Experimental Mechanics (2011) 51:1–9
DOI 10.1007/s11340-010-9437-0

The Evolving Role of Experimental Mechanics in 1-D Nanostructure-Based Device Development

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Received: 11 August 2010 / Accepted: 2 November 2010 / Published online: 30 December 2010

Abstract Future generations of transistors, sensors, and other devices maybe revolutionized through the use of one-dimensional nanostructures such as nanowires, nanotubes, and nanorods. The unique properties of these nanostructures will set new benchmarks for speed, sensitivity, functionality, and integration. These devices may even be self-powered, harvesting energy directly from their surrounding environment. However, as their critical dimensions continue to decrease and performance demands grow, classical mechanics and associated experimental techniques no longer fully characterize the observed behavior. This perspective examines the evolving role of experimental mechanics in driving the development of these new devices. Emphasis is placed on advances in experimental techniques for comprehensive characterization of size effects and their coupling, as well as assessment of device-level response.

Keywords Nanodevices · Nanowires · Nanotubes · MEMS · NEMS · Semiconducting materials · Fracture · Elasticity

This is the 8th, and final, in a series of featured review articles to celebrate the 50th anniversary of Experimental Mechanics. These articles serve to touch on both areas of mechanics where the journal has contributed extensively in the past and emergent areas for the future.

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Introduction

The International Technology Roadmap for Semiconductors (ITRS [1]) identifies emerging technologies with the potential to sustain Moore's Law. A necessary succession from planar CMOS to non-planar/dual-gate CMOS, and ultimately to novel architectures such as nano-electrical and nano-electro-mechanical systems (NEMS) is envisioned. This potential is emergent in numerous demonstrations of outstanding and often unique performance and functionality of devices constructed from one-dimensional nanostructures (e.g., nanowires, nanotubes, and nanorods). These demonstrations include ultra-sensitive chemical and biological detection (down to single viruses) [2–8], nano-scale energy harvesting [9–14], photonic waveguides [15], nano-scale transistors [16, 17], ultra-fast electrical switching [18, 19], low leakage and high on/off ratios [20], high integration levels [21, 22], and outstanding current-carrying capacity [23, 24].

The ITRS also identifies critical roadblocks currently precluding advances beyond CMOS to nanostructure-based device architectures. For example, primary among the roadblocks to NEMS are poor reliability, poor uniformity in the nanostructures from which they are constructed, and manufacturing challenges related to manipulating large numbers of nanostructures into well-ordered arrays. Understanding the mechanics of these nanostructures, and their coupling to other domains (e.g., electro-mechanical coupling), is thus critical to overcoming the roadblocks laid out by the ITRS, as well as meeting technological requirements beyond those of the semiconductor industry.

As these devices shrink exponentially and their performance continues to improve, they similarly demand increasingly powerful experimental techniques to characterize them unambiguously. Numerous theoretical and

experimental studies report a size dependence of material properties occurring with enhanced surface-to-volume ratios at the nanoscale [25–28]. The task of probing size-related effects becomes increasingly challenging as specimen sizes continue to shrink. From an applications and device development perspective, it is crucial to accurately characterize mechanical properties wherever the nanostructures experience mechanical deformation to achieve functionality. This is key to ensuring reproducibility in performance and overall device reliability. Taking nanogenerators as an example, piezoelectric nanowires are mechanically deformed to generate electrical energy [10, 12–14, 29]—therefore, it is important to know the forces required for deformation, as well as the limits to the nanostructures' deformability. Beyond the purely mechanical properties of the nanogenerator, it is also important to characterize the coupling between forces applied to the nanowires and the corresponding voltages generated across them.

Experimental characterization techniques are progressing rapidly as the field pushes for greater functionality and integration from the next generation of devices. These advances will likely be facilitated not only by exploiting the unique mechanics of one-dimensional nanostructures [30], but also their coupling to other physical domains. This perspective examines the evolution of these techniques as they facilitate development of next-generation one-dimensional nanostructure-based devices, ultimately on a mass scale. This encompasses studies ranging from characterization of fundamental mechanics and their multiphysics coupling, size effects in these properties, as well as device-level evaluation of performance metrics and failure modes.

