



Microelectromechanical Systems (MEMS)-Based Testing of Materials

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Abstract

Mechanical behavior of micro- and nanoscale materials has received considerable attention in recent years because of their widespread use in micro-/nanotechnology applications. These materials are also intriguing from a scientific standpoint because their small-size scale results in mechanical behavior that is significantly different from the behavior of macroscale materials. As a result, a variety of experimental methodologies have been developed to accurately determine the mechanical properties (modulus, strength, fracture toughness, etc.) of micro- and nanoscale materials and uncover the microscopic mechanisms that lead to those properties. Among these approaches, microelectromechanical systems (MEMS)-based platforms have proven to be highly suitable because of their capability to apply and resolve extremely small forces (nN) and displacements (nm). In addition, MEMS-based testing platforms, because of their small size, are ideal for in situ characterization in electron and scanning probe microscopes, which often have stringent space limitations. This chapter provides an overview of the development and advances in MEMS-based materials characterization with an emphasis on in situ techniques. Different actuation and sensing mechanisms as well as device configurations for various types of testing (tensile, fatigue, thermomechanical) are reviewed. Key results and insights obtained from the nanomechanical characterization of thin films, nanowires, and nanotubes using MEMS-based platforms are summarized. Finally, some of the challenges and opportunities for MEMS-based micro- and nanoscale materials characterization are discussed.

Keywords

MEMS · Mechanical testing · Thin films · Nanowires · Stress-strain response

1 Introduction

The mechanical and physical properties of micro- and nanoscale materials have been a subject of tremendous research interest since the late 1990s. This interest has been primarily driven by two factors. Micro- and nanostructures are the basic building blocks for a wide range of nanotechnology applications including flexible and stretchable electronics and energy conversion devices, and an accurate determination of their properties is essential to predict the reliability, lifetime, and performance of such devices. Another technological trend that has focused attention on nanoscale materials is the rapid miniaturization of microelectronic devices and data storage systems, where the dimensions of the smallest features are of the order of nanometers. Apart from technological considerations, micro- and nanoscale materials are also highly intriguing from a scientific standpoint. The dimensional and microstructural constraints in these materials as well as their high surface to volume ratio leads to mechanical properties that are distinct and, in many cases, superior to those of bulk materials. In particular, the deformation and failure mechanisms of micro- and nanoscale materials are significantly different from their macroscopic counterparts.

At the macroscale, the most common method to characterize the mechanical behavior is the uniaxial tensile test as it provides a wide spectrum of properties (modulus, yield and failure strength, strain hardening) and is most straightforward to interpret. However, performing uniaxial tensile tests on nanoscale materials is extremely challenging because of the small-size scale of the specimens, and these challenges manifest in all three facets of testing: specimen preparation and handling, actuation, and measurement. For example, measuring the uniaxial stress-strain response of a freestanding nanoscale thin film specimen typically requires sub- μN force resolution and sub- μm displacement resolution. The resolution requirements are even more stringent for nanowires and nanotubes, which are usually only a few μm long. Similarly, the handling, gripping, and alignment of thin film specimens for conducting a uniaxial tensile experiment are highly nontrivial tasks. Therefore, initial efforts to determine the mechanical properties of thin films focused on nanoindentation and wafer curvature measurements, which had fewer technical hurdles. In a similar spirit, for one-dimensional nanostructures such as nanowires and nanotubes, existing experimental techniques such as atomic force microscopy or nanomanipulation were adapted to perform bending, tension, or buckling experiments to measure mechanical properties.

While the abovementioned methods are capable of measuring some of the key mechanical properties (modulus, yield and fracture strength, etc.), they have one significant drawback – none of the methods is well suited to explore and quantify the microscopic mechanisms responsible for the changes in mechanical behavior at the nanoscale. Uncovering the deformation mechanisms in such materials requires in situ mechanical testing inside an electron or scanning probe microscope. Such analytical instruments, however, have small chamber sizes and hence significant miniaturization of the testing setup is required. Motivated by these requirements, microelectromechanical systems (MEMS)-based testing platforms have been increasingly used for nanoscale materials characterization. MEMS-based platforms offer a tiny footprint and can integrate sensors and actuators required to apply and measure forces and displacements with nanonewton and nanometer scale resolution, respectively. In addition, they offer tight tolerances that ensure repeatability of measurements and the ability to customize the setup for different types of mechanical testing as well as materials.

This chapter provides an overview of the development and advances in MEMS-based mechanical testing of micro- and nanoscale materials and is organized in the following manner. In Sect. 2, the most common actuation and sensing mechanisms used in MEMS testing platforms are reviewed. The challenges involved in specimen preparation and handling are discussed in Sect. 3. Next, device configurations for tensile testing that use a combination of these actuation and sensing schemes and specimen handling techniques are described in Sect. 4. In Sect. 5, MEMS platforms for fatigue and thermomechanical characterization of micro- and nanoscale materials are outlined. A few key results and mechanistic insights that have emerged from MEMS-based materials characterization are briefly discussed in Sect. 6. Finally, some of the challenges and opportunities for MEMS-based micro- and nanoscale materials characterization are discussed in Sect. 7.

Given the explosion in MEMS-based materials characterization techniques in recent years, a comprehensive review of the subject is outside the scope of this chapter. Rather, the chapter is meant to introduce the reader to the technological hurdles in micro- and nanomechanical testing, how MEMS-based platforms have been leveraged to address these hurdles and the outstanding issues that still remain. Wherever appropriate, the readers are referred to more extensive reviews that focus on one or more specific aspects of MEMS-based micro- and nanomechanical characterization.

2 Applying and Sensing Force and Displacement

At the macroscopic scale, tensile tests are typically performed on dog-bone-shaped specimens with a circular or rectangular cross section that are machined from a billet or a component. The specimens are gripped at the two ends and deformed by a servo hydraulic or electro mechanical actuator, which applies a controlled amount of deformation at a specified rate. The force on the specimen is obtained from a load cell, whereas the strain in the gage section of the specimen is measured using strain gages, extensometers, or optical measurement techniques. This setup does not translate easily to micro- and nanoscale testing because of the small specimen sizes and the minuscule forces and displacements that need to be applied and measured. As a result, many alternative actuation and sensing mechanisms have been implemented in MEMS-based testing platforms.

2.1 Actuation Mechanisms

MEMS testing platforms can be actuated externally using motorized or piezoelectric actuators or can be actuated using internal mechanisms. For on-chip actuation, the most widely used mechanisms are electrostatic and thermal actuation because of their easy integration with standard microfabrication processes. In the case of electrostatic actuation, the most common configuration is a comb drive mechanism, which consists of a series of interdigitated fingers that are actuated by applying a voltage (V) between them [1]. Figure 1 shows the simplified layout of a linear comb drive actuator/displacement sensor [2]. In a comb drive, the force generated by each set of fingers parallel to their length is given by $F = \epsilon h V^2 / d$, where ϵ is the dielectric constant of the medium (typically air or vacuum), h is the depth, and d is the perpendicular distance between the fingers. As the formula indicates, the force is independent of the overlap between the fingers and is constant for a given voltage. These actuators have a reasonably large travel range ($>10 \mu\text{m}$), but because of the large voltages (50 V or higher) required, pull-in instability of the comb structure can occur. Pull-in instability refers to the condition where electrostatic forces in the transverse direction (vertical direction in Fig. 1) become unbalanced and cause neighboring electrodes (fingers) to come into contact.

