Nanomaterial Characterization Using Actuated Microelectromechanical Testing Stages

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Nanomaterial Characterization Using Actuated Microelectromechanical Testing Stages

by

Joseph James Brown

A.B., Dartmouth College, 2000
M.S., University of Colorado at Boulder, 2008

A thesis submitted to the
Faculty of the Graduate School of the
University of Colorado in partial fulfillment
of the requirements for the degree of
Doctor of Philosophy
Department of Mechanical Engineering

2010
This thesis entitled:
Nanomaterial Characterization Using Actuated Microelectromechanical Testing Stages
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The final copy of this thesis has been examined by the signatories, and we find that both the content and the form meet acceptable presentation standards of scholarly work in the above mentioned discipline.
In this work, microfabricated mechanical systems have been created in a variety of forms and operated to perform nanomaterials characterization tests. A simplified integrated test system was developed and used to collect data from a range of materials including gallium nitride nanowires. A new force estimation approach was developed which enables estimation of the forces provided by electrothermal microelectromechanical (MEMS) actuators, and with knowledge of a material specimen cross-section area, an estimation of the engineering stress within the nanomaterial specimen.

In an expanded design, a MEMS micromanipulator probe interfaced with a removable specimen holder, also known as a test coupon, to apply strain to and acquire tensile data from carbon nanotubes grown directly on a test coupon. A novel approach for removably interfacing two microfabricated chips was created. This interface mechanism enables the test coupon to incorporate a selection of possible experiments. These test devices can be operated in vacuum or air environments, and serve as a proof-of-concept of a microsystem testbed for mechanical measurements that can be performed simultaneously with other types of measurements such as electron diffraction, piezoresistive measurement, scanning tunneling microscope observation, or optical measurements.

The goal of this thesis was the demonstration of a microsystem capable of performing tensile characterization of nanowire or nanotube specimens that are not permanently interfaced to the actuators used to apply mechanical strain, with emphasis on the overall operation and characterization of this system.
Dedication

To everyone.
Acknowledgements

Many thanks to my thesis committee and everyone who has supported me for so long. This thesis would not be possible without everyone who has touched my life, and I especially want to mention the many people who helped me in the process that led to this thesis:

Advisor: Prof. V. Bright
Thesis Committee: Prof. Y. C. Lee (U. of Colorado), Dr. P. Kabos (NIST-Boulder), Prof. C. Rogers (U. of Colorado), Prof. J. S. Bunch (U. of Colorado), Prof. Dr. Ch. Hierold (ETH-Zürich)
CU-Boulder: Prof. C. Stoldt, P. Rice, D. Miller, the faculty and staff of the Department of Mechanical Engineering
CU MEMS group: Dr. H. C. Chuang, K. Cobry, W. Krauser, B. Davidson, D. D. Luu, A.I. Baca
Other Collaborators: Prof. D. Dikin (Northwestern U.), Prof. R. Ruoff (U. Texas), Assistant Prof. G. Singh (Kansas State U.), Dr. M. Wallis (NIST-Boulder), Dr. K. Bertness (NIST-Boulder), M. Muoth (ETH-Zürich), J. W. Suk (U. Texas)
Others at NIST: N. Sanford, A. Sanders, N. Orloff, J. Beall, L. Vale, G. Hilton, J. Britton, J. Whittaker
Others at ETH-Zürich: E. Köpilä, C. Boutry, N. Wojtas, Dr. U. Lang
Family: L. and R. Brown; L. Brown and J. Beardsley; extended family including especially M. and J. Kozlowski, S. Carlson Cathey, D. and A. Bozzi
Mentors: Prof. U. J. Gibson, Dr. R. C. Dean, Jr., Dr. D. S. Lashmore
Funding for this thesis came primarily from the US National Science Foundation, NIST-Boulder, and from DARPA. From September 2007 - August 2010 I held a NSF Graduate Research Fellowship. Additional laboratory support in August 2009 - May 2010 came from the Micro and Nanosystems group at ETH-Zürich.

This research was supported by the DARPA Center on Nanoscale Science and Technology for Integrated Micro/Nano-Electromechanical Transducers (iMINT) funded by the DARPA N/MEMS S&T Fundamentals Program (Original Award #HR0011-06-1-0048, Dr. D. L. Polla, Program Manager. Renewed Award #N66001-10-1-4007, Dr. T. Akinwande, Program Manager).

Fabrication of test coupon and Universal Test Platform devices occurred in the NIST-Boulder Quantum Fabrication Facility.

Unless otherwise noted, characterization occurred at the CU-Boulder Nanomaterials Characterization Facility. Vibrometer, AFM, and most TEM characterization occurred at the laboratory of the Micro and Nanosystems Group, Department of Mechanical and Process Engineering, ETH-Zürich. Microwave and RF characterization and some optical microscopy occurred at NIST-Boulder.
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Chapter 1

Introduction

1.1 Goals of Thesis

The development of methods, microdevices, and data analysis for nanomaterial mechanical testing was the overall goal of this thesis. The devices created were structures for tensile testing of carbon nanotubes and semiconductor nanowires. These devices were subject to the constraints that mechanical testing could be combined with other tests, that the devices would operate in varying environments both inside and outside of vacuum, and that specimen handling could eventually be separated from microactuation functions. This work sought to make an operational nanomaterials characterization tool and show it could collect useful data.

Following from the thesis goal was the core design hypothesis of this thesis: The separation of a specimen-holding test coupon from an actuated microfabricated test platform would allow data collection in varied experimental environments by reducing constraints on the test platform fabrication process and on the types of nanomaterial specimens that may be tested.

Attainment of the thesis goal came through realization of the following specific technical objectives:

(1) Fabrication of a test platform with integrated probe system.

(2) Fabrication of a coupon chip that can support a nanowire or nanotube specimen.

(3) Demonstration of mechanical interfacing between the coupon chip and the test platform system, including three-dimensional alignment of mechanical components.
(4) Development of uncertainty analysis of data collected with microfabricated tensile test systems.

(5) Collection of tensile data from material specimens.

(6) Demonstration of actuated probes from the test platform effecting motion within the test coupon.

(7) Collection of nanowire or nanotube straining data from the coupon and test platform system.

(8) Demonstration of an experiment collecting tensile test data in conjunction with one other type of data, which was effected in the electron diffraction of strained carbon nanotubes.

Additional technical objectives were proposed at the start of this work, but they were unable to be fully implemented, mostly due to time constraints:

(1) Design and implementation of an on-chip displacement sensor which can collect displacement data from the integrated probe on the test platform.

(2) Development of a methodology for calibrating against a predictable, controllable standard any force and displacement data which may be obtained from a microfabricated test system.

1.2 Motivation

Advances in nanomaterials synthesis have enabled the production of materials such as carbon nanotubes (CNTs) and gallium nitride nanowires (GaN NWs) with nanoscale dimensions and a high degree of perfection. Small size scales and lack of defects allow the realization of many interesting properties, and access to these properties may be useful in design of submicron structures and devices. The design cycle for new device applications requires data collection and characterization of new materials’ relevant properties, and the uniformity of these properties, before they can be incorporated in new designs.
The generation of new designs incorporating nanoscale materials requires development of strategies for handling, fabrication, and interfacing for each new material used, and better data relating the structure of these materials and their observed properties. One example is that of the nanotube strain gauge, which may offer much greater sensitivity than a miniaturized silicon strain gauge. Use of nanotubes for a strain gauge and other force-sensing applications will require a precise cross-correlation of the structure of the nanotube with its mechanical properties and electrical properties, especially under loading. [47, 49]

Nanotubes and nanowires have garnered much attention since Iijima’s 1991 paper detailing the structure of carbon nanotubes. [57] Although the modeling of the structure of carbon nanotubes indicated they would have very high mechanical strength, stiffness, thermal conductivity, and a range of electrical properties depending on structure, these predictions did not begin to be confirmed until later in the 1990s, when a series of experiments began to derive mechanical, electrical and thermal data for these materials. [28, 116] Despite advances in understanding the physics of nanotubes, current technology for manipulating isolated nanotubes has remained cumbersome and tedious.

Because they may be nearly defect-free single crystals or they may have other size-dependent properties, tensile tests of nanomaterials specimens such as nanotubes and nanowires are of significant scientific interest. For instance, mechanical testing provides one way to probe the physics of nanoscale lattice structures, which may have significantly higher strength and strain to failure than their macroscale equivalents due to the absence of dislocation motion, and which may also manifest strain-dependent electron and phonon transport behaviors.

Gallium nitride is a direct wide-bandgap semiconductor with good thermal conductivity and notable optical, mechanical, piezoelectric, and transport properties. [92, 118, 121, 141]. GaN nanowire cantilever resonators have been shown to have a high mechanical quality factor, which makes them appealing in nanomechanical devices. [141] Recent developments in the synthesis of gallium nitride nanowires have illustrated that these structures can be created free of defects and residual strain. [5] These findings suggest that the nanowire morphology will offer numerous ap-
applications that would otherwise be unattainable in conventional epitaxial growth of this material, where it is widely recognized that GaN lattice defects interfere with optical and electrical performance. Integration of GaN NWs with active microelectromechanical system (MEMS) devices may allow development of new classes of tunable mechanical resonators, light-emitting diodes (LEDs), lasers, and switches, among other possible new sensor, transistor, and transducer technologies. Development of comprehensive mechanical data on gallium nitride nanowires (GaN NWs) will enable the realization of such new devices and applications. For instance, the ability to withstand $0.042 \pm 0.011$ strain or $7.5 \pm 3.4$ GPa tensile stress, as reported in Section 6.3, reflects exceptional strength and resilience, indicating that GaN nanowires could eventually become robust components of new micro and nanoscale mechanical systems.

Laboratory instrumentation and equipment have been key to advancing the characterization and design of structures with nanoscale features. Examples of critical advances in instrumentation include scanning electron microscopes, transmission electron microscopes, atomic force microscopes, focused ion beams, and atom probe tomography. In order to access the mechanical properties of nanoscale features, systems, or materials, mechanical manipulation is required. To some extent this has been done with piezoelectric-driven micromanipulator probes, and atomic force microscopy probes, but experiments using these technologies are slow and difficult to prepare. [41, 162] Some success in mechanical characterization of nanomaterials has been achieved with integrated microfabricated systems, but experiments with these systems are slow in setup and data collection, and for the most part confined to operation within vacuum microscope environments. [168] Force measurement with these systems remains not very standardized in approaches for measuring forces in the 50 nN - 1 mN range. Furthermore, microfabricated testers are not compatible with many nanomaterial synthesis environments due to metallization, corrosion, or risk of damage from liquid surface tension.

Several opportunities for advancement of microfabricated test systems by using discrete test coupons for specimen handling presented themselves at the outset of this project. For instance, limiting the complexity of the test coupons can accommodate difficult synthesis conditions and
perhaps increase the speed of experiment preparation. Additionally, the development of a micro-probe system interfacing to a removable specimen holder advances the catalog of MEMS and NEMS systems and applications.

The microsystems developed here provide microfabricated laboratories for manipulating and interacting with microscale and nanoscale objects. The removable coupon advances known packaging technologies to allow experiments in a wide range of environments, and to allow two microsystems to interact at one stage of processing, but then move on to separate processing trajectories. The test platform may be potentially expanded to nanoscale fabrication applications.

In eventual combination with other characterization tools such as TEM or AFM/STM imaging, the application of the ideas developed in this project may lead to data that supports fundamental understanding of dislocation motion and plastic deformation and failure in engineered materials, which is knowledge that contributes to fundamental design of new mechanical structures at all size scales. The development of new characterization tools such as modular microscale mechanical testers may eventually accelerate the pace at which nanomaterials are deployed in new electromechanical or optoelectromechanical applications, such as tunable resonators, switches, power converters, tunable microlasers and high-power LEDs with appropriate thermal management, and nanoscale sensors.

1.3 Background and State of the Art

1.3.1 Nanomechanical Testing Historical Perspective

In the development of mechanical data for nanoscale materials such as carbon nanotubes, initial challenges included materials availability, materials quality, nanofiber manipulation, and method of observation. Technologies for electron microscopy and other systems to probe molecular and atomic structure, and for nanotube synthesis have improved greatly in the last 20 years, providing advances in materials availability and quality, and in means of nanoscale observation. The development of small-scale mechanical tensile tests was required in order to begin to verify the
predicted mechanical strength of carbon nanotubes. Chief among the mechanical characterization techniques developed were the following:

**Vibration of nanofibers:** The fiber length, diameter and resonance frequency are measured, and from these the Young’s modulus can be derived. [150]

**Yarns mounted in macroscale test coupons:** A rope or yarn of entangled CNTs is mounted to a paper test coupon, with an epoxy or similar glue, and then the coupon is mounted into a tensile measurement machine, such as a dynamic mechanical analyzer. [21, 85, 93] Once it is stabilized in the machine, the sides of the coupon are cut, and subsequent mechanical testing applies the tensile load solely to the nanofibrous yarn. [82] I performed these measurements on CNT yarns in unpublished work at a private company before coming to graduate school. To some extent, the data from nanofiber yarn tensile measurements can be correlated with the bonding strength between nanofibers, and the length of contact between individual nanofibers. [111] This technique provides a rapid means to assess tensile strength, but it has the disadvantage that it measures the properties of an assembly of nanofibers, rather than an individual nanofiber.

**AFM bending tests:** By pushing across a nanomaterial specimen using an atomic force microscope (AFM) probe, some modulus and bending strength data can be obtained, but this technique is limited by the lateral resolution of the AFM. [156]

**Nanoindentation:** In an extension of the initial AFM work, a number of additional papers have developed data for assemblies of nanotubes or nanowires using nanoindentation. [79, 110] Careful experiment design will allow characterization of individual nanofibers with nanoindentation. [79] Nanoindentation appears to be a fairly rapid route to obtaining modulus data, and possibly flexural strength data. This technique has not provided much tensile strength data, electrical property assessment, or direct structure correlations.

**AFM cantilevers in SEM environments:** AFM cantilevers were integrated into a special ten-
sile measurement stage placed in a scanning electron microscope (SEM). Force applied from motion of the AFM cantilevers allowed direct assessment of the modulus and breaking strength of single wall and multiwall carbon nanotubes, providing some of the first direct confirmation of the predicted strengths of carbon nanotubes. Observed tensile strength was typically about 37 GPa (ranging from about 10 to 60 GPa), and Young’s modulus typically 1 TPa. [161,162] AFM cantilever measurements have developed as a widespread approach to nanomaterials measurement, with numerous papers published since 2000. However, the setup of these tests is tedious, and these test systems don’t necessarily provide uniaxial stress on the CNTs. [84] Frequently, the material specimens break at their contact points, where non-uniaxial stress concentrations may be expected due to bending moments. [161,162]

**TEM observations with straining stages:** Although TEM (transmission electron microscope) based straining stages have been established for observation of stressed materials, commercially available stages do not provide the force resolution (1% of 1 µN, i.e., 10 nN) needed for an individual nanotube test. The Dresselhaus group at MIT was able to use an STM (scanning tunneling microscope) probe within a TEM to simultaneously heat a nanotube and then observe dislocation motion in the nanotube induced by a stress applied by the STM probe. [55] Muoth at ETHZ ¹ and Espinosa at Northwestern University have operated microfabricated tensile stages in a TEM to perform CNT observations. [95,97,168]

A description of additional nanofiber characterization methods developed prior to 2006 may be found in Ref. [139].

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¹ ETHZ: ETH-Zürich, the Swiss Federal Institute of Technology (Eidgenössische Technische Hochschule), Zürich, Switzerland
1.3.2 Microsystems for Nanomechanical Testing

The community of researchers developing MEMS for mechanical characterization of nanofibers is relatively small. The majority of research has originated from Saif’s group at UIUC \(^2\), Ruoff’s and Espinosa’s groups at Northwestern, and Haque’s group at Penn State. Mahajan’s group has done related work. \([122,123]\) Outside of the USA, only a few other groups have contributed directly to this field, most notably several groups in Japan and Korea, and Hierold’s group at ETHZ.

Microsystems provide one means for providing a load to a tensile system, although the bonding of the nanofiber to the tensile stages remains a point of significant variation. In general, these systems make use of well-established MEMS structures to apply a tensile load to the nanofiber, and measure displacement and force. Actuation is typically accomplished with electrostatic comb drives, thermal actuators that expand upon Joule heating, or with piezocrystals. Displacement sensing is accomplished typically with capacitance measurements or vernier (microscale ruler) observation via microscopy. Force sensing typically derives from displacement measurements based on correlations of applied force and the displacement of bending beams. The mechanism by which force is transmitted to the nanofiber specimen may be direct, via a shuttle connected to actuators, and stabilized in linear motion by narrow beams that attach to the shuttle.

**Comb drives and capacitive sensing:** Some of the earliest MEMS structures for tensile testing were developed by Saif and McDonald, and later Saif and Haque. \([114,115]\) This work used comb drives both as the actuator and as the displacement sensor, detecting displacement by changes in capacitance. \([114]\) In a related system, the nanofiber is fixed at one end, and attached to a moving shuttle at the other end. Force is generated by a comb-drive connected to the shuttle, with applied force calibrated to a given applied voltage. Displacement is measured from images. Motion of the end of a beam amplifies the motion of the shuttle, and video observation of the motion of the beam allows derivation of the actual shuttle displacement and nanowire strain. \([68]\) Recently, comb drive mechanical testers were used

\(^2\) University of Illinois at Urbana-Champaign
to collect tensile data from Co nanowires. This work used comb drive electrostatic actuation as a source of applied force, but device displacements were measured with image correlation software. [163] A comb drive mechanical test structure was also built at Johns Hopkins University, but did not provide significant data. [154] Comb drive capacitive systems require a large footprint of nearly a square millimeter if they are to provide sufficient force (10s to 100s of $\mu$N) for mechanical testing of NWs and CNTs.

**Piezoactuated systems (UIUC and Penn State):** In work targeted at tensile measurements of thin metallic films, piezoactuated systems used verniers to achieve displacement measurements. [41–46] In these designs, stabilizing beams attached to a tensile shuttle, which was isolated from disturbances by a small gap at one end, and by compliant U-shaped springs. The combination of the stabilization beams, the gap and the U-springs allow linear stress loading of the tensile specimen even if the coupon is not perfectly aligned in the tensile loading system. [44] Force is measured by observation of the displacement of the four spring beams. An additional feature of this system is a specimen stabilization structure. Saif’s group added this structure to ensure that the tensile specimens would remain intact while the sample coupons were handled before testing. [41] For mechanical characterization of nanofibers, smaller loads and displacements were needed, and these structures were adapted to scale down displacements using beam buckling to achieve fine-scale displacement resolution. In this system, a piezoactuator applies a force that pushes on the system, forcing the longitudinal beams into a buckled configuration. By comparison of the displacements measured at verniers near the specimen, the specimen stress and strain were derived.

**Thermal actuation systems (Ruoff group, Northwestern and UTexas):** A system recently developed by Ruoff’s group makes similar use of mechanical leverage to develop fine control on displacement. This system generates force by Joule-heating induced thermal expansion of a silicon beam. The expansion of this beam spreads apart a thinner v-shaped beam structure, providing a tensile force to the nanofiber specimen. Displacement and force
measurements are achieved by either direct measurement of displacements of the actuator beam expansion versus stage motion using observation in a scanning electron microscope, or correlation of at least some of this motion with applied input electrical power. This system is capable of nanometer scale displacements and applied forces ranging from tens of nN up to about 20 µN. [86]

**Thermal actuators with capacitive measurement (Espinosa, Northwestern):** In this system, Joule heating causes the thermal actuators to expand to a given amount, providing a known displacement. The force corresponding to this displacement is transmitted by a moving shuttle to the nanofiber, and by the nanofiber to a second shuttle that connects to a series of springs and a MEMS capacitor. Measurement of the capacitance in the load sensor describes the sensor displacement, which correlates to a force if the spring constants of the attached beams are known. [106,168] Because actuation, sample mounting, and stress and strain measurement are fully integrated onto a single device, this system does not require the same specimen stabilization needed by the coupon developed by Haque and Saif. This system can operate in benchtop and vacuum environments. Most importantly for TEM measurement, this system does not require visual observation of displacements or verniers. Once calibrated using displacements measured from known oscillating electrical inputs provided to the thermal actuators, the system provides electrical output from the load sensor, and input displacement correlates with a given electrical input. Stress and strain data can then be obtained from electrical measurements while the TEM is used for observation of the nanofiber structure.

**Cantilever system (Hierold et al., ETHZ):** A nanoscale cantilever can be designed to actuate vertically between two static plates. A nanotube is deposited on a sacrificial SiO₂ layer, and then the cantilever and plates are deposited. Actuation was performed with an AFM tip, and electrical resistance was measured across the CNT between the cantilever and one of the plates. This system demonstrated a strong CNT piezoresistive effect, but with
significant variation between different specimens. [135,136] This test setup requires location of dispersed nanotubes, e-beam patterning and lithography, and then electrical contacting and further AFM location and manipulation.

1.3.3 Mechanical Interfacing and Integration in Transmission Electron Microscopy

Although straining stages for transmission electron microscopes are well-established in the field of materials science, and a number of stages are commercially available, far fewer systems have been documented for probing of nanowires and nanotubes. One stage, developed in Japan, was used to observe the formation, dislocation behavior, and fracture of gold nanowires. In this system, two electron-beam-milled gold wire specimens are mounted opposite each other, one on a fixed mount and the other on a mobile mount. An electromagnetic motor drives a screw drive for course positioning of the mobile mount, and a piezoactuator is used for fine motion and adjustment. Under TEM observation, the two specimens were brought into contact and then separated, forming a transitory Au NW. [71,72]

In an improvement on this system, the straining TEM stage performs electrical measurements; Oshima calls this a scanning tunneling microscope (STM) holder, which operates within a TEM to perform electrical and structural measurements on gold nanowires. [105] De Heer’s group at Georgia Tech took a similar approach to electrical measurements and TEM manipulation. They used a mercury electrode on the static part of the stage and attached a CNT sample to the moving part of the stage. [34,109,150]

In order to provide a modular system for TEM based materials observation, Haque and Saif developed structures for integration into a TEM straining stage. [43,45,46] The mechanical load is applied to the chip directly from the straining stage by means of two pins that pass through a hole at each end of the chip. [43,45,46]

Several other examples of modified TEM stages exist, most notably for performance of high-temperature and electrical conductivity measurements [138,147,165], or for scanning probe microscopy [73] or nanoindentation probing within a TEM [9,98]. Nakajima et al. demonstrate a
manipulator with multiple degrees of freedom for operation within a TEM, and a larger version for operation within an SEM. [99]

An alternative approach is to perform straining with microsystems, and interface electrically only to the microsystem from the TEM holder. Both Espinosa’s group and Hierold’s groups have implemented this approach. [95,168] This approach to mechanical device integration within a TEM provides the simplicity of eliminating the piezoactuation mechanism, but the process required for mounting the system to the electrical contacts presents a significant risk of damaging the MEMS actuator and sensor to which the nanomaterial specimen is mounted.

1.3.4 General Integration of Nanomechanical Test Systems

If the test system is not constricted to the operating space of a TEM, it can take up a larger space. The chief examples of non-TEM integration and packaging of microfabricated systems for nanomaterial testing were published by Espinosa’s, Haque’s, and Saif’s groups. In one system, electrical connections were made with wirebonds between contact pads on the device chip and the contacts of a supporting pin grid array package. [168]

In Saif and Haque’s work, benchtop device integration was accomplished by using epoxy to affix test coupon devices to a piezoactuated system. [43] This work was later expanded to a multiscale system for nanofiber characterization. Here, NWs are randomly deposited from a methanol suspension onto an oxide support grid. After focused ion beam (FIB) Pt deposition is used to affix the nanowire to the grid, the grid is cut with the FIB, and a section containing the nanowire specimen is manipulated onto a system of pin-connecting jaws. These pin connections are part of a larger buckling beam tensile system. This microfabricated device is fixed onto a stage and mechanically actuated by a beam connected to a piezocrystal. The entire system is placed within an SEM, which is used to record vernier measurements at given applied loads. Mechanical stress and strain data is derived from observations of beam bending. The authors point out that this system does not generate electrical interference and can operate in vacuum, liquid, and gas environments. They emphasize the grid pick and place technique as a method of specimen manipulation that is
widely applicable and ensures good load transfer. [26]

1.3.5 Microsystems with Piezoresistive Measurements

The approaches taken to direct electrical characterization of nanowires and carbon nanotubes are predominantly the use of two or four point electrical probes. The nanofiber is aligned across the probes, connected electrically, and the measurement is performed. Rayleigh [124] and Raman [30] scattering may be used to interrogate the electrical properties of a nanofiber, and dielectrophoresis may be used to separate fibers by their length and their structure. In contrast, the merit of the direct contact tests is that they provide an analog to the nanofiber in an electrical device application. The data and the test methodologies from direct contact tests provide progress towards the integration of nanofibers into micro and nanodevices.

Two-point electrical measurements are an appealing approach for nanofiber conductivity measurement because of the simplicity of experimental setup. Wang et al. provided a two point nanotube conductivity measurement based on contact of the nanotube with a liquid mercury electrode on a special TEM stage. [150] Other groups have demonstrated two-point conductivity or three-point transistor measurements based on nanotubes contacted to metal electrodes. [135, 136] Process flow for this approach typically involves some order of the following steps: deposit nanotubes, pattern and deposit electrodes, contact electrodes to nanotubes with evaporated metal deposits, make electrical contact to electrodes and test. The major disadvantage of two-point conductivity experiments is that, in addition to measuring the resistivity of the target specimen, they also measure the contact resistance between the specimen and the electrodes. Measurement of the conductivity of the target specimen requires interpretation of the data obtained from a two-point probe in order to remove observation of the conductivity of the contact electrodes and the contact resistance between the electrode and the specimen. In this system, the choice of “solder” metal between the electrode and test specimen may also play a significant role, in that the specimen contact resistance may be affected by the work function of the metal contact.

Four-point electrical measurements are a well-established technique in macroscale materials
measurement for characterization of electrical conductivity, and several microfabricated four-point probes have been created. [19, 107] By measuring the voltage drop across a section of material carrying a known current, the contact resistance and electrode resistance are eliminated from the measurement. The main challenge of this measurement is reproducible alignment of the material on the electrodes, to ensure the voltage drop occurs across a known distance of material. Specifying the exact distance between the two points of the voltage measurement is an additional source of error. By reducing the error due to contact resistance and electrode resistance, the four point probe measurements appear more ideal than the two-point measurements for electrical characterization.

By careful patterning of electrodes and an insulating silicon oxide, Han and Saif developed a four-point contact system that is integrated with their device for tensile measurement of micro and nanostructures. [41] They have used this device to measure conductivity in an aluminum film that was also subjected to mechanical testing, but they did not perform simultaneous mechanical and electrical measurements.

1.3.6 Nanofiber Placement

Placement of a nanomaterial specimen on a tensile stage is the first step to performing a tensile test on that specimen. A number of techniques were developed historically for manipulation of individual nanotubes and nanowires:

**Dispersion:** Because nanotubes were for a long time only available as very short structures, placement of nanotubes by deposition from liquid dispersions developed as a predominant method of nanotube placement. By controlling the concentration within the suspension, average spacings can be derived for nanotube and nanowire deposits. [26, 135, 136]

**Dielectrophoretic placement:** If nanotubes or nanowires are intended to bridge a specific gap, a technique of dielectrophoretic deposition from suspension has been developed. [20, 92] In this method, nanotubes or nanowires align with an electric field across two contacts, and a gradient in an AC electric field will attract the nanotube to the point where the electric
field is at its maximum, presumably at the two points that the nanotube is intended to bridge.

**Growth in place:** Specified CNT growth is achieved by depositing catalyst at one location where the nanotube is intended to anchor, and then subjecting the substrate and catalyst to environmental conditions that promote the chemical vapor deposition growth of nanotubes at the catalyst particles. \[51, 65, 160\] Gas flow causes the growing nanotubes to span a gap between contact points. The major disadvantage of this approach is that not all microsystems will tolerate the typically \(> 800^\circ C\) temperatures required for CVD (chemical vapor deposition) growth of carbon nanotubes.

**Flip chip placement:** Hone’s group has developed an adaptation of the prepatterned CVD approach. \[56\] By using flip chip transfer of CNTs from growth substrates to test structures, this technique allows integration of individual CNTs into larger microsystems. This technique has not been well demonstrated in preparation of samples for mechanical testing.

**Micromanipulator placement:** In this specimen placement method, a micromanipulator on a probe station may be used to place or push around nanowires, or a micromanipulator within an SEM environment selects and places NWs or carbon nanotubes. It is a tedious process which risks damaging the test system onto which the CNT or NW will be placed.

### 1.3.7 Nanomaterial Clamping

Once the nanotube or nanowire is placed onto the test structure, it must be secured to the test structure. The most common approach to clamping a nanofiber to a mechanical test system is the use of electron beam welding or deposition of clamps using ion beam induced deposition (IBID) in a focused ion beam system. In a variation of this technique, a low pressure organic vapor might be provided in an environmental SEM, and the clamps deposited by electron beam induced chemical vapor deposition (EBID). \[89, 91, 148\] Other techniques common for bonding of nanotubes and nanowires to test structures are the use of ultrasonic welding and glues such as
various epoxies. [16] For electrical tests, deposition of thin metallic layers, especially Au, Pd or Pt, is also common. [19,56,135,136]

Ideally, the method of attachment would provide good load transfer to the nanofiber without significantly altering the nanofiber structure. In reality, any bonding structure will chemically interact with the fiber. A good mechanical test therefore requires a sufficient area bonded to the fiber that deformation in the fiber dominates the measurement over deformation in the bond. Furthermore, tests wherein the specimen fractures at the bond should be seen as inconclusive data because the complex stress and strain states in the clamped regions may lead to underestimates of the ultimate tensile strength of the nanomaterial specimen. Specimens ideally will fracture where they span tensile stages. [81]
Chapter 2

Methods and Analytical Techniques

This chapter provides a review of mechanical testing of materials, focused on tensile testing, and it provides a basis for analysis of tensile test data from MEMS mechanical testers for nanomaterials. A review of methods of uncertainty analysis in MEMS mechanical tester is also included.

2.1 Tensile Testing

Mechanical tests such as tensile and compression tests are some of the most fundamental characterization techniques of materials science. By providing data on the strength, maximum strain, and Young’s modulus of materials, these tests provide fundamental data about the atomic and molecular behavior of a material, in addition to information which can be applied to mechanical designs. With control of temperature, or observation of stress and strain in relation to time or load cycling, even further information can be garnered regarding the structural behavior of materials and their limitations under different scenarios of mechanical loading.

Because compression tests require more difficult specimen preparation to prevent buckling of test specimens, and because tensile strength is usually equal or lower than compressive strength, tensile testing was the focus of work in this project.

This introduction of mechanical testing is modeled after the discussion provided by Ref. [48]. The engineering stress \( \sigma \) is given by the load \( F \) divided by the initial cross-sectional area \( A \).

\[
\sigma = \frac{F}{A} \tag{2.1}
\]
The engineering strain is given by a change in length, the instantaneous gauge length of a specimen $L$ minus the initial gauge length $L_0$, divided by the initial length of a specimen.

$$\epsilon = \frac{L - L_0}{L_0}$$  \hspace{1cm} (2.2)

Typically, a gauge length is selected in a region where the specimen has uniform cross-sectional area in order to minimize variations in strain due to different stress values at different cross-sections.

Engineering stress and strain are simplifications of the true stress and true strain experienced by a specimen. Calculation of the true stress $\sigma_{true}$ is found by dividing the load by the instantaneous cross-sectional area $A_i$ of a specimen. As a material specimen undergoes deformation in length, the constant volume of the specimen causes a reduction in the cross-sectional area.

$$\sigma_{true} = \frac{F}{A_i} = \sigma(1 + \epsilon)$$  \hspace{1cm} (2.3)

Calculation of the true strain $\epsilon_{true}$ is made through recognition that the gauge length $L$ continuously changes throughout the tensile test, therefore a given elongation at the start of the test represents a greater change in strain than in the case of an elongated specimen experiencing that much more elongation. The true strain is found with Eq. 2.4.

$$\epsilon_{true} = \ln \frac{L}{L_0} = \ln(1 + \epsilon)$$  \hspace{1cm} (2.4)

For practical tensile testing, accounting for instantaneous area change is not easily performed during the test, therefore most tensile testing approaches begin with plotting engineering stress and engineering strain, and use Eqs. 2.3 and 2.4 to derive the true stress and true strain if necessary.

At the start of most tensile tests, the change in stress is proportional to the change in strain of the test material. In this elastic region, the proportionality constant between the stress and strain, $E$, is the modulus of elasticity, also known as the Young’s modulus. In a plot of stress and strain values determined from a tensile test, $E$ can be determined from a linear regression fit to these values. The measure of $E$ that is used for design calculations is best determined by averaging
the $E$ obtained from the regression fits from numerous tensile tests.

\[ \sigma = \epsilon E \quad (2.5) \]

The resilience of a material is defined by the energy stored within the elastic region of its tensile curve, according to Eq. 2.6:

\[ \text{Resilience} = \frac{1}{2} \sigma_{\text{max}} \epsilon_{\text{max}} \quad (2.6) \]

For brittle materials, which fail without plastic deformation, the resilience is additionally equivalent to the toughness of the material, which defines the energy density that the material must mechanically absorb in order to undergo fracture.

Specimens for tensile tests are simplified structures. By choosing an experimental setup that simplifies the strain states within a specimen, the experimenter can obtain data such as the Young’s modulus, strain at failure, and stress at failure that can be applied directly to design work including failure models and predictions of deformation in more complex loading situations. Typically macroscale tensile specimens have flared ends (also known as “dogbone” specimens) that provide the location for clamping. By defining the gauge length as the narrower region between the wider ends, deformation is concentrated in the gauge length and the complex strain states in the ends of the specimen do not generally play a role in the failure of the specimen.

Most of the material specimens with nanoscale diameters that are of current interest are single crystal structures. As such, they do not provide flared ends ideal for concentrating loads in a central gauge length. These specimens are typically clamped by materials deposits such as platinum, but the clamps then introduce the possibility of deformation within the clamp, and slipping between the specimen and the clamp. The loaded force is constant through a material specimen, so measurements of stress should not be affected by clamp deformation. Measurements of nanomaterial strain should ideally be performed by defining a gauge length between fiducial points marked between the clamp locations.
2.2 Interpreting Data from MEMS Tensile Testers

Fundamentally, tensile tests require information about the load applied to a specimen, its initial cross-sectional area, its initial gauge length, and the distance by which the specimen elongates during the tensile test. The cross-section area and the gauge length are defined by direct inspection of the test specimen. The mechanical test device provides information on the load applied to the specimen during the test, and the elongation of the specimen.

For mechanical testing of specimens with nanoscale diameters and microscale lengths, measurement of the displacement of a tensile stage can provide some information about the specimen elongation, but direct observation of a gauge length of a specimen is preferable in order to eliminate the error due to slipping or deformation of clamps from the elongation measurement. Direct observation may be accomplished by electron microscopy. Use of a built-in displacement sensor would allow faster data collection and more flexibility in the test environment, even if the displacement measurements would provide more uncertainty because of including displacement due to clamp deformations.

The determination of the load force applied to the tensile specimen presents a greater challenge for microscale tensile tests. For any cross-section perpendicular to the axis of loading in a uniaxial tensile test, the applied force will be constant. So, the force in the specimen is the same as the force experienced in the tensile stage, and the force constraining the motion of any actuator that is used to apply load to a tensile specimen.

In macroscale tensile tests, the force applied to a tensile specimen is measured with a load cell. Miniaturizing such a device to a microscale system would require implementation of an additional sensor design within a tensile test microdevice. Such a design would typically require a well-characterized spring coupled with a displacement sensor to measure the displacement of the spring when subjected to the loading force. Optimally, this spring should be calibrated against known forces, or the spring constant should otherwise be determined to a high precision.

Until an appropriate load sensor can be implemented as part of the microsystems developed...
in this project, a different tactic has been taken with regards to the determination of the forces applied by tensile test microsystems. In this “expected displacement” approach, it is recognized that the displacement of a microfabricated tensile test stage represents an equilibrium value between the force provided by a MEMS actuator and the restorative forces created by any compliant beams supporting the actuator. Values that are easy to measure, namely the current and voltage supplied to an actuator, can be plotted against a measured output displacement from a test stage. A function predicting an expected displacement for any given electrical input within the range of operation of the test system can be determined by an appropriate regression curve fit between the input electrical data and the measured freely moving test stage displacement. In working with MEMS thermal actuators, electrical power, calculated by multiplying input current and input voltage, was generally used as the independent variable in this fit because power dissipation in the actuator most directly relates to the thermal strain experienced by components of the actuator.