Characterizing the Mechanics of One-Dimensional Nanostructures

As their critical dimensions shrink toward quantum limits, the physical behavior of materials no longer necessarily follows classical mechanics. Following the quest of "smaller being stronger", experimental and computational techniques have evolved rapidly over the past decade. This has allowed researchers to move beyond the realm of thin films [27, 31–33] and micro/nanopillars [26] to probe sub-100-nanometer length scales, where size-dependent material behavior becomes more prominent. As opposed to micro/nanopillars (as low as 250 nm in diameter), which can be tested using well-established nanoindentation-based methods [34–37], thinner 1-D nanostructures with larger aspect ratios pose additional challenges. Various direct and indirect experimental techniques have either been adapted from larger-scale methods or developed specifically to investigate the mechanics of one-dimensional nanostructures. These techniques can broadly be categorized in the following domains: (i) *in situ* dynamic resonance tests; (ii) Atomic Force Microscopy (AFM)-based tests (some performed *in situ* electron microscopes); (iii) micromechanical testing platforms, and; (iv) microelectromechanical system (MEMS)-based testing.

In dynamic resonance tests, the nanostructure of interest is clamped at one end to an electrode and resonated either thermally or electrostatically via an opposing electrode [38–40] [Fig. 1(a)]. An alternating voltage of tunable frequency is applied across the two electrodes. This experiment is conducted *in situ* an electron microscope such that the applied driving frequency can be tuned to match the natural frequency of the nanostructure. Since the integration time required to capture an image in electron microscopes is significantly longer than the period of the nanostructure vibration, the resonating nanostructure appears blurred in the image. The resonant frequency is identified by sweeping the driving frequency to maximize the envelope of vibration in the image. The Young's modulus can then be calculated based upon the measured resonant frequency. This method has been applied to study ZnO nanowires [39], GaN nanowires [41], and CNTs [38, 40].

In addition to resonance-based tests, atomic force microscopy (AFM) has also been applied extensively to characterize the mechanical properties of 1-D nanostructures. Here the extremely high force and displacement resolutions are exploited for finely-controlled experiments. The conventional AFM has in general been extended to mechanical characterization of 1-D nanostructures [Fig. 1(b)] in one of three configurations, all of which use the AFM cantilever as a load and displacement sensor: (i) fixed-fixed specimen configuration in lateral force mode [42]; (ii) fixed-fixed specimen configuration with load applied normal to the plane of the substrate in contact force mode [43]; (iii) singly clamped specimens where the free end is displaced in lateral force mode [44]. In each of these configurations, the specimen is loaded in bending or tension by controlling the motion of the AFM cantilever in the direction of loading. In addition, a new AFM-based technique referred to as Contact Resonance-AFM (CR-AFM) has been developed which relies on the changes in the dynamic response of an AFM cantilever when it interacts with a substrate versus when it is interacts with a nanowire [45, 46]. The differences in resonance frequency response on the substrate and nanowire can thus be used to infer the elastic properties of the nanowire. While the AFM offers high force-displacement resolution, it is limited in the sense that the direct observation of deformation or failure mechanism during the test is not possible. Therefore, researchers have also made use of the AFM cantilevers alone as load and displacement sensors to perform mechanical tests *in situ* electron microscopes [47–51]. In these experiments [Fig. 1(c)], one or both ends of the 1-D nanostructure are attached to AFM cantilevers and a piezoelectric manipulator is used for