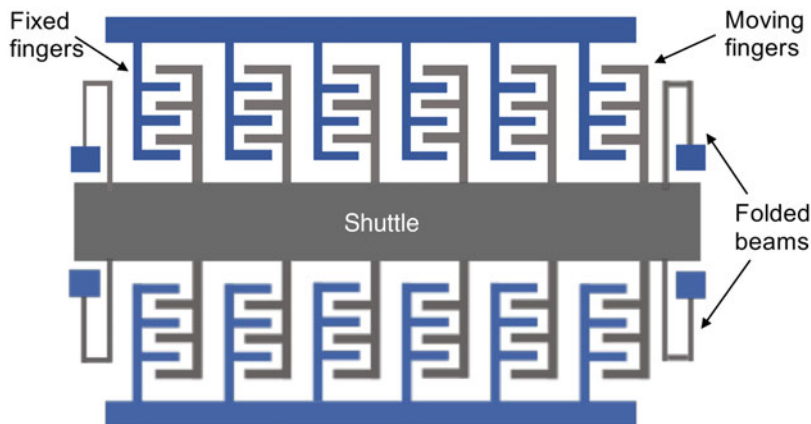


Fig. 1 Schematic of a comb drive actuator (Adapted from [2]). When a voltage is applied between the fixed and moving fingers, the shuttle is displaced horizontally and this motion can be used to deform a specimen connected to the shuttle

Thermal actuation [3–5] offers a more stable actuation method but with a typically smaller displacement range (of the order of $1\ \mu\text{m}$). In this actuation method, a DC current is passed through slender beams that are connected to a shuttle (Fig. 2a, b). The resistive heating of the beams leads to thermal expansion along the length of the beams. Because of the symmetric arrangement of the beams, this thermal expansion is converted to the linear motion of the shuttle, which is used to deform the specimen. Depending on the configuration of the beams, the actuator can be relatively compliant (Z-shaped beams) or stiff (V-shaped beams), and the force generated can vary from tens of μN to several mN . Cascaded thermal actuators [6] have been developed (Fig. 2c) that provide both large displacement (tens of μm) and large force (tens of mN). Other actuation mechanisms used in MEMS include piezoelectric and shape memory actuation.

2.2 Force/Displacement Sensing Mechanisms

Similar to actuation, several methods have been developed for measuring the specimen force and deformation at the micro- and nanoscale. For force measurement, beam flexure-based systems offer the most versatility because the transverse stiffness of the beam (proportional to t^3) can be varied over a large range by altering the thickness (t). The force is obtained by measuring the transverse deflection of the beam and multiplying it by the beam stiffness. To sense the deflection, a variety of displacement sensing techniques can be used. The simplest method is optical sensing, whereby markers on the beam (or connected structures) are imaged and the changes in displacement are extracted by tracking their motion. This method, however, requires post-processing and hence cannot provide real-time measurement of force and displacement. In contrast, capacitive sensing provides real-time

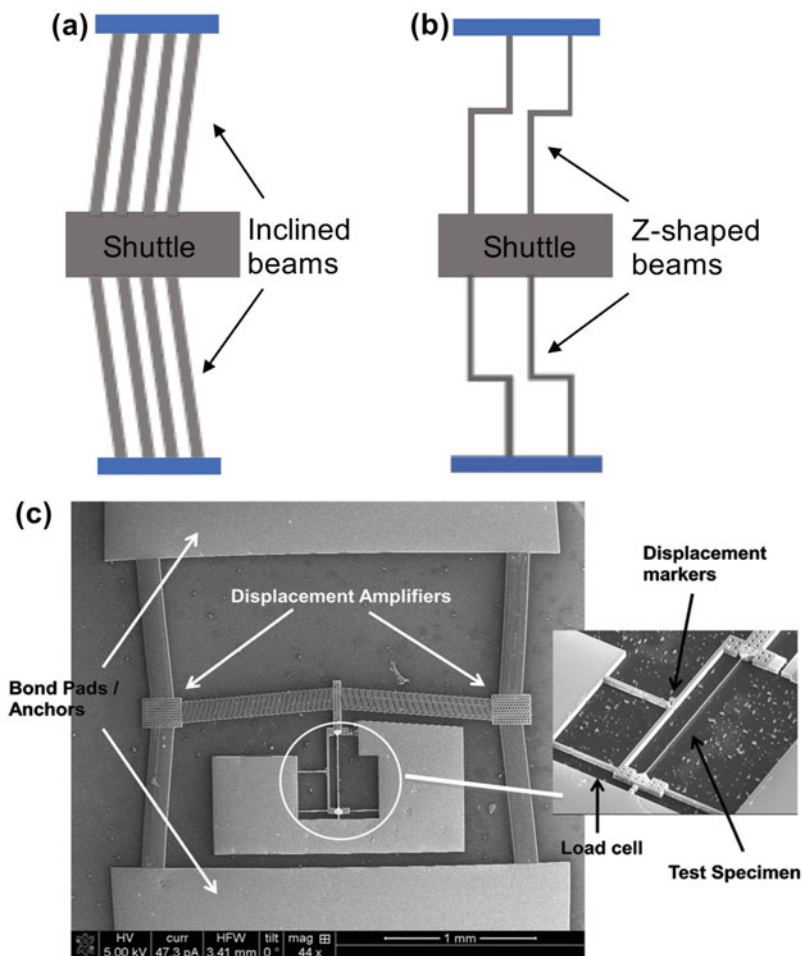


Fig. 2 (a–b) Schematics of a V-shaped thermal actuator composed of inclined beams and Z-shaped thermal actuator (Adapted from [2]). (c) A cascaded thermal actuator [6] composed of two sets of horizontal and one set of vertical V-shaped beams. The vertical V-shaped beams amplify the displacement of the horizontal V-shaped beams (Reprinted with permission from IOP publishing)

information and is the most popular method for displacement sensing [7]. In this method, the displacement/deformation of a structure results in the change in capacitance of an electrical circuit that is coupled to the structure. By measuring the change in capacitance, the displacement/deformation of the structure is deduced. In MEMS testing platforms, differential capacitance sensors rather than absolute capacitance sensors are used because the absolute capacitance changes are small and difficult to measure. Parasitic and stray capacitances often complicate the relationship between the sensor displacement and capacitance changes, but commercial

sensing modules can be used to mitigate these issues. Other displacement sensing techniques used in MEMS platforms include piezoelectric and piezoresistive sensing. In piezoelectric sensing, the displacement/deformation of the MEMS structure induces a strain (stress) in the piezoelectric sensor, which, in turn, leads to generation of electric charge. The sensor is part of an electrical circuit, the voltage output of which changes with the charge generated and is used to infer the displacement/deformation. Piezoresistive sensors work on a similar principle, except that the displacement/deformation of the structure causes a change in electrical resistance instead of electric charge generation.

2.3 Strain Measurement

The displacement sensing techniques discussed above can also be used to measure the specimen deformation or strain. For example, Haque and Saif [8] used optical measurement of the gap change between two gages connected to the ends of a thin film specimen to obtain the nominal strain in the specimen. Others have used capacitive sensing to measure the relative displacements of the ends of nanowires, nanotubes, and thin film specimens subjected to deformation and thus obtain the strain. While these techniques are adequate for the measurement of 1D strain, it is sometimes advantageous to obtain full-field measurements of the complete 2D strain tensor [9] using digital image correlation (DIC) and differential digital image tracking (DDIT). DIC relies on locating the maximum correlation coefficients (with sub-pixel accuracy) that represents the optimal fit between prescribed regions on consecutive images of the specimen. The strain in each region is extracted from pixel displacements that maximize the correlation coefficients. DDIT uses a peak-fitting algorithm to detect intensity peaks from pre-existing features on the specimen surface with even higher resolution. These methods allow the measurement of Poisson's ratio and, more importantly, the distribution of strain in the specimen.

