. Using the electrostatic loading method, we applied stresses of tens of Pa (Fig. 5), while for contact-based compression tests, stresses of kPa-MPa are typically reported [35]. Therefore, the electrostatic technique is potentially useful for non-contact mechanical testing of other fragile nanomaterials that are electrically conductive.

The application of forces to CNTs using electric fields has been demonstrated in many previous studies. Here we seek to clarify the mechanism by which the applied voltage induces forces on the CNTs in our experiments. First, when a potential difference (voltage) exists between CNTs and an electrode placed at a distance, a capacitive electrostatic force is induced. The same origin of capacitive force is commonly used in electrostatically driven nano/micro-electromechanical systems (NEMS/MEMS) devices [44, 45], whereby a voltage between two surfaces generates an attractive force typically causing one of the surfaces to move in an intended manner. Previous studies have demonstrated pulling of CNTs by electrostatic pressure [1, 46, 47] as well as rotation and alignment of CNTs in the presence of an electric field [48, 49, 50]. Electrostatic pressure applied to a CNT forest has also been reported [23, 36, 51].

An electrostrictive effect might add to the actuation by causing CNTs to elongate as they are polarized. This effect was investigated theoretically for individual CNTs [52, 53, 54] and recently was studied experimentally on CNT forests by placing them in direct contact between a pair of electrodes held at a potential dif-

ference and optionally within a liquid electrolyte [55, 56]. However, the electrostrictive effect was observed under strains significantly larger than those exist in the present study (Fig. 5) and was measured to induce electric fields of several orders of magnitude smaller than those exist in our experiment. Therefore, we conclude that the force applied in our experiments is dominated by the capacitive effect. Also, unlike the cited works [55, 56], our experiments involve non-contact application of the electric field. As a result, the magnitude of the electrostatic pressure is inversely proportional to the electrostatic gap, and the highest stress exists at the top of the CNT forest, Fig. 2. Measurement of the capacitance of CNT forests in our recent previous work [39] confirmed that the capacitance of a similar CNT forest micropillar is dominated by outer surfaces, and the electric field only penetrates several microns into the forest. Therefore, when force is applied by the electric field, the penetration depth is limited and the observed orientation change of the CNTs (see below) is restricted to the top surface where the field is greatest.

Additionally, electrostatic loading enables a wide dynamic range of operational frequencies, therefore enabling application of high frequency loads. While compression tests can apply loads as fast as 10-1000 Hz [35], electrostatic loads can readily be applied at several orders of magnitudes higher frequencies (up to the GHz range). However, while in compression tests the force is distributed uniformly over the cross-section, the electrostatic loading results in non-uniformly distributed stress on the outside surfaces of the CNT forest, as illustrated in Fig. 2(d), so that the force is transferred into the internal parts of the forest through the interactions between the CNTs. Nevertheless, since the majority of the electrostatic force is applied to the top of the forest (Fig. 2(c)), it can be roughly stated that similarly to compression tests, the force is applied mostly to the top portion of the sample. We also note that the top curvature of the sample has minor influence over the calculation of the force, since most of the charge is concentrated on the top part of the sidewalls as well as on the periphery of the top surface [39, 57].

4.2. Morphology evolution due to electrostatic loading

In order to further understand the origin of hysteresis in the CNT forest height due to cyclic electrostatic loading, DC experiments were performed

along with X-ray analysis. Note that we were not able to observe the morphology change in SEM. This can be explained due to the small strain that is induced due to the electrostatic force which is only $\sim 1m\epsilon$ (Fig. 5) and therefore difficult to detect.

Small Angle X-ray Scattering (SAXS) patterns were collected in-situ during loading/unloading of sample 3 ($D = 962\mu m$, $H = 508\mu m$), as illustrated in Fig. 7(a). The CNT pillar was mounted on a motorized sample stage in the G1 beamline at Cornell High Energy Synchrotron Source (CHESS). The X-ray beam energy was approximately 10 keV (wavelength 0.13 nm). The voltage of the probe was increased from 0 to 800 V in 100 V increments. The beam size was maintained at $500\mu m \times 20\mu m$ by means of motorized precision slits. The gap between the electrode and the CNT pillar (estimated at $100\mu m$ for this experiment) was measured using the integration of intensities on the 2D area detector, while moving the whole sample stage with respect to the X-ray beam in order to provide a complete scan. The sample-to-detector distance was selected to be $\sim 770\text{ mm}$.

We quantified the straightness of CNTs by performing an integration of intensities inside a small annulus on the 2D SAXS patterns, around the peak position. This integration over azimuthally scanned intensities is used to calculate the Hermans orientation parameter that was first introduced by Hermans to study the orientation of cellulose fibers using X-rays [58]. More details about the experimental setup and the calculation of the Hermans parameter can be found in our previous works, which also demonstrated to provide quantitative information about the morphology and dimensions of the CNTs [27, 59].

A spatial scan across the height of the CNT forest under applied DC voltage is shown in Fig. 7(b). The degree of alignment of CNTs within the sample by calculating the Hermans orientation parameter is shown in Fig. 7(c) [27, 58]. Note that voltage starts from 200V in our analysis due to challenges in measurements under low voltages during SAXS measurements. At the middle portion of the CNT pillar, the CNT alignment does not change significantly with increasing applied voltage. SAXS data at the bottom suggests a slight orientation change; however, the sensitivity of this change to the applied voltage is an order of magnitude smaller than measured for the top portion. In addition, the reliability of this measurement is challenged by substrate beneath the CNT forest that interacts with the X-ray beam. Therefore, we cannot confirm whether an orientation change happens at the bottom of the forest, and our understanding of the mechanism suggests that

Figure 7: X-ray analysis results for sample 3 ($D = 962\mu\text{m}$, $H = 508\mu\text{m}$). (a) Schematic showing the experimental setup for X-ray scattering experiments. (b) A typical SAXS intensity pattern collected by the 2D area detector. (c) Hermans orientation parameter versus the actuation voltage at the top, middle and bottom of the pillar.

the major change occurs near the top of the forest where the field penetrates.