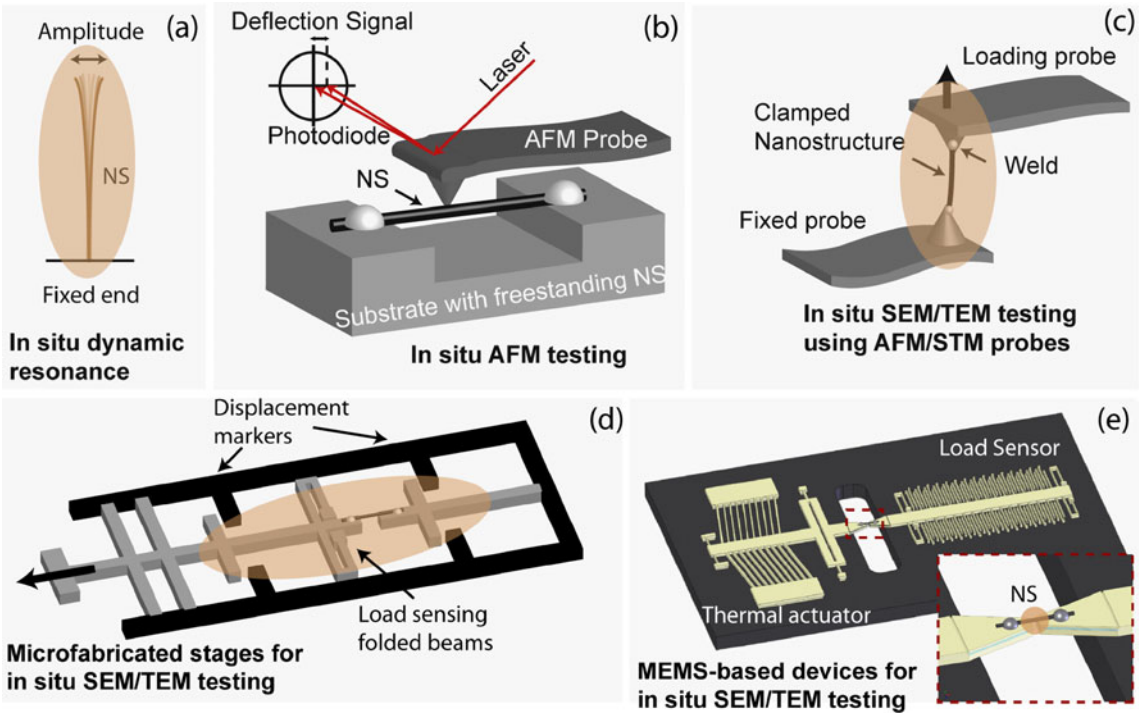

Fig. 1 Overview of experimental techniques developed for mechanical characterization of 1-D nanostructures. For *in situ* electron microscopy techniques, shaded area shows the field of view required to make a quantitative assessment of mechanical properties

controlled movement of the cantilever to apply desired displacement controlled loading. By imaging the deflection of the cantilevers using the electron microscope, the corresponding force is determined.

Beyond the use of the AFM-based techniques customized for 1D nanostructures, development of microfabricated micromechanical stages and MEMS-based testing platforms has been pursued. For example, a customized test-bed is developed with a gap between which the specimen is placed [52]. One end of the test bed is fixed and the other is attached to a customized stage used to apply displacements using a piezoactuator [53]. The force is deduced by observing the deflection of the freestanding folded beams (with known stiffness) *in situ* an SEM [Fig. 1(d)]. The displacement or the deformation of the specimen is monitored by observing the motion of the displacement markers. This technique allows uniaxial loading along with the *in situ* capability; however, the field of view is determined by the location of the displacement markers. To overcome this limitation, a nanoscale-Material Testing System (n-MTS, [54–56]) has been developed, which exploits MEMS technology to acquire load and displacement information electronically [Fig. 1(e)]. Therefore, one can have a variable field of view and observe the specimen with high resolution as desired.

The various techniques described above have built upon each other, ulitmately leading to better understanding and improved consistency of obtained results. For example, Fig. 2 shows the scatter in measured values of elastic modulus values and fracture strength reported for multi-walled carbon nanotubes over the last decade. As techniques evolved, the initially widely-scattered reported results have converged toward theoretical predictions [57]. Similar converging trends have also been observed for other nanostructures, including semiconducting zinc oxide [58, 59] and gallium nitride [60]. Figure 3 shows the elastic modulus reported for zinc oxide (ZnO) nanowires as a function of their characteristic size, using different experimental techniques. Similar to the results for carbon nanotubes, the scatter in experimental data for ZnO nanowires was also significant until recently when the size dependent elastic response was characterized using a combined experimental-computational approach [58]. The reasons for improved accuracy are discussed in the specific references.