3 Sample Preparation and Handling

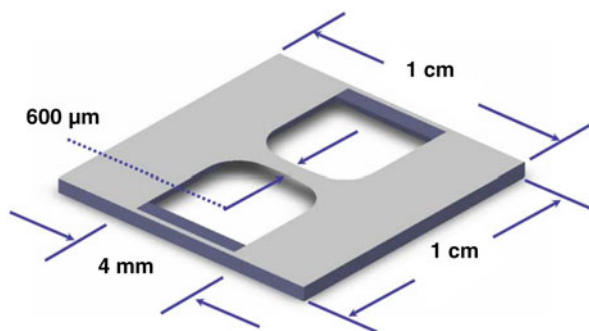
Specimen preparation, manipulation, alignment, and gripping for mechanical testing at micro- and nanoscale pose several challenges that do not exist at the macroscale. Macroscale specimens can be machined to required dimensions with traditional cutting tools, and commercially available polishing tools can be used to obtain the necessary surface finish. Such tools do not exist for manufacturing micro- and nanoscale specimens. Similarly, while hand or robotic manipulation is sufficient to transfer macroscale specimens to the testing rigs, such methods can easily damage fragile micro- and nanoscale specimens. To overcome these challenges, researchers have come up with a variety of specimen handling approaches for mechanical testing of micro- and nanosized specimens. These approaches can be broadly classified into the following three categories.

3.1 Specimen Integration with Larger-Scale Structures

In these schemes, the specimen is integrated with a larger-scale structure that is stiff enough to be manipulated directly with hand or using tweezers. This technique has been used to test freestanding thin films made of metals, alloys, semiconductors, and ceramics [10, 11]. The freestanding film specimens are obtained by a combination of additive (film deposition) and subtractive processes (etching of substrate or sacrificial layers). The specimens are anchored at one or both ends on the substrate to facilitate gripping and application of load/displacement. Fig. 3 shows a schematic of a nanocrystalline Al thin film specimen [12] anchored at both ends on a 500- μm thick silicon die. The support strips are cut before testing so that the load is applied only on the freestanding specimen. The planar dimensions of the die (10 mm by 10 mm) are large enough to be handled with tweezers, and the structure can be integrated with commercially available motorized stages and load cells for actuation and force measurement. The sample strain is usually measured using markers deposited on the specimen using optical techniques such as digital image correlation or interferometry.

While the use of support structures helps with the handling of micro- and nanoscale specimens, it is even more advantageous to co-fabricate the specimen and the testing apparatus together. Such an approach is particularly suited to thin film materials synthesized using physical or chemical vapor deposition because they can be readily incorporated into the MEMS fabrication processes. This approach circumvents the problems associated with the gripping and alignment of freestanding samples, which can cause significant measurement errors. In addition, the method allows batch processing of tens of nominally identical samples from a single wafer, which is essential to verify the repeatability of the measurements. Saif and coworkers used this approach to develop the first MEMS-based tensile testing device with co-fabricated metallic film specimens as thin as 30 nm [8]. However, the range of materials that can be co-fabricated with MEMS testing stages is fairly limited, which necessitates more general approaches as described in the following sections.

Fig. 3 Schematic of an aluminum thin film [12] integrated with a larger silicon die for tensile testing (Reprinted with permission from Elsevier)



3.2 Pick and Place Methods

These methods use micro- and nanomanipulators with needlelike probes to transfer specimens that have been synthesized separately onto MEMS testing stages. This is the most common method to manipulate whiskers, nanotubes, and nanowires for tensile testing. Because the largest dimensions of such materials are no greater than a few micrometers, locating and manipulating them also requires simultaneous high-resolution imaging. Hence, these micro- and nanomanipulators are often incorporated into a scanning electron microscope (SEM), transmission electron microscope (TEM), or dual-beam scanning electron microscope-focused ion beam (SEM-FIB) systems.

The SEM-FIB systems are particularly appealing because they can be used to attach, transfer, and test micro- and nanoscale samples in the same chamber [13]. The in situ attachment is typically done by bringing the nanomanipulator probe into close proximity with the sample and locally injecting and decomposing a precursor gas using electron or ion beam. Residual hydrocarbons present in the chamber can also be decomposed instead of the precursor gas. The decomposition results in the deposition of the desired material, which welds the probe and sample together. The sample is then transferred to the desired location in the MEMS device and welded using the same process. Finally, the probe is detached from the sample before testing using the ion beam. In some cases, the metal deposition has been directly used to grip nanowire specimens for tensile testing using nanomanipulators. However, it is important to ensure that the grips are strong enough to avoid permanent deformation during testing as this will lead to artifacts in the strain measurement. In addition to locating and attaching nanoscale specimens, the FIB can also be used to machine nanoscale samples in situ. Thus, it is possible to conduct the entire gamut of operations from specimen preparation to testing in a single setup and without exposure of the specimens to the outside environment. An example of manipulation of tin whiskers [14] in the FIB-SEM is illustrated in Fig. 4.

3.3 Alternate Specimen Handling Techniques

While pick and place techniques allow for testing a variety of nanoscale materials, the method is tedious and time-consuming and is not appropriate for testing large number of samples. Therefore, meaningful statistics about material property variations arising from either intrinsic or extrinsic effects cannot be obtained. To address this issue, several other approaches have been tried. One approach exploits the dielectrophoresis effect, which refers to the phenomenon in which a force is exerted on a dielectric material when it is subjected to a nonuniform electric field. This approach has been used to align and mount GaN nanowires onto MEMS devices by applying electric fields at specific frequencies [15]. Directed synthesis is another method where nanowires are grown in desired locations and along specific orientations by patterning growth catalyst particles [16]. However, the range of materials that has been fabricated and tested using these alternate specimen handling techniques is quite limited, and better approaches are required to accelerate materials testing at the micro- and nanoscale.

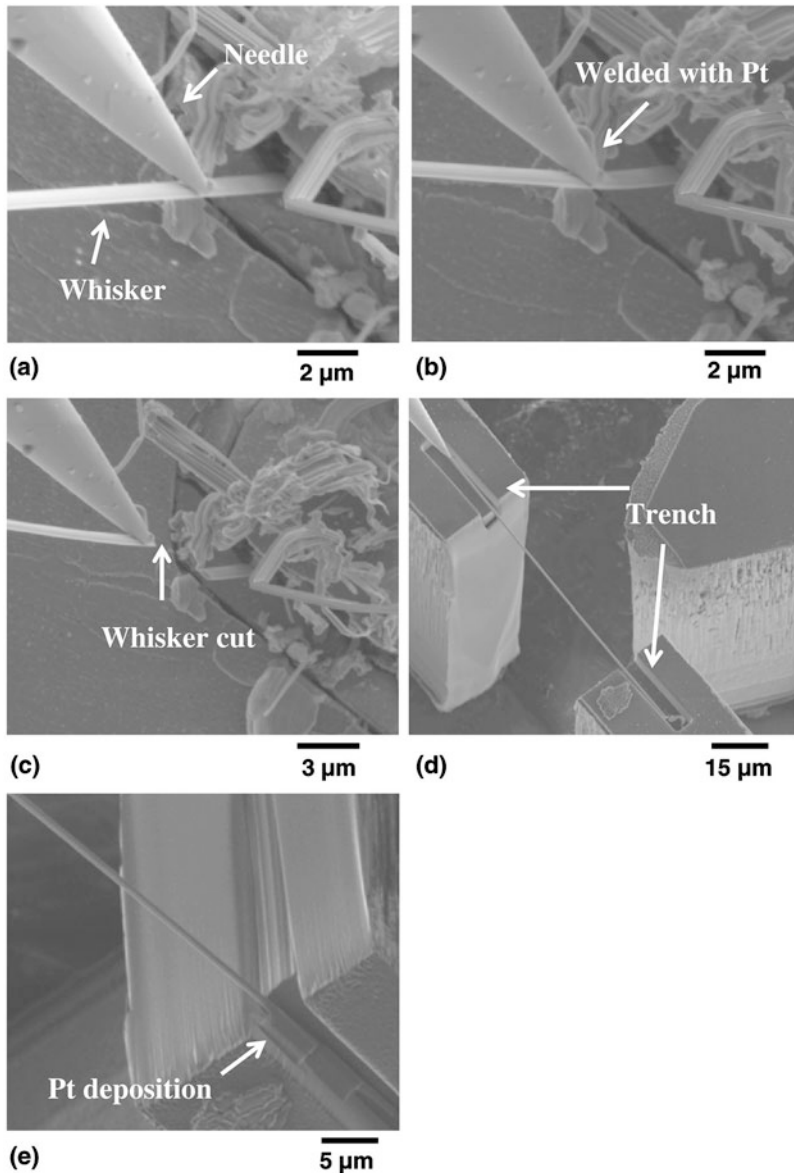


Fig. 4 Process of tin whisker lift-out [14] and placement on a MEMS device using a needle connected to a micromanipulator in a FIB-SEM. (a) Needle touching the whisker, (b) welding of the whisker to the needle using platinum, (c) cutting the whisker near welded region using FIB, (d) placement of tin whisker inside the trenches made in the MEMS device, and (e) welding the whisker in place using Pt (Reprinted with permission from Springer)