The load force experienced by the tensile specimen provides a constraint on the free motion of the actuator. In order to estimate the force applied by tensile stage constrained in motion by a tensile specimen, the assumptions are made that, around a given equilibrium point of operation, the actuator and tensile stage system exhibit a linear force versus displacement behavior, and that the spring constant for this behavior is constant and can be approximated based on bending beam calculations. For integrated testers discussed in Section 3.5, these assumptions are reasonable from the standpoint that stage motion is on the order of 2 microns, while beams in the stage suspension and thermal actuators are typically around 200 microns in length, indicating that the perturbation from the expected displacement of the tensile stage system is around 1% or less. Furthermore, calculation of the system spring constant can be made or simulated using knowledge of the elastic modulus of the material used to create the system. More precise determination of the spring constant is not necessarily required if the uncertainty in the spring constant is significantly less than the uncertainty in other measurements (such as stage displacement) used to determine the load force. The calculations in Appendix E further illustrate this point. The uncertainty due to stage displacement measurements significantly dominates over other components of the stress
The assumption of an unchanging spring constant may not necessarily be true in all constrained situations [155], and further work in microscale force calibration would be helpful for checking these assumptions. Both development of a microfabricated in-plane load sensor and development of strategies for micronewton force calibration are nontrivial research projects. For demonstration of a first-generation microfabricated test platform system they were not necessary so long as some data could be obtained from the system.

In the long term, microfabricated mechanical measurement tools should incorporate refinement of the load measurement process and a means for calibration of sub-millinewton forces in order to improve the precision of tensile test results. Several reports have used AFM measurements to attempt force measurement of in-plane motion of MEMS structures. [122, 123, 159] At least one report has also attempted to use a nanoindenter for force calibration. [36]. Some vibrometer and atomic force microscope data was collected in support of attempts at force verification in the structures developed in the following Chapters. The vibrometer data is reported in Appendix C.

Tables 2.1 and 2.2 provide a summary of the measured and calculated variables from tensile tests performed in this thesis, and the key equations used to derive the tensile test measurement results. Further discussion of the topics presented in this section may be found in Section 6.3.2.1, below.
Table 2.1: Table of symbols used in calculations. The variables at far left indicate values that were measured directly from experiments.

- **A**: nanowire cross section area
- **B**: fitting parameter found with linear first-order regression
- **D**: nanowire diameter
- **d**: actual displacement of tensile stage
- **d₀**: “expected” displacement of tensile stage
- **e**: nanowire elongation
- **ε**: engineering strain
- **F**: force on nanowire
- **I**: current flow through actuator
- **k**: microsystem spring constant
- **L**: nanowire gauge length
- **L₀**: initial gauge length of the nanowire
- **P**: actuator input power
- **σ**: engineering stress
- **V**: voltage applied to actuator

Table 2.2: The key equations used to derive tensile test measurement results.

\[
\sigma = \frac{F}{A}, \\
A = \pi D^2 / 4, \\
F = k(d_0 - d), \\
d_0 = f(P) = BP, \\
P = IV, \\
e = L - L_0, \\
\epsilon = e / L_0
\]
2.3 Uncertainty Analysis

Understanding of the precision of a measurement system requires that a quantified uncertainty value be part of the measurements obtained with the system. For microsystems to serve as tools for metrology of nanomaterials, the uncertainty in the data must be reported and used to compare and critique MEMS measurement designs and techniques. This has been accomplished in many MEMS investigations, most successfully using cantilevers or bulge testing (for examples, see Refs. [62,137]). This section provides a derivation of the uncertainty associated with data derived from MEMS tensile test devices. More discussion of the general approach to uncertainty calculation may be found in Appendix D, and example uncertainty budget calculations are included in Appendix E.

Tensile testing of nanomaterials is a field which holds great potential for new insight in physics. However, few nanomaterial tensile test reports provide a thorough examination of the uncertainty of the data obtained with MEMS or AFM tensile tests. One of the aims here was to demonstrate the application of ISO and NIST guidelines for the reporting of measurement uncertainty to results obtained with MEMS tools, and to establish standard uncertainty values associated with the data. The challenge was simply to report MEMS tester uncertainty analysis that can do the following: be applied to similar systems, establish uncertainty values on measurement results, and allow quantitative comparison between MEMS tensile metrology systems.

ISO, NIST [59,142] and various other standards organization have developed guidelines for reporting uncertainty in measurements and uncertainty propagation into the measurements. The recorded data is separated into Type A and Type B measurements, wherein the standard uncertainty $u_c$ in the Type A measurement is found according to Eq. 2.7 from the standard deviation $\hat{\sigma}$ of measurements recorded from the average of $N$ data points and the standard uncertainty in the Type B measurement is derived based on the probability that the value recorded occurs between bounds $\pm a_x$. For all of the Type B measurements recorded for this thesis, the most conservative probability distribution function was used, a square probability distribution between bounds $\pm a_x$. This means that the measured value $x_i$ occurs between $x_i - a_x$ and $x_i + a_x$, and no further information is known
of the probability of $x_i$ occurring between those bounds, so uniform probability is assumed. The standard uncertainty $u_c(x_i)$ of this measurement is found according to Eq. 2.8. [59,142]

$$u_c(x_i) = \frac{\hat{s}(x_i)}{\sqrt{N}} \quad (2.7)$$

$$u_c(x_i) = \frac{a_x}{\sqrt{3}} \quad (2.8)$$

If a measurement $y$ is derived as a function $f(x_i)$ of $n$ uncorrelated variables $x_i$, the standard uncertainty in the measurement $u_c(y)$ is found according to the root sum of squares method, Eq. 2.9. [25,59,142] In the case where $f(x_i)$ is only a product or a ratio of variables $x_i$, Eq. 2.9 simplifies to Eq. 2.10. [25]

$$u^2_c(y) = \sum_{i=1}^{n} \left( \frac{\partial f}{\partial x_i} \right)^2 u^2_c(x_i) \quad (2.9)$$

$$\left( \frac{u_c(y)}{y} \right)^2 = \sum_{i=1}^{n} \left( \frac{u_c(x_i)}{x_i} \right)^2 \quad (2.10)$$

Unless otherwise noted the standard uncertainties $u_c$ are reported here as directly stated values, ± values, or error bars without multiplication by a coverage factor, therefore they represent uncertainty values reported with approximately 68% confidence.

The standard uncertainty values were rounded to the number of significant figures appropriate to the data they describe. Generally, two significant figures are calculated for the standard uncertainty values, and for some data only one significant figure is needed. Data are rounded to the appropriate decimal place defined by the number of decimal places in the corresponding uncertainty value.

### 2.4 Uncertainty in Constrained Regressions

Regression analysis can be used to provide information about relations between variables in a given data set. Various methods exist to estimate the uncertainty in a dependent variable calculated
using an independent variable and a regression fit. [31] In some cases, such as the determination of an elastic modulus, the fit parameter itself is the output data of interest, and knowledge of the uncertainty in this parameter is important. In unconstrained regressions, several approaches have been developed for calculation of the regression parameter uncertainty. [25,31] Tensile plots where stress and strain are known to start at the origin for both variables require a constrained regression, where the regression line is set to pass through the origin. In this case, no guidance was found in literature for determination of the uncertainty in a regression parameter. This section proposes one method: the propagation of uncertainty values from each data point into the final calculated regression value.

The linear regression method finds a fit curve according to the minimization of the sum of squares of residuals between data points \((x_i, y_i)\) and the curve \((x_i, y_{ci} = f(x_i))\) that estimates for the data points. The function \(f(x_i)\) is also a function of fit parameters \(\beta_j\). Equations 2.11 and 2.12 describe the function \(f(x_i, \beta_j)\) in the case of a linear (first order polynomial) fit and in in the case of a second order polynomial fit, respectively.

\[
\begin{align*}
  f(x_i, \beta_j) &= \beta_0 + \beta_1 x \\
  f(x_i, \beta_j) &= \beta_0 + \beta_1 x + \beta_2 x^2
\end{align*}
\]

The fit parameters for the regression curve are found by minimizing \(Q\) (Eq. 2.13). Minima in \(Q\) are found by zeroing the partial derivatives with respect to the \(\beta_j\), and then solving the resulting system of equations (Eq. 2.14).

\[
Q = \sum_{i=1}^{N} (y_i - y_{ci})^2 = \sum_{i=1}^{N} (y_i - f(x_i, \beta_j))^2
\]

\[
\frac{\partial Q}{\partial \beta_j} = 0
\]

For the \(d_0\) and \(E\) fits used in Section 6.4, the fit is performed after data manipulation that has defined the \(y\)-intercept of the regression curve as the origin. Constrained forms of Equations
2.11 and 2.12 must then be used to find the regression parameters. Because the y-intercept is zero, the parameter $\beta_0$ is set equal to 0. The fit parameters are found in the same manner as for the unconstrained fits, by solving the system of equations indicated by Eq. 2.14. The resulting fit parameters have one degree of freedom less than the fit parameters given in the unconstrained cases, and calculation of the uncertainty of $f(x)$ and the uncertainty of the fit parameters $\beta_j$ can not be performed according to the same equations as are commonly given in the case of unconstrained fits (such as in Ref. [31]). An example analysis is presented here that uses the data available in Section 6.4 to rationally calculate constrained regression parameters and uncertainty values that are needed from the regression fits. The examination of correlation in the tensile test variables would require even further sophistication in the regression fitting and propagation of uncertainty.

In the case of an unweighted first order linear regression with $\beta_0 = 0$, the slope $\beta_1$ of the fit line is found according to Eq. 2.15. [75]

$$\beta_1 = \frac{\sum_{i=1}^{N} y_i x_i}{\sum_{i=1}^{N} x_i^2}$$

(2.15)

For the fit to find the elastic modulus $E$, $x_i = \epsilon_i$, $y_i = \sigma_i$, and $\beta_1 = E$. In this case, there is an uncertainty associated with each $x_i$ and $y_i$, and an estimate of the uncertainty in the slope, $u_c(\beta_1)$ is needed. This can be found by application of Eq. 2.9 to Eq. 2.15, leading to the propagation of all of the uncertainty values $u_c(x_i)$ and $u_c(y_i)$ into an combined uncertainty of the slope $u_c(\beta_1)$. The uncertainty of the slope is then described according to Eq. 2.16.

$$u^2_c(\beta_1) = \left[ \frac{\beta_1^2}{\left( \sum_{i=1}^{N} y_i x_i \right)^2} \left( \sum_{i=1}^{N} y_i x_i \right)^2 \right] + \beta_1^2 \left( \sum_{i=1}^{N} [2x_i u_c(x_i)]^2 \right)$$

(2.16)

Eq. 2.15 and Eq. 2.16 were used to find the Young’s modulus and the uncertainty in the
modulus as described in Section 6.4, Table 6.5 and Figure 6.27.

For the fit curves given by Equations 2.28 and 2.29, the uncertainty in the second-order constrained fit parameters $\beta_1$ and $\beta_2$ is not of interest for finding the results of the tensile tests, but rather the uncertainty of the estimated $d_0$ given by a specified input power $P$ is needed. In this case, the combined standard uncertainty $u_c(d_0)$ of the estimated output depends upon the uncertainty in the fit and the uncertainty produced by the propagation of the uncertainty present in the electrical power $P$. For consistency in derivation results, it should be noted that in this case $x_i = P_i$, $y_i = d_i$, and $y_c = d_0$. The independent variable $x$ and the estimated $y_c$ are fit according to the constrained relation $y_c = \beta_1 x + \beta_2 x^2$. A first approximation to the uncertainty in the fit is the square root of the variance of the fit residuals (Eq. 2.17), and the uncertainty in the constrained second-order fit relation due to uncertainty in the independent variable $x$ is found using Eq. 2.9 and given in Eq. 2.18 assuming no uncertainty in the fit parameters $\beta_j$ in this case. The combined standard uncertainty of the dependent estimated variable $y_c$ (or $d_0$) is found by taking the root of the sum of the squares of the uncertainties specified in Equations 2.17 and 2.18. The result, Eq. 2.19, was used to estimate the uncertainty in $d_0$ found as an output of the curves specified by Equations 2.28 and 2.29.

$$u^2(y_{c,fit}) = \frac{\sum_{i=1}^{N}(y_{ci} - y_i)^2}{N - 2} \quad (2.17)$$

$$u^2(y_{c,x}) = \left(\frac{\partial y_c}{\partial x}\right)^2 u_c^2(x) = (\beta_1 + 2\beta_2 x)^2 u_c^2(x) \quad (2.18)$$

$$u^2_c(y_c) = \frac{\sum_{i=1}^{N}(y_{ci} - y_i)^2}{N - 2} + (\beta_1 + 2\beta_2 x)^2 u_c^2(x) \quad (2.19)$$
2.5 Sources of Uncertainty in Experiments

The raw data (electrical measurements and displacement measurements from SEM images) collected with the MEMS tensile devices reported here was gathered under the following set of assumptions, which is reflected in the uncertainty analysis. As further development minimizes the reported uncertainties, the sources of uncertainty that are ignored according to the assumptions here should be incorporated into the combined standard uncertainty values used to interpret the tensile test data.

The spring constant of the tensile tester is assumed to exhibit linear elastic behavior, and therefore it is assumed not to change with the displacement output of the tester, and nor with the temperature changes experienced in the thermal actuator. Further discussion of the uncertainty and bounds on the spring constant can be found in Reference [10]. The uncertainty value attached to the spring constant of the system is assumed to incorporate the uncertainty in the modulus and dimensions used to calculate the spring constant. It is assumed that direct measurement of this spring constant, were this to be accomplished, would provide a value within the bounds used to find the spring constant uncertainty.

It is assumed that the curves in Section 2.6.6 can extrapolate predicted values in the tensile test region where those values cannot be measured directly. The actuator is assumed to move only in one direction, pulling the moving stage away from the fixed stage.

In the current and voltage measurements, electrical and quantization noise is minimized by averaging a large number of data points in a Type A uncertainty evaluation. Most electrical systematic errors are not propagated into the force and stress data due to the use of the expected displacement curve (Section 2.6.6).

Some sources of random error in mechanical measurement include thermal noise and vibrational eigenmodes driven by thermal noise, image focus and image resolution. These are subsumed by the uncertainty of all the displacement measurements.

Additional assumptions were made regarding sources of systematic error in mechanical mea-
urements. It is assumed that the fiducial points for displacement measurements can be located using manual line drawing. It is assumed that the SEM used for tensile testing is accurately calibrated and that the systematic error in its displacement measurements is less than the resolution that can be obtained in images from the SEM. Furthermore, it is assumed that the SEM maintains its accuracy from image to image. The calculation of strain by dividing an elongation by a gauge length removes any systematic bias in the strain due to the SEM calibration.

Within the gauge length of the fiber, a uniform diameter is assumed, as is a perfectly circular cross-section. The angle of the fiber alignment in the tensile tester, and out of the image plane, does not significantly increase the uncertainty of the force experienced in the fiber. This is discussed further in Sections 2.6.5 and 6.3, and Reference [10].

Within the gauge length of the fiber, uniaxial tension is assumed. The gauge length is chosen so as to lie between the clamping regions at the ends of the fibers. These clamping regions are subject to complex strain states, and may undergo some de-bonding and elongation, which was specifically observed in the case of Specimen 2 reported below. It is assumed that definition of a gauge length between the clamped regions removes systematic uncertainties due to slipping and deformation within the clamped regions. Furthermore, it is assumed that the platinum used as clamping material is not present on the gauge length.

2.6 Uncertainty Analysis Applied to MEMS Tester Data (Example)

The analysis and plots presented in the remainder of this chapter show the detailed uncertainty analysis performed in support of the data analysis reported in Section 6.4 regarding tensile tests performed on polymer-derived ceramic – carbon nanotube composite nanowires. Uncertainty budget examples demonstrating accounting of the stress and strain uncertainties for one PDC-CNT tensile test data point may be found in Appendix E.
2.6.1 Length Measurements

The gauge lengths of the nanofibers were measured directly from SEM images using lines drawn in ImageJ software [58]. The length data for the two nanofiber samples tested is shown in Figure 2.1 as elongation data in order to allow direct comparison between the samples. The gauge lengths used to derive strain results were defined between observable deposits of PtC clamping material. However, in the regions before the slack is removed from the specimens during the tensile tests, the gauge length measurements are not very accurate because they are measurements of a straight line approximations to curved or bowed specimens.

The elongation, $\Delta L$, is equal to the gauge length of a fiber $L$ minus an initial gauge length $L_0$, which is defined as the length of the fiber measured at the start of the test. The standard uncertainty in $\Delta L$, $u_c(\Delta L)$, is calculated (Eq. 2.21) using the standard uncertainties $u_c$ of $L$ and $L_0$. Stage displacement measurements $d$ and their uncertainties are similarly calculated, subtracting the initial separation between two fiducial points from the distance measured between the points as the test progressed. Measurements of stage separation were performed three to five times, and the displacements obtained from these measurements were averaged before being used as the stage displacement data $d$ in subsequent calculations.

$$\Delta L = L - L_0 \quad (2.20)$$

$$u_c^2(\Delta L) = u_c^2(L) + u_c^2(L_0) \quad (2.21)$$

In order to reduce the random uncertainty in the length measurements obtained from SEM images, a specific measurement procedure was introduced. A line was drawn between two fiducial points on the image, and the line length was measured. The line was preserved on the image as the next image in the series was opened. The line was compared to the fiducial points at the image. If the fiducial points had clearly separated, the line length was increased. If there was no clear change in separation of the fiducial points, the line length was not changed. The line length was
then measured, the next image opened, and this process repeated.

With this procedure, it was possible to define changes in fiber gauge length to within ±2 pixels, but the lengths used to find stage displacement were given a larger bound of ±4 or 6 pixels because the fiducial points for stage displacement measurements weren’t as clear as those used for the fiber gauge length measurements. The associated standard uncertainty for length measurements $u_c(L)$ was computed using Eq. 2.8. So, for Specimen 1 $a(L) = 33$ nm and $u_c(L) = 19$ nm and for Specimen 2 $a(L) = 22$ nm and $u_c(L) = 13$ nm. (Only gauge length data recorded at 9000× magnification was used in the final Specimen 2 tensile plot. Other data points, to the left of the vertical dotted line bisecting the Specimen 2 data in Figure 2.1, were recorded at lower magnifications and had $u_c(L) = 16$ or 18 nm.) Stage displacements were recorded with uncertainty values $u_c(d) = 55$ nm for Specimen 1 and $u_c(d) = 44$ to 53 nm for Specimen 2.
Figure 2.1: Elongation of nanofiber samples plotted versus the voltage supplied to the actuators that were driving the tensile measurement systems. For each specimen, the data points to the right of the dashed vertical line are the ones that were used to derive the tensile data. X error bars indicating $u_c(P)$ are obscured by the markers used to indicate each data point. For Sample 1 $u_c(P)/P < 2.9\%$ and for Sample 2 $u_c(P)/P < 3.5\%$. The starting gauge lengths at 0 elongation are the straight line approximations to the bowed specimens, seen in Figure 6.17 and Figure 6.20. Data to the right of the dotted vertical lines was used for Figure 6.18 and Figure 6.21.
2.6.2 Electrical Measurements

Electrical measurements during the tensile tests are reported in Figure 2.2. For each voltage data point, 1000 samples were averaged to provide the recorded value. The standard uncertainty for all voltage measurements was calculated from the measured samples according to 2.7 and found never to exceed 0.035% of the recorded voltage values.

Current was measured by recording the voltage drop across a resistor measured to have resistance $R = 386 \, \Omega$, with a standard uncertainty $u_c(R)$ of this resistance of 11 Ω. (This standard uncertainty was found using Eq. 2.8 assuming that the ±5% specification was a hard boundary on the tolerance of the resistor.) For each data point, 1000 voltage samples were averaged, yielding a Type A standard uncertainty calculation from these measurements, and the electrical current value was derived by Ohm’s law. The uncertainty in the current value $u_c(I)$ was determined according to Eq. 2.22.

$$
\left( \frac{u_c(I)}{I} \right)^2 = \left( \frac{u_c(V)}{V} \right)^2 + \left( \frac{u_c(R)}{R} \right)^2 \approx \left( \frac{u_c(R)}{R} \right)^2
$$

(2.22)

The uncertainty in the resistor used to derive current values dominates the voltage drop uncertainty by about 2 orders of magnitude.
Figure 2.2: Actuator electrical performance during the tensile tests. All voltage measurements have 0.035% or less standard uncertainty. All current measurements have standard uncertainties of 2.9% of the reported values. It should be noted from this figure that the different actuators used in testing have nearly the same resistance, and similar behavior at high input powers.
2.6.3 Strain Measurement Results

The engineering strain $\epsilon$ for each fiber specimen was calculated by dividing the elongations noted in Figure 2.1 by the gauge length $L_0$ reported for each specimen. The standard uncertainty for the strain measurements can be found according to Eq. 2.23.

\[
\left( \frac{u_c(\epsilon)}{\epsilon} \right)^2 = \left( \frac{u_c(\Delta L)}{\Delta L} \right)^2 + \left( \frac{u_c(L_0)}{L_0} \right)^2 \tag{2.23}
\]

The data presented in Figure 2.1 clearly demonstrate elongation of the fiber specimens during the course of the tensile experiments. However, the uncertainty in the length measurements obtained using line fits to SEM images leads to elongation measurements with a minimum 5.6% uncertainty $\left( \frac{u_c(\Delta L)}{\Delta L} \right)$. The uncertainty in elongation dominates the uncertainty in the strain measurement.

2.6.4 Stress Measurement Results

The calculation of the stress applied to the tensile specimens follows the same approach as has been previously described for similar test structures [11,12]. The engineering stress $\sigma$ is found according to Eq. 2.1, assuming a circular cross section. The associated standard uncertainty is then computed according to Eq. 2.24.

\[
\left( \frac{u_c(\sigma)}{\sigma} \right)^2 = \left( \frac{u_c(F)}{F} \right)^2 + \left( \frac{u_c(A)}{A} \right)^2 \tag{2.24}
\]

The load forces ($F$, Eq. 2.25) applied to the specimens were calculated according to the “expected displacement” procedure described previously [10–12], wherein a measured displacement $d$ of the tensile stage is subtracted from an expected displacement $d_0$ that is predicted from the electrical power supplied to the thermal actuator according to a fit curve. This is described further in Section 2.6.6. Figure 2.3 illustrates the discrepancies between $d_0$ and $d$ during the two tensile tests.
Since the applied force $F$ is taken as proportional to this discrepancy, Figure 2.3 also illustrates the relative progression of applied tensile force during the tensile tests.

The spring constant of the tensile system is taken as $k = 175 \pm 29$ N/m according to analysis of nearly identical MEMS testers [10]. The standard uncertainty in the applied force is calculated according to Eq. 2.26, which was derived using Eq. 2.9.

$$F = k(d_0 - d) \quad (2.25)$$

$$u_c(F) = \left[ ((d_0 - d)u_c(k))^2 + (ku_c(d_0))^2 + (ku_c(d))^2 \right]^{\frac{1}{2}} \quad (2.26)$$

The greatest sources of uncertainty in the stress measurements are the displacement measurements used to find the applied force, and also the spring constant used.
Second-Order Polynomial Fit Discrepancy

(a) Specimen 1.

(b) Specimen 2.

Figure 2.3: The discrepancies \((d_0 - d)\) between the values \(d_0\) predicted by the second order polynomial fits found in Figure 2.4(a) and Figure 2.4(b) and the measured stage motion \(d\) during and after the tensile tests. The forces experienced by the nanofibers during the tests can be found by multiplying the stage displacement discrepancies \((d_0 - d)\) during the tensile test by the test system spring constant, \(k\). At the lower power levels, the X error bars are obscured by the data markers, as in Figure 2.1. The Y error bars on these plots show the displacement measurement uncertainty for each point. These plots represent the residuals to the \(d_0\) fit curve, so fit uncertainty in \(d_0\) is not included in the Y error bars.
2.6.5 Off-Axis Loading

Reference [10] performed a detailed analysis of the uncertainties in the same type of tensile test due in particular to misalignment error. The main conclusion of this analysis was that most misalignment uncertainty is insignificant in comparison to the force uncertainty due to the uncertainty of the device spring constant. Until the spring constant can be better measured and calibrated, most of the uncertainty in stress and strain due to misalignment or out-of-plane motion does not provide a significant contribution of the stress and strain uncertainties. Only the gross difference in tensile force due to the misalignment was added using Eq. 2.9 to the force uncertainty, Eq. 2.26. The force uncertainty from misalignment \( u_{\text{misalignment}}(F) \) was calculated from Eq. 2.27, with misalignment angle \( \theta \approx 13^\circ \) for Specimen 1 and \( \theta \approx 7^\circ \) for Specimen 2. Here, the force applied by the MEMS device is \( F = F_x \), and the true tensile force in the fiber is \( F_T \).

\[
\begin{align*}
    u_{\text{misalignment}}^2(F) &= (F_T - F_x)^2 \\
    &= \left( \frac{F_x}{\cos \theta} - F_x \right)^2 \\
    &= F_x^2 \left( \frac{1}{\cos \theta} - 1 \right)^2
\end{align*}
\]  

(2.27)

2.6.6 Expected Displacement

The motion of the tensile stage after the failure of a nanofiber specimen is used to derive a relation between thermal actuator input electrical power and the freely moving displacement of the tensile stage. The use of the computerized data acquisition system enabled improved precision of the electrical measurements of the actuator during the tensile test in comparison with initial reports on similar systems. [11,12]

This allowed for improvement of the regression fit, as seen in Figure 2.4(a) and Figure 2.4(b), over the linear fit approach that was used in previous work. [11,12] Several regression fits were compared, with the constraint that they must predict zero displacement when the actuator input power is zero. Because the actuators and test devices were on separate microchips that may have
experienced slight variations in processing, the fit parameters used in calculating \( d_0 \) were calculated for the different test structures using the motion of the mechanical stages after each tensile test. The use of a nonlinear fit makes sense physically because of the complex strain states within the thermal actuator beams as they expand and bend to cause motion, and because of the temperature dependencies of the thermal and electrical conductivities.

In comparison with the linear fit, polynomial fits and a power law fit offer a large improvement in prediction of the output displacement. The \( R^2 \) (coefficient of determination) value for the second order polynomial fit was somewhat better than for the power law fit, but third order polynomial fit offered negligible improvement in the \( R^2 \) value over the second order polynomial fit for the tester used to measure Specimen 1. Therefore, the second order polynomial fit was chosen for estimation of the expected displacement parameter \( d_0 \) used in the force and stress analysis in this section. Equations 2.28 and 2.29 show the fit equations derived for the data from the testers for Specimen 1 and Specimen 2, respectively. It should be noted that the fit parameters on Figures 2.4(a) and 2.4(b) were provided by data analysis software (Microsoft Excel\textsuperscript{TM}) but that better \( R^2 \) values can be obtained if the fit parameters \( \beta_i \) are calculated with better numerical precision than is available with the regression fit functions built in the analysis software. The parameters reported in Eq. 2.28 give \( R^2 = 0.9998 \) for the Specimen 1 test and in Eq. 2.29 \( R^2 = 0.9994 \) is obtained for the Specimen 2 test.

\[
\begin{align*}
  d_0 &= \beta_2 P^2 + \beta_1 P = 0.000271P^2 + 0.01479P \\
  d_0 &= \beta_2 P^2 + \beta_1 P = 0.000305P^2 + 0.01497P 
\end{align*}
\] 

Because the fit parameters reflect a relatively good fit to the electrical and displacement data, the uncertainty in smaller displacement measurements and the uncertainty of the spring constant dominate the uncertainty in the force and stress results. Figure 2.3 illustrates the fit residuals, and the clear discrepancy from the expected displacement that arises during the tensile test.
Figure 2.4: Comparison of different regression fits to data points acquired from test stage motion after failure of tensile specimens. The displacements plotted are averages of displacement measurements that were repeated 3 times. The fit equations and $R^2$ parameters for each fit line are included on the graphs.
Chapter 3

Designs

In the design development process, constraints and specifications for tensile testing of carbon nanotubes and GaN nanowires were identified, as were constraints for performing testing in multiple environments. Design development followed an iterative process: creating ideas to meet the testing constraints, fabricating and attempting to operate these designs, and learning from fabrication and operation experiences to improve and further develop the designs. Below are presented the design specifications and constraints; reasoning behind aspects of the different integrated tester, Universal Test Platform, and test coupon designs; and examples of the most recent design iterations.

3.1 Specifications and Constraints

Because of the availability of GaN NWs in the iMINT center, and ongoing scientific interest in carbon nanotubes, these two materials were chosen to provide specifications for the force and displacement range required of the microfabricated test systems developed here. These two materials also represent the extrema of a range of nanoscale fiber sizes for which further mechanical characterization would be of scientific interest. These specifications were developed in collaboration with P. Kabos (NIST), M. Wallis (NIST), K. Bertness (NIST), D. Dikin (Northwestern U.), R. Ruoff (U. Texas), N. Sanford (NIST), and V. Bright (CU). Table 3.1 summarizes GaN NW and CNT properties used to define design guidelines.

Further constraints for carbon nanotube tests depend on the environment for the test and the method of placement of the carbon nanotubes. The nanotube flip chip technique [56] was originally
Table 3.1: Table showing typical properties expected for GaN NWs grown by K. Bertness’ group at NIST-Boulder, and for potential CNT samples. [8, 141, 161, 162] At the bottom of the table are the forces estimated to be needed for specimen failure, and the stage motion expected during a typical tensile test. Order of magnitude failure strength values are estimated as 10% of the material modulus values.

<table>
<thead>
<tr>
<th></th>
<th>GaN NWs</th>
<th>CNTs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Typical Young’s Modulus</td>
<td>300 GPa</td>
<td>1 TPa</td>
</tr>
<tr>
<td>Estimated Failure Strength</td>
<td>~30 GPa</td>
<td>~100 GPa</td>
</tr>
<tr>
<td>Typical Length</td>
<td>~10 - 25 µm</td>
<td>Up to ~300 µm</td>
</tr>
<tr>
<td>Strain to Failure</td>
<td>A few % (unknown)</td>
<td>~6 %, possibly greater</td>
</tr>
<tr>
<td>Diameter</td>
<td>50 - 500 nm, typically 120 nm</td>
<td>1 - 15 nm</td>
</tr>
<tr>
<td>Applied Force</td>
<td>~340 µN</td>
<td>~1 µN - 10 µN</td>
</tr>
<tr>
<td>Stage Motion for 20 µm sample</td>
<td>≤1 µm</td>
<td>≤3 µm (15%: remove slack, accommodate strain to failure)</td>
</tr>
</tbody>
</table>

considered for placement of CNTs, but discarded because of the risk of microsystem damage and difficulty in obtaining good CNT specimens to use to test this technique.

For growth of CNTs across a tensile stage, microsystems must withstand temperatures of at least 800°C in a non-oxidative atmosphere. Si is an adequate material for such temperatures, but metals, especially the noble metals, in contact with Si are to be avoided due to low-temperature eutectic formation (in particular, the Au-Si system has a eutectic point below 400°C).

If a TEM is to be used to observe CNTs on tensile stages, the coupon and Universal Test Platform (UTP) system must allow an electron beam to be transmitted completely through these microdevices.

### 3.1.1 Universal Testing Platform Specifications

As stated in Ch. 1, the test platform developed here should be an actuated microfabricated device whose purpose is to drive mechanical components on secondary test coupon chips used to support nanomaterials specimens. This Universal Testing Platform (UTP) should allow simultaneous, multi-property characterization of nanofiber samples mounted on the microfabricated test coupon. It should interface with the test coupon and perform mechanical tests. Also allowed
should be electrical, optical and radio frequency probing from the direction normal to the area of
the MEMS Coupon. Silicon is preferred as fabrication substrate because of available processing
knowledge. The UTP should operate primarily at room temperature, and be capable of providing
the forces specified in Table 3.1.

Although not critical for initial demonstration of the microsystem, some additional desired
features for the UTP system include the following:

- Measures overall sample temperature.

- Displacement resolution preferred at most 1% of gap displacement (e.g. 50 µm gap expands
  2.5 µm during test, 1% of 2.5 µm is 25 nm), otherwise as small as possible (currently 10
  nm at Northwestern, 0.5 nm preferred).

- Applied stress measurement resolution 50 nN or smaller. Resolution of 1% of applied
  sample load is also preferred (e.g., 100 nN load to fracture a 1 nm CNT, 1% of 100 nN is
  1 nN).

- Measurements precise to 3 significant figures.

- Contains motion stops to prevent fracture of coupon or UTP from excessive motion after
  specimen fractures.

3.1.2 Test Coupon Specifications

The MEMS Coupon is a specimen holder on which nanowire or nanotube specimens may
be placed or grown. This object should withstand a growth environment for these materials,
and should allow robust manipulation of these materials between laboratory instruments including
the transmission electron microscope, scanning electron microscope, optical microscope, and the
Universal Testing Platform. Silicon is preferred as fabrication substrate because of available mi-
crofabrication processing knowledge. The coupon contains a tensile stage that is defined by a gap
across which the nanofiber is suspended. In transmission electron microscopy and some types of
optical microscopy, beams of electrons or of light pass through the specimen. In order to allow these types of microscopy to examine the tensile specimen, the test coupon design should incorporate a window that allows electrons or light to pass completely through the test coupon.

For radio-frequency characterization, microstrip or coplanar waveguide structures are required on the test stage. A microstrip structure is essentially one strip of metal, on top of a dielectric layer insulating from a lower, wider strip of metal that serves as an electrical ground plane. At each end of these strips, three 50 \( \mu \text{m} \) square contact areas, with 125 \( \mu \text{m} \) pitch, are necessary to allow connection to the microstrip in the center and the ground plane on either side. In order to allow contact from probes in a radio frequency probe station, contact areas should be no closer than 125 \( \mu \text{m} \) from either side of the gap, although 1 mm is preferred. The width and spacing of the metal layers may be calculated to give an overall impedance of about 50 \( \Omega \), with dimensions depending on the thickness and type of dielectric used. The center conductor leads up to each side of the tensile stage gap. The specimen will connect to this top strip of metal on each side of the gap. The dielectric layer should be ideally greater than 300 nm thick, but less than 1 micron thick. A coplanar waveguide (CPW) structure is a slightly simpler configuration, consisting of a metal layer resting on a dielectric layer. In the CPW, a strip of metal provides the electrical signal to the material specimen in a similar configuration as the microstrip line. The CPW ground planes are made in the same metal layer and provide continuous metal traces flanking both sides of the signal line. Fig. 6.48 in Section 6.6 provides an illustration of dimensions of key parts of a coplanar waveguide design that has been successfully fabricated on a test coupon.

### 3.2 Opportunities for Microscale Testing

The overall approach and taken for development of the tensile test microsystems presented below emerges from the background information introduced in Ch. 1.

Many components of potential microfabricated test systems have already been created and reported. These include the following:
• Microfabricated chips for nanomaterial testing with integrated load sensors or displacement sensors

• Successful packaging and electrical interfacing to microfabricated tensile test chips using wirebonding or non-permanent pogo pin contacts

• Coupons that interface to piezoelectric actuation with measurement from verniers

• Data collection from SEM/visual inspection of moving parts

• Data collection from capacitive sensors

• Tests in multiple environments including ambient and vacuum operation

• Simultaneous piezoresistive and mechanical measurements of thin films using microfabricated test structures

The major problems encountered in previous work with tensile test microsystems have been the following:

• Slow setup of experiments, due to a number of factors:
  – Slow manipulation and bonding of individual NWs or CNTs
  – Test platform redesign for each type of material tested
  – Complicated fabrication of test platforms with integrated nanomaterial specimens

• Not much data has actually been reported from such systems, possibly due to slow setup and experimental analysis

• Force measurements with most systems have not been calibrated against known forces

• Nor has uncertainty analysis of microfabricated tensile test systems been significantly documented
From these observed problems with microfabricated tensile test systems, several opportunities for new work emerge:

- Calibration and error analysis methodology could be developed and reported for tensile microsystems.
- A device which speeds up data collection and interpretation could be created. To do this, the device would need to do the following:
  - Enable rapid tensile specimen preparation
  - Minimize the use of micromanipulators in specimen preparation and test operation, as micromanipulator positioning is a serial, slow operation
  - Automate collection of data of force and displacement applied to the tensile specimen, possibly through computerized actuator control and electronic displacement sensing
  - Keep potentially damaging specimen placement operations away from actuation and sensing
- By allowing probing from the top surface of a nanomaterial specimen, a broad range of property cross-correlation measurements could be enabled for nanomaterial characterization, such as optomechanical or microwave-mechanical tests.

