A microscale, thermally actuated, uniaxial testing stage for nanofiber materials has been designed and fabricated. Electrical separation of portions of the stage allows two-point electrical measurements simultaneously with in situ mechanical testing. Using this stage, a nanofiber consisting of a carbon nanotube (CNT) surrounded by amorphous carbon was subjected to mechanical loading and simultaneous electrical impedance characterization, which provides a means to derive fiber resistance measurements when a fiber is mechanically coupled using highly resistive contacts. Stress applied to the nanofiber was estimated using measurements of the stage displacement and the input power supplied to the thermal actuator.
As electrical power is increased to the actuator, the heat flow out of the actuator increases. Because of the polysilicon thermal resistance, the temperature rises in the actuator due to the increased heat flow. The temperature increase causes thermal expansion of the silicon, which produces force if the motion of the expanding polysilicon beams is constrained. By angling the actuator beams $1^\circ$ off of perpendicular to the shuttle that transmits force to the test stage, the expanding beams are made to cancel their off-axis motion, and they experience a buckling motion only in the shuttle axis.

Simulations were performed in order to define the behavior of the system across a range of applied input voltages. A preliminary simulation used the values in Table 1, the structure in Fig. 2, and a tetrahedral coupled-field element, SOLID98, in ANSYS 10.0 software. The simulation did not include the temperature dependence of polysilicon electrical and thermal conductivities. This simulation was repeated in COMSOL 3.4 multiphysics simulation software in order to check the results. The COMSOL simulation used 16,637 Quadratic Lagrange tetrahedral elements.

In order to estimate the force applied from the stage to a mounted nanofiber, a displacement boundary condition was assigned to the moving stage. When no displacement constraint is assigned, corresponding to the absence of a test specimen, the simulation provides a parabolic curve of displacement versus voltage, similar in shape to the measured behavior. If the displacement is plotted against the square of the voltage, which is proportional to the input power, a linear relation is evident between power and displacement, as seen in Fig. 3.

At fixed voltages, with varying displacement constraints, the moving stage exhibits a linear reaction force versus displacement. Force simulation as shown in Fig. 4 indicates the analytical estimate of 400 $\mu$N to be a reasonable order of magnitude for a zero micron displacement constraint. The actuator experimentally demonstrated $>1.60$ $\mu$m displacement, also in good agreement with the values estimated in Fig. 4.

### 2.2. Thermal design

In order to minimize the specimen temperature changes caused by the thermal actuator in Refs. [5,6], the moving stage was sep-
beams, thereby modifying the electrical resistance of each beam. For piezoresistive measurements, electrical current is carried in two beams of electron or ion current arriving at fixed and moving electrodes. The electron or ion sensor concept is based on a comparison of electron or ion current in the vicinity of the moving and fixed stage. All require further characterization. The diffraction grating beam bending sensor, and an electron emission or gas ionization sensor. These mechanisms can be seen at left in Fig. 2, and as built in the reported device. Measurement mechanisms were built into the reported device. These mechanisms can be seen at left in Fig. 2, and as built in the center of Fig. 1. They include a diffraction grating, a piezoresistive beam bending sensor, and an electron emission or gas ionization sensor. All require further characterization. The diffraction grating is designed to measure stage displacement using a reflected laser beam. The electron or ion sensor concept is based on a comparison of electron or ion current arriving at fixed and moving electrodes. For piezoresistive measurements, electrical current is carried in two of the polysilicon anchoring beams. Motion of the stage bends the beams, thereby modifying the electrical resistance of each beam.

This concept has been explored in Ref. [20], but further development is required to ensure that resistivity changes are due to piezoresistive rather than thermal effects. The successful implementation of one of the displacement sensors above can be used in combination with bending of a beam of known stiffness to measure a force output to the fiber, as in the approach taken in Ref. [8]. This direct force measurement has not yet been implemented in our design.