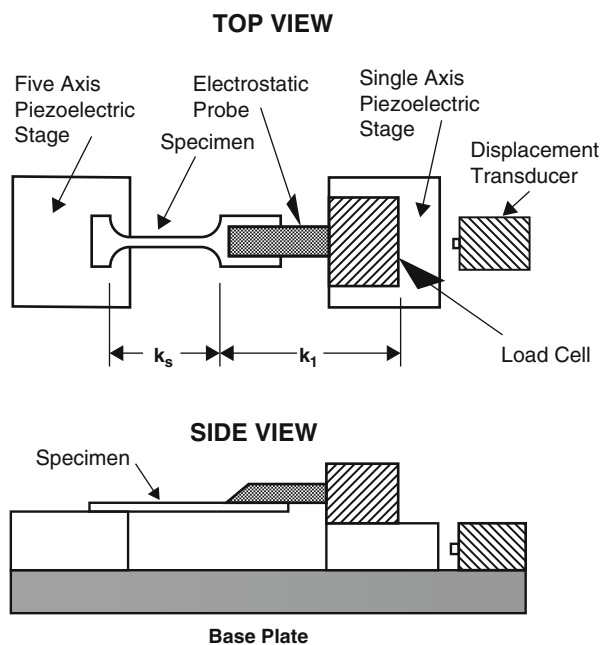
4 Device Configurations for Tensile Testing

The device configurations used in MEMS-based tensile testing can be broadly divided into four categories with increasing order of sophistication. The devices in the first category essentially provide structural support to manipulate and load the specimens, while the actuation and sensing are done externally. These represent the most basic MEMS devices for materials testing. The second category of devices integrates force and/or strain sensing but require external actuation. Devices in the third category combine on-chip actuation and sensing, while the most advanced devices (fourth category) combine on-chip actuation, sensing, and feedback control to enable pure displacement or force controlled experiments. Specific examples of each of these device configurations are described below.

4.1 Devices with External Actuation and Sensing

As mentioned above, the MEMS devices in this category essentially serve as support structures that enable easier manipulation, alignment, and loading of micro- and nanoscale samples. The first such MEMS device was developed by Read and Dally to test freestanding Ti-Al-Ti multilayer thin films deposited on silicon substrates [17]. This technique was then refined and extended to other materials by Sharpe Jr. et al. [10]. Fig. 5 shows an example of such a device to test polysilicon thin films. The specimen is gripped electrostatically, and a single axis piezoelectric stage is used

Fig. 5 Schematic of the specimen and the test system developed by Sharpe et al. [10] for tensile testing of thin films (Reprinted with permission from Springer)



to apply deformation. The force is measured using a load cell and overall displacement is measured using a capacitance based displacement transducer. If the material that is being tested has a linear elastic stress-strain response, the Young's modulus of the material can be measured directly using the force and the overall displacement, provided the stiffness of the load cell is known. However, it is more accurate to measure the strain on the specimen directly by optically tracking markers on the specimen surface.

Another development is the "push-to-pull" concept, whereby a MEMS platform is used to transform the compression exerted by an external transducer (typically a nanoindenter) into tensile deformation of the sample [18]. In these platforms, the load on the specimen is obtained by comparing the nanoindenter readout with and without the specimen, whereas the specimen strain is directly measured using imaging in a SEM or TEM. Such a push-to-pull device was developed at Hysitron Inc. [19] and integrated with their TEM nanoindentation holder to perform in situ TEM straining experiments. This device has been used to conduct in situ TEM tensile testing on nanowires and metallic thin films.

4.2 Devices with External Actuation and On-Chip Sensing

Haque and Saif developed the first MEMS device for tensile testing of thin films that integrated on-chip force and strain sensing [8]. The device introduced the co-fabrication of thin films samples and incorporated novel design features to ensure nearly perfect uniaxial tensile deformation and avoid accidental loading of the samples. A beam flexure-based force sensor was used for sample load measurement, whereas the strain was measured by tracking the movement of gages attached to the sample ends. Because the device footprint was very small (10 mm by 3 mm), it could be loaded onto standard TEM straining holders to perform in situ experiments in which the deformation mechanisms could be visualized, while the stress-strain response was simultaneously measured [20]. Later, Han and Saif [21] modified this device and introduced a simpler fabrication process that led to higher specimen yield. This design also allowed nondestructive calibration of the force sensing beams and electromechanical characterization of the thin films. Fig. 6 shows images of this device and its different functional components.

Haque and coworkers used the same sample alignment method used in [20] to develop a push-to-pull device for testing 1D nanostructures. Since the length of 1D nanostructures is usually quite small, they introduced a displacement amplification system based on beam buckling to measure the strain [22, 23]. As shown in Fig. 7, the device is comprised of a long and a short column, both of which buckle when a displacement is applied using an external actuator. By measuring the lateral displacement of the columns, both the force and deformation of the specimen can be obtained. Using this method, the deformation of the nanostructure could be amplified almost two orders of magnitude, which allowed the tensile testing of ZnO nanowires under an optical microscope [23]. A simpler MEMS platform with a leaf spring load sensor was employed by Naraghi et al. [24, 25] to measure the

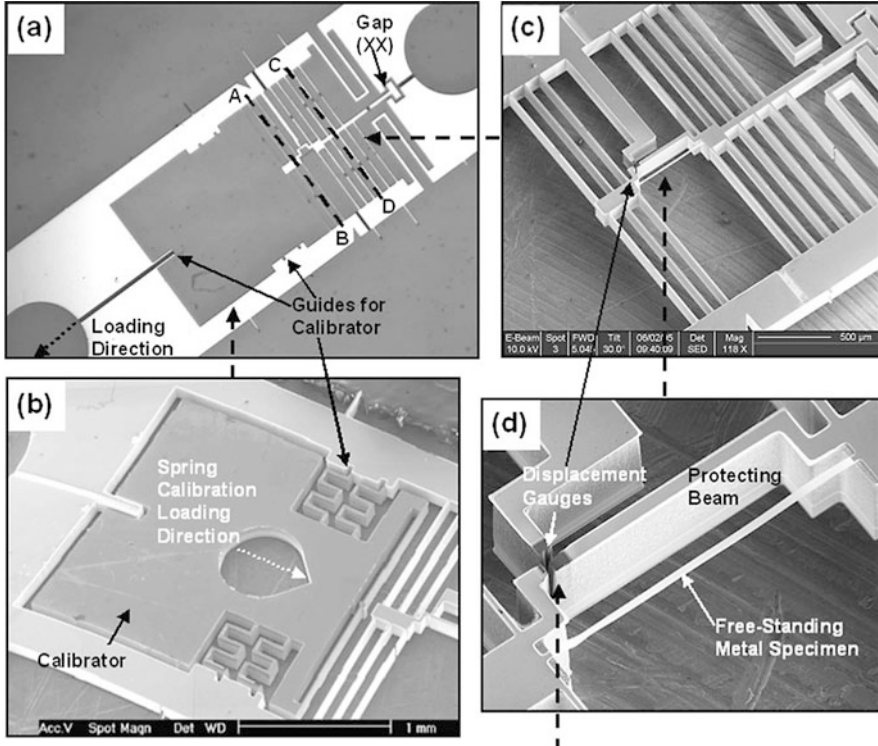


Fig. 6 MEMS tensile testing stage developed by Han and Saif [21]. (a) Optical microscope view of the entire stage with force sensing beams AB and support beams CD, (b) SEM micrograph showing a calibrator in place for measuring the stiffness of the force sensing beams, (c) SEM micrograph of the stage near the specimen and the force sensing and support beams, (d) zoomed-in view near the displacement gages (Reprinted with permission from AIP publishing)

stress-strain response of electrospun polyacrylonitrile nanofibers. In this case, both load and displacement of the sample was obtained using digital image correlation.