Increasing the speed and quantity of data collection can occur in particular due to the following factors:

- Simplifying the fabrication of the consumable components (specimen, test coupon) enables fast preparation of test specimens
- Separate specimen mounting allows fast specimen preparation processes such as dielectrophoretic NW placement or CNT chemical vapor deposition to be performed far from the test platform elements, allowing reuse of the test platform
• Use of tweezers in the laboratory environment and unidirectional micromanipulators microfabricated into the test platform to handle the test coupon simplifies manipulation of nanomaterial specimens

3.3 Multiscale Interfacing

Consideration of multiscale design was required in order to act as a human operator to interact with nanostructured materials. Fig. 3.1 provides an illustration of some of the multiple size scale thinking required. The specimen must be mounted on a test stage, which is part of a microfabricated system, which is actuated from a larger system. Electrical connections are required to operate an actuator, so a strategy for packaging the microsystem is needed, as are strategies for providing electrical connections to the chip package, and ultimately providing a source of electrical power that can be controlled by the operator of an experiment.

Design of the microfabricated structures will be discussed in the remainder of this chapter. For most experiments other than those requiring a TEM, use of a 20 pin dual inline package (DIP) ceramic chip carrier with gold wirebonds to the actuated microdevices was sufficient to power the microsystems, and the use of a piece of electrical breadboard (Fig. 3.2) was sufficient in most cases to provide electrical connections between wiring and the chip package. For operation of MEMS tensile testers within an SEM, electrical feedthroughs in the SEM wall were used to provide connections between the tensile test microsystem and the instrumentation powering this microsystem (Fig. 3.3).

Initially (for the work in Section 6.2, for example) power was directly supplied by a DC power supply, with current and voltage readings read from connected multimeters. Data acquisition with this approach quickly became a major bottleneck to performance of new experiments. Automation of this data acquisition process, and development of custom electronics to power electrothermomechanical actuators was developed as a Master’s thesis project by D. Luu. [88] This moved the data acquisition and system control to a graphical user interface running in the MATLAB computing environment (Fig. 3.4). Integrated tester thermal actuators were controlled by an op-amp current
source circuit. The UTP thermal actuators were driven by a Darlington transistor pair acting as a current sink controlled by an op-amp current control loop.

This data acquisition system was also capable of data collection from piezoresistive beams electrically linked to thermal actuators in the Universal Test Platform system. Electrical isolation was included to prevent destruction of piezoresistive beams due to common mode current flow through the beams. The piezoresistive measurements provide some information about displacement, but time constraints prevented in-depth development of these systems. In operational testing, they appeared to show some piezoresistive effect, but also present was a large temperature effect due to heating from the thermal actuators.

Figure 3.1: Illustration of multiscale interfacing challenges.
Figure 3.2: Chip packaging for 2 mm chips. Here, a MEMS chip (A) is secured and wirebonded on a 20 pin ceramic DIP chip carrier (B). Electrical interfacing to the chip carrier occurred through the breadboard (C) which was cut to the size shown to allow fitting within the SEM. The scale bar is approximate.

Figure 3.3: Operation of integrated testers at the University of Colorado (CU) Nanomaterials Characterization Facility. The computer at left runs analog electronics, connected via a feedthrough to the integrated testers operated inside the LV-SEM.
Figure 3.4: Graphical User Interface used to control UTP system. Developed by D. Luu at CU in collaboration with this project. [88]
3.4 MEMS Actuation

The limitation of tensile stage motion to in-plane motion provided a strategy to allow out-of-plane examination of tensile specimens in addition to presenting the possibility of generating uniaxial forces and the possibility for additional probe access to the tensile stages and specimens. Electrothermomechanical actuators were chosen as the motion source for most designs. Piezoelectric actuators would not be easily integrated to a test chip, and comb drive actuators generally required too large an area for sufficient forces for nanowire testing.

The general principle of electrothermomechanical actuators is that, as electrical power is increased to the actuator, the heat flow out of the actuator increases. Because of the material thermal resistance, the temperature rises in the actuator due to the increased heat flow. The temperature increase causes thermal expansion of the actuator material, which produces force if the motion of the expanding actuator beams is constrained.

Two types of electrothermomechanical MEMS actuators have been developed in past work. [38, 76, 155] Bending arm or U-type actuators achieve motion by differential expansion of a hot arm and a cold arm, and move in an arc of motion unless symmetrically paired and linked with a compliant structure to a suspended shuttle between the actuator pair. Chevron or V-type thermal actuators rely on the buckling of pairs of angled beams to achieve a linear displacement. All beam elements of the V-type thermal actuators contribute to the actuator force and displacement output, whereas the cold arms of the U-type actuators oppose and constrain the motion of the hot arms, so V-type actuators were selected to provide force and displacement output for the tensile test Microsystems developed below.

By angling the actuator beams 1° off of perpendicular to the shuttle that transmits force to the test stage and by placing the beams in pairs symmetric across the axis of motion, the opposing beams are constrained from off-axis motion and their bending moments at the shuttle also cancel. They experience a buckling motion only in the shuttle axis. Using V-actuator angled geometry and the solid mechanics plane strain condition, the maximum force output of a V-type actuator can be
easily estimated. The plane strain condition approximates the case where the stage is constrained not to move even as the beams in the actuator are forced to thermally expand due to Joule heating. For actuators for the integrated testers, the actuator design was adapted from a design developed by D. Miller, formerly a student at the University of Colorado. Some designs also used $3^\circ$ angles.

The largest drawback to the use of electrothermomechanical MEMS actuators is that they may require a significant power dissipation from the chip on which they are supported. The substrate chip temperature may rise until heat transfer matches power dissipation from the thermal actuator. Because this project aimed to demonstrate the operation of new devices, the advantages of size and force output from thermal actuators were deemed more important than potential chip temperature problems. If the actuator rises to too high a temperature, the material comprising the actuator will soften, creep, and eventually fail, creating a definite limit to the displacement or force that may be generated by a thermal actuator.

### 3.5 Integrated Tester Design and Fabrication

In order to develop an understanding of the practical issues involved in nanomaterial testing (such as challenges of specimen preparation, instrumentation, and data analysis), integrated MEMS tensile test systems were developed, consisting of a fixed stage and a moving stage directly linked to a microfabricated actuator.

These designs provide an easily fabricated means of obtaining MEMS tensile testers for nanomaterials. Key features of the designs are one fixed stage and one suspended moving portion of the tensile stage, connected to a MEMS actuator that pulls on the moving stage to increase the separation of the portions of the stage. The fixed stage is anchored to a silicon nitride substrate, and the moving stage is connected by a shuttle to a thermal actuator. Stabilizing beams are placed in opposing pairs along the length of the moving stage in order to reduce or eliminate off-axis motion and ensure that the stage only moves in the direction provided by the actuator. Connections to wirebond pads provide additional electrical access to the moving stage.
Designs were built from released polysilicon using the PolyMUMPS prototyping service. Moving parts are fabricated from double layer POLY1 and POLY2 polycrystalline silicon by creating a large region of the POLY1 layer, removing OXIDE2 from this region using the POLY1_POLY2_VIA mask, and then defining the desired pattern in the POLY2 layer. Patterning of the POLY2 layer additionally patterns the underlying POLY1 layer.

Comb drive actuation was initially examined for integrated tensile testers, but was abandoned because of the footprint required for sufficient force output to apply strain to nanowires. Electrothermomechanical actuators were selected to provide actuation to the test systems. Figure 3.5 shows one of the first successful test systems, which incorporates some extra displacement measurement features that were not fully explored. Attached to one side of the thermal actuator is an optical grating intended to modify the angle of reflection of an incident laser, and at the end of this grating are components that were intended to function as electron field emission sensors. The capability of measurement of stabilizing beam piezoresistance was incorporated in all integrated tester designs, but data was not easily collected from these beams due to the tendency of electrical signals from the thermal actuator to interfere with and occasionally overheat and destroy the stabilizing beams when resistance measurements were attempted. Eventually a strategy for resistance measurements of these beams with sufficient electrical isolation was developed by M.S. student D. Luu as part of his thesis project, which was completed in May 2010. In particular, the design at far right in Fig. 3.6 was used for most of his data collection and experimentation.

Additional integrated tester designs are seen in Figures 3.6 and 3.7. Fig. 3.6 shows several designs that never were put to extensive use, mostly because new work with the UTP and coupon system had been started by the time these were ready for use. Also, for the design at middle and left, this design was abandoned because it required a much larger footprint than the designs in either of Figures 3.5 or 3.7. The designs using the V-type electrothermomechanical actuators had been shown to be effective for nanomaterial tensile testing by the time that an initial design problem in the U-type bending arm actuator tester was corrected. In the end, the design seen in Fig. 3.7 was capable of providing mechanical test data from nanoscale materials placed on these
testers. The small footprint of these testers meant that from a given fabrication run of 15 chips many more of these devices could be prepared with material specimens than with the other designs in Figures 3.5 and 3.6.

Figure 3.5: The initial design for integrated MEMS tensile testers using a V-type (chevron) electrothermomechanical actuator to drive motion of the tensile stage.
Figure 3.6: Additional integrated tester designs. The design seen at middle and left uses U-type bending arm electrothermomechanical actuators. The design at right contained a doubled actuator and extra stabilizing springs in an attempt to provide a design capable of allowing better direct collection of displacement data indicative of specimen strain and applied force.

Figure 3.7: The final design for integrated tester devices. Six of these devices can fit on one 2 mm × 2 mm chip.
3.6 Coupon and UTP System Designs

Based on the specifications and constraints discussed above, the test coupons and Universal Test Platform (UTP) designs were created following these guidelines:

- At the test stage the system should supply a force within one or two orders of magnitude of the force required for the nanomaterial specimen fracture (\(\sim 400 \mu \text{N} \) for GaN NWs, \(\sim 10 \mu \text{N} \) for CNTs).

- Uniaxial motion must be supplied to the nanofiber in order to minimize errors due to bending moments and clamp failure.

- Frictional forces cannot be introduced to the measurement. Coupon and universal test platform (UTP) surfaces cannot be allowed to slide or experience shear traction. All mechanical interfacing should be performed with normal forces.

- Specimen preparation must be confined to simple structures.

- Test coupons should be customizable for harsh environment (e.g., high temperature) preparation.

- The test coupon should allow electrical and optical access to the specimen, and should be able to be customized for specific tests (e.g., RF, TEM).

- The coupon should be robust. It must be able to be handled within a laboratory environment without special equipment.

- The mechanical interface between the coupon and the UTP must be robust. It must not be a permanent joint, and it must not be easily damaged as the coupon is mounted in the UTP.

- The coupon and UTP test system must be able to accommodate vertical misalignment. They should be able to derive measurements from the coupon even if it is not perfectly aligned in the UTP.
Planning an idealized user experience based on the following steps also helped guide the design process:

(1) Put UTP in environment for specific test (e.g., TEM, Raman, RF).

(2) Put specimen on coupon.

(3) Put coupon in UTP (handle with tweezers).

(4) Run mechanical portion of test from computer.

3.6.1 Design of Chip Interfacing

The non-permanent mechanical interface between the coupon and test platform chips enables a variety of coupon designs to interface with a well-designed test platform system, and is a design approach that has not been seen in other work with microsystems capable of testing individual nanofibers.

For successful mechanical interfacing, only normal forces and small surface areas can be used by the probes on the test platform, as contacting that uses shear forces may also lead to stiction that could damage the test platform. So, devices are confined to motion within the plane of a device layer anchored to a substrate. The contacting microprobe must contact the edge of the coupon device layer, so vertical alignment between these two structures is critical.

A number of active vertical (out-of-plane) alignment systems were considered, before a simpler design was chosen (Figure 3.8). The bulk part of the test coupon is inserted into a deep well formed within the Universal Test Platform chip. Tabs protruding from the top of the coupon rest on the surface of the test platform. The coupon aligns vertically to the test platform probe simply by resting on the test platform. As long as the thickness of the coupon device layer is thicker than the sacrificial layer removed from the test platform, the microprobe should contact the coupon. By placing test coupons face up rather than flipping them into place, the coupon frontside is available for optical and electrical access.
The in-plane alignment was a more difficult question to resolve, because a tolerance had to be left between the coupon substrate and the test platform well wall in order to allow for misalignment as the coupon is placed into the well. In practice, this wasn’t as critical as anticipated because the deep etch processes used in coupon and test platform fabrication slightly overetched the vertical walls in the coupon and test platform devices, thereby increasing the misalignment tolerance between the two chips. For designs, a gap of 10 microns was chosen at each edge, allowing 0.5% of the overall dimension of the 2 mm coupon for imprecision in manufacture due to possible angles in the sidewalls and misalignment in placement.

Several passive alignment mechanisms were designed, namely posts and springs for the tabs on the coupon to abut (these may be seen in Fig. 3.8). Additionally, an active alignment mechanism was designed, wherein actuators would push circular probes into V-shaped tabs, pushing the coupon against posts at the back of the well, and laterally aligning the coupon so that both edges of the V would contact the circular tab. The effectiveness of these alignment methods is discussed in Ch. 5.
3.6.2 Active Interfacing, Coupon Actuation, and UTP Displacement Amplification

To avoid error created by friction and the possibility of undesired stiction, all actuated mechanical interfacing between the UTP and test coupon for experimental measurements must be accomplished with normal forces between contacting surfaces. This can be accomplished through pull-tab and sliding tab interfaces, or through compressive interfaces. Because interfaces involving tabs are not inherently robust—they present a risk of fracture as a coupon is being positioned onto the UTP system—they were set aside in favor of compressive interfaces. Similarly, other mechanisms for interfacing two chips such as electrostatic clamping, glueing and unglueing, soldering and desoldering were possible design directions, but they were not pursued because they did not appear to be inherently robust enough to allow significant reuse of test platform chips.

If a 20 µm misalignment is allowable according to the chip interfacing design, then the test platform must probe the test coupon with a probe that can move more than the 20 µm misalignment distance, and yet provide the 400 µN of force needed for fracture of a nanowire. Electrostatic comb drives might be capable of this displacement, but a very large area would be required in order to access that magnitude of force from a comb drive. Thermal actuators can provide several millinewtons of force, but keeping them within a 300:1 beam aspect ratio and below the fracture stress of Si limits them to about 5 to 10 µm of output displacement. In order to meet the specifications of >20 µm displacement and >400 µN force, necessary to perform a tensile test on a removable coupon, a displacement amplification transmission system was designed. The displacement amplification system is a requirement of the mechanical interface design selected above and the need to have a non-permanent mechanical interface. After consideration of many other interfacing systems, it became evident that displacement amplification was the only workable approach to providing a test probe system separate from a specimen holder.

The displacement amplification system was designed by considering several arrangements of bending beams and simulating their maximum displacement and maximum force outputs using Coventorware software [23]. In order to prevent frictional wear and unpredictable stiction behavior,
the displacement amplification system was not allowed to use pivoting or pinned joints, and all anchor points were clamped joints created by joining of a moving compliant beam with an anchored fixed region. By constraining the internal stresses to less than the tensile strength of Si, and seeking maximum force and displacement outputs, the basic beam network design seen in Figure 3.9 was arrived at after about 10 - 20 iterations of simulation, which had started with a wide variety of brainstormed compliant displacement amplification structures (Fig. 3.10). With every UTP fabrication run, additional arrays of compliant probe designs were fabricated (such as in Fig. 3.11), allowing comparison of the survivability and effectiveness of probe designs. Although the final probe designs generally displayed good robustness, they were observed to experience more stiction to the underlying substrate if extra springs were attached at the ends of the probes (as seen at the ends of the design in Fig. 3.11). Because piezoresistivity in spring deformations provides one means to detect displacement, extra springs were sometimes added at the end of probe designs in order to explore this possible sensing modality.

Figure 3.9: The three displacement amplification transmission designs that were implemented in the UTP designs. The top row shows designs as created in L-edit CAD software. [140] The bottom row shows meshed solid models constructed using Coventorware software. [23]
Figure 3.10: Initial designs considered for compliant displacement amplification structures.

Figure 3.11: Test array of transmission designs.
3.6.3 UTP Designs

The design of the test platform for performing experiments on the test coupon structures centers on the use of a microfabricated probe which interfaces mechanically with the coupon, and the mechanical alignment and interfacing between the coupon chip and the test platform chip. Metal pads were placed around the periphery of the designs to allow wirebond connections to actuator and piezoresistor sensing structures. Six variations on the test platform were designed and fabricated, with dimensions of 4.5 mm × 4 mm, 3.5 mm × 4 mm, and 2.5 mm × 4 mm (Figure 3.12). The smallest of these layouts place the tensile specimen exactly at the center of the chip, and were designed especially to be used in constrained spaces such as within a TEM stage.
Figure 3.12: The six different Universal Test Platform chip designs implemented in the last fabrication round. Dimensions of the different layouts are 4.5 mm × 4 mm, 3.5 mm × 4 mm, and 2.5 mm × 4 mm. The general concept for all designs is that passive alignment structures present general positioning of test coupons (green regions) and that actuated mechanical probes extend from the test platform chips to contact the test coupon structures.
3.6.4 Test Coupon Designs

Because the test coupon must allow for several microns deformation of the test specimen, it must include fixed and moving stages and a network of supporting springs in its device layer. Attachment of a test specimen will modify the overall spring constant of this network. Measurement of force and displacement at the UTP will allow observation of the coupons effective spring constant and extraction of the tensile behavior of the fiber. Relations between force and displacement inputs and outputs will have the form of some transfer function that is proportional to elastic modulus $E$ and device thickness $t$ because each component of the function will have a spring constant proportional to $Et$. The motion of each beam will be a combination of bending and compressive or tensile responses. For the coupon layout, layouts symmetric about the axis of motion are best at providing uniaxial motion, although asymmetric layouts can be adapted to provide nearly uniaxial motion.

The test coupon design is an optimization problem restricted by the constraints identified here and by the techniques and materials available for microfabrication processing. A key implication is that the coupon must consist of some sort of device layer that is a small region of a larger chip that is used for handling in a laboratory. The device layer could be a thin ($3.5 \, \mu m$) Si layer on top of handle wafer ($100$ or $300 \, \mu m$) in an SOI (system-on-insulator) wafer, or it could be a thicker layer in a monolithic coupon. Lithographic constraints of a beam aspect ratio less than $300:1$ and the need for devices that are thicker than they are wide in order to prevent out-of-plane vibrations restrict the active test area to about $500 \, \mu m \times 500 \, \mu m$ or less, and discussions and experience have set the total coupon chip size at $2 \, mm \times 2 \, mm$. This is about the minimum size that can be easily handled with tweezers in a laboratory setting.

One finding from design simulations using Coventorware software was that measurement resolution may be limited by deformation of a fixed stage under stress. For instance, $400 \, \mu N$ loaded in a concentrated region, as is required for GaN NW tests, causes the fixed stage region of the design to deform $8 \, nm$. This means that measurements on nanowires may only be accurate
to ±10 nm at best, but carbon nanotubes might allow better accuracy because they require less deformation force. Another finding was that the fundamental eigenfrequency of ~21 kHz (for the coupon design in Figure 3.13) is much larger than frequencies encountered in typical handling within a laboratory setting, so specimens handled on test coupons are not likely to be disturbed by the handling of the test coupons.

For electrical measurements in a radio frequency probe station, the test coupon needed to accommodate coplanar waveguide metal patterns on top of an insulating layer. The insulating layer was chosen as silicon dioxide, as this could easily be formed by thermal oxidation of wafers before any subsequent processing, and it has a higher dielectric constant than silicon nitride, which was an important parameter for the impedance design. Microstrip designs required a highly conductive ground plane underneath a dielectric layer; the doped Si was insufficient. The coplanar waveguide design was chosen because it offered simpler fabrication than the microstrip design. All of the coplanar waveguide metal is formed in one metal deposition step. Calibration of the impedance analyzer used to record measurements from a coplanar waveguide requires that calibration lines (Fig. 3.14) be patterned along side the test coupons in the wafer used to produce the test coupons. For generation of coupons that can withstand CNT growth temperatures, the waveguide metal deposition step is omitted.

A solid model of a test coupon with alignment tabs, compliant test stage region, metal waveguide, and regions for tweezers to grip from the sides may be seen in Fig. 3.13. The device layer of this design was later updated to the variations shown in Fig. 3.15. In contrast to the device layer shown in Fig. 3.13, the four designs at the center and left of Fig. 3.15 place the ground planes of the coplanar waveguide structures on fixed regions which do not move, allowing the ground planes to form a continuous path on either side of the waveguide center conductor.
Figure 3.13: A solid model of a test coupon design, showing alignment tabs, shuttle and device layer in blue, handle wafer in green, and yellow metal coplanar waveguides.

Figure 3.14: Example of calibration line design required to identify background-level radio frequency transmission and reflection signals.
Figure 3.15: Six different coupon designs implemented in the last round of fabrication. There are 3 different compliant test stages, and two different alignment tab arrangements, giving six total combinations. The red regions indicate the coupon device layer. The yellow-grey and dark red regions indicate the deep well locations etched from the wafer backside. The purple regions indicate the metal patterns used as radio frequency coplanar waveguides.
Chapter 4

Fabrication

This chapter describes the methods used to fabricate the various microsystems used in this thesis.

4.1 Process Overview

Integrated testers such as those reported in sections 6.2, 6.3, and 6.4 were fabricated using the well-documented PolyMUMPS prototyping service. [14,108] After fabrication, chips supporting these testers were subdiced and the moving parts were released using these HF (hydrofluoric acid) release process steps:

1. Use solvents (acetone, isopropanol, water) to remove protective photoresist and clean the device surface.

2. Remove SiO$_2$ layers by immersion in HF 48\%$_{(aq)}$ for times ranging from 2 minutes 45 seconds to 3 minutes 30 seconds.

3. Transfer to water, 4:1 (volume:volume) methanol:water mixture, and finally methanol in order to remove trace HF.

4. Drying of devices using supercritical CO$_2$.

Test platforms and test coupons were fabricated using custom processes that were developed using the NIST-Boulder Quantum Fabrication Facility.
Generalized Universal Test Platform (UTP) Fabrication Process:

1. Start with SOI (system-on-insulator) wafer
2. Deposit metal with evaporator and pattern with lift-off
3. Etch device layer with DRIE (deep reactive ion etching)
4. Etch deep well with RIE (reactive ion etching) to clear the SiO$_2$, then DRIE for removal of $\sim 500 \mu m$ of Si all the way through the SOI handle wafer layer
5. HF etch and CO$_2$ drying to release moving parts
6. Packaging and wirebonding to create electrical connections to UTP chip

Generalized test coupon fabrication process:

1. Start with double-side polished Si wafer
2. Grow thermal oxide, 1 $\mu m$
3. Pattern and etch (RIE) oxide layer
4. Pattern and etch (DRIE) alignment holes all the way through the wafer
5. Deposit metal with evaporator and pattern with lift-off
6. Use DRIE to pattern silicon devices in frontside
7. Etch deep wells on backside to define coupon chips and support frame
8. Separate from backing wafer used in DRIE by soaking in solvent, clean off excessive photoresist using O$_2$ asher.

Full descriptions of the UTP and coupon fabrication processes may be found in Appendix A. Table 4.1 provides a summary of the fabrication data from the most recent, most successful
fabrication runs for the UTP and for the test coupon. Fig. 4.1 provides examples of the fabricated coupon designs, with Fig. 4.2 showing the passive compliant structures implemented in these designs. An automated stepper was used for patterning of the metal and device layers in the UTP chips; Fig. 4.3 shows the pattern that the stepper repeated over the UTP wafer frontside surface. The full wafer scale patterns for the test coupon masks and for the UTP devices may be seen in Fig. 4.4. For patterning the deep etch step for the UTP fabrication process, use of a contact aligner was required because the photoresist was too thick for use in the stepper. The alignment mark pattern in Fig. 4.5 was used to align the deep etch pattern to structures formed in the UTP wafer device layer. An example of a fabricated UTP chip may be found in Fig. 4.6.

The UTP and coupon processes here should be considered as specific to the fabrication facility at NIST-Boulder. Use of a different fabrication facility may entail significant changes in many aspects of these processes:

**Photoresist:** Selection and processing of photoresists is dependent upon what is available and allowed in a given facility.

**Oxygen Plasma Cleaning and Ashing:** Some facilities (for example, the facilities at ETH) do not allow exposed metal surfaces in $O_2$ asher. A dry oxidation process is very necessary for removal of photoresists after deep etching.

**DRIE Restrictions:** At NIST, metal hard masks are not allowed for deep etching. At other laboratories, this approach is allowed as a means of protecting devices fabricated with bulk machining in a Bosch Process/DRIE system.

**Supercritical Drying:** Laboratories range in restriction of the types of samples that may be placed in $CO_2$ supercritical dryers. Devices that have been subjected to deep etching processes for long durations may shed some particles. This is not acceptable in some locations.
Table 4.1: Yield of fabrication processes. Note that more test coupon chips could fit on a wafer but the fabricated designs reserved several regions for radio frequency calibration line patterns.

<table>
<thead>
<tr>
<th></th>
<th>UTP</th>
<th>Test Coupons</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total number of devices per wafer</td>
<td>$147^a$</td>
<td>384</td>
</tr>
<tr>
<td>Number surviving fabrication</td>
<td>19</td>
<td>$&gt; 70$</td>
</tr>
<tr>
<td>Number surviving fabrication, HF release, and CO₂ drying</td>
<td>8</td>
<td></td>
</tr>
<tr>
<td>Overall yield</td>
<td>5.4%</td>
<td>$&gt; 18.2%$</td>
</tr>
<tr>
<td>Wafer type</td>
<td>SOI, 20 μm device layer</td>
<td>DSP Si</td>
</tr>
<tr>
<td>Wafer cost</td>
<td>$180 - 200$</td>
<td>$20$</td>
</tr>
<tr>
<td>Si material cost per functional device (excluding other processing costs)</td>
<td>$25$</td>
<td>$0.29$</td>
</tr>
</tbody>
</table>

^a UTP designs per instance: 7 (plus 1 test array of actuator probes). Number of instances on wafer: 21. Total number of device chips (including probe array chip and UTP chips): 168.

Figure 4.1: Images of test coupons as fabricated in Si frames before separation of individual coupons. Coupons are approximately $2 \text{ mm} \times 2 \text{ mm}$ in size and are secured in place with tabs of Si that connect to the frames. The holes at cross-points of the frame are used for alignment between the front and back sides.

Figure 4.2: Images of suspended compliant mechanisms of some of the different test coupons. These optical microscope images make clear the different layers on the frontside of the coupons. Bright pale yellow is Au, the red layer is SiO₂, the grey layer is Si.
Figure 4.3: The 11 mm × 11 mm design which was repeated 21 times across the SOI wafer using the automated stepper. Multiple UTP designs and layouts were included, as well as an array of displacement amplification designs (lower left).
Figure 4.4: Images of wafer-level masks for test coupons (left) and for UTP devices (right).
Figure 4.5: The alignment mark pattern which was used to align the mask for the deep well pattern to the structures created in the UTP device layer. The deep well patterning step required the use of a contact aligner due to the thickness of the photoresist required. In this pattern, the red color indicates the location of a structure standing in the UTP device layer, and the width of each of the large red lines crossing at the center is 10 \( \mu \text{m} \). Gray areas indicate open regions of the UTP deep well mask.
Figure 4.6: Example of fabricated 2.5 mm × 4 mm UTP chip. Chip thickness is about 500 – 530 µm.
4.2 Process Development

Fabrication process development was a key step limiting production of test platform and coupon systems. As with most process development, these fabrication processes required significant iteration to work through the attendant challenges. Perhaps the most significant pitfalls were those related to photoresist chemistry, deep reactive ion etching, and device handling, all described in the subsections below. Description of additional minor fabrication issues and their resolutions may be found in Tables 4.2 and 4.3.

4.2.1 Photoresist

For fabrication at NIST-Boulder,\(^1\) negative tone photoresist is not allowed. In order to pattern the microprobe with a high degree of precision, it was necessary to pattern it in the SOI wafer device layer using the ASML stepper [2] at NIST before fabrication of the deep well for coupon positioning. Because of this, the device layer structures required protection during the deep well machining in order to prevent the deep well etching plasma from destroying the device layer structures. Use of a metal hard mask is not allowed at NIST, so a thick positive photoresist recipe was developed.

The chief pitfalls in working with positive photoresist derive from issues of diffusion within the photoresist layer. Most common positive photoresists are phenol-formaldehyde resins with a DNQ (diazonaphthaquinone) photo-sensitive dye and solvents that adjust the viscosity of the resin. After spinning a photoresist onto a fabrication wafer, or using some other form of deposition, the photoresist is cured or “baked” on a hot plate. This sets up the thermoset polymer and drives out some of the embedded solvents. Baking too rapidly can crack the photoresist or create embedded bubbles due to too much thermal stress or too rapid solvent diffusion. Baking also drives out H\(_2\)O, and time must be allowed for water to diffuse back into the photoresist layer. More time must be allowed for thick photoresist layers than for thin photoresist layers, and higher atmospheric

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\(^1\) National Institute of Standards and Technology
humidity should allow faster photoresist rehydration than dry ambients. [80]

The presence of water in the photoresist layer is essential for patterning of the photoresist. During exposure, the water reacts with the DNQ to break down the DNQ and release N₂ gas, which then diffuses out of the photoresist layer. [112] Because of the N₂ evolution during photoresist exposure, exposure steps must be carefully performed so that N₂ is not generated faster than it can diffuse out of the photoresist. If this condition is not met, bubbles can form that damage the photoresist pattern. Because most contact aligners have a fixed power output, the exposure of the photoresist can be moderated by cycling the exposure several times and adjusting the duration of exposure and rest time during each cycle. For thick photoresist layers, a long total exposure time is needed because the dye at the bottom portion of the layer is not fully exposed until the dye in upper portions has been bleached to oblivion.

After the photoresist has been exposed, it is developed by immersion in a hydroxide solution which attacks the thermoset. The DNQ is an inhibitor to this reaction, therefore exposed areas are removed because they no longer contain DNQ, whereas unexposed areas remain in place. [112] If a thick positive photoresist layer fails to fully develop, it usually indicates insufficient UV exposure or insufficient hydration of the photoresist layer before exposure.

4.2.2 Deep Etching and Device Handling

To etch through the handle wafer with DRIE requires 2 to 4 hours of processing time. The reactive etching plasma can during this time attack photoresist layers at about 1/100th the rate at which it attacks exposed Si. This can occasionally lead to breaching of the photoresist layer and destruction of underlying Si devices, and such a process was typically biggest inhibitor to UTP device yield. At ETH, such problems are prevented by evaporating several nm of Al onto devices as a hard mask before ICP (Inductively-Coupled Plasma)/DRIE processing. The Al oxidizes to Al₂O₃ instantaneously upon exposure to air. Such a process is not approved for use at NIST without photoresist masking, but further UTP processing could use both the Al and photoresist protective layers to prevent Si device layer damage.
Deep etch processes also have a tendency to increase polymerization and bonding of the photoresist layers, making them difficult to remove after processing has finished. Soaking devices for several days in standard clean room solvents like acetone, methanol, isopropanol, or PG Remover was insufficient to remove this recalcitrant photoresist. I found that a long exposure to O₂ plasma in a plasma ash (more than 10 minutes, at 50 W) can help to remove intransigent photoresist.

HF release of devices on SOI wafers must be carefully timed. Regions of device-layer silicon used to anchor freely moving parts are held in place by the embedded silicon dioxide layer, so overetching in HF will result in loss of these anchoring points. For the UTP devices, 6.5 minutes of immersion in concentrated (49%) aqueous hydrofluoric acid was usually sufficient for release of moving parts while preserving the anchor points. For devices that had an embedded oxide layer of 0.5 microns, this was generally insufficient sacrificial layer thickness to allow for the free suspension of the UTP probe. After fabrication of the displacement amplification structure, it was found that a 7 µm device layer with 0.5 µm oxide layer was insufficient to prevent stiction of the probe to the substrate, but with a 20 µm device layer with a 1 µm oxide layer stiction was not usually a problem. Even with CO₂ supercritical drying, vibrations induced in handling were sufficient to lead to pulldown and stiction of the probe. Use of 1 micron and 4 micron sacrificial oxide thicknesses was generally more effective in creating freely moving devices.

Peeling of deposited metal layers was another processing risk in the UTP and test coupon fabrication processes. In particular, the HF release step can significantly weaken or damage metal layers. In general, use of a 15 nm Ti adhesion layer rather than a 5 nm layer seemed to improve metal adhesion. Gold layers were sufficiently thick with 200 nm thickness. Aluminum should be avoided in any HF processes due to near-complete etching and removal of aluminum in HF.

As seen in Figures 4.7 and 4.8, overetch of beams and springs is a major risk in DRIE processing for MEMS fabrication. This risk can be mitigated by making designs as laterally thick as possible. One way this was performed in the test coupon designs was to replace springs 3 microns wide with folded springs designed to be 6 microns wide. Although the individual beam elements were then 8 times stiffer than the narrow beams, the increase in stiffness was mitigated by reducing
the overall number of attachment springs (from 8 springs to 4), and then by assembling beams in
series, making a folded spring pattern. In this folded, serpentine spring pattern, the overall spring
stiffness is equal to the stiffness of each component beam divided by the number of beams linked
in the spring. For instance, a folded spring with 5 flexures attached in a serpentine manner has
overall $k$ 5 times lower than the $k$ of one individual flexure. Although this design is more intricate,
each micron added in the beam thickness makes the design more robust and likely to survive the
device deep etch process.

All bulk micromachining deep etch processes require that the wafer being processed be sup-
ported on a backing wafer. In general, this was done with backing wafers comprised of sapphire or of
heavily oxidized (1 - 2 $\mu$m SiO$_2$ thickness) silicon wafers. The wafer being processed was supported
on the backing wafer with Crystalbond 509 wax, which melts at temperatures around 115°C, and
which can be removed in acetone. Because of a large amount of fenestration in the coupon wafer,
the acetone easily removed the backing wax from the test coupon wafer. The test coupon wafer is
easily separated from the backing wafer after one or two hours of soaking in acetone. In contrast,
penetration of the acetone under the much larger UTP chips usually required soaking at least an
order of magnitude longer (i.e., overnight).

A notable feature of the UTP processing is that fabrication only occurs on the frontside of the
processed wafers. Although this required the development of a thick positive photoresist process,
confining processing of SOI wafers to only frontside processing provided some helpful advantages
over processing including backside processing.

Frontside and backside processing was originally considered for the UTP fabrication process,
but backside processing presented significant problems. First, aligning to the frontside while being
supported on a backing wafer was not a simple task, especially given that precise alignment was
required in order to place the UTP probe at the edge of the deep well regions. Secondly, if the
frontside was processed first, it was not easy to sufficiently protect from the deep etch plasma, and
furthermore it was easily damaged in the backing wafer attachment and detachment processes. If
the frontside was processed after the backside, gas bubbles would be trapped in the deep wells
which could damage the UTP devices during front side deep etching, which exposed large silicon oxide membrane areas in the deep etcher low vacuum environment. Additionally, the automated stepper machine at NIST could not be used for patterning of the device and metal layers because wafers supported by backing wafers are thicker than the range allowed in the stepper. In contrast, fabrication from the frontside only allowed straightforward alignment between the device layer and the deep well structures, and handling of the wax backing was minimized.

In order to minimize processing times, deep etching was employed to preempt the need for any dicing. By designing deep wells surrounding the UTP chips, etching completely through the UTP chips separated all the chips from each other. For the test coupons a support frame was designed to surround each coupon in order to individually separate each test coupon and to keep them organized within the wafer even after all processing was completed. Si tabs were used to allow the coupons to remain in the device layer to attach the coupon to the support frame, and pressing on the coupon with tweezers allowed removal from the fabrication wafer.