2.4. Force calibration

Calibration of force applied to the nanofiber specimen can be derived indirectly from measurement of actuator input power and stage displacement. The simulation shown in Fig. 4 indicates that at any given applied voltage, there is a linear relation between force and displacement. In essence, the beams in the actuator and the beams that stabilize the stage act as a spring. At every input power level, the spring and actuator system equilibrates itself to accommodate the expansion of the beams within the actuator and a steady-state thermal dissipation of power from the actuator. Displacement perturbation in the vicinity of this mechanical equilibrium exhibits a linear force versus displacement behavior, which can be described with a spring constant $K$. Force $F$ can be estimated by comparing measured stage displacement $d$ with the displacement $d_0$ that is expected for a given power input:

$$F = K(d_0 - d)$$

The simulation in Fig. 4 estimates $K = 253 \, \mu N/\mu m$. When an analytical estimate is performed, modeling the spring constant as the sum of the spring constant of perpendicular clamped–clamped beams of varying lengths, and including the beams in the actuator, a value of $293 \, \mu N/\mu m$ is obtained for $K$, which is in the vicinity of the value from the simulation. If the temperature dependence of polysilicon electrical and thermal conductivity is included in the simulation, as in Ref. [14], the force versus displacement behavior diverges from linearity, although it is still linear in the vicinity of the free stage displacement. The magnitude of slope of these curves increases at higher electrical power inputs, indicating that the spring constant $K$ is power dependent and that $K$ from the constant conductivity simulation likely underestimates the highest forces.

The expected displacement $d_0$ can be estimated from calibration based on the input power to the actuators. By measuring the behavior of a freely moving system, the interplay of electrical, thermal, and mechanical behavior is incorporated into the fitting parameters that relate $d_0$ to the input power $P$. ($P=I^2 V$, the current flowing through the actuator times the voltage drop across the actuator.) As seen in Fig. 3, measurements of displacement $d_0$ versus power $P$ on unconstrained stages give linear fits according to the following equation:

$$BP = d_0$$

From these measurements, the linear regression fitting constant $B$ lies in the range of 0.0024 to 0.0034 ($\mu m/W$), with $R^2$ (coefficient of determination) values ranging from 0.82 to 0.97. For the data presented in Fig. 3, Devices 1 and 2, a power law fit provides a slight improvement to these $R^2$ values (respectively, 0.985 and 0.947 for the power law fit versus 0.974 and 0.954 for the linear fit). Because the fitting improvement is only slight for these data series and because the power law fit does not improve the fit to the data from Device 3, the linear fit appears to serve adequately for the data presented here. With improvement in measurement, power-law curve-fitting remains a strategy to further refine the force calibration.

Current and voltage supplied to the actuators were predominantly measured using two point electrical measurements. A
comparison of two point and four point measurements found less than 3% mismatch in actuator resistance values between these methods. From these measurements, it can be inferred that the resistance in the actuator circuit is dominated by the actuator and its connections to the pads on the chip, and not bond pad contact resistance or line resistance from the power supply.

2.5. Electrical characterization

Although two gold connections for the fiber specimen have been provided on the moving stage, they are electrically linked by the underlying polysilicon layer. Two separate electrical contacts were defined on the fixed side of the stage, effectively enabling a three-point conductivity measurement. In practice, the difficulty of carbon nanofiber placement limited actual connections to only two: one on the moving stage, and one on the closest portion of the fixed stage. The fiber was mechanically clamped to the stage with amorphous carbon deposits that also served as electrical connections. The amorphous carbon contacts present a high electrical resistivity, giving contact resistances $R_1$ and $R_2$ values on the order of several megohms.

The structure of the contacts consists of a highly resistive material in a thin layer between two more conductive materials. In essence, these contacts are capacitors with lossy dielectrics. These capacitors lie in series with the fiber specimen, whose resistance is of interest in relation to strain behavior. Capacitors in a series circuit create a high-pass filter. At low frequencies and for direct currents (dc), current must flow directly through the contacts and is therefore limited by the high resistance of the amorphous carbon contacts. As the frequency of an alternating current (ac) increases, the capacitors at the fiber contact points will begin to behave as if the contacts were shorted. Current at higher frequencies will capacitively couple across the contact points and thereby minimize the effect of the contact resistance on the impedance. (Fig. 6) The fiber resistance and the reactance due to the contact capacitances will therefore dominate the measured impedance.