4.3 Devices with On-Chip Actuation and Sensing

Zhu and Espinosa [26, 27] developed the first MEMS testing platform with an on-chip actuator and load sensor. Two types of actuation schemes were employed – a thermal actuator for controlling the applied displacement and a comb drive actuator for controlling the applied force, as shown in Fig. 8. The key improvement in this design was that the load sensor used differential capacitive sensing and hence could provide real-time measurements, unlike optical measurements. Also, the small device footprint allows for integration with TEM holders for in situ measurements. Since then, other researchers have developed similar platforms for testing nanowires [28].

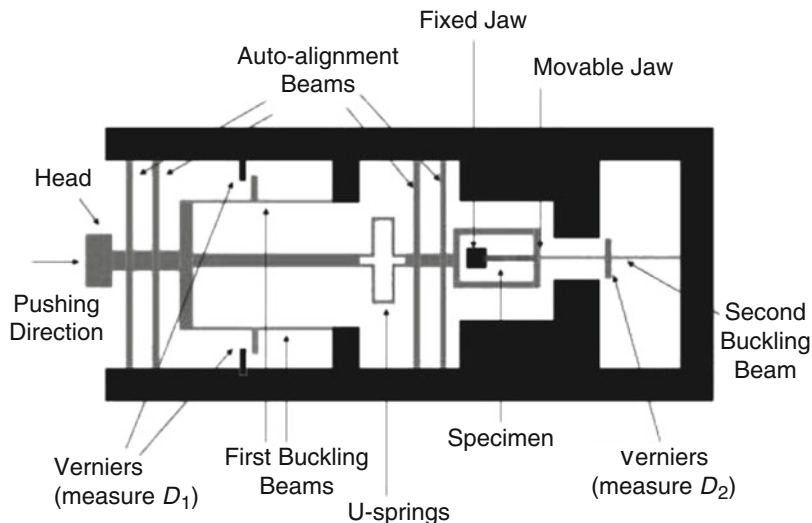


Fig. 7 Schematic of the MEMS tensile tester for nanowires developed by Desai and Haque [22]. The darker structures in the schematic are fixed, while the gray structures are movable. The device is loaded by applying a displacement to the “head” toward the right. The two sets of buckling beams amplify the axial displacements and lead to high resolution of the force (~ 10 nN) and displacement (~ 1 nm) applied on the specimen (Reprinted with permission from Sage Publications)

4.4 Devices with Feedback Control

All the MEMS tensile testing devices discussed in Sects. 4.2 and 4.3, which include an on-chip load sensor, use flexible beams (load/force sensor) that are in series with the sample (e.g., see Fig. 6) to measure the sample force. This inevitably means that the applied displacement is partitioned between the sample deformation and the deflection of the force sensing beams. Therefore, when there is stress relaxation in the sample, it leads to a reduction in the beam deflection and increase in sample deformation (strain) until a new equilibrium is reached. As a result, a true strain or strain rate controlled experiment cannot be performed with these devices. One obvious way to circumvent the problem is to employ an external load sensor with a high stiffness, but this essentially precludes in situ experiments because of the large size of such sensors. A better approach is to use feedback control to keep the displacement of the force sensing beams constant. Pantano et al. [29] introduced a MEMS device (Fig. 9) with an additional electrostatic actuator to control the displacement of the force sensing beams. In their scheme, a feedback voltage is used to control the output of the electrostatic actuator, which ensures that the deflection of the load sensing beams remains zero at all times. Therefore, the specimen experiences the entire applied displacement, which enables true strain or strain rate controlled experiments. Since the force sensing beams do not move, the force on the sample is not directly sensed from their displacement. Rather, the force

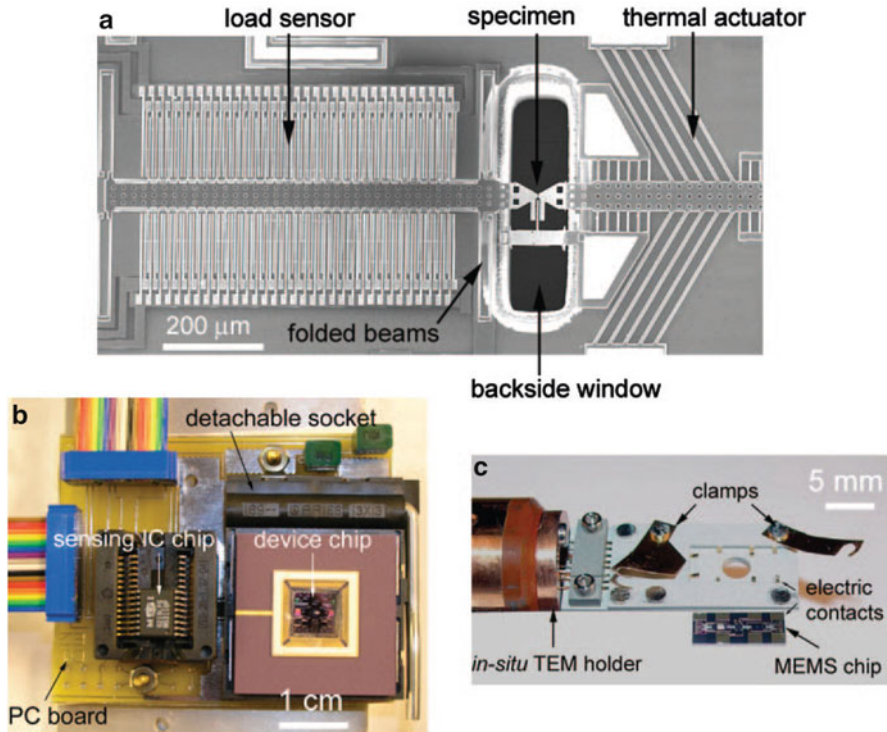


Fig. 8 In situ SEM and TEM tensile testing device developed by Zhu and Espinosa [27]. The setup includes an actuator, load sensor, and specimen. Four folded beams support the load sensor. (a) Testing device used for in situ TEM studies. (b) Experimental setup for in situ SEM testing. MEMS device chip is positioned on a printed circuit board. The setup is connected to a power supply, a digital multimeter, and a computer outside the SEM by means of a chamber feedthrough. (c) In situ TEM holder (containing a feedthrough and eight electric contact pads) along with a 5 mm by 10 mm MEMS chip (Copyright National Academy of Sciences)

on the sample is obtained from the force exerted by the electrostatic actuator, since these forces have to be equal for the force sensing beams to have zero deflection.