The coupon designs rely on tabs that protrude from the bulk part of the coupon over the deep well areas that define the bulk parts of the coupon. Because of this, backside processing was required in order to create these suspended tabs. The coupon wafer was supported on a backing wafer during the deep etching steps, requiring development of a strategy to align the two sides. By generating holes all the way through the wafer to use to orient the backside pattern, alignment to precision within ±15 or 10 µm was achieved. Initially, SOI wafers were used to create the test coupon structures, but solid Si wafers were ultimately used because they required no HF release, allowed fewer processing steps, and cost an order of magnitude less, allowing the test coupons to be created as inexpensive consumable chips for materials testing. (Table 4.1) For the use of solid Si wafers, the selection of double side polished wafers is important because the surface roughness of the backside propagates through the deep etch wells. If the unpolished backside of a single side polished wafer is subjected to deep etching, the rough surface will eventually damage the device layer compliant structures before the deep wells are finished, reducing the overall yield of usable test coupons.
Figure 4.7: One of the major pitfalls in deep etch fabrication on SOI wafers is that running the deep etch process too long will undercut desired patterns. Here, the plasma ions charged the oxide layer, creating a lateral repulsion that undercut some of the thinner Si device layer beams.

Figure 4.8: Cross-section of one of the narrower beams in a typical displacement amplification mechanism on a fabricated UTP device. The beam remains just barely attached to the underlying SOI silicon oxide layer. Image b.) shows a magnified view of the attachment point seen at the bottom of the beam in image a.). The cross-section was generated using a gallium focused ion beam.
Table 4.2: Issues in UTP Fabrication.

<table>
<thead>
<tr>
<th>Problem Encountered</th>
<th>Method Solved</th>
</tr>
</thead>
<tbody>
<tr>
<td>Device stiction after release</td>
<td>Use 1 (\mu)m oxide layer and 20 (\mu)m device layer rather than 0.5 (\mu)m oxide and 7 (\mu)m device layer</td>
</tr>
<tr>
<td>Loss of Al metallization during HF etch</td>
<td>Better surface cleaning using (O_2) asher before</td>
</tr>
<tr>
<td></td>
<td>Use Au, Ag, or Cu as metal for pads</td>
</tr>
<tr>
<td>Alignment marks on wafer are obstructed by masks</td>
<td>Iteration of UTP designs</td>
</tr>
<tr>
<td></td>
<td>Use stepper for alignment of metal and device layers</td>
</tr>
<tr>
<td>Photoresist edge bead near deep well</td>
<td>Pattern device layer before deep well rather than after</td>
</tr>
<tr>
<td></td>
<td>Use (SiO_2) deposited with PECVD or other conformal coating to protect the device layer while etching the deep well</td>
</tr>
<tr>
<td>Photoresist degrades or does make correct pattern</td>
<td>Careful adherence to processing steps for different types of photoresists</td>
</tr>
<tr>
<td></td>
<td>Checking developing time when working with new photoresist</td>
</tr>
<tr>
<td></td>
<td>Photoresist recipe development as described above</td>
</tr>
<tr>
<td>Artifacts appear in mask patterns</td>
<td>Iteration of UTP designs to minimize acute angles</td>
</tr>
<tr>
<td></td>
<td>Modification of parameters for conversion of mask file to file for operation of pattern generator at NIST</td>
</tr>
<tr>
<td>Photoresist will not be removed</td>
<td>Remove photoresist before heating chips to 115°C (required to remove chips from wax bonding to sapphire wafer used in DRIE processing)</td>
</tr>
<tr>
<td></td>
<td>Minimize time photoresist is exposed to 115°C temperatures</td>
</tr>
<tr>
<td></td>
<td>Frequent use of (O_2) RIE and (O_2) asher to remove organic “scum” after cleaning with acetone</td>
</tr>
</tbody>
</table>
Table 4.3: Additional issues in test coupon fabrication.

<table>
<thead>
<tr>
<th>Problem Encountered</th>
<th>Method Solved</th>
</tr>
</thead>
<tbody>
<tr>
<td>Misalignment between frontside and backside patterns</td>
<td>Create an array of alignment holes that passes all the way through the wafer, serving as an alignment reference on both sides</td>
</tr>
<tr>
<td>Failure of SiO$_2$ membranes surrounding coupons</td>
<td>Eliminate embedded SiO$_2$ by using standard Si rather than SOI wafer</td>
</tr>
<tr>
<td></td>
<td>Monitor deep etch endpoint to create suspended structures</td>
</tr>
<tr>
<td>Large numbers of coupons require much time to handle</td>
<td>Stabilize coupons with connections to surrounding frames in handle wafer</td>
</tr>
<tr>
<td></td>
<td>After DRIE, use solvents to remove backing wax, and slide coupon wafer off of backing wafer without pulling upwards/vertically</td>
</tr>
<tr>
<td>Coupons too small to handle</td>
<td>Worked with chips of different sizes (in mm): $1 \times 1$, $1 \times 2$, $2 \times 2$</td>
</tr>
<tr>
<td></td>
<td>The 2 $\times$ 2 chips were found to be about the minimum that could be easily handled.</td>
</tr>
<tr>
<td>Dielectric layer and metal layers are needed for electrical experiments with coupons</td>
<td>Before patterning, grow SiO$_2$ on wafers that will require metallization</td>
</tr>
<tr>
<td></td>
<td>Evaporate and lift-off Ti and Au for metal patterns</td>
</tr>
</tbody>
</table>
Chapter 5

Device Characterization

This chapter describes testing of the key ideas incorporated within the Universal Test Platform and test coupon designs. Operational and mechanical performance of the integrated testers is reported in Chapter 6, Section 6.2, and improved upon in Chapter 6, Section 6.3. Advancement of the fabrication processes through the challenges discussed in the previous chapter was required to create devices that fully implemented UTP probe designs with three-dimensional alignment between the UTP chip and the removable test coupon chips. Upon successful fabrication of the Universal Test Platform and test coupon designs, joint operation of these structures was demonstrated using a probe station.

5.1 Probes and Actuators in UTP Designs

Multiple probe and displacement amplification structures were designed, and only a few of these were implemented on the UTP chips. Arrays of test designs were fabricated in addition to the UTP designs (Fig. 5.1). The thermal actuator designs (Fig. 5.2) driving the UTP probe motion were not significantly different from design to design, and were easily operated in a laboratory environment. On the test probe arrays, three different displacement amplification mechanisms were constructed, all of these worked to transmit motion to the probe ends. Fig. 5.3 shows an example of motion in part of one of these designs.

A more significant design variation was the inclusion of extra flexure designs meant to generate displacements indicative of force and displacement applied by the UTP probe, rather than
displacement alone. These more complicated designs can be seen in the upper right of Fig. 5.1, versus the simpler designs in the lower left of that figure. In general, the simplified designs were easily actuated and demonstrated the capabilities for very large (> 30 µm) displacements. The more elaborate designs tended to easily pull-down to the fabrication substrate, resisting any motion due to stiction over a large area. Furthermore, even if the designs were not pulled down, the UTP probe output was insufficient to move these designs much more than about 15 or 20 µm.

Figure 5.5 shows a portion of one test actuator and probe system. The compliant transmission design demonstrates that actuator motion of less than 5 µm creates probe motion output that can be greater than 40 µm (Fig. 5.5).

Figure 5.1: Fabricated array of varying displacement amplification test structures. This image shows a fabricated version of the design seen in Fig. 3.11.
Figure 5.2: Example of fabricated thermal actuator on test chip of probe designs.

Figure 5.3: Overlaid images of UTP actuator and displacement amplification at different points of operation. In the left portion of this image, the displacement amplification structure compliant joints change location, demonstrating actuated motion.
Figure 5.4: Example of fabricated displacement amplification mechanism in UTP device layer.
Figure 5.5: Demonstration of displacement amplification operation using test chip of probe designs. a.) Before actuation. b.) Probe has moved over 40 microns as indicated by vernier. The pitch of the vernier teeth is 10 µm.
5.2 Passive Three-Dimensional Alignment Between Two Microfabricated Chips

Resolution of the fabrication issues described earlier led to a good demonstration of the non-permanent interfacing between coupon and UTP chips. Test coupon chips were placed into the test platform well using tweezers, and they were easily removed either using tweezers or by inverting the UTP chip and letting gravity pull out the coupon chip.

Figures 5.6 and 5.7 provide two examples of test coupon chips successfully aligned within two different UTP designs. Out of plane alignment (Z direction) is achieved by the coupon alignment tabs resting on the UTP chips handle layer. In-plane (X, Y, and $\theta$) alignment is achieved by interaction of the coupon alignment tabs with structures patterned in the UTP device layer.

Using tweezers or a micromanipulator probe, it was possible to slide the coupon around within the well, but lightly tapping on the surface supporting the UTP chip, sending transient vibrations through the two chips, proved to be a sufficient means of manipulating the coupon within the UTP. Although the interaction of the bulk portion of the test coupons with the UTP wells was sufficient for general alignment of the two chips, frequently upon dropping the coupon into place in the UTP chip, further manipulation was required to obtain adequate self-alignment between the two chips. The tabs protruding from the coupon chips can come to rest upon device layer structures in the UTP, but with the light tapping mentioned above, the coupon will reposition itself so that the tabs rest upon the handle layer of the UTP, and the device layer structures provide a general X, Y, and $\theta$ alignment for the coupon.

Care must be taken when placing the coupon within the UTP, because excessive misalignment between the two structures will result in a canted placement of the coupon, with the coupon alignment tabs stuck somewhere down in the UTP deep well. In this case the coupon must be completely removed and replaced. Probably the best strategy for placing the coupon using tweezers is to grasp the coupon with the tweezers, place the tips of the tweezers at the bottom of the UTP well, and release the tweezers letting the coupon drop into place on the UTP chip. For the UTP
chips that are only 2.5 mm wide (Fig. 5.7) this strategy is not possible because the test platform chip does not allow for sufficient room for the tips of tweezers to pass through the chip. In this case, greater care is required in placement.

Figure 5.6: Passive interfacing of a test coupon chip in a 4 mm × 4.5 mm UTP chip. The inset images show points of XY passive alignment.
Figure 5.7: Passive interfacing of a test coupon chip in a 2.5 mm × 4 mm UTP chip. The images around the periphery show some of the points of XY passive alignment.
Active Interfacing and Coupon Actuation

Active alignment of the test coupons within the Universal Test Platform for actuation of the coupons did not operate as initially planned. As is seen in Figures 5.6 and 5.7, on either side of the probe on the UTP chips are actuated structures intended to actively clamp and align the test coupons. In practice, these were not needed. These alignment probes served as passive alignment structures in initial alignment of the test coupons. When working with coupons without test specimens, or with rigid test specimens like GaN nanowires, alignment for actuation of the coupon was attained by the interaction of the UTP probe and the passive stops at the opposing side of the coupon (see Fig. 5.7, for example). The probe pushed the whole coupon towards the stops, and then the interaction of the alignment tab and the stops led to alignment of the coupon with the axis of motion of the probe. Test coupons supporting CNT specimens were examined in a transmission electron microscope, in which case silver paint typically used in electron microscopy was used to bond the test coupon to the UTP, regardless of the relative positions of the backstops and passive alignment structures.. The active alignment probes may yet be useful in cases where delicate specimens such as carbon nanotubes need to be manipulated, and when the permanent bond attained using the silver paint must be avoided.

In Fig. 5.8 micromanipulator probes in a probe station can be seen in contact with a UTP system, which was used to demonstrate the active interfacing between the UTP and different test coupons. Figures 5.9, 5.10, and 5.11 show fabricated versions of three different test coupon designs. The designs seen in Figures 5.9 and 5.11 were successfully actuated by the UTP probe.

Figures 5.12 and 5.13 show interfacing and actuation of the first coupon design (Fig. 5.9). The compliant mechanism in Fig. 5.9 provides motion to a tensile stage via a rigid member that directly links the UTP probe motion to the specimen stage motion. Figure 5.14 shows motion at the test stage of one of the coupons of the design shown in Fig. 5.9.

Interfacing between the UTP probe and the third test coupon design (Fig. 5.11) is seen in Fig. 5.15. The compliant mechanism seen in Fig. 5.11 achieves reversal of actuation motion,
meaning that the tensile stage region moves in the opposite direction from the region which is impacted by the UTP probe. The tensile stage motion from this structure is significantly smaller than the stage motion achieved with the first design.

Figure 5.8: Demonstration of UTP probe actuation of test coupon. Micromanipulator probes in the upper right of the image supply electrical current to the thermal actuator on this UTP chip.
Figure 5.9: Magnification series of the first test coupon design. The compliant mechanism in this design was most effective for mechanical operation when interfaced to the UTP.

Figure 5.10: Magnification series of the second test coupon design. The compliant mechanism in this design was not effective for mechanical operation when interfaced to the UTP.
Figure 5.11: Magnification series of the third test coupon design. The compliant mechanism in this design was not used for tensile tests, but it did demonstrate reversing motion. The motion of the test stage was in the opposite direction of the motion applied with the UTP probe.

Figure 5.12: Demonstration of UTP probe actuation of test coupon. a.) Before actuation. b.)UTP probe in contact with coupon. c.)UTP probe has actuated coupon.
Figure 5.13: Close-up view of UTP probe actuation of test coupon. a.) UTP probe in contact with coupon. b.) Coupon compliant mechanism has been displaced about 2 or 3 microns to the left. Scale is indicated by the vernier teeth on the probe; the pitch of the teeth is $10 \, \mu\text{m}$. 
Figure 5.14: Coupon design 1 tensile stage. a.) Before operation, gap between points $\approx 3 \mu m$. b.) After stage actuation, the stage gap is larger, with the gap between points $\approx 8 \mu m$.

Figure 5.15: UTP probe in contact with the compliant mechanism from the third test coupon design. For size reference, the nearly-square suspended region at the image center is about 100 $\mu m$ wide.
Chapter 6

Experimentation

This chapter describes the experimental procedures and results obtained with the microsystems developed for this thesis. Sections 6.2 and 6.3 are reproductions of published papers. All remaining sections are as yet unpublished. The work in this chapter demonstrates the application of microelectromechanical systems in the characterization of various types of material specimens with nanoscale cross-sections.

6.1 Specimen Preparation

Specimen preparation in the work described below was performed in collaborations. Dr. Gurpreet Singh (currently at Kansas State University) has helped me with using micromanipulators within SEM environments to place fibers with CNT cores onto tensile test stages. [10, 11] Alicia Baca has placed all of the nanowire specimens I have used in tensile tests using a dielectrophoretic method. [10, 11] Her work was part of a Master’s thesis completed in May 2010 on the subject of electrode structures to optimize dielectrophoretic nanowire placement. [3] Matthias Muoth at ETHZ grew carbon nanotubes on the test coupon structures developed in this thesis.

Until July 2008, electron-beam induced deposition (EBID) of carbon from the ambient SEM environment was used to clamp nanomaterial specimens to test structures. This is performed simply by placing the SEM in spot mode for several minutes. After that point in time a dual beam SEM/Focused Ion Beam (FIB) system became available at the University of Colorado Nanomaterials Characterization Facility, which allowed deposition of platinum-carbon clamps using Ion Beam
Induced Deposition (IBID), in which gallium ions dissociate a platinum organometallic gas.

After the clamp failure seen in Section 6.3, Fig. 6.17, the clamping procedure for GaN NWs was refined to include the following steps:

(1) Scan ion beam across all bonding surfaces at low beam current in order to remove surface contaminants.

(2) Use low beam current to deposit a thin Pt layer on all bonding surfaces.

(3) Use higher beam currents to build up Pt near NWs.

(4) Deposit successive thick layers of Pt to blancket the clamping regions and provide good mechanical reinforcement.

The best example of the result of this procedure may be seen in Fig. 6.1. Unfortunately, the specimen seen in this image was too large for good data to be acquired in the force ranges that could be obtained using integrated testers.

Figure 6.1: Example of good clamping created using Pt deposited using FIB IBID on a GaN NW specimen resting on a polysilicon MEMS mechanical test stage.
6.2 Microsystem for Nanofiber Electromechanical Measurements

The work in this section has been published in references [12, 13] and was performed in collaboration with J. W. Suk, G. Singh, A. Baca, D. Dikin, R. Ruoff, & V. Bright.

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.

6.2.1 Introduction

Electromechanical measurements on nanoscale fibers such as nanotubes and nanowires are of interest to enable integration of these materials into sensors and other microdevices. [65, 135] Uniaxial loading is desirable in mechanical testing to ensure uniform loading throughout a fiber specimen [161, 162], and to this end several microdevices have been developed to perform uniaxial mechanical testing on a nanofiber. [63, 64, 69, 86, 87, 154, 167] Furthermore, electrical coupling to a specimen can allow piezoresistive and electrothermomechanical characterization. The novelty of the device presented here lies in the ability to perform electrical measurement of an electrically conducting or semiconducting nanoscale fiber specimen under mechanical loading. This work also explores the use of alternating current (AC) electrical measurements as a novel means to bypass contact resistance in two-point electrical measurements of a fiber specimen mounted on a microsystem. This paper is an expansion of a paper presented at the 2008 Solid State Sensors, Actuators, and Microsystems Workshop in Hilton Head, SC, USA. [13] For mechanical tester microsystems
such as those in references [63, 64, 69, 86, 87, 154, 167], measurement of the forces generated with the system remains a challenge. The current paper builds on ref. [13] by including a demonstration of a new force calibration approach based on the discrepancy between expected and measured displacements.

6.2.2 Design and Simulation

The MEMS tensile test system developed here consists of a moving stage and a fixed stage, as seen in Figure 6.2. These components are built from released polysilicon using the PolyMUMPS prototyping service. [108] The fixed stage is anchored to a silicon nitride substrate, and the moving stage is connected by a shuttle to a thermal actuator, and it is stabilized to uniaxial motion by a set of opposing beams which are anchored at their ends to the nitride substrate.

Figure 6.2: Left: Diagram of actuator and stage system. The moving stage at left is linked by bending beams to rectangular anchor points. A thermal actuator pulls the moving stage away from the fixed stage, at right. Center: SEM image of overall system. Right: Close-up of stage showing mounted carbon nanofiber, 120 nm diameter, before loading.
6.2.2.1 Mechanical Design

Stage actuation is realized by thermal expansion through Joule heating of a set of angled beams, as has been previously explored in [76, 155, 166]. Beams symmetrically connected to the stage ensure uniaxial motion and also serve as heat sinks. Using the actuator geometry and a plane strain condition in a solid mechanics mathematical analysis, the thermal actuator was estimated to provide up to 400 $\mu$N force output. The plane strain condition approximates the case where the stage is constrained not to move even as the beams in the actuator are forced to thermally expand due to Joule heating.

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 6.1, the structure in Figure 6.3, 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.
Table 6.1: Polysilicon material properties used as simulation inputs. [94, 104, 128]

<table>
<thead>
<tr>
<th>Material Properties of Polysilicon</th>
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<tbody>
<tr>
<td>Thickness</td>
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<tr>
<td>Electrical Resistivity</td>
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<td>Thermal Conductivity</td>
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<td>Poisson Ratio</td>
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<td>Coefficient of Thermal Expansion</td>
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<td>Yield Strength</td>
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<td>Density</td>
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Figure 6.3: Layout for simulation. The moving stage is the pointed part at the right of the device, the thermal actuator is at the center, and the springs and large grating at the left were added to test several indirect means of displacement measurement.
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 Figure 6.4.

At fixed voltages, with varying displacement constraints, the moving stage exhibits a linear reaction force versus displacement. Force simulation as shown in Figure 6.4 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 Figure 6.5.
Figure 6.4: Data and linear regression fit lines for three test platforms, used to derive values for $B$, the proportionality constant between input power and unconstrained displacement. Also plotted in this figure is data from a simulation of displacement versus input voltage squared, indicating a linear relation for the simulated system with constant thermal and electrical conductivities. The simulated data has been scaled by a factor of 2 to fit on the plot of experimental data, because the simulated data does not include losses due to the polysilicon connections between the contact pads and the actuator on the chip.
Figure 6.5: Simulation output of the reaction force at the tip of the stage for different values of actuator voltage and constraints on the tip displacement.
6.2.3 Thermal Design

In order to minimize the specimen temperature changes caused by the thermal actuator in [86, 87], the moving stage was separated from the actuator and mechanically anchored. For example, at 15 V the simulation predicts a stage temperature of 229°C, as seen in Figure 6.6. This is significantly lower than the actuator temperature of 437°C, but it demonstrates a need for further optimization of the thermal design.

![Figure 6.6: Thermal simulation results from ANSYS simulation. Plot of maximum temperature change, which is located in the actuators, and the temperature change of the tip of the moving stage at various voltage inputs to the actuator.](image)
6.2.3.1 Mechanical Measurements

Tensile measurement is enabled by fixing a material specimen across the gap between the moving and fixed portions of the stage. The experiment reported here uses scanning electron microscope (SEM) observation as a direct means of measuring the stage displacement and carbon nanofiber strain. Microscopy requires the interpretation of micrographs in order to derive strain data, which can be a slow process. Sensors could provide a faster approach to acquisition of strain data, and to that end, several indirect strain measurement mechanisms were built into the reported device. These mechanisms can be seen at left in Figure 6.3, and as built in the center of Figure 6.2. 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 [151], 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 [167]. This direct force measurement has not yet been implemented in our design.

6.2.3.2 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 Figure 6.5 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.

$$F = k(d_0 - d)$$  \hspace{1cm} (6.1)

The simulation in Figure 6.5 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 [155], 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 $P$. ($P = IV$, the current flowing through the actuator times the voltage drop across the actuator.) As seen in Figure 6.4, measurements of displacement $d_0$ versus power $P$ on unconstrained stages give linear fits according to the following equation:

$$BP = d_0$$  \hspace{1cm} (6.2)

From these measurements, the linear regression fitting constant $B$ lies in the range of 0.0024 to 0.0034 ($\mu m/mW$), with $R^2$ (coefficient of determination) values ranging from 0.82 to 0.97. For the data presented in Figure 6.4, 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.

6.2.3.3 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 3 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 megaohms.

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. (Figure 6.7) 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 $R_s$ 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 $R_s$ has a value on the order of kilo-ohms, so $R_s$, $R_1$, 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.

6.2.3.4 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.
Figure 6.7: Circuit diagram for a fiber specimen with amorphous carbon contacts. Each contact has contact resistance $R_1$ and $R_2$, and contact capacitance $C_1$ and $C_2$. For fiber piezoresistive measurements, the resistance of the fiber $R_s$ must be resolved.
6.2.4 Experiment

The microdevice was fabricated from polysilicon with Au contacts using the PolyMUMPs service. [108] The polysilicon layers were released using a 3 minute etch in 48% HF$_{\text{aq}}$ followed by supercritical CO$_2$ drying.

Carbon nanotube 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 hours in a toluene solution and dried to form a mat. A 3-axis piezoelectric micro-manipulator (Kleindiek$^{TM}$) 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 [132]. The nanofiber was then transferred and bonded at its ends to the MEMS tensile stage by performing electron beam induced deposition (EBID) for 10 minutes in a JEOL JSM-6480LV SEM operated in spot mode with 30 kV 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.

6.2.5 Results and Discussion

6.2.5.1 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 [152]. Furthermore, observation (Figure 6.8) of actuator electrical resistivity during the car-
bon 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 [152]. ¹ Because the actuator electrical resistance varies during an experiment, calibration based on voltage inputs alone is insufficient to predict the unconstrained displacement of the stage, which depends on the thermal resistance, power dissipation, and thermal expansion of the actuator beams.

Figure 6.8: During the experiment, the polysilicon actuator shows a linear increase in electrical resistance with increasing input power, with a small resistivity hysteresis upon reduction of the input power.

¹ In hindsight, this hysteresis may have been mostly a thermal effect similar to the behavior observed with UTP operation in a TEM (Section 6.5.1).
6.2.5.2 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 kΩ 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 Figure 6.9. As the moving stage was allowed to return to its original position, the nanofiber buckled upon itself as seen in Figure 6.10, indicating that the failure seen in Figure 6.9 was not a complete fracture. In Figures 6.11 and 6.12 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.
Figure 6.9: Tensile observation. Left: Slack removed from nanofiber before significant loading, $R = 5.01$ kΩ (measured at 100 kHz). Center: Nanofiber just before failure, $\epsilon = 5.3\%$. $R = 4.98$ kΩ. Right: Failure of an outer layer. $R = 4.98$ kΩ. Reactances ranged from $X = -54.7$ kΩ to -57.0 kΩ. No significant trends in resistance or reactance were observed for this sample during the tensile test.

Figure 6.10: Image of the nanofiber after the moving stage has returned to its original position.
Figure 6.11: Strain determined from extension of the nanofiber from its length when completely unloaded. Following from the origin, the three marked inflection points indicate (1) where the fiber had straightened, (2) a point where the specimen resistance changed spontaneously under a constant strain, and (3) the point just after failure, seen at right in Fig. 6.9, where the fiber began to be unloaded.

Figure 6.12: Estimates of stress and strain applied to the carbon nanofiber during loading and unloading. These estimates were derived from the input power applied to the test device actuator, and the measured displacement of the stage. Error bars show 6% error based on the uncertainty in the $d_0 = BP$ curve fit.
Using the force calibration approach defined above, the stage displacement and actuator input power were mapped into estimates of the force applied to the carbon nanotube. In order to calculate the engineering stress applied to the fiber, the fiber diameter was measured using high-resolution SEM imaging, as seen in Figure 6.13. For the value of $B$, the fitting parameter from Device 3, which was from the same PolyMUMPs [108] fabrication run as the device used to obtain the carbon nanofiber data in Figure 6.4, 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, $B = 0.00557 \mu m/mW$, 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.
Figure 6.13: High-resolution SEM micrograph of the failure “neck” in the carbon nanofiber. In this image it is evident that a 43 nm carbon nanotube core has pulled out from the 140 nm carbon fiber, which appears to have fractured during the mechanical testing.
Figure 6.12 shows the stress-strain response as the fiber straightened and started to take the load along its length. Because typically the outer diameter $D_o >> D_i$, the inner carbon nanotube diameter, for these nanofibers, the loading stress can be estimated by assuming a coreless geometry [132], using $D_o = 140$ nm (Figure 6.13). The initial portion of the curve shows the typical linear elastic deformation behavior observed in carbon nanotubes (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 $\sim 350$ GPa. This value falls well within the experimental values reported in the literature [162,167]. It should be noted that the CNT 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 until the outer shell eventually fractured and the inner nanotube nearly pulled out (Figure 6.13). As seen in Figure 6.10, the fiber buckled when the stage was returned to a point of zero input power, and this has manifested as a small amount of compressive stress observed in the final data point of the tensile test in Figure 6.12.

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 ($0.38 +/- 0.06 \mu$m) 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. [134] using the same carbon nanofibers and clamping techniques.

6.2.6 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.

6.2.7 Acknowledgements

This research was supported by DARPA Award # HR0011-06-1-0048 and the DARPA Focus Center for Integrated Micro/NanoMechanical Transducers (iMINT Center) at the University of Colorado at Boulder, the University of Texas at Austin, and Northwestern University. This research is based upon work supported under a National Science Foundation Graduate Research Fellowship, which supports J.J. Brown. The authors wish to thank NIST-Boulder and Paul Rice at the University of Colorado for help with the nanotubes and the micromanipulator. Additionally, the authors thank Dr. Christofer Hierold, and Dr. Alain Jungen, ETH Zurich, Micro and Nanosystems, for many fruitful discussions about subjects related to this research.
6.3 Tensile Measurement of Single Crystal Gallium Nitride Nanowires on MEMS Test Stages

The work in this section is reproduced from reference [10] and was performed in collaboration with K. Bertness, A. Baca, R. Ruoff, D. Dikin, & V. Bright. Part of this work was originally published in Ref. [11].

Direct tensile tests were performed on \( n \)-type (Si-doped) gallium nitride single crystal nanowires that were grown by nitrogen plasma-assisted molecular beam epitaxy and which are essentially free of defects and residual strain. Nanowires were integrated with actuated, active microelectromechanical (MEMS) devices using dielectrophoresis-driven self-assembly and platinum-carbon clamps created using a gallium focused ion beam. For one nanowire, failure strain of 0.042 ± 0.011 was found. Most nanowire specimens appeared to demonstrate tensile strength in the range of 4.0 ± 1.7 GPa to 7.5 ± 3.4 GPa. Failure modes included clamp failure, transverse (nanowire \( c \)-plane) fractures, and insufficient force from the MEMS test actuator.

6.3.1 Introduction

Gallium nitride is a direct wide-bandgap semiconductor with good thermal conductivity and notable optical, mechanical, piezoelectric, and transport properties. [92, 118, 121, 141]. GaN nanowire cantilever resonators have been shown to have a high mechanical quality factor, which makes them appealing in nanomechanical devices. [141] Recent developments in the synthesis of gallium nitride nanowires have illustrated that these structures can be free of defects and residual strain. [5] These findings suggest that the nanowire morphology will offer numerous applications that would otherwise be unattainable in conventional epitaxial growth of this material. Integration of this material with active MEMS structures may allow development of new classes of tunable mechanical resonators, LEDs, lasers, and switches, among other possible new sensor and transducer technologies. Development of comprehensive mechanical data on gallium nitride nanowires (GaN
will enable the realization of such new devices and applications. For instance, the ability to withstand $0.042 \pm 0.011$ tensile strain or $7.5 \pm 3.4$ GPa tensile stress, as reported here for one specimen, reflects exceptional strength and resilience for what might at first be considered a brittle material. This paper significantly expands upon the preliminary GaN tensile study given earlier. [11]

The use of microfabricated structures enables mechanical testing of materials in small quantities or for which bulk sample preparation is difficult. Additionally, the integration of nanomaterial specimens on a MEMS mechanical tester serves as a test case for chip and wafer- scale integration of microtechnologies with a nanoscale material that has specific synthesis requirements. The data produced from these experiments are a step towards the thorough characterization of defect- free, single crystal GaN. There remain large sources of uncertainty associated with this mechanical system.

6.3.2 Experiment

6.3.2.1 MEMS Test Device

The MEMS mechanical test structure (Figure 6.14) is a simplification of the one reported in references [12], consisting of a fixed stage electrically isolated from a moving stage that is laterally stabilized and actuated using a buckling beam thermal actuator. This test device was fabricated using the PolyMUMPS surface micromachining service. [108]

Stage motion is measured directly from scanning electron microscope (SEM) micrographs. The force applied by the stage can be computed using the approach described in [11–13], which is repeated here. The actual stage displacement $d$ is compared to an expected displacement $d_0$. The force applied $F$ is calculated according to Eq. 6.3 using the discrepancy between these displacements multiplied by the spring constant $k$ of the system, which is calculated as discussed below.

$$F = k(d_0 - d) \quad (6.3)$$
Figure 6.14: (Top) Microfabricated tensile test structure consisting of electrically isolated moving and fixed stages. During a tensile test the moving stage moves away from the fixed stage. Thermal actuator beams are angled 89° from the direction of motion. (Bottom) Simplified schematic of the device seen above. The thermal actuator consists of an array of angled beams, which expand as current flows through them. The actuator pulls the moving stage, which is laterally stabilized by pairs of opposing beams.
During tensile tests, the MEMS test device was wirebonded to a 20 pin ceramic dual in-line package (DIP) chip carrier, and operated within a JEOL JSM-6480LV SEM. The test devices were connected to power electronics using an electrical feedthrough. Nanowire lengths were measured directly from SEM images. The equations used to map the nanowire length, stage displacement, and actuator power measurements into tensile curves of stress and strain are listed in Table 2.2.

In order to map experimental data onto force measurements using Eq. 6.3, a linear fit between free-moving displacement $d_0$ and input power $P$ was used, $d_0 = BP$. The fit parameter $B$ was calculated from a linear regression of $d_0$ and $P$ data measured as output and input, respectively, of a freely moving test structure. Similar to prior work on other v-shaped thermal actuators, the $d_0$ vs. $P$ curve as observed here for freely moving test systems is usually linear or slightly curved. [4,38,130]

For a freely moving stage, displacement depends on thermal strain, which derives from temperature change and thermal expansion coefficients in the actuator beams. Due to the thermal resistance of the beams, the temperature profile of the beams depends upon the power dissipated within them, which can be measured by multiplying the current and voltage supplied to the actuator. Fitting to measured experimental curves allows the fit parameters to incorporate the nonuniform actuator temperature profile, the nonlinear thermal expansion and temperature-dependent changes in the electrical and thermal conductivities.

Many papers have reported investigations of v-type thermal actuators similar to the ones we have used here. [4,38,106,130,152,153,167] These investigations have shown relatively linear force vs. displacement behavior within several microns of the free-moving displacement.

Most attempts to calibrate the forces produced by a thermal actuator or other structures within a polycrystalline silicon device layer rely on the bending of a suspended beam formed from the same material and subjected to the same processing as the actuator. This approach relies on assumptions of Young’s modulus $E$ and dimensions of a beam in order to predict a spring constant $k$ for that beam. [155] Some work has been done to verify $k$ derived in this method with an alternate measurement derived from the resonance frequency of a suspended structure. [106] Better force
measurement procedures are needed to verify the forces experienced by MEMS structures and this remains an active area of research.

The simulated behavior of systems that include v-type thermal actuators indicates that, as the power into the actuator increases, the force and displacement produced by the actuator also increase, but at rates that depend on the initial constraints of the actuator. [4,155] In Ref. [155] this manifests as the “load line” trajectory of a given microactuated system. In Refs. [4] and [155], it is seen in measurements that the simulation holds mostly true when \((d_0 - d)\) is near 0, but at higher deviations from \(d_0\) the force produced is significantly less than what is specified by simulation, due to a mode of beam buckling in the \(xy\) plane.

When temperature dependence of material properties used for simulation is neglected [12], simulation shows a clear linear relationship between \(F\) and \(d\) at a given input power, with \(k = F_x/d\). This relation is easily related to predictions from beam mechanics as discussed below. For the force estimation discussed here, \(k\) is taken to be a constant value. This is the spring constant value valid for small perturbations of the spring and actuator system at \(P = 0, d_0 = 0, \) and \(F_x = 0\). At higher input power levels, \(\left|\frac{\partial F_x}{\partial d}\right|_{F_x=0}\) increases, indicating that Eq. 6.3 may systematically underestimate \(F\). [4] Because high tensile strength and high elastic modulus are generally desirable, important properties, caution guides the observer to choose methods with systematic error such that, if it cannot be eliminated, it underestimates properties reported as the result of an experimental procedure. Due to the temperature dependencies in constitutive parameters, the assumption of beam bending with a constant \(k\) underestimates the actual force outputs, subject to the variation and accuracy of \(d, d_0, \) and \(k\). In the absence of more traceable characterization of thermal actuators under varying input powers, displacements, and forces, the method used here provides a lower bound to forces and stresses derived from measurements taken with this MEMS system. Because the degree of the systematic underestimate is not known, this uncertainty is not included in the reported force, stress, and modulus values below.
6.3.2.2 Uncertainty

The uncertainties \( u(x_i) \) associated with the data presented here can be determined using similar mathematical analysis as that developed in Ref. [155], and following the guidelines described in Ref. [59]. Under this approach, uncertainties in measured data are evaluated statistically as the standard deviation of the mean of a data set (Type A evaluation) or according to a factor determined by an estimated probability distribution function for a given measurement (Type B evaluation). [59] For instance, where a bound of \( \pm a \) is given on a measurement, the uncertainty \( u \) is evaluated from \( u^2 = a^2/3 \). [59] A combined standard uncertainty \( u_c \) for a measurement with multiple sources of uncertainty is found from the square root of the sum of the squares of the component uncertainties. In measurements that are derived from other data, as \( y = f(x_i) \), the \( u_c(y) \) can be found from the square root of the sum of the squares of the partial derivative of each \( x_i \) times each \( u(x_i) \). [59, 155]

For uncertainty values reported throughout Section 6.3 as \( \pm mn \), this value \( mn \) represents the 95% confidence interval, which can be found by multiplication of the standard uncertainty \( u(x_i) \) by a coverage factor of approximately 2. In the graphs in this section, error bars show values of standard uncertainty \( u(x_i) \) for each variable with no coverage factor included.

6.3.2.3 Test Device Spring Constant

The maximum test system displacements \( d \) recorded here were about 3 \( \mu m \) and the minimum beam length was \( L_B = 98.5 \mu m \). So, \( d/L_B = 0.0304 \). This is within the regime of small displacement approximation, so conventional solid mechanics beam deflection calculations for clamped beams can be used to approximate the spring constant \( k \). In turn, \( k \) can be used to relate external force and displacement of a system of bending beams as represented by the test system in Fig. 6.14.