Characterizing Electro-Mechanical Coupling in One-Dimensional Nanostructures

Coupling between physics domains (e.g., electro-mechanical coupling) enables additional degrees of device functionality. Strong size effects in this coupling can further be leveraged to achieve greater gains in performance from nanostructure-based devices relative to larger counterparts. For example, the piezoresistive response of silicon nanowires was shown to be two orders of magnitude greater than that of bulk

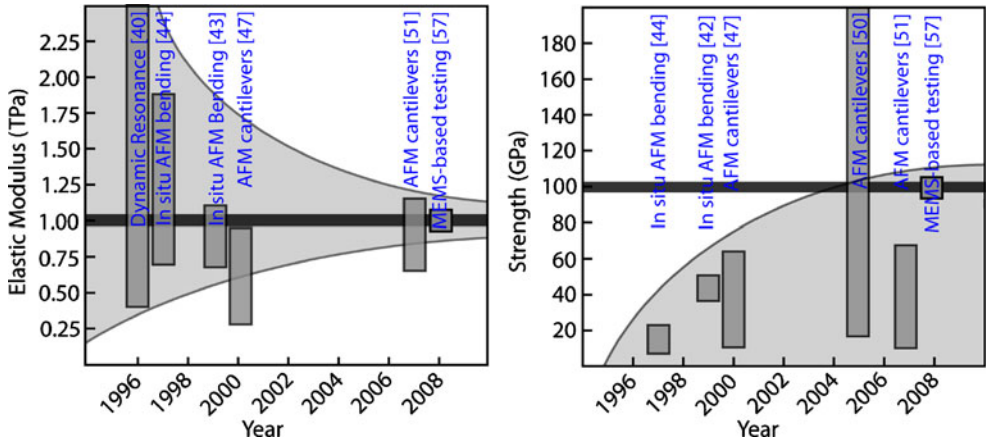

Fig. 2 Elastic Modulus and fracture strength values reported for MWCNTs over the last decade showing the trend converging to theoretical predictions

silicon [61]. Thus by using silicon nanowires, a new level of sensitivity in electromechanical strain sensors can be achieved that would otherwise not be possible with micro-scale silicon components. Given the multiphysics coupling and the potential for strong size effects, purely mechanical experimental techniques must further be adapted to simultaneously characterize electrical response or input.

Coupling Through Electronic Band Structure

In carbon nanotubes, the mechanical and electrical responses are coupled through the band structure. Straining the graphene lattice can either open or reduce the bandgap, changing the overall conductance of the nanotube [62–65]. Whether the conductance increases or decreases depends on the chirality of the CNT [62]. In the case of most metallic CNTs (with the exception of armchair chiralities whose symmetry is maintained under tensile strain), tensile strain opens the bandgap, resulting in a significant increase in resistance. For semiconducting CNTs, depending on their specific chirality, conductance will either increase with tensile strain through creation of a dip in the energy bands, or decrease due to further opening of the bandgap [62]. The bandgap of various nanowires has similarly been demonstrated to be affected by strain [66, 67].