5 MEMS Platforms for Fatigue and High-Temperature Testing

As described in Sect. 4, many MEMS device configurations have been employed to perform monotonic tensile testing on micro- and nanoscale specimens. While the uniaxial tensile test can reveal a number of key material properties, it is also important to investigate the behavior of micro-/nanoscale materials under different loading conditions for both scientific and technological reasons. For instance, nanostructured metallic thin films that are used as structural components in MEMS

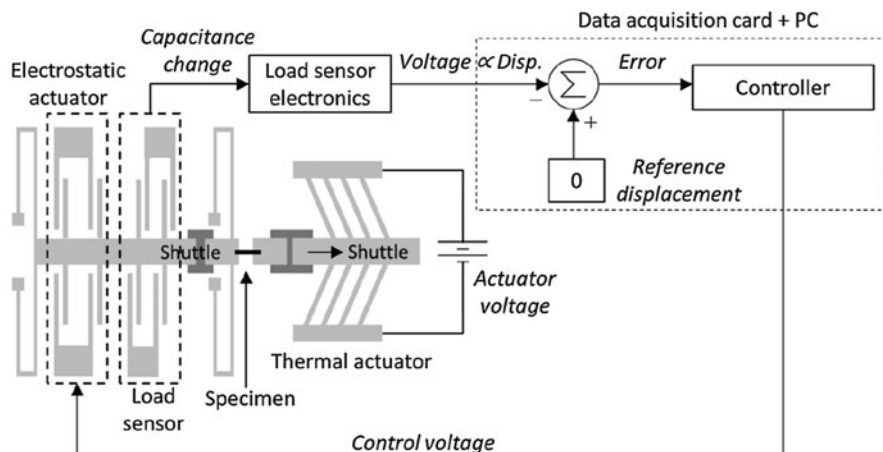


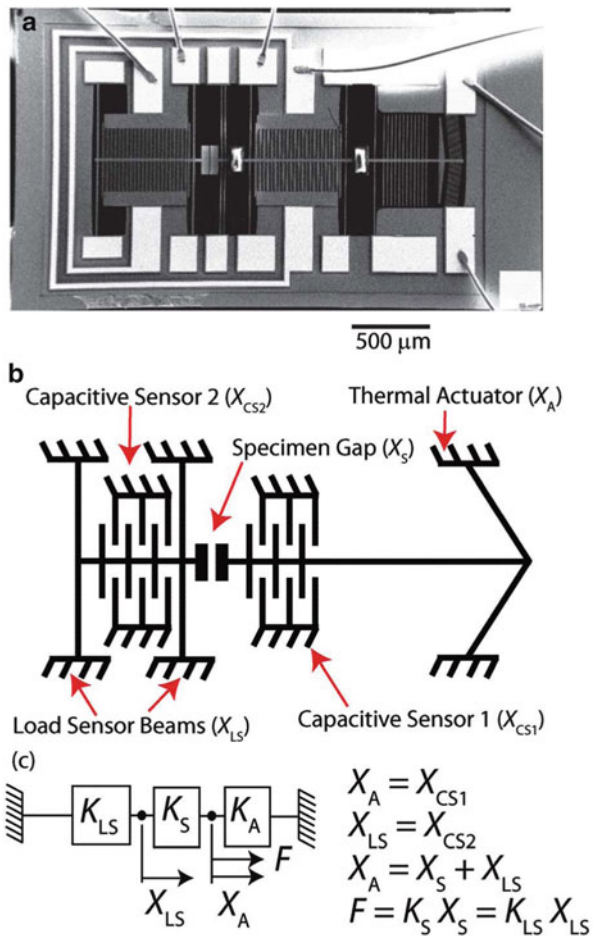
Fig. 9 Schematic of the closed-loop scheme implemented by Pantano et al. [29] for feedback control using capacitive sensing (Reprinted with permission from Springer)

or as interconnects in microelectronic devices undergo cyclic deformation either due to applied mechanical loading or thermally induced stresses. Therefore, it is essential to understand their fatigue behavior to both predict failure as well as to make design/processing changes to increase their fatigue resistance. Similarly, small-scale devices made from ceramics/semiconductors, which are used in aerospace and automotive applications, often operate at elevated temperatures, and it is necessary to characterize their high-temperature behavior to ensure reliable performance. Motivated by these considerations, researchers have developed MEMS stages for probing the cyclic fatigue and thermomechanical behavior of small-scale materials.

Pierron and coworkers [30] developed a MEMS testing platform for studying the cyclic tensile load-unload behavior of freestanding metal films. Their platform (Fig. 10), which is approximately 3 mm by 1 mm in dimension, comprises of a thermal actuator and two capacitive sensors to track the specimen elongation and load. The advantage of using two capacitive sensors is that neither load nor strain measurements require high-resolution imaging and hence the same platform can be used for both in situ TEM and ex situ experiments. This is important because it allows one to study the effect of environmental conditions (air exposure vs. vacuum) on the fatigue behavior. In addition, comparing the behavior during ex situ and in situ TEM experiments can reveal if specimen exposure to the TEM electron beam causes anomalous changes in the behavior [31–33]. High cycle in situ TEM fatigue experiments have also been conducted on nanoscale thin films by employing a push-to-pull device [34].

MEMS-based platforms have been used to probe the high-temperature deformation behavior of nanowires and thin films using both tensile and bending experiments. Chang and Zhu have developed a Si MEMS thermomechanical stage that incorporates an on chip heater based on Joule heating to test nanowires and other 1D nanostructures under tension from room temperature to about 325 °C [35]. By

Fig. 10 (a) SEM image of the MEMS platform devised by Pierron and coworkers [30] for fatigue testing. (b) Corresponding schematic, with a description of its components and their displacements (e.g., X_A : displacement of thermal actuator). (c) Lump model of the MEMS device with corresponding governing equations (Reprinted with permission from RSC publishing)



adopting a symmetrical design, they ensured that the temperature is same on both ends of the specimen, which avoids temperature gradients along the 1D specimens. Multiphysics simulations were used to predict the temperature distribution in the device and temperature measurements using Raman spectroscopy was used to validate the simulations. Kang and Saif have developed an SiC-based MEMS platform for in situ uniaxial testing of micro-/nanoscale specimens [36]. This platform also uses the Joule heating mechanism but also incorporates a bimetal type sensor which allows in situ temperature measurement (Fig. 11). Furthermore, because the platform is fabricated from SiC, significantly higher temperatures (up to 700 °C) can be sustained. Apart from the above examples, MEMS devices have also been integrated with external heaters to perform elevated temperature mechanical testing on nanowires and metallic films [37].