Significant sources of uncertainty in \( k \) include processing variations in beam dimensions and Young’s modulus, and dependence of the spring constant on the given state of the actuator. This latter uncertainty is understood from the discussion above to be at a minimum for \( P = 0, d_0 = 0, \) and \( F_x = 0 \), and will not be included in the following uncertainty estimate.
Beam etch and width variation from batch to batch was measured from SEM images of one device seen before HF release and two devices after HF release (Table 6.2). For each device, the average and standard error were determined. The error due to pixel resolution in the source image proved to be more significant as a source of error than the uncertainty between measurement values, and therefore the uncertainty due to image resolution and was included with the standard error to give a combined standard uncertainty value.

Table 6.2: Measurements obtained to estimate \( u_c \) for beam widths.

<table>
<thead>
<tr>
<th>Beam Width (( \mu m ))</th>
<th>Combined Standard Uncertainty ( u_c ) (( \mu m ))</th>
<th>Number of Measurements Averaged</th>
<th>Image Resolution (pixels/( \mu m ))</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.001</td>
<td>0.084</td>
<td>6</td>
<td>8</td>
<td>Unreleased</td>
</tr>
<tr>
<td>3.030</td>
<td>0.064</td>
<td>9</td>
<td>10</td>
<td>Released</td>
</tr>
<tr>
<td>2.93</td>
<td>0.13</td>
<td>8</td>
<td>4.75</td>
<td>Released</td>
</tr>
</tbody>
</table>

The maximum uncertainty in Table 6.2 was used as the value for in-plane dimensional uncertainty in the spring constant calculation. For the uncertainty due to thickness, a calculation based on manufacturing tolerance was used. The MEMS foundry specifies thickness of 3.50 \( \mu m \) with a tolerance of 0.25 \( \mu m \). [14] Again using a Type B [59] evaluation, 

\[
\frac{u_c^2}{3} = \left( \frac{0.25}{3} \right)^2, \text{ so } u_c = 0.14 \, \mu m.
\]

From 6 sets of measurements across 3 different chips and devices on a tilted SEM stage, a device thickness value of 3.36 \( \mu m \) with \( u = 0.08 \, \mu m \) was found. This measurement is within the foundry specification, but it does not include uncertainties due to tilt, so the foundry specification of 3.50 \( \mu m \) with \( u = 0.14 \, \mu m \) was used for the thickness in calculation of \( k \). The in-plane Young’s modulus of MUMPS polycrystalline silicon has been well characterized, and \( E = 162 \pm 14 \, \text{GPa} \), determined from beams of similar dimension [127], was used as an input to the calculation of \( k \). Assuming this value reported uncertainty according to standard practice of 95% confidence, the standard uncertainty is found by dividing by a coverage factor of 2, giving \( u(E) = 7 \, \text{GPa} \). [155]

By adding the individual spring constants computed according to the bending of each of the rectangular cross-section beams suspending the tensile test structure, it is readily verified that 

\( k = 175 \, \mu N/\mu m \). The combined standard uncertainty for the spring constant of each component
beam is then determined according to the root sum of squares of the individual uncertainties [59,155], and the total uncertainty in $k$ was found by the arithmetic addition of each of these beam uncertainty values in order to give a maximum estimate of the uncertainty in $k$. This was found to be $u(k) = 29 \, \text{N/m}$.

Finite element simulations in CoventorWare software [23] corroborated the derived value of $k$. Additionally, we have used an atomic force microscope to do a preliminary measurement of $k$ on a suspended structure with an identical array of beams and found an order of magnitude in the range specified here for $k$. (From the AFM measurement, $k = 150 \, \text{N/m}$ with unknown $u(k)$.) Traceable measurement of $k$ remains an ongoing aspect of work with MEMS test structures, but for the calculations below the calculated $k$ and its corresponding $u(k)$ were used for derivation of force measurements.

6.3.2.4 Nanowire Placement

Dielectrophoresis has been previously demonstrated [8,92] as a means of integrating nanowires to microfabricated structures in a wafer-level assembly technique. This method is less time-consuming than using micromanipulators to place individual nanowires. As seen in Figures 6.15 and 6.16, we have demonstrated that this self-assembly approach can be used to place nanowires on active MEMS devices.

The dielectrophoretic nanowire placement is performed using a modified probe station. Two electrical probes are placed in contact with electrical pads that allow an alternating (AC) electrical field to be applied across the fixed and moving stages. The stages taper to opposing points, where the nanowire placement is desired. Consequentially, there is a gradient in the amplitude of the electric field between these stages, with the maximum field at the location where the stages are closest. The nanowires polarize in the presence of an electric field, causing them to align parallel to the field lines. In the presence of the electric field gradient, nanowires are swept towards the field maximum, causing the nanowires to bridge the fixed and moving stages.
Figure 6.15: The mounted GaN NW Specimen #1, 200 ± 17 nm in diameter, was bonded to an unreleased tensile stage using Pt-C deposits formed using IBID.
Figure 6.16: (Upper left) GaN NW Specimen 2. (Upper right) GaN NW Specimen 3 as originally tested, after clamping and before an attempt to reduce the nanowire cross section. (Lower left) GaN NW Specimen 4. (Lower right) GaN NW Specimen 5 just after failure.
The c-axis oriented, Si-doped (n-type) GaN nanowires were synthesized by nitrogen plasma assisted molecular beam epitaxy as described in Ref. [5]. A nanowire-liquid-suspension (roughly 2 mL in isopropanol) was formed by brief (2 - 5 minutes) sonication of a substrate supporting grown nanowires. The suspension was dispersed by a syringe that was positioned over the MEMS tester using a probe station micromanipulator. For the dielectrophoretic nanowire placement, a voltage of 10 V peak-to-peak amplitude was applied at 60 - 75 kHz to the opposing sides of the stage. While the electric field was applied, about 0.1 µL of the nanowire suspension was dispensed onto the stage. As the solvent evaporated, the surface tension of isopropanol and dielectrophoretic electrical forces caused the positioning of one or more nanowires across the gap between the moving and fixed stages. The dielectrophoresis parameters were derived through experimentation with voltage amplitude and frequency, and the values above were found to be most effective for the given stage and nanowires. This dielectrophoretic self-assembly process functions successfully for both unreleased and released MEMS structures. Solvent evaporation did not cause released MEMS structures to adhere to the fabrication substrate above which they were suspended.

### 6.3.2.5 Nanowire Clamping

The nanowire specimens were clamped (Figure 6.15) to the MEMS test structures using Pt-C deposits formed in a FEI NOVA 600i scanning electron beam and focused ion beam microscope (SEM-FIB) by ion-beam induced deposition (IBID). This is similar to the approach taken in prior work. [100,101,143] For specimens 1 - 3, after deposition of the contacts, the test structure was released in aqueous HF and dried using supercritical CO2. For specimens 4 - 6, the test structure was released and dried first, and then the nanowires and clamps were deposited onto the devices. GaN survives HF etching without significant damage. Occasionally the IBID Pt deposition process can leave extra platinum deposited on the nanowire specimen, as seen for specimen 2 in Figure 6.16.
6.3.2.6 Measurements

Measurements of nanowire length and of stage motion were recorded from SEM images of the nanowire using ImageJ software. [58] Nanowire elongation $e$ was computed directly from measurements of a specimen gauge length $L$ defined between the clamps as observed in electron micrographs. Definition of the gauge length in this manner removes the effect of clamp deformation and specimen rotation from the recorded measurements of nanowire elongation and strain.

For specimens 1 – 3, current and voltage supplied to the MEMS actuator were measured using Hewlett-Packard 34401A multimeters or a Hewlett-Packard 3425A Universal Source. For specimens 4 – 6, the MEMS actuator was controlled by a National Instruments USB-6259 Data Acquisition System (DAQ) D/A converter feeding a signal to an op-amp current source circuit. The current passed through the actuator and a known resistor, and voltage follower circuits were used to read the voltage from nodes on each side of these loads. The voltage followers provided outputs to the A/D converter of the DAQ, which acquired and averaged 100 samples for each data point that was recorded. Current was measured from the voltage drop across the known resistor.

6.3.3 Analysis

Stress was calculated by dividing the force applied by the cross-sectional area of the nanowire. The cross section area is found by measuring the nanowire diameter $D$ from high-resolution images captured with the SEM capabilities of the FEI NOVA 600i dual beam microscope. For the calculations used in this paper, a circular cross-section of a diameter $D$ has been assumed for all nanowire specimens. In reality, these are single crystals with imperfect hexagonal cross-sections. In the estimate of axial stress $\sigma$, a perfect hexagon of edge length $D/2$ can be inscribed into a circle of diameter $D$. The area of the hexagon is 17.3% less than the area of the circle. Alternately, a circle of diameter $D$ can inscribe a hexagon with edge length $D\sqrt{3}/2$, underestimating the hexagon area by about 10%. The larger systematic uncertainty, 17.3% of a measured area, was treated as boundary, so $u_{\text{(geometry)}}^2 = (0.173A)^2/3$. This was included in the combined uncertainty for area
measurements derived from nanowire diameter measurements.

6.3.3.1 Misalignment

In order to consider the motion and forces present within the nanowire specimen when it is not perfectly aligned with the axis of stage motion, it is helpful to set up multiple coordinate systems. Motion coincident with the motion of the tensile stage can be set in the Cartesian \((x, y)\) system, with the nanowire end on the moving stage undergoing displacement \(\delta x\). For misalignment analysis, an additional coordinate system may be specified coincident with the axis of the undeformed nanowire. For two-dimensional misalignment, this is a rotated Cartesian system \((x', y')\) with the \(\hat{x}'\) direction coincident with the nanowire and the \(\hat{y}'\) direction perpendicular to the nanowire. In three dimensions a cylindrical coordinate system \((x'', \theta'', r'')\) is chosen, with the \(\hat{x}''\) direction coincident with the nanowire and the \(\hat{r}''\) direction perpendicular to the nanowire. The misalignment angle between the nanowire axis and the stage motion direction \(\hat{x}\) is defined as \(\theta\).

In two dimensions, the end of the nanowire specimen on the moving stage experiences motion \(\delta x\) in the \(\hat{x}\) direction. This may be transformed to motion in the \((x', y')\) coordinate system according to 
\[
\delta x' = \delta x \cos \theta \quad \text{and} \quad \delta y' = \delta x \sin \theta .
\]

In the case of three-dimensional misalignment, the out-of-plane misalignment distance \(\Delta Z\) must also be considered with a small modification to \(\theta\). For a specimen with actual gauge length \(L_{\text{actual}}\) and misalignment angle \(\gamma\) in the \(xy\) plane, there is an out-of-plane angle \(\beta\) such that
\[
\beta = \sin^{-1}(\Delta Z/L_{\text{actual}}) = \tan^{-1}(\Delta Z/L_0),
\]
if \(L_0\) is measured as only a distance in the \(xy\) plane. The transformation of a vector in \((x, y)\) into the three-dimensional nanowire coordinate system can be found by two successive Cartesian rotations (first about \(\hat{z}\), then about \(\hat{y}'\)) followed by conversion from Cartesian to cylindrical coordinates \((x'', \theta'', r'')\). This gives misalignment angle \(\theta\) as specified by Eq. 6.4. The extensional displacement \(\delta x'\) and the perpendicular displacement \(\delta r''\) of the nanowire end are specified by Eq. 6.5 and Eq. 6.6, respectively. For typical values such as \(\Delta Z = 500\) nm (approximately the case when one end of the nanowire rests on the silicon stage, and the other end rests on a metal pad on the silicon stage) and \(L_0 = 10\ \mu m\), \(\beta\) is found equal to
2.86°. The value $\cos \beta = 0.9988$, which is less than 0.2% modification to most calculations, so the effect of three-dimensional misalignment is not included in the uncertainty analysis below.

$$
\theta = \cos^{-1}(\cos \gamma \cos \beta) \quad (6.4)
$$

$$
\delta x'' = \delta x \cos \theta = \delta x \cos \gamma \cos \beta \quad (6.5)
$$

$$
\delta r'' = \delta x \sin \theta \quad (6.6)
$$

### 6.3.3.2 Clamp Deformation

If there is no deformation within the clamp, the motion $\delta x$ is identical to the stage displacement: $\delta x = d$. Because all forces opposing the motion of the MEMS test system are transmitted through the tensile specimen, they can be determined without the need to examine the complex stress state within the clamp in great detail. Strain is measured from nanowire gauge lengths specified between the end clamps on each nanowire specimen, therefore strain within the clamps does not contribute to the uncertainty of the strains recorded for the nanowire specimens.

### 6.3.3.3 Tensile Forces in Misaligned Specimens

Stress values in the nanowires are estimated in Table 6.3 assuming fully uniaxial loading of the nanowires. The misalignment of the nanowires in several of the tensile tests creates the likelihood that that the actual stresses in the nanowires are somewhat larger than those estimated in Table 6.3. If the ends are treated as pinned rather than clamped, allowing the stress nonuniformities created by the bending moments at the clamps to be ignored, a free body diagram can be used to estimate the tensile force $F_T$ in each nanowire. The tensile stage force $F_x$ is calculated as discussed above, using Eq. 6.3. The sum of forces in the $\hat{x}$ direction equals zero: $F_x - F_T \cos \theta = 0$, rewritten as Eq. 6.7. The difference between $F_T$ and $F_x$ was computed and included in the uncertainty for the stress values in Table 6.3, but not in the reported stress values. Even for a 20° misalignment, only a 6% discrepancy is found, which is much less than the uncertainties that propagate from $u(k)$, $u(A)$, and $u(L)$ due to distance measurement uncertainty.
\[ F_T = \frac{F_x}{\cos \theta} \]  

(6.7)

### 6.3.3.4 Rotation

When bending is ignored, there is some rotation of the nanowire specimen due to displacement of the nanowire end. If the nanowire initial length is \( L_0 \) and the nanowire is displaced a distance \( \delta x \) in the direction of tensile stage motion \( \hat{x} \), the nanowire stretches to final gauge length \( L \) and rotates an angle \( \alpha \) (Eq. 6.8). If rotation of the specimen is included in the analysis, Eq. 6.7 becomes Eq. 6.9. Eq. 6.9 is maximized when \( \epsilon = 0 \) and \( \theta \) is maximized. In this case, Eq. 6.9 reduces back to Eq. 6.7 and rotation does not contribute to the force uncertainty. For specimens 1 and 6, \( \epsilon > 0 \) and \( \theta > 0 \), so Eq. 6.9 can be used to compute a value different from Eq. 6.7. For both specimens 1 and 6, the difference between \( F_T \) from Eq. 6.9 and \( F_T \) from Eq. 6.7 is only about 0.1%, so rotation due to elongation was not included in the uncertainty analysis for the data presented in Tables 6.3 and 6.4.

\[ \alpha = \theta - \sin^{-1} \left( \frac{L_0 \sin \theta}{L} \right) = \theta - \sin^{-1} \left( \frac{\sin \theta}{1 + \epsilon} \right) \]  

(6.8)

\[ F_T = \frac{F_x}{\cos(\theta - \alpha)} = \frac{F_x}{\sqrt{1 - \frac{\sin^2 \theta}{(1+\epsilon)^2}}} \]  

(6.9)

### 6.3.3.5 Bending

The nanowire ends are clamped and if it is assumed there is no deformation within the clamps, the nanowire is not subject to rotation. In this case, bending must be assumed to account for all motion that is not elongation of the nanowire. For a first analysis, motion in the \( \hat{y}' \) direction is taken as pure bending, and motion in the \( \hat{x}' \) direction is taken as pure elongation. The nanowire is assumed to be isotropic and have a constant circular cross section along its length. The bending motion
results from a force $F_B$ in the $\hat{y}'$ direction that has a component in the original $\hat{x}$ direction, opposing the stage motion. Because the sum of forces in the $\hat{x}$ direction equals zero, $F_x = F_T \cos \theta + F_B \sin \theta$.

Motion $\delta x'$ results from $F_T$ (Eq. 6.10) and bending can be described (Eq. 6.11) by the beam equation for a slender beam with one end clamped and one end guided (free to displace but not to rotate), with applied force $F_B$, displacement $\delta y'$, and bending moment of inertia for a circular cross section $I_0 = \pi D^4/64$.

\[
\delta x' = \frac{F_T L_0}{AE} \quad (6.10)
\]

\[
\delta y' = \frac{F_B L_0^3}{12EI_0} = \frac{16F_B L_0^3}{3\pi ED^4} \quad (6.11)
\]

\[
\frac{\Delta F_T}{F_T} = \frac{3D^2 \tan^2 \theta}{4L_0^2} \quad (6.12)
\]

The systematic error in the tensile force due to presence of the bending force can be examined using Eq. 6.12. For $L_0 = 10\mu$m NW, $D = 200$ nm, and $\theta = 10^\circ$, Eq. 6.12 yields $\Delta F_T/F_T = 10^{-5}$. Force error due to bending is a very minor effect and is therefore not included in uncertainty calculations.

If maximum stress within the nanowire is considered, this occurs where the maximum bending stress is added to tensile stress. The bending moment $M$ within the nanowire is given by Eq. 6.13. The maximum bending stress $\sigma_{x',B}$ is at $(x', y') = (0, -D/2)$ or $(L_0, D/2)$. Axial bending stress is given by Eq. 6.14.

\[
M = \frac{1}{2} F_B L_0 - F_B x' \quad (6.13)
\]

\[
\sigma_{x',B} = \frac{-Ey'}{EI_0} = \frac{-y'M}{I_0} \quad (6.14)
\]
Using Eqs. 6.13 and 6.14, and relating the bending stress at the locations of maximum stress to the tensile stress due to elongation, $\sigma_{x',T}$, the maximum bending stress can be found:

$$Max.\sigma_{x',B} = \frac{3D\sigma_{x',T}\tan\theta}{L_0} \quad (6.15)$$

For the example values above, this means that $\sigma_{x',B}/\sigma_{x',T} \approx 0.01$. In other words, bending stress nonuniformity can add about 1% error to the estimated tensile stress. This was not a significant contribution in comparison to the uncertainties due to force and area, so it was omitted from the calculation of combined stress uncertainty.

### 6.3.3.6 Strain Uncertainty Due to Misalignment

Measurement of fiber length is repeated for each data point, with gauge length defined between the clamped regions. Therefore, uncertainty from in-plane rotation is not present in the elongation measurement used to derive the strain. Out-of-plane rotation of the nanowire specimen may lead to an overestimate of the actual strain. This can be evaluated following an approach similar to Eq. 6.9 or methods published for AFM tensile test misalignments [27, 84]. For the typical $\beta = 2.86^\circ$ found earlier, and with a very large in-plane strain of $\epsilon = 0.1$, this strain can be found to overestimate the actual strain by 0.3%, which is negligible in comparison to the other uncertainties in the strain. Uncertainty due to out-of-plane misalignment is therefore not included in $u(\epsilon)$ and $u(\epsilon)$.

Bending of the nanowire due to the clamped ends may have distorted the elongation measurement, which was performed using straight lines drawn on SEM images. The $u(\epsilon)$ related to the bending is estimated as follows. A displacement $\delta x$ of one end of a nanowire misaligned $\theta^\circ$ will lead to a measured elongation $e_m = L - L_0$. The actual elongation $e_a$ and hypotenuse $\delta x$, with angle $\theta$ and between sides $e_a$ and $\delta x$ define a right triangle that is inscribed within a circular sector of radius $L_0 + e_m$. Using this geometry, $e_m$ can be related to the actual elongation $e_a$ in the
presence of bending according to Eq. 6.16. When divided by $L_0^2$, a relationship (Eq. 6.17) between measured and actual strains, $\epsilon_m$ and $\epsilon_a$, is derived.

$$\epsilon_a^2 \tan^2 \theta = (\epsilon_m + L_0)^2 - (\epsilon_a + L_0)^2$$

(6.16)

$$\epsilon_a^2 \tan^2 \theta = (1 + \epsilon_m)^2 - (1 + \epsilon_a)^2$$

(6.17)

For $\theta = 10^\circ$ and $\epsilon_a = 0.1$, the value $\epsilon_m = 0.10014$ is found. Therefore, $\epsilon_m$ overestimates $\epsilon_a$ by 0.14%, and bending error in strain measurements can be omitted from the uncertainty calculation. Out-of-plane bending does not significantly modify this calculation.

There is additional localized strain variation from bending due to internal stress $\sigma_x$ variations: $\pm \epsilon_x = \sigma_x/E$, and this tends to add a similar percentage of uncertainty as the bending stress calculated in Eq. 6.15 contributes to the stress uncertainty. For example, for specimen 1, bending variations contribute $u(\epsilon) = 1.1\%$. Because measurement resolution uncertainties are about an order of magnitude larger or more, this localized bending uncertainty was not included in $u(\epsilon)$.

### 6.3.4 Results

Tensile tests were performed on several nanowire specimens. These results are summarized in Tables 6.3 and 6.4, including the results of three tensile tests, specimens 4 - 6, performed after the initial results reported elsewhere [11]. From Table 6.3 it should be noted that the nanowire specimens are capable of withstanding significant strain.

Specimen 1 (Fig. 6.17) stands out in both the measured result (Fig. 6.18) and the accuracy of its strain measurement because its relatively long $10.13 \pm 0.08 \mu m$ length and narrow $200 \pm 17$ nm diameter enabled it to stretch significantly under the load provided by the microfabricated test system. The tensile stress applied to the nanowire when the clamp failed was $7.5 \pm 3.4$ GPa.
Table 6.3: Tensile test data for six GaN nanowire specimens. A discussion of Young’s modulus uncertainty values is located at the end of Section 6.3.4.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Maximum $\epsilon$</th>
<th>Maximum $\sigma$</th>
<th>Young’s Modulus $E$</th>
<th>Failure Mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.042 ± 0.011</td>
<td>7.5 ± 3.4 GPa</td>
<td>210 GPa</td>
<td>Clamp Failure</td>
</tr>
<tr>
<td>2</td>
<td>0.031 ± 0.018</td>
<td>2.1 ± 0.6 GPa</td>
<td>c-plane fracture</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>0.022 ± 0.012</td>
<td></td>
<td>Insufficient force</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>0.0123 ± 0.0076</td>
<td>4.0 ± 1.7 GPa</td>
<td>Insufficient force</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>0.01 ± 0.01</td>
<td>4.7 ± 2.2 GPa</td>
<td>c-plane fracture</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>0.0103 ± 0.0036</td>
<td>7.1 ± 4.0 GPa</td>
<td>250 GPa</td>
<td>c-plane fracture</td>
</tr>
</tbody>
</table>

Table 6.4: Additional data for the six nanowire specimens. Angle $\theta$ of misalignment from stage motion direction, nanowire diameter $D$, linear fit parameter $B$, nanowire tensile test gauge length $L_0$, & maximum power $P_{\text{Max.}}$ reached during tensile test before observable damage to the MEMS actuator.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>$\theta$</th>
<th>$D$ (nm)</th>
<th>$B$ ($\mu$m/mW)</th>
<th>$L_0$ ($\mu$m)</th>
<th>$P_{\text{Max.}}$ (mW)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9.2° ± 0.4°</td>
<td>200 ± 17</td>
<td>0.0280 ± 0.0011</td>
<td>10.13 ± 0.08</td>
<td>72 ± 4</td>
</tr>
<tr>
<td>2</td>
<td>11.6° ± 1.4°</td>
<td>450 ± 20</td>
<td>0.0267 ± 0.0013</td>
<td>4.58 ± 0.06</td>
<td>96 ± 6</td>
</tr>
<tr>
<td>3</td>
<td>1.4° ± 1.0°</td>
<td>±34</td>
<td>0.0267 ± 0.0013</td>
<td>9.29 ± 0.08</td>
<td>81 ± 4</td>
</tr>
<tr>
<td>4</td>
<td>14.1° ± 0.8°</td>
<td>380 ± 20</td>
<td>0.02066 ± 0.00034</td>
<td>10.85 ± 0.06</td>
<td>134 ± 14</td>
</tr>
<tr>
<td>5</td>
<td>19.2° ± 1.4°</td>
<td>344 ± 30</td>
<td>0.02066 ± 0.00034</td>
<td>5.85 ± 0.04</td>
<td>155 ± 16</td>
</tr>
<tr>
<td>6</td>
<td>13.56° ± 0.19°</td>
<td>Min. 151 ± 30</td>
<td>0.0264 ± 0.0011</td>
<td>13.97 ± 0.08</td>
<td>62.746 ± 0.038</td>
</tr>
</tbody>
</table>

Figure 6.17: Specimen 1, GaN nanowire under tensile load before (left) and after (right) failure of the bottom clamp (inset) at the interface between the clamp and the fixed stage. The area of the failed platinum/carbon clamp is approximately 3 $\mu$m$^2$. The additional nanowires and membrane-like material seen in these images are located underneath the suspended nanowire and MEMS test stage structures, and they were not in contact with the moving parts in this mechanical test.
Figure 6.18: Tensile test results for GaN NW Specimen 1. The regression line corresponds with a Young’s modulus of 210 GPa.
The expected displacement $d_0$ used in force calibration for the specimen 1 test device was derived from motion of the stage after the specimen clamp failed (Fig. 6.19). This is a new development in tester calibration beyond the approach reported in Refs. 7 & 8, in that the fitting parameter between $d_0$ and $P$ is derived from the same device which was used to perform a mechanical test. After specimen 4 and specimen 6 fractured, they were used to provide fitting parameters (in Table 6.4) for the test devices on which those specimens were mounted.

Figure 6.19: Raw data from GaN NW Specimen 1 tensile test. Device input power and output displacement during the initial tensile test and then after the failure seen in Fig. 6.17.
Specimen 2 did not appear to fracture during the tensile test, but subsequent high-resolution SEM examination and FIB cross sectioning showed that the nanowire developed two fractures, one near each clamp.

FIB cross-sectioning also revealed that specimen 3 (Fig. 6.16) appeared to be two nanowires that had joined together during synthesis. This specimen had a large cross-sectional area of about $510 \times 893 \text{ nm} (0.46 \pm 0.04 \mu\text{m}^2)$ and the force available from the actuator (about $340 \pm 120 \mu\text{N}$ according to experimental data, giving a maximum tensile stress in this specimen of about $0.7 \pm 0.3 \text{ GPa}$) was insufficient to cause failure of this structure in the tensile test reported in Tables 6.3 and 6.4. The FIB was used to reduce the cross section of this specimen to about $250 \times 290 \text{ nm}$. With a reduced cross-section, the specimen spontaneously buckled, likely due to damage or heating induced by FIB milling. When tensile stress was applied, failure occurred with more than $2.4 \text{ GPa}$ stress applied to the resulting nanowire. This stress was estimated from the nanowire cross section and the assumption of uniform loading. In reality, the specimen remained curved as it failed, indicating the presence of residual stress nonuniformity in the nanowire cross section and a higher internal stress than is estimated with simple uniaxial tension.

The tensile tester MEMS device used to test specimen 4 was unable to provide sufficient force to induce failure of nanowire specimen 4 (Fig. 6.16). The MEMS actuator in the device used to test specimen 4 failed due to too much applied power. The nanowire specimen 4 did not fail during this tensile test. The maximum stress value reported in Table 6.3 reflects the highest value of force that was applied to this nanowire before the thermal actuator underwent observable plastic deformation.

Specimen 5 demonstrated very little elongation before it failed. It can be seen in Figure 6.16 that the failure mode was another fracture perpendicular to the $c$-axis, although the edge of this fracture surface appeared to have been made irregular by some excess deposited platinum.

Elongation and failure were observed in specimen 6, Fig. 6.20. However, the presence of the taper of specimen 6 meant that the stress and strain were not uniform over the length of the nanowire. Although there was the same force transmitted over the length of the nanowire, the stress
at the thick end was significantly less than the stress at the thin end. Assuming the elastic modulus was uniform throughout this specimen, the thin end experienced more stress and strain than the thick end. The nanowire failed at the thin end, indicating that an estimate of the maximum stress at failure (specimen 6, Table 6.3) should be found by dividing the force applied by the area at the thin end. The specimen 6 nanowire is obviously a tapered shape, which can be approximated as a conical prism with circular cross section. Because elongation is measured for a gauge length that includes this significant taper, and this elongation value is used for computation of strain for this specimen, the calculation of stress used to compute the Young’s modulus $E$ must also incorporate the effects of the taper. The elongation $e$ relates to the tensile force $F_T$ according to Eq. 6.18.

$$e = \frac{4F_T L_0}{\pi ED_1D_2} \quad (6.18)$$

Here, $D_1$ and $D_2$ are the nanowire diameters at either end of $L_0$ and a circular cross section is assumed. This elongation is equivalent to that of a uniform diameter cylinder with diameter $D_0$. For computation of $E$, the strain based on $e/L_0$ must be compared to a stress calculated using $D_0$, so a moderated stress $\sigma_{mod}$ (Fig. 6.21) is calculated from Eq. 6.19.

$$\sigma_{mod} = \frac{F_T}{A} = \frac{F_T}{\pi D_0^2/4} = \frac{F_T}{\pi D_1D_2/4} \quad (6.19)$$

As seen in Table 6.3, $E = 210$ GPa was found for specimen 1 and $E = 250$ GPa was found for specimen 6. These are the slopes of the unweighted linear regression lines seen in Figs. 5 and 6, with the $y$-intercepts set to 0. The standard error of the regression for each of these values was $u = 10$ GPa, giving 95% confidence values of ±20 GPa. However, this value does not include the uncertainties of the stress and strain data points used to compute the regression line. When these values are added, the combined uncertainty of the derived modulus $E$ could be as large as $u_c(E) = 60$ GPa for specimen 1 and $u_c(E) = 120$ GPa for specimen 6.
Figure 6.20: (Left) GaN NW Specimen 6 just before failure. Note the significant taper in this nanowire specimen. (Right) Specimen 6 just after failure. (Inset) Close-up view of the nanowire fracture, indicating a fracture mostly perpendicular to the long, c-axis of the nanowire.

Figure 6.21: Tensile test results for GaN NW Specimen 6, using $\sigma$ computed according to Eq. 6.19. The regression line corresponds with a Young’s modulus of 250 GPa.
6.3.5 Discussion

The robust nature of specimen 1 in withstanding a $0.040 \pm 0.017$ engineering strain is perhaps the most notable result of this work. Image superposition confirms the strain measurement for this specimen. Observation of the specimen after the clamp failure indicates that it did not exhibit failure, nor did it experience plastic deformation. The estimated maximum stress applied to this nanowire, $7.5 \pm 3.4$ GPa appears to be a reasonable value in that the ultimate failure of specimens 3, 4, and 6 occurred for stress values of a similar order of magnitude.

The ability to withstand a large strain, as reported here for specimen 1, reflects an exceptional strain tolerance and resilience for what might at first be considered a brittle material, but it is not entirely surprising given that this measurement was performed on a single crystal of a covalent material with a hexagonal lattice. These material characteristics are all known to increase mechanical strength. The lack of apparent extended and point defects in the nanowires also contributes to their strength.

Tensile strength and strain at tensile failure have not been widely reported for GaN, although there has been at least one report of compressive yield strength of 15 GPa in a GaN film. [103] Resonant frequency measurements on GaN NWs similar to those used here measured $c$-axis Young’s modulus $E_3 = 250 - 350$ GPa. [141] Resonance experiments on other $a$-axis [120] oriented GaN NWs found $E_1 = 227 - 305$ GPa. [153] AFM 3-point bending on other $c$-axis GaN NWs found $E_3 = 218 - 400$ GPa, with a possible observation of diameter dependence of the Young’s modulus. [17] Elastic properties of GaN thin films and bulk crystals have been widely reported. Nanoindentation of epitaxially-grown GaN films with unspecified orientation found Young’s modulus values in the range of 210 - 295 GPa. [54, 74, 103] Other types of experiments on bulk GaN crystals (ultrasonic, Brillouin scattering) have provided elastic constants $C_{11}, C_{12}, C_{13}, C_{33}$ that can be used to find $E_3$, giving $E_3$ of 161 GPa (ultrasonic measurement) to 362 GPa (optical measurement). [15, 24, 120, 158]

The modulus results mentioned above are in the correct range to fit with previously-reported values of the GaN $E_3$ but the uncertainties from this test system may be too large to make a
definitive statement about values of $E$ measured here. However, the maximum stress and strain values reported in Table 6.3 have 95% confidence intervals that are defined well enough to point to GaN NWs as resilient, high-strength materials.

Measurements from the JEOL SEM used to observe the tensile experiments are limited to the resolution of images obtained from this instrument. For the magnifications used for the data collected here, each pixel corresponds to about 20 – 30 nm. However, as noted in the 95% confidence values reported for the nanowire gauge lengths $L_0$ in Table 6.4, about ±40 nm is the best accuracy obtained from this instrument. Typical values were more on the order of ±80 nm in the measurements reported here due to the focal resolution of this SEM. Therefore, large displacements and large specimen gauge lengths will be more accurately recorded with SEM observation of MEMS testers than measurements of smaller distances. The nanowire gauge lengths are recorded in Table 6.4 to allow comparison of these measurements.

The failure shear stress of the specimen 1 clamp-microdevice interface was about $80 \pm 30$ MPa, which was much lower than might be expected for an interface with good bonding. After this failure occurred at the clamp-tester interface, potential sources of material that would interfere with good bonding between the clamp and the MEMS tester were noted.

It was observed that HF release after clamping nanowires to the microfabricated test stages using IBID can leave a membrane-like residue visible on the substrate of the test device (visible in Fig. 6.17). This residue can also form in instances where devices are subjected to SEM imaging followed by HF release, but it is not seen in cases where HF release is performed before imaging. This residue may originate from chemical decomposition of residual solvent adsorbed to the working surfaces, or of miscellaneous organic contamination from other molecules that were suspended in the solvent or present on other surfaces in the electron microscope. It appears to generate a resilient layer as a result of the SEM process. Alternatively, this may be errant Pt-C deposited as a byproduct of the IBID clamping procedure. In applications where a good surface bond is desirable, such as the clamping of a nanomaterial specimen, FIB removal of potential bonding surface contamination is a quick and straightforward step, although some of the ablated material
will nevertheless be redeposited. So, after the test on specimen 1 resulted in clamp failure rather than nanowire failure, the clamping procedure was modified. The gallium beam was used to remove an estimated 10 nm of material from the surface of the nanowire and the surrounding areas before clamp deposition. Furthermore, larger clamping areas of platinum were generated. Clamp failure was not observed in any subsequent tests.

The power at which the MEMS test structures begin to exhibit failure appeared to vary from chip to chip, likely dependent upon variations in processing of the chips holding these structures. For instance, devices used for earlier experiments reported in Ref. [11] displayed the onset of creep for input power the range of 60 - 90 mW. However, the device used to test specimen 4 withstood up to $134 \pm 14$ mW before it began to fail. This suggests that the fit parameter that correlates free displacement with input power is not a universal parameter but rather one that depends upon chip processing, most likely related to the HF release etch time. Therefore, at least one measurement of $B$ must be made on each chip that is used for nanowire tensile testing. The four devices on each chip have identical structural configurations and identical processing histories, so the $B$ value found with one device can be used for others on the chip.

The eventual thermal failure of the MEMS test structures manifests in several ways: the actuator resistance varies over time, SEM imagery shows discoloration in the vicinity of the hottest part of the actuator, and upon cessation of input power, the moving stage returns to a point closer to the fixed stage than when it began. These observations appear to indicate that the highest input powers also lead to plastic deformation of the thermal actuator.

### 6.3.6 Conclusion

This work has demonstrated that dielectrophoretic self-assembly of GaN nanowires can place nanowires on active MEMS devices without the need to separately manipulate individual nanowires. With careful design of suspended structures, this self assembly process can operate on released structures without pulling these suspended structures into contact with the substrate. IBID platinum-carbon clamps can secure individual nanowires to a polysilicon surface for mechanical loading of
the nanowires, but the interface between these clamps and the surface can fail with a shear stress on the order of $80 \pm 30$ MPa, therefore clamps must be deposited over a sufficient area to withstand the force applied to the nanowire specimen. Clamping is a key failure point and limiting fabrication step. Length measurements from SEM images are another significant limitation.