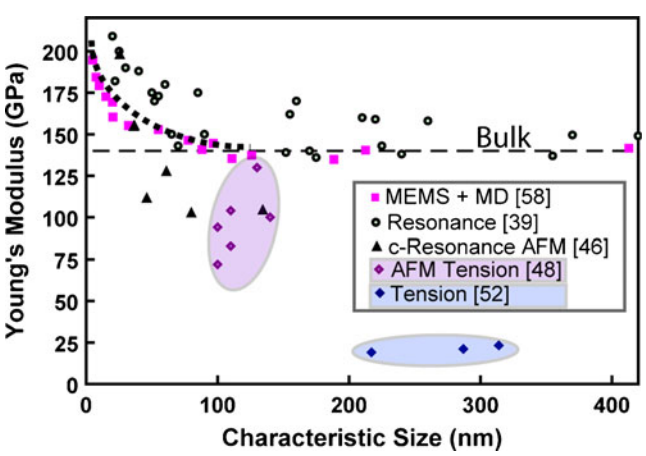

Fig. 3 Young's modulus of ZnO nanostructures as a function of their characteristic size obtained using different experimental techniques

The effect of strain on the bandgap and conductivity of carbon nanotubes has most commonly been assessed through AFM-based techniques, and supplemented with computational analyses [62–65, 68]. These techniques leverage the high force and displacement resolution of the AFM. Other techniques used two- and four-probe measurements to identify local barriers created by sharp bends in the CNT [69]. Pioneering AFM-based techniques employed sharp AFM probes to deflect single-walled carbon nanotubes suspended in a fixed-fixed configuration between two metal contacts [63, 68] (similar to that shown in Fig. 1(b)). The large changes in conductance with strain were believed to be the result of either large local deformation at the point of the sharp probe contact resulting in formation of local sp^3 bonds [63], or alteration of the bandgap [70, 71]. Later work, which used a similar AFM-based technique with the additional ability to use the AFM as a gate electrode, demonstrated experimentally that this was indeed the result of strain-induced alteration of the bandgap [62]. This enabled simultaneous characterization of conductance as a function of both strain and gate voltage. Subsequent comparison to computational/theoretical results enabled estimation of the chirality of the CNT sample, as well as the strain-bandgap relation. Later work sought to reduce spurious effects (e.g., the effect of the sharp probe locally deforming the CNT), by using more advanced constraints. For example, by fixing the CNT at one end [64] (or both [65]) to a rigid support and the other end (or center) to a flexible beam, the flexible beam could be deflected using the AFM probe to strain the CNT, rather than directly contacting the CNT with the probe.

Coupling Through Piezoelectric and Piezoresistive Effects

Piezoelectric or piezoresistive effects provide another potential mechanism by which the mechanical and electrical response of the nanostructure maybe coupled. Since the

mechanical properties, particularly fracture stresses and strains, improve significantly (5-6% strain at failure as opposed to <1% for bulk materials), 1D nanostructures also constitute ideal candidates for devices where mechanical deformation is inevitable to achieve functionality. In addition to high deformability, the miniature sizes of these nanostructures offer an additional advantage over their macroscale counterparts, i.e., the forces required to deform them are small enough to be derived from renewable sources of energy like flowing water, wind energy, etc. Therefore, the conversion of vibrational and mechanical energy into electricity using piezoelectric nanoscale materials is emerging as an effective route alongside the more extensively studied solar and thermal based methods [72–76]. This approach, in the long-term, can lead to the development of self powered devices, which gain their energy from the environment through self sustained energy resources. Increasingly, this area of research is being explored by taking advantage of piezoelectric phenomena to harvest and store energy using nanoscale materials [12, 29, 77]. Still, we lack a fundamental understanding of the enhancement in piezoelectric effects which can be achieved by reducing the characteristic size scales of the materials, particularly piezoelectric nanowires.