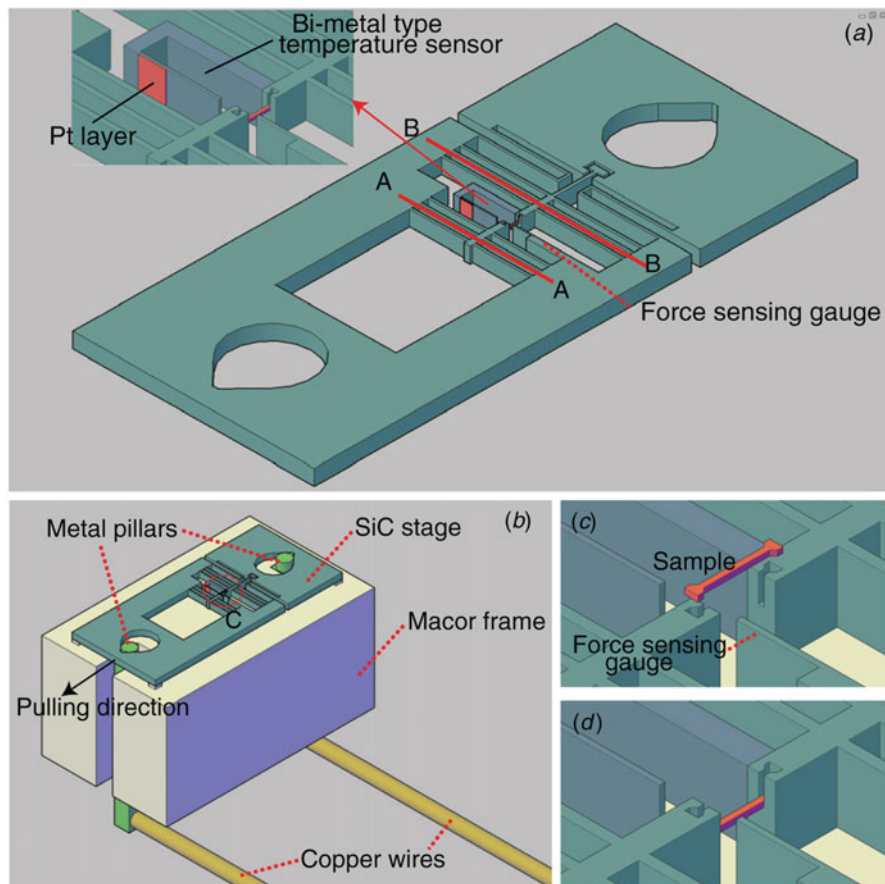


Fig. 11 Three-dimensional schematics of the SiC MEMS stage developed by Kang and Saif [36]. (a) Overall view of the SiC stage. A zoom-in view of a bimetal-type temperature sensor is shown in the inset. (b) Experimental setup for in situ uniaxial tests. The stage is thermally isolated from an SEM sample holder by the macor heat-insulating frame. The metal pillars are connected to copper wires so that a voltage can directly be applied to the stage for resistive heating. (c and d) Enlargement of area marked by C in (b), which show a sample before and after assembly with the stage, respectively (Reprinted with permission from IOP publishing)

6 Selected Results

In the previous sections, we have discussed different MEMS platforms for conducting various types of mechanical tests on micro- and nanoscale materials. In this section, we focus on the mechanical properties and behavior of micro-/nanoscale materials. The purpose is to highlight certain unusual behaviors exhibited by these materials, some of which were discovered using MEMS-based platforms.

6.1 Size-Dependent Elastic Modulus of Nanowires

It has long been known that plastic properties of crystalline materials are size dependent, with the yield strength increasing with decreasing specimen dimension or microstructural size. In contrast, the elastic properties, which depend on the strength of atomic bonds, are expected to be fairly insensitive to specimen or microstructural length scales. Nevertheless, significant changes in the elastic modulus have been reported in both metallic and ceramic nanowires as the diameter is reduced below 100 nm. Figure 12a, for instance, shows an increase in Young's modulus of ZnO nanowires from approximately 140 GPa to 160 GPa as the diameter is reduced from 100 nm to 20 nm [38]. An even higher increase in Young's modulus has been reported for Ag nanowires [39]. The changes in Young's modulus have been mainly attributed to the increased influence of surface stresses and atomic rearrangements of surface atoms. While the surface atoms have a lower coordination number compared to atoms in the bulk, which leads to a reduction in modulus, electron redistribution can compensate for this effect and lead to a net increase in the modulus of certain surfaces compared to the bulk. As a result, the modulus of very small diameter (<30 nm) nanowires, where the surface effects predominate, can be higher than the bulk modulus.

6.2 Reversible Plasticity in Nanostructured Metals

Plastic deformation is conventionally considered to be irrecoverable. Surprisingly, nanostructured metals exhibit the capability to recover a significant fraction of their plastic strain after unloading [40]. This unusual strain recovery is time dependent and thermally activated. Fig. 12b provides an example of plastic strain recovery in a nanocrystalline aluminum thin film after unloading. Similar strain recovery has also been observed in nanocrystalline copper films [41] as well as bulk aluminum with a bimodal nanostructure [42]. In certain cases, strain recovery occurs both during and after unloading [43]. The plastic strain recovery is driven by large internal stresses induced by the highly heterogeneous deformation of the nanocrystalline microstructure, and several mechanisms including dislocation motion, grain boundary sliding, and diffusional creep have been proposed to explain this phenomenon. Pentatwinned Ag nanowires (Fig. 12c) have also been shown to exhibit plastic strain recovery after unloading [44]. In this case, the twin boundaries act as barriers to dislocations, which results in pileup of dislocations. When the external stress is released, the back stress from the pileup induces the reverse motion of dislocations, leading to the recovery of plastic strain.

6.3 Strain Rate Sensitivity of Nanostructured Metal Films

Face-centered cubic (FCC) metals such as Cu, Au, and Al show low strain rate sensitivity (SRS) at room temperature, i.e., their flow stress does not change

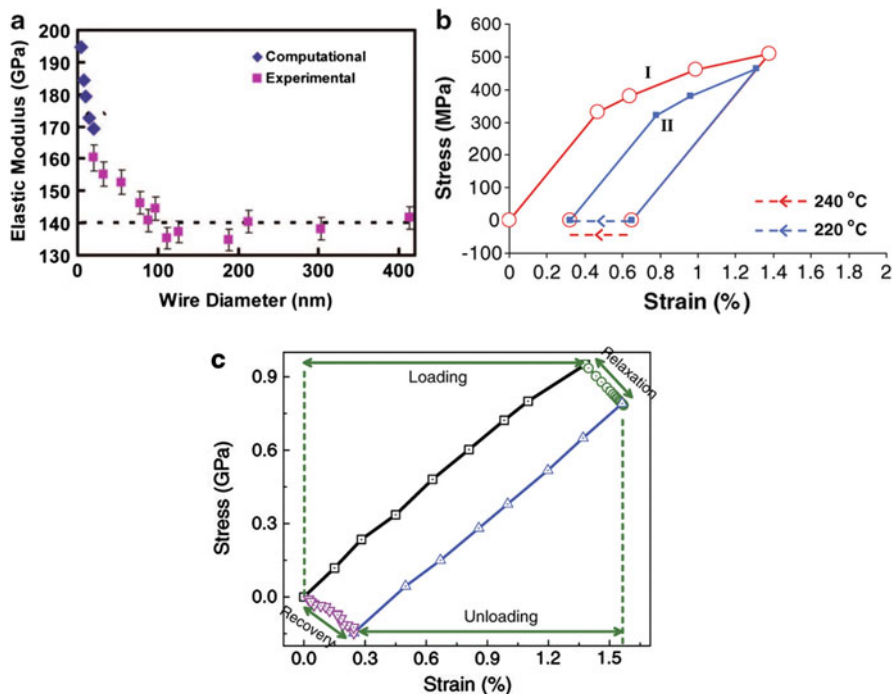


Fig. 12 (a) Variation of Young's modulus of ZnO nanowires with wire diameter [38]. The dashed line shows the experimentally reported bulk value of ~ 140 GPa (Reprinted with permission from ACS publications). (b) Plastic strain recovery in a nanocrystalline aluminum thin film [40]. The dashed lines indicate the recovery of strain after unloading at two different temperatures. (c) Plastic strain recovery in a penta-twinned Ag nanowire [44] (Reprinted with permission from Springer Nature)

significantly with an increase in the rate at which they are deformed. The SRS exponent (m), which quantifies the rate dependence of flow stress, is usually below 0.01 for microcrystalline (mean grain size $> 1 \mu\text{m}$) FCC metals. However, when the grain size is substantially reduced below $1 \mu\text{m}$, m increases significantly [45, 46]. The increase in SRS happens because the dominant deformation mechanism changes from intragranular dislocation plasticity in the microcrystalline regime to grain boundary-mediated plasticity in the nanocrystalline/ultrafine-grained regime. In effect, nucleation of dislocations at grain boundaries, pinning/depinning of dislocations at grain boundaries, grain boundary sliding, etc. become the rate-controlling process. This increased SRS is illustrated in Fig. 13a, where the yield stress of nanocrystalline gold films increases by more than 100% as the strain rate is increased from $10^{-6}/\text{s}$ to $10/\text{s}$ [46]. High strain rate sensitivity has been reported in randomly oriented, ultrafine-grained aluminum films and has been attributed to time-dependent grain rotations [47].