The MEMS test structures presented here have a moving stage that can be displaced by up to about $3 \mu$m, but clamping of a tensile specimen across the moving and fixed stage creates a constraint which prevents motion of the moving stage, instead loading the tensile specimen. Measurement of specimen strain from SEM images removes the effect of clamp deformation from the tensile curve, but the clarity of tensile data was limited by image resolution. Measurements of electrical power are readily automated. Repeated tensile tests indicate that single crystal GaN appears capable of withstanding uniaxial strain at least 0.01 and as much as $0.040 \pm 0.017$. Furthermore, GaN NW tensile stress may reach the range of $\sim 4$ to $7$ GPa without material failure.

6.3.7 Acknowledgements

J.J. Brown is supported by a National Science Foundation Graduate Research Fellowship. The authors thank Dr. Norman Sanford of NIST-Boulder, Boulder, CO, USA, for many helpful discussions, and Matthias Muoth and the ETH-Zürich Micro and Nanosystems group for help with AFM measurement. FIB machining and SEM imaging were performed at the Nanomaterials Characterization Facility of the University of Colorado at Boulder.
6.4 MEMS Tensile Characterization of SiCNO-CNT Composite Nanowires

This section reports results produced in collaboration with V. Bright, G. Singh, C. Hierold, and R. Mahajan. Figures 6.22 through 6.24 were recorded by G. Singh (now at Kansas State) when he was working with R. Mahajan at Virginia Tech, Blacksburg, VA, USA.

Polymer-derived silicon carboxynitride coated multiwall carbon nanotubes were strained using a microelectromechanical (MEMS) tensile test system within a scanning electron microscope. These composite nanowires were synthesized by pyrolysis of an organosilicic polymer coating on a nanotube scaffold.

Experiments obtained modulus measurements of $21.2 \pm 4.1$ GPa and $28.8 \pm 10.4$ GPa for the two tensile specimens tested. The maximum stress observed at failure was $1.97 \pm 0.81$ GPa, and the failure strain exceeded $0.0212 \pm 0.0035$.

Tensile tests were performed on silicon carboxynitride–multiwall carbon nanotube (SiCNO–MWCNT) composite nanowires, structured with MWCNT core and SiCNO polymer-derived ceramic (PDC) shell. This composite nanowire (NW) material shows potential for customizable properties such as piezoresistivity. [66,164] Microelectromechanical systems (MEMS) for mechanical testing provide one means to interrogate such nanowires, and have been under development by several groups. [1,10,12,37,40,70,154,163] These systems achieve actuation for tensile tests using comb drives, electrothermal actuators, or externally from such devices as piezoelectric actuators.

With specification of precursor polymer composition and processing conditions, PDC electrical conductivity can be varied from that of a complete insulator ($\sigma_{dc} < 10^{-10}(\Omega\text{cm})^{-1}$) to a semiconductor [22] while piezoresistive gauge factors [164] may range as high as 4000. Furthermore, Young’s modulus for SiCN can be specified from $\sim 80$ to $140$ GPa. [22,60] Properties of bulk PDC SiCN and SiCNO are well known, [22,113,125] but little data is available on mechanical properties of PDC nanowires (NWs). Consequentially, there is interest in understanding the evolution
of PDC properties under varying dimensional constraints. [66,131] Unique properties of PDCs are usually attributed to the presence of nanosize crystalline domains (∼1 – 2 nm) and graphene-like free carbon. Dimensional constraint may interact with these nanoscale domains to significantly modify the properties of PDCs in NWs, in comparison to bulk PDCs.

The synthesis of CNTs with specified properties remains a challenge. Composite PDC-CNT NWs may provide one route to the generation of nanoscale devices with repeatable, customizable material properties. The chemical precursors to PDCs have been shown to wet CNT surfaces, allowing the generation of nanoscale PDC structures on CNT scaffolds, [126] producing composite NWs with repeatable processing and fairly predictable properties. PDC-CNT NWs have recently been explored for applications in multifunctional sensors. [131] More mechanical, thermal, and electrical data is needed in order to realize new PDC-CNT designs.

Synthesis of SiCNO-MWCNT nanowires was achieved by controlled pyrolysis. [126, 131] MWCNTs prepared using Fe catalyst [131] were dispersed in acetone containing 10% (by volume) of poly(ureamethylvinyl)silazane. The mixture was dried in air and subsequently pyrolyzed in N₂ at 1100°C for 4 hours.

Transmission electron microscopy (FEI Titan 300), Fig. 6.22, indicated a nanowire structure without large crystalline domains. X-ray energy dispersive spectroscopy, Fig. 6.23, and x-ray photoelectron spectroscopy (PHI Quantera), Fig. 6.24, revealed the presence of Si, C, N and O in nanowires.

MEMS tensile test devices, [10] Fig. 6.25, consisting of fixed and moving mechanical stages fabricated from polycrystalline silicon, were used to perform tensile tests. The moving stage is suspended by beams that stabilize linear motion that originates from a chevron-type electrothermomechanical actuator. [155] NWs were mounted across the tensile stages (Fig. 6.26) using a micromanipulator probe within a dual (electron and focused ion) beam microscopy system. [12, 131] PtC clamps, generated by ion beam induced deposition, [10, 163] secured the nanowires to the MEMS stages.
Figure 6.22: TEM micrograph of a typical example of the PDC-CNT composite nanowire material used as tensile samples in this section.

Figure 6.23: TEM energy dispersive spectroscopy (EDS) of an individual composite nanowire. Intensity peaks confirm the presence of Si and C in the NW, and indicate the additional presence of some N and O. The Cu signal is due to the TEM grid. The Y-axis is a relative scale of signal intensity. Units of the X-axis are kilo-electron-volts.
Figure 6.24: X-ray photoelectron spectroscopy (XPS) results confirming the presence of Si, C, and O in the elemental survey of the composite nanowire material. Y-axis scale is given in $10^4$ counts per second. The embedded data table shows a compositional analysis (Right hand column = atomic %) based on the peaks present in the spectrum.
Figure 6.25: Proceeding clockwise from upper left, this is a series of progressively higher magnification images showing PDC-CNT Specimen 1 supported on a MEMS tensile tester before commencement of the tensile test.
Figure 6.26: (a) PDC-CNT Specimen 1 just before failure. (b) Specimen 1 fracture surface, from the left clamped region in (a). (c) PDC-CNT Specimen 2 just before failure. (d) Specimen 2 after experiment, illustrating failure of the clamping material. In (a) and (c), the white line at the image center illustrates the gauge length.
Gauge lengths were defined between the clamped regions in order to remove systematic uncertainty due to clamp deformation. Gauge lengths were measured using lines drawn between fiducial points in SEM images. The tensile tests clearly demonstrated elongation of the fiber specimens. Actuator voltage and current were measured, with 1000 samples averaged per data point, minimizing noise error. The standard uncertainty for all voltages was $\leq 0.035 \%$. Current was calculated from the voltage drop across a resistor with $R = 386 \pm 11 \Omega$. The use of computerized data acquisition improved electrical measurement precision in comparison with earlier work. [10,12]

Analysis of this tensile test microsystem has been previously described. [10] The forces $F$ applied to the specimens were calculated [10, 12] according to $F = k(d_0 - d)$, where measured displacement $d$ of the tensile stage is subtracted from expected free-moving displacement $d_0$. The previously obtained [10] spring constant of the MEMS testers was $k = 175 \pm 29 \text{N/m}$. After the failure of each specimen, each actuator was operated to observe the free-moving stage displacement, allowing second-order polynomial regression to relate $d_0$ to input electrical power.

The MEMS test system demonstrated a 3.2 $\mu$m range of motion, using $\leq 85 \text{mW}$ and $\leq 5 \text{V}$. Motion during tensile tests was $\leq 1 \mu$m. The maximum force obtained during the tensile tests was $47 \pm 13 \mu\text{N}$. The standard uncertainty $u(x_i)$ of each measurement $x_i$ was found according to standard recommendations and propagated to measurement results using the root sum of squares method. [59, 142] Misalignment uncertainty was insignificant in comparison to $u(k)$ and displacement uncertainty, [10] which dominated the stress uncertainty. The gauge length uncertainty yielded elongation measurements with $u \geq 5.6\%$, which dominated the strain uncertainty.

Both specimens exhibited slack at the start of their respective tensile tests. To derive tensile curves from straightened fibers, strain was evaluated using the gauge lengths after the specimens were straightened under partial loading of the nanowires. The stress values applied at these points were used to define zero-points for the stress, and were subtracted from the subsequent stress measurements, so that the tensile plots, Fig. 6.27, would show only the changes in strain due to stress added beyond the point where the specimens were straightened. The Young’s Modulus $E$ for each specimen was obtained using first order linear regression.
Figure 6.27: Tensile plots obtained for the PDC-CNT specimens. (a) Specimen 1. Gauge length = 7.783 ± 0.033 μm. (b) Specimen 2. Gauge length = 3.647 ± 0.013 μm. Error bars indicate the standard uncertainty for each stress and strain value.
Table 6.5: SiCNO-CNT composite nanowire tensile test results.

<table>
<thead>
<tr>
<th></th>
<th>Specimen 1</th>
<th>Specimen 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Young’s Modulus, $E$ (GPa)</td>
<td>28.8 ± 10.4</td>
<td>21.2 ± 4.1</td>
</tr>
<tr>
<td>Strain at failure, $\epsilon_f$</td>
<td>0.0212 ± 0.0035</td>
<td>0.088 ± 0.005</td>
</tr>
<tr>
<td>Stress at failure, $\sigma_f$ (MPa)</td>
<td>630 ± 300</td>
<td>1970 ± 810</td>
</tr>
<tr>
<td>Diameter, $D$ (nm)</td>
<td>230 ± 12</td>
<td>150 ± 9</td>
</tr>
</tbody>
</table>

Specimen 1 failed as a fracture in one of the clamped regions (Fig. 6.26), where complex strain states are typically present, with stress concentrations and strains greater than in the main part of the fiber. Because of this, the stress and strain reported in Table 6.5 for Specimen 1 represent lower bounds on the fracture strength and the strain to failure. In Specimen 2, much larger stress and strain were found at specimen failure, but these are also lower bounds because the PtC clamp material failed, not the NW. Clamp failure is not surprising; it has been previously observed in testing GaN NWs. [10] The strains reported in Table 6.5 are very high, considering that SiCN is a hard ceramic at bulk scale. These values are comparable to observed MWCNT failure strains. [162]

Typically the Young’s Modulus is determined from the mean of regression parameters obtained from multiple tensile samples, and the uncertainty $u(E)$ can be evaluated as the standard deviation of the mean. Such an approach is somewhat impractical in experiments requiring micromanipulation of individual nanofibers. Here, values for $u(E)$ were obtained by propagation of stress and strain uncertainty through the constrained first order regression.

The SiCNO-MWNT composite nanowire tensile tests indicate $E$ on the order of 20 – 30 GPa, with a maximum value of 28.8 ± 10.4 GPa (Table 6.5). The modulus values obtained for each specimen overlap in uncertainty, indicating that the values are mutually consistent. These SiCNO-MWNT NW modulus values are lower than $E$ of individual MWCNTs but comparable to similar dimension Si-based amorphous NWs and carbon nanofibers reported in literature (Table 6.6), indicating that the disordered nature of the SiCNO coating may be dominant in determining $E$ of PDC-CNT composite nanowires.

These experiments demonstrated the integration of composite nanowires with active MEMS
Table 6.6: Comparison of modulus measurements for similar materials reported in literature.

<table>
<thead>
<tr>
<th>Material</th>
<th>$D$(nm)</th>
<th>$E$(GPa)</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$ NW$^a$</td>
<td>50</td>
<td>57</td>
<td>3-point bending</td>
</tr>
<tr>
<td>SiO$_2$ NW$^b$</td>
<td>70</td>
<td>26 ± 3.1</td>
<td>Bending in TEM</td>
</tr>
<tr>
<td>SiC/SiO$_x$ composite$^b$</td>
<td>83</td>
<td>52 ± 8.2</td>
<td>Bending in TEM</td>
</tr>
<tr>
<td>Vapor-grown C NW$^c$</td>
<td>&gt; 80</td>
<td>~ 25</td>
<td>3-point bending</td>
</tr>
<tr>
<td>Vapor-grown C NW$^d$</td>
<td>88 – 129</td>
<td>69.2</td>
<td>MEMS tensile test</td>
</tr>
<tr>
<td>C NWs$^e$</td>
<td>125 – 230</td>
<td>11 – 59</td>
<td>Bending in TEM</td>
</tr>
</tbody>
</table>

$^a$ From ref [102]. $^b$ From ref [149]. $^c$ From ref [78].
$^d$ From ref [70]. $^e$ From ref [133].

devices. The results of MEMS tensile straining on composite nanowires have provided bounds on the failure strength and the strain to failure for SiCNO-MWCNT NWs, in addition to characterization of the Young’s Modulus of these NWs. SiCNO-CNT composite nanowires show potential for very good fracture strength and strain to failure, elastic modulus, and resilience. Electromechanical and thermomechanical nanowire experiments may be contemplated using similar test systems.

Acknowledgements: This research was supported by the DARPA Center on Nanoscale Science and Technology for Integrated Micro/Nano-Electromechanical Transducers (iMINT), funded by the DARPA N/MEMS S&T Fundamentals Program (Award #N66001-10-1-4007, Dr. T. Akinwande, Program Manager). Tensile tests were performed at the CU Nanomaterials Characterization Facility, Boulder, CO. J. J. Brown is supported by a National Science Foundation Graduate Research Fellowship. Thanks to D. D. Luu (CU) for electronics; Dr. R. S. Ruoff (UT - Austin), Dr. D. A. Dikin (Northwestern U.), and S. Shah (USAFA) for useful discussions; S. Priya (VT) for lab use; and Kansas State University for a travel grant. Thanks also to all VT NCFL staff and all CU IMINT and NCF staff.
6.5 Carbon Nanotube Straining in a Transmission Electron Microscope

Work in this section was performed in collaboration with Prof. C. Hierold and M. Muoth at ETH-Zürich. Motivations for this work included the idea of developing MEMS tools for varying experimental environments, and also the measurements [47,136] indicating that carbon nanotubes could be powerful piezoresistive strain sensors. Furthermore, techniques are needed that present the opportunity for scalability in data collection. With significantly more data on CNT failure, much of the physics of CNTs could be better described and verified. We operated the UTP and test coupon designs within a transmission electron microscope in order to demonstrate several points: the applicability of these designs to the constrained TEM environment, the ability to control mechanical interaction with structures having nanoscale dimensions, and to provide proof-of-concept of separating nanomaterial specimen preparation from actively-controlled structures for mechanical actuation.

Data collection scalability was not specifically addressed in these experiments. However, the improvements in the speed of specimen handling and preparation that are enabled by use of test coupons suggest that a larger study of nanomaterial failure behavior is within reach with several more UTP and coupon design iterations.

Carbon nanotubes were prepared on the MEMS test coupons according to the recipe in use at Prof. Hierold’s group at ETH. Ferritin molecules were deposited from nonstandardized aqueous solution by immersion of the coupon chips within a droplet of this solution and then drying of the chips on a heated surface. The solution was prepared by dissolving ferritin in water, followed by sonication, and then centrifugation to remove clumps and allow fractionation of the solution with well-dispersed ferritin. CNTs were grown using 850 °C pyrolysis of methane. [32,96]

The presence of CNTs was confirmed with SEM imaging. Fig. 6.28 shows an example of CNTs on a test coupon surface, and Fig. 6.29 demonstrates that CNTs may be successfully grown to span the fixed and moving stages on a test coupon.
Figure 6.28: Image of CNTs grown on the surface of a test coupon using ETHZ facilities and recipe.
Figure 6.29: Image of CNTs grown across the gap in a test coupon using ETHZ facilities.
The Universal Test Platform chip was mounted (Fig. 6.30) in a printed circuit (PC) board specially designed for interfacing to the electric feedthrough TEM stage used at ETH. Standard electron microscopy silver paint was used to secure the chip to the PC board. Electric connections were made through the feedthrough stage to computer-controlled analog electronics used to drive the thermal actuator on the Test Platform chip. A coupon on which several CNTs were observed to span the tensile stage area was selected for testing, and it was inserted in the UTP chip with alignment similar to that seen in Fig. 5.6.

The TEM stage insertion process requires physical inversion of the TEM stage. Because gravity is used to achieve the Z-direction alignment of the coupon within the UTP, inversion of the UTP presents the likelihood that the test coupon will fall out of place. In most laboratory settings, this is a convenient means to remove the coupon and reuse a UTP chip, but this is undesirable within the TEM environment. A small quantity of silver paint was used to fix the test coupon to the UTP in order to prevent loss of the coupon during the TEM stage loading process. The prepared coupon and UTP chip may be seen in Fig. 6.31.

Using the TEM (FEI model CM12), the test coupon was located in the TEM image, as was the tensile stage on the test coupon (Fig. 6.32). Two carbon nanotubes on the tensile stage were identified for straining. The first test focused on straining the CNT marked with “1” in Fig. 6.32, and the subsequent test was performed with TEM observation of the CNT marked with “2” in that image.

The UTP probe was operated and observed to contact the coupon stage. The computer-based instrumentation system recorded electrical current and voltage supplied to the actuator. Plotting the current and voltage data from the TEM experiments (Fig. 6.33), it is immediately evident that the UTP actuator resistance fluctuated significantly during operation in the TEM. For operation straining the CNTs, the input power was changed in small increments, indicated by the dense locations of data points seen in Fig. 6.33.

In powering up to and powering down from the power levels needed for actuation of the coupon, larger current steps were taken, and large variations in the actuator voltage can be observed.
These variations were not observed to the same extent during prior operational testing of the UTP chip using a probe station, and a change in thermal management of the UTP chip provides a likely explanation for this observation. In probe station testing, the UTP chips were supported on a metal surface capable of significant heat dissipation. When a glass coverslip was inserted under a UTP chip, the actuator quickly burned out at what had been a previously acceptable power level. Similarly, the primary mechanism of heat dissipation from the UTP chip within the TEM is thermal conduction in the polyimide PC board. The poor thermal conduction in polyimide in comparison to, say, aluminum presents the likelihood that slow thermal transients may interfere with stable operation of the UTP actuator. The actuator was observed to slowly change resistance over slow (multi-minute) time spans, and these changes were documented by repeating computerized data collection without changing the control output to the actuator.

Figure 6.30: UTP chip mounted on a chip carrier for TEM usage. TEM chip carrier was provided by M. Muoth and C. Hierold, ETHZ.
Figure 6.31: UTP chip mounted on a chip carrier for TEM usage.

Figure 6.32: TEM images of the test coupon stage used to support CNTs for the testing reported here.
Figure 6.33: Current and voltage response of the actuator during two tensile tests. Top: Test of Specimen 1, linear fit is to the region of dense data points at upper right of this image, which is where the actuation for tensile straining of the CNT occurred. Bottom: Test of Specimen 2. In both images, the test platform probe is in contact with the test coupon in the region where current exceeds about 135 mA.
6.5.1 TEM CNT Straining Results

The first tensile specimen appeared to move its attachment points slightly as it was partially loaded and unloaded. (Fig. 6.34) Slack was removed from the specimen, electron diffraction images obtained, and the specimen was loaded until it reached a failure point. The left end of the CNT specimen failed or detached from the actuated stage. Upon retraction of the stage, the CNT reattached to the tensile stage (Fig. 6.35). The stage was advanced and retracted several times, resulting in repeated detachment and reattachment of the CNT specimen. This behavior was recorded in several images, and also in a video file.

The second tensile specimen (Fig. 6.36) was subjected to mostly increasing loading, with strain held constant several times for durations of several minutes in order to allow acquisition of more TEM electron diffraction images. From Fig. 6.37, we see that the second CNT specimen had an inner diameter of about 6 ± 1 nm, and an outer diameter of about 15 ± 2 nm. The diffraction patterns reported in Section 6.5.2 below show that CNT Specimen 2 is a double-walled carbon nanotube, therefore some of the CNT diameter observed from imaging is likely due to amorphous carbon deposits during the CNT synthesis.

The electronic signal output and thermal actuator current, voltage, and power measurements recorded by the data acquisition system during loading, failure, and reattachment of CNT Specimen 1 may be seen in Figures 6.38 and 6.39. The large control oscillations were performed to extend and retract the tensile stage, and where recorded in images, attachment and detachment of the CNT is noted. Similar data for the test of Specimen 2 is included in Fig. 6.40. As described earlier, the fluctuations in the thermal actuator current and voltage measurements are likely to be due to slow thermal transients.

Strain was recorded for both specimens (Fig. 6.41) by selecting a gauge length at the start of image series in which magnification was unchanged, and measuring changes in this length according to the procedure described in Section 6.4. The gauge length of Specimen 1 was \( L_0 = 3.413 \pm 0.011 \mu\text{m} \) and was defined by the apparent attachment points of the CNT (Fig. 6.42). The maximum
strain value for Specimen 1 was $\epsilon_{Max.} = 0.0510 \pm 0.0032$. Length measurements were bounded by ±4 pixels for the images from the Specimen 1 test.

Using Specimen 2 $L_0 = 7.999 \pm 0.013 \mu m$, measured between the Y-junction at one extremity of the Specimen 2 CNT, and the apparent attachment point at the other end, strain for a continuous series of increasing actuator power was derived and plotted (Fig. 6.41). Length measurements were defined by boundaries of ±6 pixels for the images from the Specimen 2 test. Specimen 2 endpoints are visible in Fig. 6.43. The maximum strain obtained for Specimen 2 is $\epsilon_{Max.} = 0.2388 \pm 0.0023$. Above these recorded strain levels, the test platform actuator was pushed to its limit, eventually breaking the CNT at an unknown level before failure of the thermal actuator itself. This appears to be a very high recorded strain, but this might be explained by a telescoping failure mode. During progression of the tensile experiment, CNT Specimen 2 appeared to have some variation in outer diameter along the length of the CNT. Progression of the tensile experiment appeared to increase the size of the narrow regions on Specimen 2. It is also possible that slippage of the CNT specimens due to insufficient clamping led to an overestimation of the CNT strain values.

For both specimens, image calibration error introduced by changing magnification and focus of the TEM created significant jumps in image scaling that prevent estimation of applied stresses according to the methods described in Sections 6.2, 6.3, and 6.4. This behavior is evident in some of the jumps seen in the stage displacement and specimen length data plotted in Fig. 6.44.

The minimum UTP stage displacement step seemed to be on the order of the uncertainty in the displacement measurements. Although displacement steps were on average 27 nm for the steps in the test of Specimen 1 and 52 nm for the Specimen 2 test steps, minimum values observed were 11 nm and 6 nm, respectively. The displacement measurement uncertainty was $u(d) = 16$ nm for the Specimen 1 test, and 18 nm for the Specimen 2 test. This result is similar to what can be observed by examining the data for the integrated tester. In Section 6.3, the displacement uncertainty is 54 nm, which also appears to be the same order of magnitude as the observed displacement steps.
Figure 6.34: One end of CNT TEM Specimen 1 can be seen to shift in these images of the early operational testing of this specimen. Scale bar was unavailable for these images, which are at different magnifications, but the stage gap in both images is about 2.7 $\mu$m.
Figure 6.35: Image of CNT TEM Specimen 1 after failure and reattachment of the left end.
Figure 6.36: Image of CNT TEM Specimen 2 before loading.
Figure 6.37: Close-up of CNT TEM Specimen 2.
Figure 6.38: Illustration of signals measured during the test of CNT TEM Specimen 1. Data have been scaled to allow for comparison between signals. Points labeled “A” mark detachment of the CNT, and points labeled “B” mark reattachment of the CNT.

Figure 6.39: Electrical measurements during operation of the test stage in the TEM while manipulating CNT Specimen 1. Left: Control signal specified by computer and sent to actuator. Center: Measured actuator current. Right: Measured actuator power.
Figure 6.40: Illustration of signals measured during the test of CNT TEM Specimen 2. Data have been scaled to allow for comparison between signals. Gaps in the stage displacement measurement indicate regions where images were not recorded, preventing measurement of the stage gap. However, the data acquisition system recorded electronic data in these regions. The arrow at the upper right indicates where the UTP thermal actuator began to fail.

Figure 6.41: Strain versus power dissipated in actuator for the two CNT TEM specimens. Left: Test of Specimen 1. Right: Test of Specimen 2. “A” indicates the point after diffraction image plate 9 was collected, “B” indicates the point at which diffraction image plate 10 was collected.
Figure 6.42: Magnified images of the ends of CNT TEM Specimen 1 before failure of the specimen.

Figure 6.43: Magnified images of the ends of CNT TEM Specimen 2.
Figure 6.44: Stage gap and CNT lengths measured during the test of Specimen 2, plotted versus power dissipated in the test platform actuator. Points marked “A” indicate the boundaries on the measurements recorded between diffraction image plates 5 and 6, and points marked “B” indicate boundaries on the measurements recorded between diffraction image plates 9 and 10. The CNT length is less than the stage gap because one of the fiducial points of the Specimen 2 CNT was a junction with an additional CNT, seen in Figures 6.36 and 6.43.
The key points from this work are the following:

- By creating UTP and coupon devices with voids passing completely through these devices, it was possible to demonstrate use of these devices in a transmission electron microscope environment.

- As a proof of the concept of separating specimen preparation from device actuation, growth of CNTs was performed on test coupons, avoiding the need to place individual CNTs on the tensile stage using a micromanipulator. Subsequent to the CNT growth, we used the UTP and coupon design to impart mechanical signals to CNTs observed within the TEM.

- In contrast to most other microdevices used for TEM experiments, the UTP and coupon design can provide displacements of 3 µm or greater.

- Control signals generated at a computer interface were converted to mechanical signals experienced by multiple nanotube specimens.

- Large strains were applied to two CNT tensile specimens using the UTP and coupon system. For one specimen, snap-off/snap-on behavior was repeatedly observed. For the second specimen, a very large strain was obtained, probably indicative of telescopic CNT failure.

This work suggests the following steps for future work:

**Distance Calibration:** Structures for distance calibration standards are needed to reduce systematic errors between images taken in the TEM with slightly varying focus and magnification.

**Packaging Thermal Design:** The device packaging design requires some engineering for the specific application chosen for operation of the UTP. In the case of a TEM, some thermal engineering is required, as is a better mechanism to prevent loss of the test coupon chip during the TEM stage loading process.
**Specimen Clamping:** Clamping of CNT specimens requires further attention for tensile tests performed in a TEM. Verification of the clamping of the CNT is needed in order to obtain accurate strain values.

**Actuation:** Due to the large thermal transients suspected of interfering with measurements and displacement stability, other mechanisms for UTP device actuation should be reconsidered.

**Displacement Sensing:** As mentioned in previous sections, on-chip in-plane displacement sensing could be helpful to improve the precision and quantity of stage displacement measurements. Although piezoresistive displacement sensing was implemented in the UTP design, it was not a central focus of work, and further development of this design could be useful.
6.5.2 TEM Diffraction Patterns

The electron diffraction pattern of an individual carbon nanotube can be used to identify the chirality of the nanotube. [50, 77, 95, 97] These patterns are formed as a collimated electron beam is scattered by a nanotube, resulting in periodic summation and cancelation of electron waves. Because these patterns are angle dependent, it may be expected that strain within a carbon nanotube may affect the bond angle between carbon atoms in the nanotube.

The Universal Test Platform and coupon system provide one means to begin to probe this hypothesis. During each of the tensile tests described in Section 6.5.1, several diffraction images were acquired, at various applied strain states. For Specimen 1, the diffraction images, seen in Fig. 6.45, were acquired first before any load was applied to the CNT specimen, and then two images were acquired at approximately the same level of applied strain. From these diffraction patterns, the CNT can be identified as a single wall carbon nanotube, with one of two possible chiralities: either chirality \( (n, m) = (33, 23) \) with diameter \( D = 3.82 \) and chiral angle \( \theta = 24.11^\circ \), or chirality \( (n, m) = (36, 25) \) with diameter \( D = 4.16 \) and chiral angle \( \theta = 24.06^\circ \).

For Specimen 2, three images seen in Fig. 6.46 were acquired where the CNT strain was modified, either with a step up or step down of actuator power, during the image acquisition. For two of these diffraction patterns, the applied step did not appear to detectably modify the diffraction pattern. However, in one of the stepped diffraction patterns, several of the diffraction maxima appear to be shifted slightly.

Several additional diffraction patterns were applied with constant actuator power, with a series acquired of images recorded at generally increasing strain values (Fig. 6.47). Superimposition of these images shows small amounts of distortion of the electron maxima seen in the patterns. In particular, images 5 and 9 will not superimpose identically over each other if they are scaled proportionally in the X and Y directions. This observation could be explained by changes in the CNT strain. Initial chirality characterization indicates that the CNT Specimen 2 is a double-walled.

\[\text{Determination of chirality, diameter, and chiral angle for CNT Specimens 1 and 2 was made by M. Muoth at ETH-Zürich.}\]
carbon nanotube, with chiral angles $\theta_{\chi,1} = 13.9 \pm 0.1^\circ$ and $\theta_{\chi,2} = 19.0 \pm 0.1^\circ$.

The performance of the actuator may have shifted slightly during the exposure times, increasing the blur of the resulting diffraction images. Furthermore, systematic errors in image scaling due to modification of focus during the experiment with the second specimen prevent association of specific strain values with specific acquired diffraction images. However, the change in strain can be defined between two sets of image plates. The first set, marked with the points labeled “A” in Fig. 6.44, showed $\Delta \epsilon = 0.0109 \pm 0.0023$ between image plates 5 and 6. The second set, marked with the points labeled “B” in Fig. 6.44, showed $\Delta \epsilon = 0.1053 \pm 0.0023$ between image plates 9 and 10. As mentioned in Section 6.5.1, telescopic elongation of the CNT may be responsible for some of this observed strain, rather than deformation within the CNT macromolecular structure.

The collection of these TEM diffraction patterns demonstrates the following:

- Multiple types of TEM experiments are possible using the UTP and test coupon system.
- Mechanical actuation can be used in conjunction with TEM diffraction to explore the effects of the mechanical signal on a nanostructured material.
- TEM electron diffraction patterns appear to provide some observation of strain change effects on macromolecular structure.
Figure 6.45: TEM electron diffraction patterns taken during a tensile test performed on carbon nanotube Specimen 1. The image at top left shows a diffraction pattern recorded before the CNT was subjected to any loading. The two lower images show the diffraction pattern after slack was removed from the CNT. These two images are repeated exposures at a constant strain level.
Figure 6.46: TEM electron diffraction patterns taken during the tensile test performed on carbon nanotube Specimen 2. Upper Left (image plate 3): CNT was subjected to strain, and then the strain was increased slightly during the exposure of the diffraction imaging plate. No jump effect is discernable in this image. Upper Middle (image plate 4): CNT was subjected to strain, and then the strain was reduced slightly during the exposure of the imaging plate. A discontinuity in the pattern was visible at the top of this image, which is shown magnified in the lower image. Such a discontinuity is not visible in the other diffraction images taken of this CNT at constant strain. Upper Right (image plate 7): CNT strain was reduced slightly during the exposure of the imaging plate. No jump effect is discernable in this image. Lower Image: Magnification of the portion of the Upper Middle/plate 4 image showing the diffraction image discontinuity.
Figure 6.47: TEM electron diffraction patterns for CNT Specimen 2, progressing from left to right, top to bottom. Superimposition of these images shows small changes with increasing strain values. Top Left: plate 1. Top Right: plate 2. Middle Left: plate 5. Middle Right: plate 6. Bottom Left: plate 9. Bottom Right, plate 10.
6.6 Radio-Frequency Electrical Probing of GaN Nanowires on Universal Test Platform

Work in this section was performed in collaboration with Dr. Pavel Kabos and Dr. Mitch Wallis at NIST-Boulder. Dr. K. Bertness of NIST supplied the nanowire specimens, and A. Baca used dielectrophoretic self-assembly to place nanowires on the waveguide test coupons.

Part of demonstration of the Universal Test Platform requires demonstration of the universality of this microdevice. An additional setting for operation of this device is that of a radio-frequency probe station, which provides an example of operation in a normal, air-filled environment.

The transmission and reflection of radio-frequency (RF) signals through crystalline material specimens is affected by the anisotropic electron band structure and Brillouin zones of the material specimen. Microfabricated microwave waveguides such as microstrip or coplanar waveguide structures may be used to supply radio-frequency electrical signals to materials specimens. [18,67] The RF signal generated by an impedance analyzer may be transmitted to the microfabricated waveguide by means of an RF probe station. The chief distinction of an RF probe station from standard probe stations for microdevice analysis is that each signal probe is flanked on opposing sides by probes providing ground connections. By controlling the physical dimensions of the coplanar waveguide, an impedance may be specified that allows impedance measurements within the dynamic range of the impedance analyzer. An additional application of such waveguides is as the basis for a radio frequency scanning tunneling microscope.

The ability to strain nanomaterial specimens in combination with examination of radio frequency electrical transmission and reflection through the specimen may allow a new avenue to probe the effect of material strain on the electron structure of a material. Optical spectroscopy has provided one means to accomplish this; it is well known that strained crystals may experience shifts or broadening of optical absorbance peaks. RF transmission may provide a complementary analytical technique.
Creation of a coplanar waveguide device that can apply strain to a nanomaterial specimen in addition to allowing electrical interfacing to RF probes in a probe station may therefore be of interest from the standpoint of development of a new experimental technique. The UTP and test coupon designs provide one means to apply strain to a nanomaterial specimen. Unlike most integrated tester designs, the test coupon is of large enough size that it can accommodate a coplanar waveguide structure (requiring about 250 \( \mu \)m in overall width to allow the RF probes to contact ground planes on the waveguide. By patterning of metal on the top, electrically insulating surface of the test coupon designs, coplanar waveguide designs can be achieved (Fig. 6.48).

A Universal Test Platform chip was secured in a standard 20-pin ceramic DIP chip carrier, and wirebonds were used to provide electrical interfacing between the UTP chip and the chip carrier. In prior SEM work, a piece of breadboard was used to provide electrical interfacing between analog driver electronics and similar chip carrier packages. However, for the radio-frequency probe station available at NIST-Boulder, this presented too high of a profile on the stage, and the wiring interface was easily mechanically disturbed. For these reasons, a custom PC board (Fig. 6.49) was assembled to provide electrical interfacing between the wiring originating from driver electronics and the chip carrier holding the UTP chip.

Using metallized test coupons, probes from a standard probe station were contacted to the waveguide central conductors and supplied with a \( \sim 65 \) kHz signal. Following the dielectrophoretic methods reported above (Section 6.3), gallium nitride nanowires were deposited onto several test coupons, spanning the waveguide conductors on the tensile stage regions of the test coupons. Using the FIB-SEM, organic residue on the ends of the NWs was reduced, and Pt-C clamps were deposited on the ends of the nanowire specimens, making electrical contact with the waveguide center conductors.
Figure 6.48: Optical image of the coplanar waveguide on one of the fabricated test coupons. The pale yellow color indicates the presence of gold. The underlying red substrate is SiO$_2$ and the visible grey parts are Si.

Figure 6.49: A custom PC board prototype for holding the chip carrier in the RF probe station. The DIP switches connect to all the chip carrier pins through 1 MΩ resistors to a common connection to prevent charge accumulation (which is more important in SEM environments).
For RF measurement experiments, a test coupon chip was placed in the packaged UTP chip, and the structure supporting this package was then placed on the stage of the RF probe station (Figures 6.50 and 6.51). Using computer-based actuation control, the UTP probe was extended, nudging the back of the test coupon into contact with the motion stop structures on the UTP chip. RF probes were placed in contact with both ends of the waveguide structure on the test coupon (Fig. 6.52), and connected to an impedance analyzer which measured S-parameters for transmission and reflection in the frequency range from 100 MHz to 40 GHz.

Although S-parameter ($S_{11}$, $S_{12}$, etc.) transmission and reflection data was able to be obtained for a nanowire specimen, it was unclear that this represented a difference from background levels. More stringent calibration will be required before proceeding with further RF measurements.

The UTP probe was able to be actuated 40 $\mu$m in the laboratory environment in order to contact the test coupon (Fig. 6.53). Actuation beyond this level was not attempted, and will not be until the RF measurements are better calibrated.

Although this experiment has not yet yielded RF measurements in correspondence with nanowire strains, several results may still be noted:

- A coplanar waveguide has been created on a passive microsystem, the test coupon, and this has been interfaced to the actuated UTP.

- Dielectrophoretic self-assembly was able to place NW specimens across suspended coplanar waveguide center conductor structures.

- UTP operation may occur outside of a vacuum environment, and probe actuation of 40 $\mu$m or greater is possible as a mechanical output from this device.
Figure 6.50: Close-up view of RF measurement attempt. RF probes contact the top of the test coupon.