The electrical connection to the moving stage is made by a bending polysilicon beam with a gold layer patterned on top of the beam. The conductivity of the gold dominates that of the polysilicon, so current flows predominantly in the gold layer. Therefore, the piezoresistive change experienced by the polysilicon beam can be neglected. The circuit probing the nanofiber resistance $R_3$ and the contact resistances $R_1$ and $R_2$ has some inherent resistance of about 5 Ω. However, the small cross-section of the nanofiber implies that $R_3$ has a value on the order of kilo-ohms, so $R_3$, $R_1$, and $R_2$ dominate the circuit resistance.

The circuit to the fiber specimen has inductance $L$ which may reduce the magnitude of the observed reactance. Inductive reactance $X_L$ is proportional to angular frequency $\omega$ and inductance. $(X_L = \omega L)$ Capacitive reactance $X_C$ is inversely proportional to angular frequency $\omega$ and capacitance $C$. $(X_C = 1/\omega C)$ When both inductance and capacitance are small, and the ac frequency is kept relatively low (below the MHz range), the capacitive reactance will have a much larger value than the inductive reactance, and therefore dominates the reactance measurement.

2.6. Electrical connections

Three electrical circuits are tied together by the moving stage. These are the thermal actuator circuit, the nanofiber resistance measurement circuit, and, if desired, a displacement measurement circuit through bending polysilicon beams. If these circuits are not tied together at any other point, for instance if no more than one of the circuits is grounded, then the three circuits can operate independently.

3. Experiment

The microdevice was fabricated from polysilicon with Au contacts using the PolyMUMP’s service. [13] The polysilicon layers were released using a 3 min etch in 48% HF$_{aq}$ followed by supercritical CO$_2$ drying.

Carbon nanotube (CNT) core carbon nanofibers were synthesized at the University of Colorado in Boulder using Fe catalyst in a chemical vapor deposition (CVD) reactor at 725 °C; they were subsequently ultrasonicated for 3 h in a toluene solution and dried to form a mat. A three-axis piezoelectric micro-manipulator (Kleinindeck$^{\text{TM}}$) with a tungsten microscopy probe was used for separating an individual nanofiber from the mat. The synthesis and nanotube separation procedures were the same as the ones detailed in Ref. [21]. The nanofiber was then transferred and bonded to its ends to the MEMS tensile stage by performing electron beam-induced deposition (EBID) for 10 min in a JEOL JSM-6480LV SEM operated in spot mode with 30kV acceleration voltage. The device substrate was bonded to a chip carrier, with wire-bonding for electrical connections, and this system was mounted in the SEM.

A variable dc power supply provided electrical current to the thermal actuator. Voltage and current supplied to the actuators were measured using a Hewlett Packard 34401A multimeter. Using an Agilent 4263B LCR meter, the nanofiber resistance and reactance were observed during mechanical loading. The stage displacement and nanofiber length were extracted from SEM images using ImageJ software. High-resolution scanning electron microscopy was performed using a JEOL JSM-7401F field emission SEM, operating at 1.8 kV acceleration voltage.

4. Results and discussion

4.1. Actuator

During operation of the actuator at constant voltage, the electrical resistivity was sometimes observed to decline after the voltage was set at each new level, and similar fluctuation was observed in Ref. [22]. Furthermore, observation (Fig. 7) of actuator electrical resistivity during the carbon nanofiber mechanical test described below indicated that the actuators experienced increased resistance with increasing electrical power inputs, and different electrical resistance values during unloading versus loading, possibly due to piezoresistive effects on the actuators, as has been elaborated in Ref. [22]. Because the actuator electrical resistance varies during an experiment, calibration based on voltage inputs alone is insufficient to predict the un constrained displacement of the stage, which depends on the thermal resis-
tance, power dissipation, and thermal expansion of the actuator beams.

4.2. Carbon nanofiber

Before loading, dc resistance across the carbon nanofiber was $>10 \text{ M}\Omega$. However, measurement at 100 kHz found 5.0 kΩ resistance and $-55.5 \text{k}\Omega$ reactance, indicating that contact resistance dominates the dc measurement. The negative reactance indicates that the reactance is dominated by the capacitance. If the capacitance is inferred from the total reactance, the measured reactance corresponds to an inline capacitance of 28.7 pF, or 57.4 pF at each contact if the contacts are assumed to be identical. Measurement at 10 kHz gave 44 pF inline capacitance. No significant trends in the nanofiber resistance or reactance were observed in the subsequent tensile test, likely indicating that current in the fiber was predominantly carried by outer layers of the fiber without a great degree of piezoresistance.