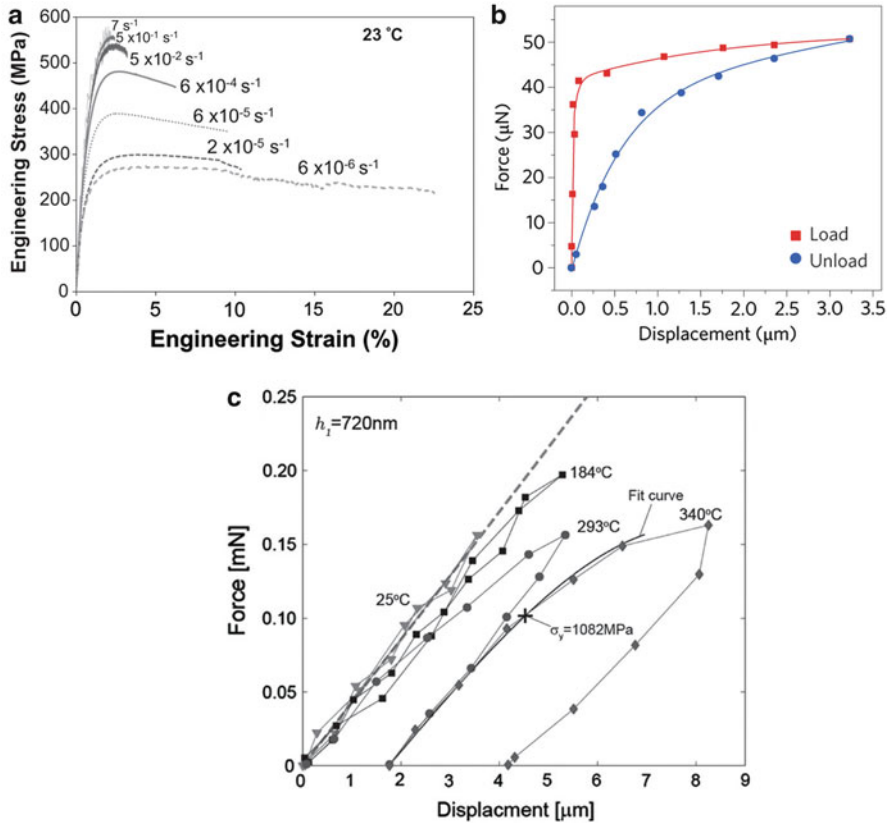


Fig. 13 (a) Stress-strain response of a nanocrystalline gold film [46] at different strain rates near room temperature (23 °C) (Reprinted with permission from Elsevier). (b) Load-unload behavior of a single crystalline ZnO nanowire subjected to compression/buckling [49]. The hysteresis in the stress-strain response indicates the presence of large anelastic deformation (Reprinted with permission from Springer Nature). (c) Stress-strain response of a silicon microbeam with a thickness of 720 nm at different temperatures [50]. The beam exhibits plastic deformation at temperatures greater than 293 °C (Reprinted with permission from Wiley)

6.4 Large Anelasticity in Single Crystal Nanowires

Anelasticity refers to the time-dependent deformation recovery of materials after the applied load has been removed. Unlike elastic deformation, which is instantaneously recovered, anelastic deformation leads to dissipation of mechanical energy [48]. While there are many sources of anelasticity (relaxations of point defects, dislocations and grain boundaries, thermoelastic relaxation, etc.), single crystals typically exhibit very little anelastic deformation at the macroscopic scale. In contrast, it has been shown that single crystalline ZnO (Fig. 13b) and p-doped Si nanowires exhibit significant anelasticity (more than 10,000 times that of bulk single crystalline

materials) with recovery times in the order of tens of minutes [49]. This large anelasticity occurs due to migration of point defects, driven by gradients in the applied stress. When the applied stress is released, the point defects migrate back and lead to time-dependent deformation recovery. Single crystalline ZnO wires, in particular, showed an extremely high damping merit index, which could prove useful for damping applications in micro- and nanoscale structures and devices.

6.5 Size-Dependent Brittle to Ductile Transition in Silicon

Most materials, with the exception of certain face centered cubic metals, become brittle at low temperature. Conversely, even intrinsically brittle materials, for example, semiconductors and ceramics, start to deform plastically when the temperature is sufficiently increased. This brittle to ductile transition (BDT) can either occur over a broad or narrow range of temperatures. Bulk silicon exhibits a sharp transition at around 545 °C, but it was not clear if the BDT temperature of silicon remained the same at small specimen dimensions. This is important because silicon is the most widely used material in MEMS, and if it undergoes a BDT and starts deforming plastically, its electrical and mechanical properties can change dramatically. To address this issue, Kang and Saif used the thermomechanical MEMS testing platform described in Fig. 11 to perform elevated temperature bending tests on silicon beams with thicknesses varying from 720 nm to 8.7 μm. They found that as the specimen size decreased the BDT temperature (Fig. 13c) also decreased, with the 720 nm thick specimen undergoing notable plastic deformation even at 293 °C [50]. They explained the reduction in BDT temperature in terms of a model which takes into account the increased contribution of surface dislocation nucleation, which has a lower energy barrier compared to bulk dislocation nucleation, to plastic deformation. Chang and Zhu have reported an even lower BDT temperature (~125 °C) in silicon nanowires with a diameter of 60 nm [35].

7 Future Challenges in MEMS-Based Materials Testing

In this chapter, we have reviewed different aspects of MEMS-based testing of micro- and nanoscale materials. As described in the previous sections, the significant challenges that exist in specimen fabrication, handling, and gripping at small scale have been successfully overcome using innovative strategies and advances in nanomanipulation and high-resolution imaging. A surfeit of new techniques has also been established to apply and measure forces and displacements with nanonewton and nanometer resolution, respectively. These capabilities have enabled the measurement of stress-strain response and discovery of unexpected mechanical behavior in nanostructured thin films, micro-/nanopillars, and nanowires. It is quite reasonable to expect that these unique behaviors will be exploited to enhance the performance of micro- and nanoscale systems in the future.

While tremendous progress has been achieved in the characterization of nanoscale materials using MEMS, considerable challenges remain. One particular aspect that needs to be studied at much greater depth is the behavior of these materials under

cyclic/long-term loading conditions. As is well known, a large majority of structural failures at the macroscale occur due to fatigue. As nanoscale materials are incorporated more and more into nanotechnology applications such as energy harvesting and flexible and stretchable electronic devices, where they are subjected to intermittent loading over long periods of time, it is likely that the fatigue response will dictate their performance and lifetime. However, currently available MEMS platforms are not particularly well suited to probe the cyclic deformation behavior of nanoscale materials. Specifically, current MEMS platforms are not capable of cycling the nanoscale specimens over a constant stress or strain range during the entire period of the experiment, which is required to quantitatively characterize their fatigue behavior. The development of MEMS testing platforms with feedback control [29] provides a route to performing such tests, but better stability and faster response is still needed.

Similarly, significant hurdles remain with respect to obtaining meaningful statistics on the property variations of certain nanoscale materials. For instance, 1D nanomaterials like nanowires or nanotubes are typically manipulated using pick and place methods for testing. When pick and place methods are used, the boundary conditions applied on the specimens during testing can sometimes change, and it is also possible to accidentally load the sample during manipulation. Therefore, the mechanical properties obtained can vary significantly. Unfortunately, these manipulation methods are highly time consuming, and hence it is not possible to test enough specimens to obtain rigorous bounds on mechanical properties. This problem becomes particularly acute for properties like fatigue strength, which show significant scatter even in macroscopic samples for which standard testing protocols exist. Co-fabrication of 1D nanostructures with MEMS testing platforms, which has already been used for testing of thin films, would be one way to overcome this problem, but this would require the development of new synthesis/fabrication processes.

These challenges aside, the future of MEMS-based materials testing remains highly promising, and advances in high-resolution imaging (e.g., dynamic TEM) and spectroscopy techniques are likely to open up possibilities to probe the fundamental properties of nanoscale materials at even greater depth.

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