Figure 6.51: Setup for UTP operation in RF probe station at NIST-Boulder.
Figure 6.52: Microscope camera view of RF probes in contact with the coplanar waveguide. The probes are visible as the black, three-pointed objects on the left and right of this image. This image was acquired by photographing the RF probe station microscope camera monitor using a digital camera.

Figure 6.53: Microscope camera views of UTP probe actuation in the RF probe station. For distance scale, each notch plus tooth on the vernier is 10 \(\mu\)m. These images are slightly distorted because they were acquired by photographing the RF probe station microscope camera monitor using a digital camera. a.) Before actuation, displacement = 0 \(\mu\)m. b.) During actuation, displacement = 40 \(\pm\) 2 \(\mu\)m.
Chapter 7

Conclusions

This thesis developed methods and devices for mechanical testing of nanomaterials. An integrated microfabricated tester was generated and used to perform tensile tests on gallium nitride and carbon-based nanowires. The integrated tester design used electrothermomechanical actuation to achieve a compact footprint. Tests using these devices led to development of methods of analyzing image-based displacement data from tensile test microsystems, including uncertainty analysis. Furthermore, a new method of estimating the uncertainty in a constrained regression parameter was identified, allowing quantification of uncertainty in Young’s modulus values obtained from individual tensile tests.

In the Introduction, Chapter 1, it was posited that the separation of a specimen-holding test coupon from an actuated microfabricated test platform would allow data collection in varied experimental environments by reducing constraints on fabrication processes and on types of nanomaterial specimens that may be tested. This design hypothesis has been supported by the work performed in fabrication and operation of the Universal Test Platform and test coupon microsystems. In the generation of test coupons, it was shown that the fabrication process can be adapted to include or omit metallized layers. Test coupons with metallization were used to prepare GaN NW test specimens, and test coupons without metallization allowed the growth of carbon nanotubes across mechanically actuated test stages. It was demonstrated that the Universal Test Platform chip could mechanically interface to test coupons. Controllably actuated mechanical interfacing was accomplished in air and vacuum test environments.
Nanomaterials characterization microdevices have been designed, fabricated, and operated. These tools, both integrated mechanical testers and the test platform/test coupon system have been used to collect mechanical data from nanostructured materials including gallium nitride nanowires, composite nanowires, and carbon nanotubes.

7.1 Accomplishments

The work presented in this thesis has generated the following technical contributions to microsystem and nanosystem mechanical engineering:

**Force Estimation in MEMS:** A new approach to force estimation has been developed and published. [12]

**Nanomaterial Tensile Data:** New tensile data on GaN nanowires and CNT composite fibers has been obtained and published. [10]

- Repeated tensile tests indicate that single crystal gallium nitride appears capable of withstanding uniaxial strain at least 1% and as much as 4% strain and more than 6 GPa normal stress without failure.
- These tests may include some of the first reported tensile failures in GaN NWs.
- Bounds on the failure strength and the strain to failure for SiCNO-MWCNT NWs were obtained, in addition to characterization of the Young’s Modulus of these NWs.

**Nanowire Placement on Suspended Structures:** Dielectrophoretic self-assembly was used to place nanowire specimens across suspended coplanar waveguide center conductor structures. Using micromanipulator probe placement, integration of composite nanowires with active MEMS devices was demonstrated.

**Fabrication Processing:** Two new fabrication processes were developed, each containing unique approaches to microsystem fabrication.
• For the UTP fabrication process, a process allowing only frontside processing completely through an SOI wafer was created, with the concomitant development of a thick positive photoresist process for embedding and protecting device-layer MEMS parts during deep etching of a well through the substrate wafer.

• For the test coupon fabrication process, backside alignment marks were created by deep etching completely through the wafer from the frontside. This allowed alignment of frontside and backside patterns to accuracy better than \( \pm 10 \, \mu \text{m} \).

• For the test coupon fabrication process, the use of frontside and backside deep etching allowed the generation of suspended compliant structures without the requirement of \( \text{CO}_2 \) supercritical drying. A wafer-scale process for production of ready-to-use test coupon chips with suspended moving parts was demonstrated.

**UTP Probe:** A new MEMS manipulator system with integrated displacement sensing was developed and operated.

• Actuation of 40 \( \mu \text{m} \) or greater is possible as a mechanical output from this device.

• The UTP microsystem was operated in vacuum (TEM) and air environments.

• Tensile stage motion of 3 \( \mu \text{m} \) or greater was demonstrated within a TEM.

**MEMS Mechanical Interfacing:** A new type of microsystem packaging was demonstrated using non-permanent mechanical interfacing between two MEMS structures.

• With careful design of passive alignment elements, and active interfacing from the UTP probe, the test coupon chips were able to be aligned and manipulated by the UTP probe, and then subsequently replaced with different chips.

• Compressive interfacing of structures experiencing in-plane motion was demonstrated as an effective means for non-permanent mechanical interfacing to compliant structures on the test coupons.
Because of this design, it was possible to grow carbon nanotubes on test coupons without requiring that the UTP actuator be subjected to the CNT growth environment.

**CNT Mechanical Signaling:** Control signals generated at a computer interface were converted to mechanical signals experienced by multiple nanotube specimens.

**Carbon Nanotube Manipulation:** Large strains were applied to two CNT tensile specimens using the UTP and coupon system. For one specimen, snap-off/snap-on behavior was repeatedly observed. For the second specimen, a very large strain was obtained, probably indicative of telescopic CNT failure.

**Actuated Coplanar Waveguide:** A coplanar waveguide has been created on a passive microsystem, the test coupon, and this has been interfaced simultaneously to the actuated UTP and to a radio-frequency probe station.

**Misalignment of Rigid Specimens:** A set of formulas and analytical approaches for estimation of uncertainty in tensile tests due to misalignment of rigid specimens was developed and reported in a publication. [10]

**Regression Uncertainty:** A formula for propagation of stress and strain uncertainty to the uncertainty of the regression parameter of a constrained linear regression was created. This allows the Young’s modulus value obtained from an individual tensile test to be reported with an associated uncertainty value.

**Electron Diffraction of CNTs:** TEM diffraction data was obtained on variably strained carbon nanotubes, and may show strain-dependent effects in electron diffraction patterns.

**7.2 Publications**

The work presented in this thesis has generated the following peer-reviewed publications:
7.2.1 Peer-Reviewed Journal Publications


7.2.2 Conference Papers


7.3 Next Steps

There are many directions in which this work could be continued, enhanced and developed. This project achieved many milestones, including demonstration of radio-frequency interfacing to a movable coplanar waveguide, and straining of carbon nanotubes under observation within a transmission electron microscope. However, these achievements fell short of some of the ambitions for this project. These identify perhaps the most important short-term directions for further development:

In-Plane Displacement Sensing: Piezoresistive displacement sensing was incorporated in all designs, but the sensing design needs further development, including compensation for
temperature fluctuations. Mechanical interfacing and actuation for in-plane tensile tester motion were at the center of design development, and motion sensing was not explicitly needed so long as displacement and specimen length data could be obtained from imaging. Automated collection of displacement data will help to increase the speed and number of future materials tests.

Fabrication Processing: The fabrication process for the test coupons is time consuming due to the alignment hole deep etch, and better or options for backside-to-frontside alignment may improve the fabrication speed by removing the alignment well deep etch step. Methods to improve the test coupon yield should also be explored. Because the UTP devices are fabricated from a much more expensive starting wafer than the test coupons, the low yield of the UTP devices is unacceptable for bringing these devices into broader usage, even if it was sufficient to provide proof-of-concept first generation devices reported in this thesis. The chief source of UTP fabrication trouble is the frontside etch through the deep well used to hold the test coupons. The device layer devices would be better preserved if they were first coated with a protective layer before proceeding with the deep well processing.

MEMS Force Calibration: The tensile measurements reported above rely on constitutive material properties and spring constant calculations related to the measured dimensions of fabricated structures in order to estimate the forces applied to tensile specimens. A more direct measurement and measurement standard for micronewton-scale forces is needed in order to provide greater accuracy in reported fracture strength and elastic modulus measurements. Development of a force standard in the range of nanonewtons to millinewtons remains a significant challenge of its own. To some extent, the use of atomic force microscopy and laser vibrometer measurements were explored in the present work as mechanisms for force calibration of microsystems, but they were not developed to the point of yielding conclusions.

Data Processing Speed: The analysis of each microscale tensile test remains a slow process.
The use of computerized electrical measurement sped the rate of data acquisition. However, without computerized stage displacement measurement, a broader study of nanomaterial mechanical properties will continue to be a time-consuming proposition.

**Broader Studies:** At the outset of this thesis work, it appeared possible that within the scope of this work could be included the correlation of mechanical nanomaterials measurements with piezoresistivity, optical spectra, and information about nanomaterial structure. Because research focused on the performance of basic mechanical tests, and the development of operational apparatus, these additional experiments did not fit within the scope of this work. Furthermore, attempts at DC piezoresistive measurements of nanoscale materials usually resulted in vaporization of nanoscale specimens, indicating that further sophistication of measurement electronics may be needed before pursuing significant piezoresistivity studies.

The device characterization experiences also suggest the following short-term courses of action:

**Distance Calibration:** Structures for distance calibration standards are needed to reduce systematic errors between images taken in the TEM with slightly varying focus and magnification.

**Packaging Thermal Design:** The device packaging design requires some engineering for the specific application chosen for operation of the UTP. In the case of a TEM, some thermal engineering is required, as is a better mechanism to prevent loss of the test coupon chip during the TEM stage loading process.

**Specimen Clamping:** Clamping of CNT specimens requires further attention for tensile tests performed in a TEM. Verification of the clamping of the CNT is needed in order to obtain accurate strain values.
**Actuation:** Due to the large thermal transients suspected of interfering with measurements and displacement stability, other mechanisms for UTP device actuation should be reconsidered.

**Coupon Placement:** Although the test coupons were designed to be a size that could be handled with tweezers in a laboratory setting, placing the test coupons into the UTP structure was more difficult and imprecise than anticipated. The coupon can be removed by simply flipping the UTP upside-down and using gravity to pull out the test coupon. However, damage to the UTP and to test coupons could be prevented if a different means was devised to place the test coupons into the UTP.

On a longer time scale, there are many directions which could develop from this research project:

**Probe Systems:** The probe system created for the UTP demonstrates a large displacement output for a microfabricated device. Perhaps it could be used in other applications, such as experimentation with biological cells. Alternatively, perhaps the displacement amplification structure could be scaled down by several orders of magnitude to help create “large-displacement” nanoscale actuators using rigid macromolecular structures.

**Nanoindenter Force Calibration:** One preexisting tool that may provide a means of force calibration of mechanical tester microsystems is a nanoindenter. These have been used to perform tensile tests using compliant passive microfabricated structures [36], and similar experiments could be performed on test coupons, or to verify the actual force versus displacement versus input power behavior of an electrothermomechanical actuator.

**Force Calibration Using Nanowire Standards:** An additional method for calibrating measurements of forces in the range of micronewtons could make use of nanowire specimens as force standards. Gallium nitride nanowires are resistant to oxidation and generally exhibit good chemical stability. They can be synthesized as defect free structures, and techniques
for diameter control of these nanowires are improving. In comparison to other types of semiconductor or metal nanowires, single crystal gallium nitride nanowires may therefore be useful as specimens for force calibration of microscale tensile testers.

**Nanoscale Experiments:** The ability to mechanically manipulate and interact with multiple nanoscale specimens presents the opportunity to develop many routes for experimentation on strained materials specimens. Opportunities include piezoresistive and optical spectroscopy measurements. However, the UTP and coupon system could also be adapted to create lateral probe for field effect experiments, and there may be opportunities to be explored in cryogenic, corrosive, high-temperature, and radioactive environments.

**Nanofabrication:** The UTP and coupon system provides a means of manipulating small specimens, or at least moving a stage with nanoscale displacements. It is conceivable that such nanomanipulation capability could in combination with a materials deposition or etching process form the basis for a user-controlled system for fabrication of nanoscale structures.

### 7.4 Concluding Remarks

New actively operated actuated microdevices have been fabricated. These devices have been used to interact mechanically with nanostructured materials such as carbon nanotubes and gallium nitride nanowires, and tensile data has been obtained from these materials. The microsystems developed in this thesis may form a basis for new work in nanomaterials characterization, and microscale and nanoscale fabrication.
Bibliography


A.1 Thick Positive Photoresist

This is the general recipe for 30 - 70 μm thick positive photoresist.

(1) H₂O removed from surface
- hot plate 115 C, 5 minutes
- Or, time in O₂ asher

(2) Apply P20, 4000 rpm

(3) Low viscosity resist: 220-3
- cover whole wafer before spinning
- Spin on 2500 rpm
- Bake 5 mins

(4) Thick photoresist (PR) first layer 220-7
   (a) spin 280, wait 1 min
   (b) Spin up 1000 rpm
   (c) Wait 20 mins for leveling and diffusion
   (d) Bake 95°C, 2 mins
   (e) Bake 115°C, 2 mins
   (f) Remove edge bead
   (g) wait 2 h
   (h) Baking steps need further optimization. Minimize bubbles from too much solvent evaporation. Minimize cracking.

(5) Thick PR second layer 220-7
   (a) Spin on 2500
(b) Wait 5 mins, spin again
(c) Wait 5 mins
(d) Bake 95°C, 2 mins
(e) Bake 115°C, 2 mins
(f) After this bake, the PR layer is set up.

(6) Rehydration: wait > 11 h

(7) Exposure multiexpose, 5 cycles, 60 sec rest time

(8) Wait for reaction, 60 mins. Diffusion of N\textsubscript{2} out, H\textsubscript{2}O reaction with DMQ

(9) Develop MF 701

NO POSTBAKE. Keep wafer exposed to air as much as possible. Leave wafer compact ajar while rehydrating for 11 h.

A.2 Universal Test Platform (UTP) Fabrication Process

Last updated on February 22, 2010

(1) SOI wafer:
   (a) 500 µm <100> Si handle wafer, >1 Ω-cm
   (b) 1 µm silicon oxide layer,
   (c) 20 ± µm thick <100> doped Si device layer, p/B, 0.001-0.005 Ω-cm
   (d) All processing occurs on frontside with device layer.
   (e) Identify frontside (FS), mark backside (BS).
   (f) Use resistance probes to Identify FS. BS has oxide surface, and will not conduct, nor will it machine alignment marks.
   (g) Measurement in 2W setting clearly distinguishes between conducting and non-conducting surfaces
   (h) Watch out because probes will create surface damage, and also there can be native oxide that prevents measurement, so may need to jiggle wafer under probes a little

(2) Clean wafer
   (a) Sonicate in H\textsubscript{2}O, 10 minutes, fs down
   (b) Rinse with ISO, dry with N\textsubscript{2}
   (c) Spin ACE, ISO, MET, ISO (acetone, isopropanol, methanol, isopropanol)
   (d) Sonicate in ACE, 10 minutes, fs down
   (e) Rinse with ISO, dry with N\textsubscript{2}
   (f) Cycle once in rinser-dryer

(3) AXIC O\textsubscript{2} RIE Si\textsubscript{02_fast}, 5 minutes
(4) Pattern stepper alignment marks.

(a) Apply P20 and SPR 660 in autospinner spinner: “304” speed, 35 sec, 3.5 sec PR spread time. Clean autodispenser with swab before use, and reset spinner dispense time to 2.5 sec after use.

(b) Hot plate 95°C, 1 minute

(c) Stepper:
   (i) ALIGN mask
   (ii) job file “jbrown”
   (iii) level “pg”
   (iv) exposure 230 mJ/cm²

(d) Hot plate 110°C, 1 minute

(e) Develop using developer spinner 60 sec. MF26A developer.

(f) O₂ asher 5 minutes
   (i) Turn on switches on RH side back to front
   (ii) Turn on the 2 power switches
   (iii) Vent chamber
   (iv) Put chips on top of a new wafer in the front area
   (v) Vent off, solenoid on, vac to 0.15 Torr
   (vi) Turn on gas #1 (O₂)
   (vii) Operate with power toggle, see plasma. “50 W”
   (viii) Off: power, gas
   (ix) Vent on, then solenoid off
   (x) Turn off: solenoid to vent, then power, then front to back side valves

(g) DRIE “marks” process

(h) Clean off photoresist:
   (i) Solvents: ACE, ISO
   (ii) O₂ asher 5 minutes
   (iii) Marks should be clearly evident to the eye and easy to find under microscope. Cool appearance when viewed with dark field optics. ¹
   (iv) Rinse in wafer rinser-dryer

(5) LOR photoresist for Metal Pattern:

(a) Clean wafer in asher, 2 minutes

(b) Check LOR spinner to get 2500 rpm

(c) Use LOR 3A. Pour on and let sit a little. Cover the whole wafer.

(d) Spin 2500 rpm, 45 sec ²

¹ Note: Alignment marks must be clearly visible to the naked eye on the bare wafer before processing further. If necessary confirm by patterning an SPR-660 layer using the stepper. LOR patterning and metal deposit must be performed relatively close in time. The LOR will erode if stored for long times and give ragged edges on the patterns.

² Shield splatter from hot plate.
(e) Let sit 1 minute to relax edge bead
(f) Wipe off backside with EBR PG, dry with N₂
(g) Hot plate, 150 °C, 5 minutes
(h) Cool on metal block next to hot plate
(i) Wrap in foil
(j) Clean up and move on to Bay 3
(k) Apply SPR 660 in autospinner: 2500 rpm ("238" setting), 35 sec. Again, 3.5 sec PR spread time. NO P20.
(l) Hot plate 95 °C, 3 minutes
(m) Stepper:
   (i) 0300 mask
   (ii) job file “jbrown”
   (iii) level “metal”
   (iv) exposure 210 mJ/cm²
(n) Hot plate 110 °C, 5 minutes
(o) Develop using developer spinner 45 sec. MF26A developer. ³

(6) Metal evaporation (NIST ONO system): 15 nm Ti, 200 nm Au
Run “reference motors” recipe frequently to prevent mechanical problems. Always verify ok transfer.
(a) Start LL vent (Vent the load lock)
(b) Drop in wafer holder
(c) Start LL pump, and “Degas” after pump gets to mTorr region.
(d) “Run Recipe”: Plasma Clean Vacuum Fast: 5 minutes Ar plasma, 75 W
(e) Open SQS sigma
   (i) Edit>Process>choose process
   (ii) Edit process thicknesses and rates
        Ti 0.1500 kÅ, 1.0 Å/sec
        Au 2.0000 kÅ, 5.0 Å/sec
(f) “Start Sample Load”
(g) “Run Recipe” Ti, then Au
(h) Sample Unload
   Notes:
   Report errors to GCH, JWB
   Click on green boxes to make steps happen
   LRP=linear rack & pinion
   Elevation: 1=0, 2=1.22, up for clean
   Red=problem, green=ok, blue=recipe in process

³ Develop time works very well.
To abort the plasma etch, don’t press “Abort” just turn off the RF and wait for the cycle to end.

(7) Lift-off (LOR removal steps, 090616, originally from W. Altman):
   (a) Spin ACE, ISO, N$_2$ in Bay 4
   (b) Sonication for 30 minutes, Bay 0, Nano PG remover. Wafer is face down in beaker, held by PTFE spider
   (c) Rinse with H2O, ISO, dry with N$_2$
   (d) Spin ACE, ISO, N$_2$
   (e) 10 min sonication in ACE, fs down
   (f) Rinse ACE, ISO, N$_2$
   (g) Spin ACE, ISO, N$_2$
   (h) One cycle in the wafer rinser-dryer
   (i) Inspect. Check for ok metal pattern using microscope

(8) ECR nitride deposit
   (a) deposit ~ 100 nm of SiNx
   (b) Use ECR (PECVD) machine
   (c) Recipe 56 seconds “SiNx_rfcln” (~ 1.8 nm/sec)

(9) Device Layer Nitride Pattern with SPR-660
   (a) Autospin P20 and SPR-660
      (i) Prime the PR pump in order to get good coverage
      (ii) Set PR spin speed to “250” and set back to “300” when done, dispense time “3.5”
   (b) Hot plate 95°C, 1 minute
   (c) Stepper exposure:
      (i) 0100 mask
      (ii) job file “jbrown”
      (iii) level “device”
      (iv) exposure 275 mJ/cm$^2$
   (d) Hot plate 110°C, 1 minute
   (e) Developer spinner, 60 seconds, MF-26A
   (f) AXIC “jasnitride.prc”
      (i) Process until endpoint
      (ii) About 8 minutes
      (iii) Aim endpoint monitor into a well region

(10) Device Layer Silicon Pattern with 3 µm SPR220-3
    (a) Clean wafer surface
(i) Solvents: ACE, ISO
(ii) O₂ asher, 5 minutes

(b) Apply P20 in spinner: 4000 rpm, 40 s

(c) Apply SPR220-3 with manual spinner:
   (i) 2500 rpm, 40 sec for ~ 3 µm thickness
   (ii) Spread PR over the whole wafer before turning on the spinner.

(d) Hot plate 115 °C, 90 seconds

(e) Stepper:
   (i) 0100 mask
   (ii) job file “jbrown”
   (iii) level “device”
   (iv) exposure 275 mJ/cm²

(f) Develop using developer spinner 60 sec, MF26A

(g) Check in microscope

(h) DRIE:
   (i) Recipe “specbnod”
   (ii) For 25 µm device layer, use 23 – 32 cycles
   I seem to get my beam widths about 1 µm less than what I patterned
   Recipe “specbibo” 2 – 3 cycles to clean off last bits of Si in inconvenient areas,
   especially after oxide is about 2/3 exposed
   Make sure to check that Si is cleared out of bottom of fine feature
   Watch as oxide is cleared away, don’t use excess cycles.
   (iii) Clean off photoresist:
      (iii.a) Solvents: ACE, ISO
      (iii.b) O₂ asher 3 minutes
      (iii.c) Make sure to check that the wafer is clean

(11) Deep Well Patterning

(a) Attach to backing wafer using wax (CrystalBond 509 Wax, 115 °C preferred) on hot-plate. Clean up wax on wafer with ACE before proceeding. Don’t do anything else until the wafer is attached to the backing wafer. This step will create bubbles in the photoresist if performed after the photoresist deposit, expose, and develop steps

(b) Deposit thick (30 µm) photoresist and pattern for deep well etching
   For all photoresists in this step, start at low spin speed and then increase speed to stated value
   With thick photoresist, one key to avoiding bubbles is to slowly cover the wafer. Do not get coverage too quickly.
   (i) Apply P20 in spinner: 4000 rpm, 40 s
   (ii) Apply SPR220-3 with manual spinner:

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4 On February 5, 2010, this recipe was measured to produce photoresist layers with thickness 45 µm to 65 µm.
(ii.a) Spin 2500 rpm, 40 sec for \(\sim 3\) \(\mu\)m thickness
(ii.b) Spread PR over the whole wafer before turning on the spinner.
(ii.c) Hot plate 115\(^\circ\)C, 5 minutes

(iii) Apply SPR220-7 with 9 \(\mu\)m recipe

This step does a thick layer that embeds all the device layer structures in photoresist

The extra spinning and rest time helps to smooth out the layer

(iii.a) Pour on SPR220-7
(iii.b) Let rest 15 - 30 seconds
(iii.c) Spin 280 rpm, 15 seconds
(iii.d) Rest 45 seconds
(iii.e) Spin up: 1000 rpm, 40 sec
(iii.f) Wait 10 minutes
(iii.g) Spin: 1000 rpm, 40 sec
(iii.h) Wait 10 minutes
(iii.i) Clean up backside and edge
(iii.j) Hot plate: 95\(^\circ\)C, 2 min
(iii.k) Hot plate: 115\(^\circ\)C, 2 min
(iii.l) Spin 2500 rpm, use ACE to remove edge bead \(^5\)
(iii.m) Rehydrate 120 minutes or more

(iv) Apply SPR220-7 with 7 \(\mu\)m recipe

(iv.a) Pour on SPR220-7
(iv.b) Let rest a few seconds
(iv.c) Spin up: 2500 rpm, 40 sec
(iv.d) Wait 5 minutes
(iv.e) Spin: 2500 rpm, 40 sec
(iv.f) Wait 5 minutes
(iv.g) Clean up backside and edge
(iv.h) Hot plate: 95\(^\circ\)C, 2 min
(iv.i) Hot plate: 115\(^\circ\)C, 2 min
(iv.j) Rehydrate 11 hours or more

(c) Suss Contact Aligner exposure

(i) 0200 mask
(ii) Prog 7 with “MultiExpose” selected
(iii) Make sure to correctly orient label and wafer flat
(iv) exposure 48 seconds, 5 cycles, 30 seconds between cycle

(d) Wait: at least 60 minutes

(e) Moisten wafer before developing in order to prevent small bubbles

(f) Develop by hand, 6 – 15 minutes, MF-26A

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\(^5\) Edge bead removal works better here than after deposit of the thick photoresist. The next spinning step doesn’t make a big edge bead as a consequence, and also the photoresist seems to survive better overall. (100206)
(g) O$_2$ Ash, 5 – 7 minutes
(h) Remove oxide in wells using (RIE) AXIC:
   (i) “sio2_fast” until endpoint (25 nm/min oxide, 51 nm/min photoresist)
   (ii) Aim endpoint monitor into a deep well
   (iii) For 1 $\mu$m oxide, 45 min etch time.
   (iv) For 4 $\mu$m oxide, 180 min etch time.
(i) DRIE well with vertical sidewalls:
   (i) Recipe “specbnod”
   (ii) For 500 $\mu$m handle wafer, use 500 - 550 cycles (100119)

(12) Separate devices
   (a) Soak in ACE
      (i) Wafers face down, held by spider
      (ii) Devices will separate and fall to bottom of beaker
   (b) “Filter” devices with wiper, rinse with ACE, ISO
   (c) Sort and store

(13) Release devices:
   (a) Strip all photoresist
      (i) Solvents: ACE, ISO, MET, ISO
      (ii) dry
   (b) Etch for 6.5 minutes in concentrated (43%) aqueous HF
   (c) H2O rinse
   (d) MET:H2O 4:1 5 minutes
   (e) Hold in Methanol
   (f) Dry using supercritical CO2
   (g) Clean off remaining organic residues using 3 minutes of exposure to O$_2$ RIE

(14) AXIC nitride etch
   (a) In order to expose the metal pads
   (b) recipe “jasnitride.prc”
   (c) Process time $\sim$ 8 minutes

(15) Packaging:
   (a) Set in desired packaging
   (b) wirebond electrical connections to the pads on the test platform
A.3 Coupon Fabrication Process


(1) Si wafer, 380 \( \mu m \), DSP, \(< 100 >\)

Processing will occur on both frontside and backside. Side of processing will be noted below with “fs” or “bs.”

(2) Grow thermal oxide, 1 \( \mu m \).

Note: Lift-off resist (LOR) patterning and metal deposit must be performed on the same day. The LOR will erode if stored for long times and give ragged edges on the patterns. LOR must have 95°C bake immediately before exposure.

(3) LOR photoresist for Metal Pattern (positive resist, W. Altman’s recipe, 090604.):

(a) Clean wafer in asher, 2 minutes
(b) Check LOR spinner to get 2500 rpm
(c) Use LOR 3A. Pour on and let sit a little. Cover the whole wafer.
(d) Spin 2500 rpm, 45 sec
(e) Let sit 1 minute to relax edge bead
(f) Hot plate, 150°C, 5 minutes
(g) Cool on metal block next to hot plate
(h) Wrap in foil
(i) Clean up and move on to Bay 3
(j) Apply SPR 660 in autospinner: 2500 rpm (“238” setting), 35 sec, 3.5 sec PR spread time. NO P20.
(k) Hot plate 95°C, 3 minutes
(l) Suss contact aligner exposure
   (i) Program 5, exposure for 10.7 seconds.
   (ii) Pattern with mask UTP 003
(m) Hot plate 110°C, 5 minutes
(n) Develop using developer spinner 30 sec. MF26A developer.

(4) Metal evaporation (NIST ONO system): 15 nm Ti, 200 nm Au

(a) Start LL vent (Vent the load lock)
(b) Drop in wafer holder
(c) Start LL pump, and “Degas” after pump gets to mTorr region.
(d) “Run Recipe”: Plasma Clean Vacuum Fast: 5 minutes Ar plasma, 75 W
(e) Open SQS sigma
(i) Edit > Process > choose process
(ii) Edit process thicknesses and rates
   - Ti 0.1500 kÅ, 1.0 Å/sec
   - Au 2.0000 kÅ, 5.0 Å/sec
(f) “Start Sample Load”
(g) “Run Recipe” Ti, then Au
(h) Sample Unload
(i) Notes:
   (i) Report errors to GCH, JWB
   (ii) Click on green boxes to make steps happen
   (iii) LRP = linear rack & pinion
   (iv) Elevation: 1=0, 2=1.22, up for clean
   (v) Red = problem, green = ok, blue = recipe in process
   (vi) To abort the plasma etch, don’t press “Abort” just turn off the RF and wait for the cycle to end.

(5) Lift-off (LOR removal steps, 090616, Wendy):
   (a) Spin ACE, ISO, N₂ in Bay 4
   (b) Sonication for 30 minutes, Bay 0, Nano PG remover. Wafer is face down in beaker, held by PTFE spider
   (c) Rinse H₂O, ISO
   (d) Dry with N₂ gun
   (e) 10 min sonication in ACE, wafer face down
   (f) Rinse ISO, N₂ dry
   (g) Spin ACE, ISO, N₂
   (h) Process one cycle in wafer rinser-dryer
   (i) Inspect. Check for ok metal pattern using microscope

(6) Oxide Patterning with 3 µm PR
   (a) Clean surface for good bonding: O₂ asher 3 minutes
   (b) Apply P20 in spinner: 4000 rpm, 40 s
   (c) Apply SPR220-3 with manual spinner:
      (i) 2500 rpm, 40 sec.
      (ii) Spread PR over the whole wafer before turning on the spinner.
   (d) Hot plate 115°C, 90 seconds
   (e) Suss contact aligner exposure
      (i) Program 3, exposure for 30 seconds
      (ii) Pattern with mask COUP005
   (f) Develop using developer spinner 30 sec, MF26A
(g) Check in microscope

(h) AXIC RIE “Si02_fast” until endpoint: About 45 minutes for ~ 1 µm oxide

(i) Clean off photoresist:
   (i) Solvents: ACE, ISO
   (ii) O<sub>2</sub> asher 5 minutes

(7) Alignment Well Patterning

(a) Attach to backing wafer using wax: CrystalBond 509 Wax, 115 °C

(b) Deposit thick (7 µm) photoresist and pattern for deep well etching
   (i) Solvents: ACE, ISO
   (ii) Adhesion promoter: P20, 4000 RPM, 40 sec
   (iii) Pour on SPR220-7
   (iv) Let rest a few seconds
   (v) Spin: 2500 rpm, 40 sec
   (vi) Wait 5 minutes
   (vii) Hot plate: 95 °C, 1 min 40 sec
   (viii) Hot plate: 115 °C, 2 minutes
   (ix) Rehydrate 60 minutes or more

(c) Suss contact aligner exposure
   (i) Program 7, exposure time 48 seconds
   (ii) Mask is COUP0600

(d) Wait: at least 35 minutes

(e) Developer spinner, 90 seconds, MF26A

(f) Check in microscope

(g) DRIE 400 µm well with vertical sidewalls: “specbnod” 400 cycles

(8) Device Layer Processing with 1 µm PR

(a) Sonicate in acetone, 5 minutes, in order to remove wax from alignment wells

(b) Rinse ACE, ISO

(c) Spin ACE, ISO

(d) Apply P20 and SPR660 in autospinner
   (i) Prime the PR pump in order to get good coverage
   (ii) Set PR spin speed to “250” and set back to “300” when done

(e) Hot plate 95 °C, 1 minute

(f) Expose as normal for SPR 660
   (i) (Contact aligner channel 2, program 2, 7 sec)
   (ii) Pattern with mask COUP001

(g) Hot plate 110 °C, 1 minute

(h) Develop using spinner 60 sec, MF26A
(i) Check in microscope
(j) DRIE: 30 cycles, “specbnod”

(9) Deep Well Patterning

(a) Spin on SPR220-3 to fs and bake 2 min, 115°C
(b) Hot plate 115°C, separate wafers
(c) Wax onto new backing wafer, on hot plate, 115°C
(d) Attach coupon wafer fs to backing wafer fs
(e) Sonicate in acetone, 2 minutes, in order to remove wax from alignment wells
(f) Spin ACE, ISO to clean surface
(g) Deposit thick (7 µm) photoresist and pattern for deep well etching
   (i) Adhesion promoter: P20, 4000 RPM, 40 sec
   (ii) Pour on SPR220-7
   (iii) Let rest a few seconds
   (iv) Spin: 2500 rpm, 40 sec
   (v) Wait 10 minutes
   (vi) Hot plate: 95°C, 1min 40sec
   (vii) Hot plate: 115°C, 2 minutes
   (viii) Rehydrate 60 minutes or more
(h) Suss contact aligner exposure
   (i) Program 7, exposure time 48 seconds
   (ii) Mask is COUP0200
   (iii) Mask should have been printed with “invert data” box checked on pattern generator (PG)
   (iv) Put mask with mask label farthest away from where all the other mask labels are aligned
(i) Wait: at least 35 minutes
(j) Develop in spinner 90 seconds, MF26A
(k) Check in microscope
(l) AXIC RIE: “sio2_fast” until endpoint (40 - 45 minutes)
(m) DRIE 360 µm well with vertical sidewalls
   (i) Process “specbnod” 380 cycles.
   (ii) Stop when device pattern is evident.
   (iii) Endpoint must be carefully observed

(10) Separate and store coupon wafer:
(a) Soak in acetone until wafers separate (1 – 2 h?)
(b) Squirt ACE between wafers to accelerate separation
(c) Soak coup wafer alone in fresh acetone to clean
(d) Soak in isopropanol
(e) Air dry
(f) \( \text{O}_2 \) asher 10 minutes, or until clean
(g) Store coupon wafer and store backing wafer

A.4 File Conversion for Pattern Generator

Important: Rotated Instances are not allowed. To fix, flatten top cell before export from L-edit to gds.

Also, “wires” can sometimes cause errors. To remove these, select all, then Draw>Merge, then Draw>Convert>Fracture Polygons in L-edit.

To avoid other errors, do not use acute angles, and make sure all line segments are > 2 \( \mu \text{m} \).

Directions for Processing Reticle Files

1. In L-edit, export tdb file to GDSII (fracture if layout contains abnormal shapes and angles, 1 database unit = 0.001 \( \mu \text{m} \))
2. Use SSH to move gds file to grumpy server
3. Type “xic” and open file “A.gds” then select “save as”, hit <enter>. This saves a backup file.
4. Go to “Convert” > “Convert” and change file output formed to “xic”. Select “A.gds” file from dialog box.
5. Close xic, open xic, and open xic file (top cell). “Expand all”

Note: Filled in part is where chrome will be etched away.

Making a GDS file

1. Create your layout using xic using a scale of 1 \( \mu \text{m} \) = 1 lambda
2. From within xic (on grumpy) choose Attributes > Save Tech to save the xic_tech technology file.
3. Exit xic
4. From the command line on grumpy type “xictechmod s” to add stream numbers to your xic_tech file and to create a xic_tech.clay file for later.
5. Restart xic and load your file
6. Select Convert > Export Control and save your file in GDSII format.

Running Reticle
(1) On grumpy (but in the same directory as above) run either
(2) Reticle myfile.gds for 4 inch contact aligner masks, or
(3) reticle5 myfile.gds for 5 inch stepper masks
(4) Select the desired options for layers, inversions, etc.
(5) Click on either Make PG files to have one layer run at a time or Batch PG Files to have them all made in one pass
(6) Exit reticle. You should now have file s of the formname_layer.int
(7) “Delete intermediate files”? yes

Notes:
Check layers using xic, then return to reticle to upload files to PG.
GDS # shows up in hexadecimal XX00, e.g. 40 = 2800
For stepper, choose ASML-5000, die width 11 mm, die height 11 mm
4 in: 1.1 Tool Suss S, 1.2 die width 75, 1.3 die height 75, barcode, name

Pattern Generator (PG) Notes  
Use 90° angles when possible. Need to play with settings in reticle.
PG min flash, min 1.5 \( \mu \text{m} \), typ 2 \( \mu \text{m} \)
Turn off overlap (improves significantly)
Flash combiner: combine flashes = yes, Error 0.1
Cats parameters, layer by layer adjust
Resolution 0.1, PG error 0.1, Grow 0.0
Don’t select rotated fracture or mirror
PG aperture needs occasional calibration with test pattern
CATS parameters: Vertex alignment of desired and fractured pattern typ 0.01 \( \mu \text{m} \), res-box alignment resolution 0.001 \( \mu \text{m} \) typ, pg min flash – min aperture size for boxes. Min = 1.5 \( \mu \text{m} \)
Rotated fracture and overlap are ok unless the reticle program halts.