As the nanofiber was loaded, first it straightened and simultaneously elongated, until direct tensile load was applied to the entire length of the fiber. Failure eventually occurred in the outer layers of the fiber as seen in Fig. 8. As the moving stage was allowed to return to its original position, the nanofiber buckled upon it as seen in Fig. 9, indicating that the failure seen in Fig. 8 was not a complete fracture. In Figs. 10 and 11 it can be seen that only a portion of the strain could be recovered as the fiber was unloaded, indicating that the nanofiber experienced a plastic deformation.

Using the force calibration approach defined above, the stage displacement and actuator input power measurements were mapped into estimates of the force applied to the CNT. In order to calculate the engineering stress applied to the fiber, the fiber diameter was measured using high-resolution SEM imaging, as seen in Fig. 12. For the value of $B$, the fitting parameter from Device 3, which was from the same PolyMUMPs [13] fabrication run as the device used to obtain the carbon nanofiber data in Fig. 3, was used. The value of $B$ was scaled to account for a greater expected displacement than in the calibration device due to beams that did not survive the HF release process. The fitting parameter, $B = 0.00557 \mu\text{m}/\text{mW}$, had a 6% standard error from the calibration data for Device 3, and most of the other sources of error in the related measurements (such as stage displacement, voltage, and current measurements, and variation in the dimensions of the beams of the released actuators) were also within this amount. Better understanding of the accuracy of this force calibration method will be enabled by additional calibration experiments measuring the microsystem spring constant and the expected displacement of devices subjected to different etch conditions during the release step of processing.

Fig. 11 shows the stress–strain response as the fiber straightened and started to take the load along its length. Because typically the outer diameter $D_o \gg D_i$, the inner CNT diameter, for these nanofibers, the loading stress can be estimated by assuming a coreless geometry [21], using $D_o = 140 \text{ nm}$ (Fig. 12). The initial portion of the curve shows the typical linear elastic deformation behavior observed in CNTs and similar materials. A linear fit to the initial portion of the curve (up to 4% strain) yields a slope or Young’s modulus value of $\sim 350 \text{ GPa}$. This value falls well within the experimental values reported in the literature [3,8]. It should be noted that the CNTs used in this study had amorphous coating (as thick as 30 nm) surrounding its outer shell, due to excess reaction gas pyrolysis during the CNT synthesis. The amorphous carbon greatly degrades the mechanical properties as compared to pristine CNTs (which can have a Young’s modulus value as high as 1 TPa).

Further extension of the nanofiber to $\sim 5\%$ strain resulted in permanent damage to its outer shells, giving rise to plastic behavior...
Subtraction of the nanofiber length change from the stage displacement provides a measurement of slippage or elastic deformation in the clamps. In the elastic portion of the tensile curve, the displacement in the clamps increases, but then remains approximately constant (0.38 ± 0.06 μm) in the plastic portion of the tensile curve while nanofiber strain increased. It is not surprising that there should be some deformation within the clamps, because clamp failure was commonly observed in bending experiments reported by Singh et al. [23] using the same carbon nanofibers and clamping techniques.

5. Conclusions

A stage for electromechanical testing of micro- and nanoscale fibers has been designed, simulated, and fabricated. The stage was used for electrical measurements continuously during a mechanical tensile test of a carbon nanofiber.

The use of ac two-point impedance measurements has been demonstrated as a means to bypass the high contact resistance from mechanical welds to electrically conducting tensile specimens.

For carbon nanofiber testing, specimens were placed using a micromanipulator and bonded using amorphous carbon deposited in an SEM. The development of faster approaches to displacement measurement and to nanofiber placement is needed to improve the quality of mechanical characterization data of nanoscale fibers. By measuring the stage displacement and actuator input power, the stress applied to a carbon nanofiber during a tensile test was estimated. A typical telescopic mode of failure involving breaking of outer shells was realized.

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References


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