A great deal of work has individually addressed the electrical [78–80] or mechanical [56, 81–86] behavior of nanostructures. However, electromechanical coupling and its potential size-effects, have not been thoroughly addressed. Due to difficulties in sample manipulation at the nanoscale, electrical measurements are typically based on positioning electrical connections in contact with as dispersed or as grown samples on a substrate, with minimal sample manipulation. Some examples of this are electrical measurements based on conductive atomic force microscopy on individual NWs [78], conventional electrical measurements on forests of NWs [80], and microfabrication of electrodes on as-deposited NWs for electrical measurements [79]. While these approaches are suitable for electrical measurements, they cannot be adapted for *electromechanical* stimulation and measurements. A few initial experimental approaches have been applied to measure the piezoelectric properties of NWs beginning to address the challenge of *coupled* electro-mechanical measurements. The majority of these are again AFM-based methods, most notably utilizing the technique of piezoresponse force microscopy (PFM) [87–90]. While such AFM based techniques are straight forward to apply experimentally to as grown nanostructures, they do not provide a well defined method of measuring and interpreting piezoelectric properties. *Limitations of these methods include: (i) determining the electrical* properties of the AFM tip/specimen contact; (ii) loading configurations which do not allow uniaxial straining measurements (typically leading to complex and poorly defined stress distributions); and (iii) significant systematic errors in the calibration of force sensors. Alternative methods based on measuring the shift in resonance frequency of NWs have also been presented which report order of magnitude increases in piezoelectric constants for ZnO [91]. Resonance based methods, however, also lead to non-uniaxial deformation as well as complexities in analysis and data interpretation. The piezoelectric coefficients determined from various studies show a large scatter for a given characteristic size, as summarized in Fig. 4.

This emphasizes the need for development of alternative experimental techniques to achieve unambiguous characterization and interpretation of the piezoelectric effect. One such technique is based on augmentation of the MEMS-based platform developed by Espinosa and co-workers. By insulating the platform on which the specimen is mounted from remaining electronic circuitry required for mechanical loading, the specimen's electrical response is simultaneously probed white it is being mechanically deformed. With this modification to the MEMS-based uniaxial tensile testing set-up, one can probe the electromechanical response in a straightforward manner with minimal assumptions. Figure 5 shows a set of I–V curves obtained on doped and undoped GaN nanowires as a function of applied uniaxial tension. The non-linear electrical response suggests non-ohmic behavior which highlights another challenge of obtaining good electrical contacts with the specimen and to ensure that the actual material properties are not influenced by the behavior of electrical contacts. In this vein, further modifications are envisioned allowing for four-point electrical measurements.

Device Level Testing and Analysis of Failure Modes

As outlined by the ITRS, improving the reliability of devices constructed from 1-D nanostructures represents a

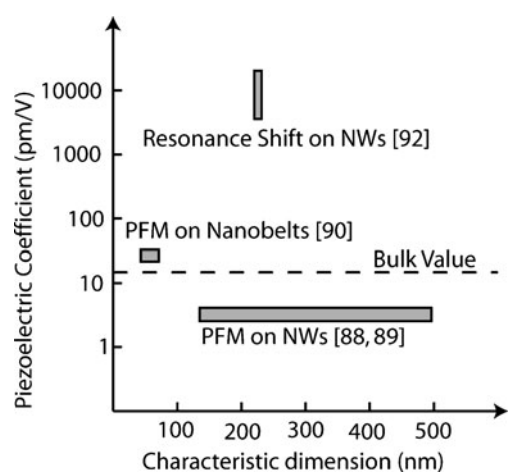

Fig. 4 Plot showing the scatter in piezoelectric coefficients for different characteristic sizes of zinc oxide nanostructures using different experimental techniques

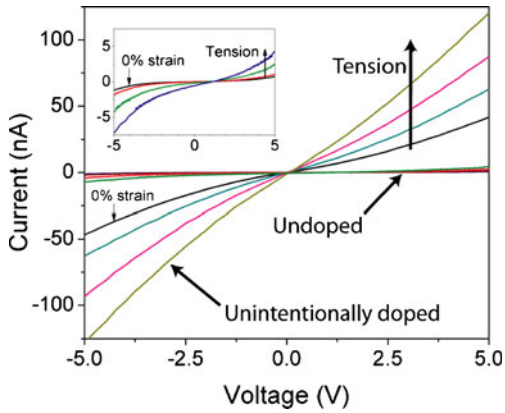

Fig. 5 I–V response of doped/undoped GaN nanowires as a function of applied uniaxial tension [92] (By permission from *MRS Bulletin*, www.mrs.org/bulletin)

critical challenge in their development [1]. Beyond comprehensive knowledge of the fundamental behavior of the nanostructures themselves (as described above), this requires an analysis of the conditions encountered by the nanostructures during normal operation (e.g., within a NEMS device) and the corresponding modes of failure.