---

6 From J. Britton, 9/24/08.
Appendix B

Some Additional Interesting Images

Figure B.1: SiO$_2$ layer on Si from a test coupon structure, with anisotropic etching evident in the top SiO$_2$ layer due to time in the O$_2$ asher or the DRIE system. Image taken by M. Muoth at ETH-Zürich using e-beam writer SEM.
Figure B.2: Silicon nitride with small Au patterns after exposure to unknown etch process.
Figure B.3: Silicon nitride with small Au patterns after exposure to unknown etch process.
Figure B.4: Early test coupon prototype using SOI wafer for fabrication substrate. Si has been cleared from both sides of the embedded oxide, which subsequently buckled due to internal stress.
Figure B.5: SEM image of part of a polysilicon thermal actuator after thermal failure of the actuator.
Appendix  C

Force Calibration in Suspended Microfabricated Structures

Many of the existing approaches for determining the force applied to a system do so by measuring the displacement of a well-characterized spring. In the context of force measurement using MEMS structures, many approaches can be designed for displacement measurement, but knowledge of the spring constant of a given structure is required in order to extrapolate a force from a measured displacement.

One method for determining some information related to the lumped spring constant of a system is the measurement of a resonant frequency $f_0$. The natural frequency of a lumped system provides information about the ratio of a spring constant and the oscillating mass, according to Eq. C.1.

$$f_0 = \frac{\omega_0}{2\pi} = \frac{1}{2\pi} \sqrt{\frac{k}{m}}$$  \hspace{1cm} (C.1)

This equation can be rewritten as Eq. C.2 to provide an approximation of $k$ if $m$ is known. Here, $m$ is calculated from the density and volume of masses that are subjected to oscillation.

$$k = m\omega_0^2 = m(2\pi f_0)^2$$  \hspace{1cm} (C.2)

The resonant frequency can be observed for MEMS structures with $f_0$ below several MHz using a vibrometer. As the vibrometer scans through driving frequencies, a laser interferometer
component of the vibrometer observes the magnitude of the vibrational displacement of suspended parts.

At ETH-Zürich, a Polytec vibrometer (Figure C.1) was available for data collection. Using this device, a set of resonant frequency measurements was performed on an array of suspended microfabricated structures. The goal of this work was to find experimental $f_0$ from which $k$ could be calculated and compared to other methods of derivation of $k$ for a given suspended structure.
Figure C.1: Polytec vibrometer system (ETH-Zürich) which was used to observe resonant frequencies of suspended structures. The tested chip is placed in a chamber with vacuum maintained by the pump seen at the foreground.
C.1 Experiment

Test chips were fabricated using the PolyMUMPS process and suspended parts were released using HF etching and CO$_2$ supercritical drying. Initial vibrometer measurements observed the in-plane motion of polysilicon masses suspended from bending beam springs of varying sizes. The test chips were placed in a vacuum chamber pumped to several mTorr pressure, and located under the vibrometer microscope as seen in Figure C.2. As seen in Figure C.3, the chips were clamped so that the in-plane motion could be observed from the vibrometer optics, and the chips could be supplied with vibrations from a piezoelectric chip that was scanned through a range of driving frequencies. A laser was aimed at suspended parts during scans through defined frequency ranges, and resonance data recorded. The location of a given resonant peak was determined using a Gaussian fit to the peak that was present due to the suspended structure. In cases where multiple peaks were observed, spectral comparison between the suspended structures and the nearby substrate was used to observe if a peak vanished when examining a substrate only. Such a peak would be defined then as the main resonant mode of the suspended structures. Resonant spectra were recorded several times, and the results averaged to find $f_0$. The uncertainty of the resonant frequency was in this case determined using a Type A evaluation. The values of $f_0$ and $u(f_0)$ are reported in Table C.1.
Figure C.2: Polytec vibrometer system (ETH-Zürich) in operation. The display at center shows the view through the microscope optic. The arrow indicates a suspended structure viewed edge on, used for resonant frequency observations.
Figure C.3: Test chip in the vacuum vibrometer system. The arrow indicates the location of the 2 mm square test chip.
C.2 Results

Table C.1: Resonant frequencies for the designs measured with the Polytec vibrometer.

<table>
<thead>
<tr>
<th>Design</th>
<th>Sample Size $N$</th>
<th>Measured $f_0$ (kHz)</th>
<th>$u(f_0)$ (kHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>d1-1</td>
<td>3</td>
<td>11.4</td>
<td>0.7</td>
</tr>
<tr>
<td>d1-2</td>
<td>5</td>
<td>298</td>
<td>5.5</td>
</tr>
<tr>
<td>d1-3</td>
<td>5</td>
<td>267.4</td>
<td>4.9</td>
</tr>
<tr>
<td>d2-1</td>
<td>4</td>
<td>93.8</td>
<td>4.6</td>
</tr>
<tr>
<td>d2-2</td>
<td>3</td>
<td>213</td>
<td>37</td>
</tr>
<tr>
<td>d2-3</td>
<td>4</td>
<td>26.2</td>
<td>1.1</td>
</tr>
</tbody>
</table>

For several devices, the vibrometer was operated to record vibration spectra without adding driving modes from the piezoelectric actuator. In these cases, resonant peaks were still observed for many devices, as evident in Figure C.4.
Figure C.4: Vibrometer data gathered from 3 different suspended structures in vacuum with no applied driving perturbations. Resonant peaks are visible at the right of each frequency spectrum in the left hand column, clearly standing out from the noise spectra.
Appendix  D

Reporting Mechanical Measurements Obtained with MEMS Systems

When reporting data collected through experimental measurements, a statement of the measurement uncertainty values should be included as part of the measurement data. Texts on measurement statistics provide conflicting approaches for the reporting of measurement uncertainty and the propagation of uncertainty to derived quantities. This Appendix summarizes a general methodology for uncertainty analysis in MEMS measurement systems based on international guidelines for reporting measurements.

The ISO guidelines for reporting uncertainty in measurement provide a framework for reporting the uncertainty in experimental data. [59, 61] Guidelines from the US National Institute of Standards and Technology (NIST) and the UK Accreditation Service provide additional clarity. [142, 144–146] From these documents, all systematic uncertainties are included in measured data and a combined standard uncertainty value is established for each measured quantity. For measurement results that are derived from data that is measured directly, the uncertainties in the measured data are propagated into a standard uncertainty for the derived measurement result.

D.1 Type A vs. Type B Measurements

As mentioned above in Section 2.3, the ISO standard distinguishes between Type A and Type B measurements. This is a different type of distinction than systematic versus random error. Type A measurements are those where a population of measurements can be collected, allowing derivation of statistical data such as expectation value and standard deviation from that population.
For Type B measurements, repeated measurements are not possible, so it becomes necessary for
the observer to define bounds on the measurement and the probability distribution of the measured
value existing within those bounds. Type B uncertainties also typically include systematic error
estimates, and tolerances of measurement equipment.

D.2 Procedure

For recording uncertainty in measurements and measurement results, a procedure can be
derived from the ISO guidelines and the additional American and British comments. For this
procedure, raw data is collected with a measurement instrument. Examples include the distances
measured from an SEM image, the voltages measured with a multimeter, and the temperature
measured with a thermometer. In all of these cases, an observer is required to interact with a piece
of instrumentation to record data.

D.2.1 Data Measurement

(1) Consider the equipment providing the data that will be collected. Identify the following:

(a) The type of measurement (A or B) that will be collected

(b) Sources of uncertainty. This includes the tolerances of the measurement equipment,
and errors in calibration of the equipment.

(c) The probability distribution function of these sources of uncertainty.

(2) The measurement $x_i$ is recorded with the uncertainty in the measurement given as $u_m$.

(a) For Type A measurements of $n$ independent samples $x_{i,k}$, record the measurement
value as the average value over the $n$ samples. The measurement uncertainty $u_m$ is
the standard deviation of the mean of these samples. The number of degrees of freedom
$\nu_i$ of the measurement is defined in most cases according to Eq. D.3, and provides
information about how well the uncertainty $u_m$ estimates the actual uncertainty. [142]
\[ x_i = \bar{x}_i = \frac{1}{n} \sum_{k=1}^{n} x_{i,k} \]  
(D.1)

\[ u_m(\bar{x}_i) = \sqrt{\frac{1}{n} \left( \frac{1}{n-1} \sum_{k=1}^{n} (x_{i,k} - \bar{x}_i)^2 \right)} \]  
(D.2)

\[ \nu_i = n - 1 \]  
(D.3)

(b) For Type B measurements, record the measurement value \( x_i \), with bounds \( x_i \pm a \), and a measurement uncertainty based on what is known about the bounds and the probability distribution function. The degrees of freedom value \( \nu_i \) for Type B uncertainty evaluations is usually taken to approach infinity.

Examples:

In the case where the observer is 100% sure that the measurement value is somewhere between \( \pm a \), with no further information available about the likely value of the measurement, a square probability distribution function is assumed. This gives \( u_m(x_i) \) as follows:

\[ u_m = \frac{a}{\sqrt{3}} \]  
(D.4)

For cases where a measurement can be defined with 95% confidence between \( \pm a \), with a normal distribution of probability around the measured value the measurement uncertainty \( u_m \) is defined according to Eq. D.5.

\[ u_m = \frac{a}{2} \]  
(D.5)

Systematic and instrument uncertainties should be recorded in the same way as these Type B measurements.
(3) For each measurement, add the measurement uncertainty plus instrument uncertainties using the sum of the squares of the uncertainties to get the combined standard uncertainty \( u_c \). If the measurement calibration is unknown, then a large tolerance is needed. So, if measurement bounds are \( \pm b \),

\[
   u_t = \frac{b}{\sqrt{3}}, \quad u_c = \sqrt{u_m^2 + \frac{b^2}{3}}
\]

If the measurement calibration is known, record the data with the calibration included, but add the uncertainty of the calibration.

- Do not include in \( u_c \) the sources of uncertainty like the measurement resolution which are automatically included in \( u_m \) just by virtue of taking a measurement.
- Do use measurement resolution to check the value obtained in the measurement. If the uncertainty value is less than the resolution, use uncertainty from the resolution.

\[
   u_0 = (\text{interval length})/2
\]

(4) Report collected data as discussed below in Section D.3.

(5) Use standard combined uncertainties \( u_c \) in calculations of uncertainties in derived measurement results.

D.2.2 Propagation of Uncertainty in Measurement Results

A measurement result derives from mathematical manipulation of measured values. [31] In order to find the uncertainty of a measurement result, the uncertainty must be propagated from various uncertainty components \( u(x_i) \). To do so, measurand \( y \) is defined as a function of input variables \( x_i \).

\[
   y = f(X) = f(x_1, x_2, \ldots, x_i)
\]

The first order Taylor series approximation of \( y \) around \( \bar{X} = \{\bar{x}_1, \bar{x}_2, \ldots, \bar{x}_i, \ldots, \bar{x}_N\} \) gives
the following:

\[
\Delta y = (y - \bar{y} | \mathbf{x} = \bar{x}) \approx \sum_{i=1}^{N} \frac{\partial f}{\partial x_i} (x_i - \bar{x}_i)
\]

(D.7)

The variance in \(\bar{y}\) is the expectation value of the square of \(\Delta y\), when \(f\) and the partial derivatives of \(f\) are evaluated at the expectation values for the various \(x_i\).

\[
(\Delta y)^2 = (y - \bar{y} | \mathbf{x} = \bar{x})^2 \approx \left( \sum_{i=1}^{N} \frac{\partial f}{\partial x_i} (x_i - \bar{x}_i) \right)^2
\]

(D.8)

\[
= \sum_{i=1}^{N} \left( \frac{\partial f}{\partial x_i} \right)^2 (x_i - \bar{x}_i)^2 + 2 \sum_{i=1}^{N-1} \sum_{j=i+1}^{N} \frac{\partial f}{\partial x_i} \frac{\partial f}{\partial x_j} (x_i - \bar{x}_i) (x_j - \bar{x}_j)
\]

(D.9)

\[E (x_i - \bar{x}_i)^2 = u^2 (x_i)\] and \(E (x_i - \bar{x}_i) (x_j - \bar{x}_j)\) gives the covariance between \(x_i\) and \(x_j\). The result for the uncertainty of \(y\) is then given by Eq. D.10. [59]

\[
u^2 (y) = \sum_{i=1}^{N} \left( \frac{\partial f}{\partial x_i} \right)^2 u^2 (x_i) + 2 \sum_{i=1}^{N-1} \sum_{j=i+1}^{N} \frac{\partial f}{\partial x_i} \frac{\partial f}{\partial x_j} u (x_i) u (x_j) \rho_{ij}
\]

(D.10)

The last term in Eq. D.10 is used when variables are correlated. It tends to reduce the overall value of uncertainty. If all of the variables are independent, then this equation can be truncated to Eq. D.11. When \(f\) is simply the arithmetic addition of a number of quantities, as is the case when calibration values are added to a measurement reading, the partial derivatives reduce to one, and the combined uncertainty of the measurement is simply the square root of the sum of the squares of individual uncertainty estimates.

\[
u^2 (y) = \sum_{i=1}^{N} \left( \frac{\partial f}{\partial x_i} \right)^2 u^2 (x_i)
\]

(D.11)

The number of effective degrees of freedom \(\nu_{eff}\) of \(u(y)\) is typically calculated using the “Welch-Satterthwaite” formula, Eq. D.12, where the uncertainty contributions \(u_i(y)\) are defined as in Eq. D.13 by the uncertainty \(u(x_i)\) of particular sources of uncertainty \(x_i\) multiplied by an associated sensitivity coefficient. [142]
\[ \nu_{eff} = \sum_{i=1}^{N} \frac{u_i^4(y)}{\nu_i} \]  
\[ u_i(y) = \left| \frac{\partial f}{\partial x_i} \right| u(x_i) \]  

(D.12)  
(D.13)

In summary, the standard uncertainty of the measurement result \( y \) is obtained using partial derivatives evaluated at the expectation values for a measurement, and the uncertainties of those measurements. There can be several equally acceptable variations to this strategy, and occasionally it is necessary to consider derivations using terms from the second order Taylor approximation. What is described here should be taken as an initial view to this problem. Some situations may require deeper analysis, in particular those with nonlinear functions or with unusual probability distribution functions.

### D.3 Reporting Data

All data recorded should also be recorded with a discussion of how the measurement and the uncertainty in the measurement were obtained. In general, each measurement should be recorded with the following information included:

- The measurement value
- A value for standard combined uncertainty
- Units for the measurement and uncertainty
- Degrees of freedom in the measurement
- The type of measurement should be recorded
- For Type B measurements, the probability distribution function should be recorded
- For correlated variables, the correlation coefficients should be reported
If it is desirable to place the measurement within a specified confidence interval, an expanded
uncertainty $U$ should also be reported. $U$ is typically the product of a coverage factor $k$ multiplied
by the standard combined uncertainty for a measurement. The coverage factor $k$ is most typically
the Student $t$ parameter. For infinite degrees of freedom and 95% confidence, $k = t_{\infty,95} \approx 2$.

The ISO and NIST guidelines recommend preparation of uncertainty budgets as a means to
convey the detailed calculations of the uncertainty components in a measurement result. Appendix
E provides an example of uncertainty budget calculations.

**D.3.1 Plus/Minus**

It is not always convenient or necessary to report data with the level of detail used in un-
certainty tables. [59] Frequently, data is seen reported as a value ± an interval with a range of
confidence. In this case, the text should still explain the coverage factor within the interval, and
the confidence level estimated for the given data. The text should still clearly describe the method
by which measurement and its uncertainty were obtained, and the units should still be defined for
the measurement. For example:

"$Y = 36.006 \pm 0.022 \ \mu m$"

"The reported expanded uncertainty is based on a standard uncertainty multiplied by a coverage
factor of $k = 2$ [or something else if $\nu$ is small], providing a level of confidence of $\sim 95\%$."

The ISO guidelines [59] Section 7 provides more examples of reporting data. The key consid-
eration is that there be a clear report of the method by which the measurement was obtained, and
that a combined standard uncertainty is reported which can be used in subsequent calculations by
a reader of the report.
D.3.2 Significant Figures

Generally, the guidelines recommend using not more than two significant figures in the reported uncertainty, with the comment that sometimes it is useful to retain 3 significant figures during some calculations. [59] The standard uncertainty value should be rounded to two significant figures, but there is no general guidance as to whether it should always be rounded up. The reported measured value can retain as many significant figures as is needed to be bounded by the reported uncertainty value.

D.3.3 Random Error vs. Systematic Error

Many authors draw a distinction between random error, which involves variations within a set of measurements, and systematic errors, which reflect a repeated bias in a measurement. Generally, a systematic error can be corrected with a calibration. It is generally important to work with calibrated instruments because a measurement may be precise but not accurate. So, the random error in an uncalibrated measurement may not reflect the true uncertainty in the measurement. The ISO guidelines request that all measurements be performed on calibrated instruments, but they also recognize that occasionally this is not possible. When a calibration cannot be performed, a much larger uncertainty is included in the measurement. For calibrated measurements, an output value is defined in response to an instrument reading. In this case the result of a measurement is offset according to the relation defined by the calibration, and uncertainty propagates accordingly, as shown in the following equations:

\[ x_i = m_i x_i' + b_i \] (D.14)

\[ u^2(x_i) = m_i^2 u^2(x_i') + u^2(b_i) \] (D.15)

When equipment calibration cannot be performed, the observer can usually make a judgment as to the magnitude of possible deviation from the measured value, and this judgment can be used in a Type B evaluation of uncertainty.

The ISO guidelines do not provide much guidance for cases where calibrations cannot be
performed. They do state, however, that systematic errors should be added and propagated just like any other error, using the root sum of squares summation of uncertainties (Eq. D.11). This is easiest performed if the systematic error is defined as existing with equal probability between two bounds separated by interval $2a$, in which case Eq. D.4 can be used to provide the uncertainty. If the probability density function for a systematic error can be defined beyond the bounded, flat probability, and if the systematic error dominates the combined uncertainty in a measurement, then error propagation becomes more complicated.

An alternative view of uncertainty analysis and propagation within a measurement adds uncertainties with the following formula [119]:

$$u(y) = \sum_{i=1}^{N} \left| \frac{\partial f}{\partial x_i} \right| u(x_i)$$  \hspace{1cm} (D.16)

This formula provides an upper bound on the uncertainty in a measurement. This summation is generally avoided in the ISO guidelines, in part because it overestimates the measurement uncertainty and it does not provide a standard uncertainty for propagation to derived measurements. For further discussion please refer to sections E.3 – E.5 in the ISO guidelines. [59]

D.4 Resources

The references listed below are helpful in clarifying the statistical approaches required for reporting measurement results. In particular, Reference [59] establishes guidelines for reporting, and Reference [90] provides a more detailed discussion of curve fitting and application of statistical analysis to measured data.


Appendix E

Microscale Tensile Testers Example Uncertainty Calculation

This appendix provides a set of detailed uncertainty budget tables illustrating the calculation of uncertainty in stress and strain for one tensile test data point. Specifically, the calculation is performed for the uncertainty values of the final data point before tensile failure of the polymer-derived ceramic carbon nanotube composite nanowire Specimen 1 from Section 6.4. This data point is reported in Fig. 6.27(a), and as the failure stress and strain seen in Table 6.5.

The tables presented below illustrate the uncertainty calculation method described in Appendix D. Furthermore, the uncertainty budget format allows a detailed examination of the relative importance of various sources of uncertainty in the results from MEMS test systems, and specifically the integrated test systems used for the PDC-CNT tests.

These uncertainty calculations were performed according to the ISO method, with uncertainties reported as 68% confidence values. Because of the large amount of information presented, the uncertainty budget tables are broken into pairs of tables. In the first table in each pair, the uncertainty of specific variables or sources of uncertainty is calculated. In the second table of each pair, calculations are provided for the stress or strain uncertainty contribution due to each uncertainty source.

The specific data point for which these sources are calculated was originally reported (in Section 6.4) as strain $\epsilon = 0.0212 \pm 0.0035$ and stress $\sigma = 0.63 \pm 0.30$ GPa. The strain uncertainty calculation in Tables E.1 and E.2 reproduces the strain uncertainty value reported in Section 6.4. The stress uncertainty calculated in Tables E.3 and E.4 is slightly less than the value reported in
Section 6.4 (i.e., $u_c(\sigma) = 0.28$ GPa vs. $u_c(\sigma) = 0.30$ GPa, respectively). The reason for this is that the original calculation used cross-section area uncertainty $u(A)$, which was rounded up when calculated and subsequently propagated into the stress uncertainty. In Tables E.3 and E.4, the specimen diameter uncertainty $u(D)$ is used rather than the area uncertainty $u(A)$, and use of the diameter uncertainty leads to the slightly smaller $u_c(\sigma)$ value.

The strain is calculated on fiber gauge length $L_0 = 7.783 \pm 0.019 \mu$m. The corresponding stress value is 630 MPa, which is the additional loading applied to a fiber after straightening. The total stress including the force required to straighten the fiber is 740 MPa. The uncertainty in the force and stress must be calculated using the full force applied to a specimen, so $\sigma = 740$ MPa is used to calculate the parameters in the Tables E.3 and E.4.

The effective degrees of freedom $\nu_{eff}$ value is reported with the stress uncertainty based on calculation using Eq. D.12, in which the Type A uncertainty evaluations limit the overall value for $\nu_{eff}$. Because the degrees of freedom for Type B evaluations approach infinity ($\nu_i \rightarrow \infty$), $\nu_{eff}$ for the strain uncertainty also approaches infinity and is therefore not reported in Table E.2.

The calculations in Tables E.1 – E.4 incorporate the most relevant sources of uncertainty. As these sources are reduced with future work, other laboratory environmental variables may start to impact the uncertainty calculations. For the moment, it is mostly distance measurement uncertainties which dominate the stress and strain uncertainty results. The 31% stress uncertainty due to test stage displacement measurements provides the starkest example. Other sources of uncertainty such as the polysilicon elastic modulus and the test device dimension uncertainties may be supplanted by a more direct calibration measurement of the test device spring constant or output forces.
Table E.1: Strain uncertainty budget Part 1: Sources of strain uncertainty.

<table>
<thead>
<tr>
<th>Uncertainty Component, $x_i$</th>
<th>Standard Uncertainty Calculation</th>
<th>$u(x_i)$ Value</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gauge Length Uncertainty</td>
<td>$\frac{a(L)}{\sqrt{3}} = 0.019\mu m$</td>
<td>$\pm 2\text{ pixels} = \pm a(L)\mu m = 0.033\mu m$, Type B, square</td>
<td></td>
</tr>
<tr>
<td>Misalignment</td>
<td>$</td>
<td>\epsilon_m - \epsilon_a</td>
<td>= 1.9E - 5$</td>
</tr>
<tr>
<td>Bending</td>
<td>$\frac{3D\sigma\tan \theta}{EL_0} = 5.3E - 4$</td>
<td>Eq. 6.15 and Section 6.3.3.6</td>
<td></td>
</tr>
</tbody>
</table>

Table E.2: Strain uncertainty budget Part 2: Strain uncertainty calculation.

| Uncertainty Component, $x_i$ Value | $u(x_i)$ Calculation | $|\frac{\partial \epsilon}{\partial x_i}|$ Value | Contribution to $u(\epsilon)$ | $u_i(\epsilon)$ Value | Percent, $\frac{u_i(\epsilon)}{\epsilon}$ |
|-----------------------------------|----------------------|-----------------------------|-----------------------------|-----------------------|------------------------|
| Gauge length uncertainty          | $0.019\mu m$         | $\frac{\sqrt{L^2 + L_0^2}}{L_0} = 0.1836\mu m^{-1}$ | $u_L(\epsilon) = 3.5E - 3$ | 13%                   |
| Misalignment                      | $1.9E - 5$           | 1                           | $u_{misalign}(\epsilon) = 1.9E - 5$ | 0.072%                |
| Bending                           | $5.3E - 4$           | 1                           | $u_{bending}(\epsilon) = 5.3E - 4$ | 2.0%                  |
| Combined Standard Uncertainty $u_c(\epsilon)$ | $u_c^2(\epsilon) = \sum u_i^2(\epsilon) = 1.25E - 5$ | $u_c(\epsilon) = 0.0035$ | $\frac{u_c(\epsilon)}{\epsilon} = 13\%$ |
Table E.3: Stress uncertainty budget, Part 1: Sources of stress uncertainty.

<table>
<thead>
<tr>
<th>Uncertainty Component, $x_i$</th>
<th>Standard Uncertainty</th>
<th>$u(x_i)$ Value</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber diameter, $D$</td>
<td>$\frac{a(D)}{\sqrt{3}}$</td>
<td>0.01 $\mu$m</td>
<td>$\pm a(D) = \pm 0.02$ $\mu$m, Type B, Square</td>
</tr>
<tr>
<td>Cross-section shape variation</td>
<td>$\sigma \left</td>
<td>\frac{A_{\text{actual}} - A}{A} \right</td>
<td>$</td>
</tr>
<tr>
<td>Stage separation, $x$</td>
<td>$\frac{a(x)}{\sqrt{3}}$</td>
<td>0.039 $\mu$m</td>
<td>$\pm 4$ pixels = $\pm a = \pm 0.068$ $\mu$m, Type B, square</td>
</tr>
<tr>
<td>Specimen rotation</td>
<td>$\sigma \left</td>
<td>\left( \cos \theta \right)^{-1} - \left[ \frac{1 - \sin^2 \theta}{(1 + \epsilon)^2} \right]^{-\frac{1}{2}} \right</td>
<td>$</td>
</tr>
<tr>
<td>Bending</td>
<td>$\sigma \frac{3D \tan \theta}{L_0}$</td>
<td>0.015 GPa</td>
<td>Eq. 6.15</td>
</tr>
<tr>
<td>Misalignment</td>
<td>$\sigma \left</td>
<td>1 - (\cos \theta)^{-1} \right</td>
<td>$</td>
</tr>
<tr>
<td>Polysilicon elastic modulus, $E_{ps}$</td>
<td>$u(E_{ps}) = \frac{14}{2}$</td>
<td>7.0 GPa</td>
<td>$E_{ps} = 162 \pm 14$ GPa, Normal</td>
</tr>
<tr>
<td>Lateral device dimensions</td>
<td>$u(dims) = \sqrt{u_p^2 + u_m^2}$ = 0.12 $\mu$m</td>
<td>$u_p$(pixels): Type B, square. $u_m$(measured): Type A, normal.</td>
<td></td>
</tr>
<tr>
<td>Device thickness</td>
<td>$u(t) = \sqrt{u_p^2 + u_m^2}$ = 0.14 $\mu$m</td>
<td>$u_p$(pixels): Type B, square. $u_m$(measured): Type A, normal.</td>
<td></td>
</tr>
<tr>
<td>Variance of $d_0 = f(P)$ regression fit</td>
<td>$\frac{k}{A} \left[ \frac{\sum(y_i - \bar{y})^2}{N-2} \right]^\frac{1}{2}$</td>
<td>0.064 GPa</td>
<td>$N = 14$</td>
</tr>
<tr>
<td>Actuator voltage sampling</td>
<td>$u_s(V) = \left( \frac{\text{Var}(V)}{N} \right)^\frac{1}{2}$</td>
<td>$4.00E - 6$ V</td>
<td>Normal, Type A, $N = 1000$</td>
</tr>
<tr>
<td>Actuator current sampling</td>
<td>$u_s(I) = \left( \frac{\text{Var}(I)}{N} \right)^\frac{1}{2}$</td>
<td>$1.11E - 5$ A</td>
<td>Normal, Type A, $N = 1000$</td>
</tr>
<tr>
<td>Resistor used to determine current</td>
<td>$\frac{a(R)}{\sqrt{3}}$</td>
<td>11 $\Omega$</td>
<td>Square, Type B, $a(R) = 386 \Omega \times (\pm 5%)$</td>
</tr>
</tbody>
</table>
Table E.4: Stress uncertainty budget, Part 2: Calculation of stress uncertainty.

| Uncertainty Component, $x_i$ | $u(x_i)$ Value | Sensitivity Coefficient, $\left| \frac{\partial \sigma}{\partial x_i} \right|$ | Contribution to $u(\sigma)$ Value, $u_i(\sigma)$ | Percent, $\frac{u_i(\sigma)}{\sigma}$ |
|-------------------------------|----------------|---------------------------------|-------------------|------------------|
| Fiber diameter, $D$ | 0.01 $\mu$m | $\frac{2\sigma}{D} = 6.4$ GPa/µm | 0.064 GPa | 8.6% |
| Cross-section shape variation | 0 | 1 | 0 GPa | 0% |
| Stage separation, $x$ | 0.039 $\mu$m | $\sqrt{\frac{2k}{A}} = 5.9$ GPa/µm | 0.23 GPa | 31% |
| Specimen rotation | 0.020 GPa | 1 | 0.020 GPa | 2.7% |
| Bending | 0.015 GPa | 1 | 0.015 GPa | 2.0% |
| Misalignment | 0.019 GPa | 1 | 0.019 GPa | 2.6% |
| Polysilicon elastic modulus, $E_{ps}$ | 7.0 GPa | $\left| \frac{d_0 - d}{A} \right| \left| \frac{\partial k}{\partial E_{ps}} \right| = 0.0045$ GPa | 0.032 GPa | 4.3% |
| Lateral device dimensions | 0.13 $\mu$m | $\left| \frac{d_0 - d}{A} \right| \left| \frac{\partial k}{\partial (\text{dims})} \right| = 0.88$ GPa/µm | 0.11 GPa | 15% |
| Device thickness | 0.14 $\mu$m | $\left| \frac{d_0 - d}{A} \right| \left| \frac{\partial k}{\partial t} \right| = 0.21$ GPa/µm | 0.029 GPa | 3.9% |
| Variance of $d_0 = f(P)$ regression fit | 0.064 GPa | 1 | 0.064 GPa | 8.6% |
| Actuator voltage sampling | $4.00E-6$ V | $\frac{k}{\frac{\beta_1}{A} + 2\beta_2 V I} I = 0.832$ GPa/V | 3.33 kPa | 0.00% |
| Actuator current sampling | $1.11E-5$ A | $\frac{k}{\frac{\beta_1}{A} + 2\beta_2 V I} V = 152$ GPa/A | 1.69 MPa | 0.228% |
| Resistor used to determine current | 11 $\Omega$ | $\frac{k}{\frac{\beta_1}{A} + 2\beta_2 V I} \frac{V I}{\frac{\beta_1}{A} + 2\beta_2 V I} = 5.33E-3$ GPa/Ω | 0.0586 GPa | 7.92% |

Combined Standard Uncertainty $u_c(\sigma)$: $u_c^2(\sigma) = \sum u_i^2(\sigma) = 0.0795\text{ GPa}^2$

\[ u_c(\sigma) = 0.28\text{ GPa} \]

\[ \frac{u_c(\sigma)}{\sigma} = 38\% \]

Effective Degrees of Freedom, $\nu_{eff} = 4100$
In order to explore the opportunity to improve measurement precision with better calibration, the stress uncertainty budget developed above was re-examined with Type B uncertainty sources removed. The strain uncertainty contains only Type B evaluation, so it was not recalculated. Most of the distance measurements used in calculating $u_c(\sigma)$ required Type B uncertainty estimates. The device dimension uncertainties were originally calculated in Table E.3 as a combination of pixel uncertainty (a Type B evaluation) and repeated measurements performed on images of typical devices (a Type A evaluation). For Table E.5, only the Type A uncertainties were included. The polysilicon Young’s modulus uncertainty was kept in this calculation because it reflects results from a previously reported measurement experiment (Ref. [127]). Sources of systematic error were otherwise omitted from the $u_c(\sigma)$ calculation in Tables E.5 and E.6.

The result is that random errors that were able to be quantified using Type A evaluations provide a stress uncertainty of $u_c(\sigma) = 0.078$ GPa, or 11%. This is significantly less than the $u_c(\sigma) = 0.28$ GPa, or 38%, found in Table E.4. This indicates that significant improvement in measurement precision may be possible with further improvements in distance resolution and minimization of systematic errors.
Table E.5: Stress uncertainty budget, Part 1: Type A uncertainties.

<table>
<thead>
<tr>
<th>Uncertainty Component, ( x_i )</th>
<th>Standard Uncertainty Calculation</th>
<th>( u(x_i) ) Value</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polysilicon elastic modulus, ( E_{ps} )</td>
<td>( u(E_{ps}) = \frac{14}{2} = )</td>
<td>7.0 GPa</td>
<td>( E_{ps} = 162 \pm 14 ) GPa, Normal</td>
</tr>
<tr>
<td>Lateral device dimensions</td>
<td>( u(dims) = u_m(N = 8) = )</td>
<td>0.032 ( \mu )m</td>
<td>( u_m(\text{measured}): ) Type A, normal.</td>
</tr>
<tr>
<td>Device thickness</td>
<td>( u(t) = u_m(N = 7) = )</td>
<td>0.062 ( \mu )m</td>
<td>( u_m(\text{measured}): ) Type A, normal.</td>
</tr>
<tr>
<td>Variance of ( d_0 = f(P) ) regression fit</td>
<td>( \frac{k}{N} \left[ \frac{\sum(y_i - y)^2}{N-2} \right] = )</td>
<td>0.064 GPa</td>
<td>( N = 14 )</td>
</tr>
<tr>
<td>Actuator voltage sampling</td>
<td>( u_s(V) = \left( \frac{\text{Var}(V)}{N} \right) = )</td>
<td>4.00( E - 6 ) V</td>
<td>Normal, Type A, ( N = 1000 )</td>
</tr>
<tr>
<td>Actuator current sampling</td>
<td>( u_s(I) = \left( \frac{\text{Var}(I)}{N} \right) = )</td>
<td>1.11( E - 5 ) A</td>
<td>Normal, Type A, ( N = 1000 )</td>
</tr>
</tbody>
</table>
Table E.6: Stress uncertainty budget, Part 2: Calculation of $u_c(\sigma)$ due to Type A uncertainties only.

| Uncertainty Component, $x_i$ | $u(x_i)$ Value | Sensitivity Coefficient, $\left| \frac{\partial \sigma}{\partial x_i} \right|$ | Contribution to $u(\sigma)$ Value, $u_i(\sigma)$ | Percent, $\frac{u_i(\sigma)}{\sigma}$ |
|-----------------------------|----------------|---------------------------------|-----------------------------|--------------------------|
| Polysilicon elastic modulus, $E_{ps}$ | 7.0 GPa | $\frac{d_A - d_A}{A} \left| \frac{\partial k}{\partial E_{ps}} \right| = 0.0045$ | 0.032 GPa | 4.3% |
| Lateral device dimensions | 0.032 $\mu$m | $\frac{d_A - d_A}{A} \left| \frac{\partial k}{\partial \text{dims}} \right| = 0.88$ | 0.028 GPa | 3.8% |
| Device thickness | 0.062 $\mu$m | $\frac{d_A - d_A}{A} \left| \frac{\partial k}{\partial t} \right| = 0.21$ | 0.013 GPa | 1.7% |
| Variance of $d_0 = f(P)$ regression fit | 0.064 GPa | 1 | 0.064 GPa | 8.6% |
| Actuator voltage sampling | $4.00E-6$ V | $\frac{k}{4} \left[ \beta_1 + 2\beta_2 VI \right] I = 0.832$ | 3.33 kPa | 0.00% |
| Actuator current sampling | $1.11E-5$ A | $\frac{k}{4} \left[ \beta_1 + 2\beta_2 VI \right] V = 152$ | 1.69 MPa | 0.228% |

Combined Standard Uncertainty $u_c(\sigma)$: $u^2_c(\sigma) = \sum u^2_i(\sigma) = 0.00607$ GPa$^2$

$u_c(\sigma) = 0.078$ GPa

$\frac{u_c(\sigma)}{\sigma} = 11\%$

Effective Degrees of Freedom, $\nu_{eff} = 24$