CNTs at the top, on the other hand, experienced a more significant morphology change. We hypothesize that the applied electrostatic tension force initiates CNT-CNT sliding, which enables the release of a stored strain (Fig. 6(c)) and reorganization of the CNTs at the top to be slightly more aligned with the applied field. This observation well agrees with other studies that have demonstrated that CNT-CNT interactions dominates the mechanics of CNT forest under strains smaller than 5% [60]. It also agrees with other studies that demonstrated stiffening of CNT networks due to alignment of CNTs [61]. We note that the stiffening (measured increase in Young's modulus) of the CNT forest was observed despite of the softening effect related to electrostatic actuation [62]. Strictly speaking, applying a voltage to electromechanically actuated devices has a softening effect due to reduction in the effective spring constant of the mechanical elements, a phenomena often referred to as electrostatic negative spring [63]. This therefore also implies that the observed stiffening is due to a morphological change of the CNTs.

The source for the stored energy is the fact that CNTs, which become mechanically coupled to one another during the growth process due to van der Waals forces, grow at different rates [64]. Therefore, CNTs that grow faster would experience compression, while CNTs that grow slower would experience tension. The observed alignment (Fig. 7(c)) is consistent with our DC measurements, which measured very small hysteretic strain, due to extension in the top region of the forest. Moreover, it is well known that individual CNTs can be aligned by electric fields [48, 49, 50]. Knowing that the CNTs at the top of the forest are randomly oriented, we hypothesize that the electrostatic field influences slight reorientation of the CNTs toward the vertical direction, as demonstrated schematically in Fig. 8. The hysteretic nature of this field-induced alignment suggests that the process relieves residual strains within the tangled CNT network at the top of the forest, which arise because CNTs exert forces on one another during the growth process. The influence of the field is here limited by the penetration depth into the forest [39]. Therefore the measured change in Young's modulus (Fig. 5), originates mostly from the morphological change of the top portion of the forest, where the electrostatic pressure gets its maximal value and CNTs change their orientation.

Moreover, the measured increase in quality factor (i.e., reduction in material damping), shown in Fig. 6(b), further indicates that AC loading modifies organization of the CNTs within the top of the forest. Note that although

Figure 8: Schematic of the morphological change in CNT forests due to the applied electrostatic forces. CNTs from the top portion become straighter due to the electrostatic loading.

different dissipation mechanisms in individual CNTs have been investigated [65, 66], dissipation due to motion at CNT-CNT contact sites is considered to be dominant in CNT forests [67, 68, 69]. Specifically, stick-slip friction between individual CNTs and detaching (unzipping) of individual CNTs that are initially held together by strong van der Waals forces have been analyzed [70]. Thus, the measurement of higher quality factor is consistent with our observation of straighter individual CNTs, which suggests that due to the applied voltage, CNTs within the top layer rearranged in a manner that the CNT-CNT friction area is reduced (for example, due to smaller number of CNT-CNT contact points). Therefore the dissipated energy is reduced with sequential loading cycles. It is also possible to assume that more aligned CNTs dissipate less energy than tortuous ones.

5. Conclusion

For the first time, we studied the static and dynamic mechanical response of CNT forests under electrostatic loading. Apparent stiffening of the CNT forests after cyclic loading is attributed to morphological change of straightening of the individual CNTs at the top of the pillar in which the electric

field penetrates, along with relaxation of residual strains accumulated during the growth process. Unlike common solid materials such as silicon that are used in NEMS/MEMS devices, CNT forests are much softer and exhibit significant energy dissipation due to internal damping by stick-slip of CNT contact sites. The low stiffness and wide bandwidth of CNT forests under resonance may suggest their use as soft resonant elements that measure small forces over large contact areas, or that electrostatically detect changes in surface conditions of CNTs such as adsorption of molecular species. Thorough understanding of those properties will enable new applications that leverage the capability to design and fabricate CNT forest patterns using conventional lithography and CVD methods.

Appendix A

Measurement uncertainty

The uncertainty in Young's modulus calculation was derived using the method of absolute limit of error, which is a conservative approach that assumes that errors exist in the experiment accumulate. Young's modulus is a function of the following parameters

$$E = \frac{\sigma}{\varepsilon} = \frac{FH}{Au} \quad (\text{A.1})$$

where σ and ε are the stress and strain, respectively. In addition, F , H , A and u are the applied force, micropillar height, cross section area and measured displacement, respectively. According to the absolute limit of error method, the uncertainty is

$$\Delta E = \left| \Delta F \frac{\partial E}{\partial F} \right| + \left| \Delta H \frac{\partial E}{\partial H} \right| + \left| \Delta u \frac{\partial E}{\partial u} \right| + \left| \Delta A \frac{\partial E}{\partial A} \right| \quad (\text{A.2})$$

where ΔF , ΔH , ΔA and Δu are the measurement uncertainties of the force, height, cross section area and displacement, respectively. Note that since uncertainty related to force measurements as well as to geometrical measurements are relatively small, the majority of the uncertainty is originated from the displacement measurement. Therefore, the measurement uncertainty can be approximated as the simplified term below

$$\Delta E \cong \left| \Delta u \frac{\partial E}{\partial u} \right| \quad (\text{A.3})$$

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