Characterization at the device level is inherently more complex. Advanced device architectures lead to complex loading states, mixed or ill-defined boundary conditions, and in many cases, a highly dynamic response. For example, nanoelectromechanical devices composed of one-dimensional nanostructures can change states at Gigahertz rates or faster [18, 19, 93]. Consequently, they experience extreme (and coupled) mechanical, electrical, and thermal loading in a period of nanoseconds or less. Thus, while the primarily quasi-static techniques described above are directly applicable in the design phase of these devices, unique implementations or altogether different methods are required at the device level.

Electromechanical characterization of NEMS constructed from one-dimensional nanostructures is most commonly conducted *in situ* the scanning electron microscope (SEM) [21, 94, 97–101]. This enables high resolution imaging along with the full suite of electronic characterization that would otherwise be available *ex situ*. For example, SEM imaging is often used to determine physical device state (e.g., open or closed switch), and correlate this state to corresponding current-voltage measurements [21, 94, 97–99, 101]. Perhaps more importantly in the context of improving reliability, this also enables direct imaging of device failure [94, 99]. For example, a parametric study into the design space of freestanding carbon nanotube-based NEMS switches was conducted *in situ* the SEM [94]. As shown in Fig. 6, this enabled identification of prevalent failure modes (e.g., irreversible stiction between the carbon nanotube and electrode, and ablation of the tip of the nanotube) and mapping of their point of onset within the design space.

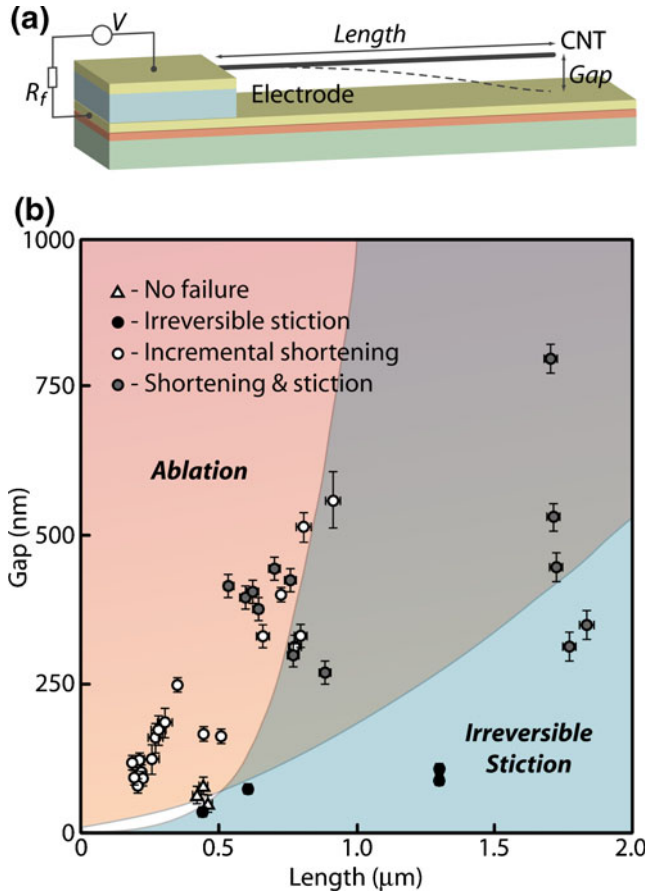

Fig. 6 Results from *in situ* electromechanical characterization of carbon nanotube-based nanoelectromechanical switches [94]. (**a**) Schematic of the cantilevered nanotube device consisting of a single carbon nanotube cantilevered over an electrode. (**b**) Map of various failure modes identified through *in situ* characterization within the geometric design space. The highly limited remaining region (*white*) in which failure is avoided highlights the need for further developments to improve the robustness of these NEMS devices. Potential solutions include the use of alternative electrode materials [94], electrode surface coatings [95], and more complex actuation schemes [21, 22, 96, 97] (By permission from Wiley)

However, the limited speed of imaging using electron microscopy effectively limits assessment of mechanical device state and failure modes to quasi-static analyses (though the electrical measurements are not limited in speed). For example, the switching time for these devices is on the order of nanoseconds, versus an integration time of seconds for high-resolution SEM imaging. As a result, the device state before and after actuation is captured, but the transient dynamics of the switching event itself (where, for example, the most extreme mechanical deformations occur) are lost. As the spatial and temporal resolution of electron microscopy continues to advance [102, 103], it may become possible to visualize these highly dynamic events directly. In addition, as discussed further below, *in situ* characterization using alternative tools (e.g., Raman spec-

troscopy) will provide richer data beyond basic imaging to correlate with corresponding electrical measurements.

Concluding Remarks

As the dimensions of one-dimensional nanostructures continue to decrease, textbook mechanics begin to break down in describing their behavior. Advances in experimental mechanics, such as those described herein, are thus critical to fully understanding and leveraging the outstanding properties of these unique structures. The rapid rate of development in this field is evident in examining, for example, the evolution of techniques used to characterize the modulus and strength of carbon nanotubes. Within the past decade, reported values for the modulus have converged from more than an order of magnitude of scatter to well within 10% of the theoretically-predicted modulus [57]. A similar scenario occurred in the characterization of NWs [58, 59]. Given these powerful experimental techniques, a number of intriguing size effects have been identified in the modulus, fracture properties, and electromechanical coupling of one-dimensional nanostructures.

Ongoing developments will continue to advance our understanding of the fundamental properties of smaller and smaller nanostructures. In turn, this will push the development of novel devices leveraging these nanostructures to set new benchmarks in performance. Evolving *in situ* techniques in particular will continue to provide rich insight into fundamental and device-level characteristics.

With continued improvements in the spatial and temporal resolution [102, 103], *in situ* electron microscopy tests will advance from providing quasi-static imaging of overall nanostructure response to capturing the dynamic response with atomic resolution. The *in situ* combination of MEMS-based characterization tools and high-end microscopy techniques together can provide further insights on atomic scale phenomena relevant at small scales. Moving beyond DC MEMS-based techniques, incorporation of AC fields will enable more detailed investigations into electromechanical coupling and their size effects.

Beyond electron microscopy, other conventionally bulk analysis tools are being pushed to characterize smaller and smaller structures. Incorporating these techniques into *in situ* analyses provides a new level of comprehensive characterization. For example, Raman spectroscopy provides a wealth of information including chemical composition, bond structure and defect density, orientation, strain, pressure, and temperature [104–113]. Near-field Raman spectroscopy pushes the resolution further, going beyond the diffraction limit to map properties in one-dimensional nanostructures. Hybrid techniques combining FTIR, photoluminescence, and/or fluorescence imaging provide even more flexibility. Finally, further in the future, it maybe possible to conduct *in situ* tests using angle-resolved photoemission spectroscopy (ARPES) measurements. This will enable direct characterization of electronic band structure and, for example, how it is affected by strain during cyclic operation [114].

Thus, as dimensions continue to shrink and performance demands grow, the role of experimental mechanics in nanoscale device development continues to evolve. Conventional macroscale techniques designed to provide well-defined boundary and loading conditions have been scaled down to micro-scale tools and loading stages. In the future, these will evolve further to provide the same level of comprehensive analysis at the device level to characterize failure modes and their fundamental causes. Ultimately, experimental mechanics will play a crucial role in the development of next-generation sensors and electronics promising revolutionary performance.

Acknowledgments This work was supported by the National Science Foundation through awards No. CMMI-0555734, DMR-0907196, and the Nanoscale Science and Engineering Initiative under NSF award EEC-0647560, the Army Research Office though awards No. W911NF-08-1-0061 and W911NF-09-1-0541, and the Office of Naval Research through award N00014-08-1-0109.

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