Quantitative In-situ Nanomechanical Characterization of Metallic Nanowires

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This paper reviews recent studies on in-situ quantitative mechanical characterization of metallic nanowires with diameters from a few nanometers to hundreds of nanometers, with particular emphasis placed on tensile loading geometry. Critical challenges and pitfalls in manipulating, clamping, and quantitatively testing nanowire specimens, with drastically different dimensions, are discussed. Two general experimental strategies are employed: microelectrochemical systems-based technology for testing of larger-diameter metal nanowires (D ~ 30–300 nm), and in-situ transmission electron microscopy-atomic force microscopy platform for testing of ultrathin metallic nanowires (D < 20 nm). Size-dependent mechanical behaviors of gold nanowires, as well as the transition of different deformation mechanisms at corresponding length scales, are clearly revealed.

How would you…

…describe the overall significance of this paper?

This paper offers an overview of advances in quantitative mechanical characterization of one-dimensional metallic nanostructures, especially for metallic nanowires with diameters ranging from hundreds of nanometers to only a few nanometers. Ultrahigh mechanical strength and size-dependent mechanical behaviors are demonstrated with the aid of recently developed highly sensitive measurement techniques.

…describe this work to a materials science and engineering professional with no experience in your technical specialty?

When a crystal's dimension reduces to nanometer length scale, the strength of the material is expected to approach the ideal strength of a defect-free crystal. The plastic deformation and the ultrahigh strength attained in these nanoscale entities were predicted to be controlled by surface dislocation nucleation rather than by dislocation multiplication/interaction as in bulk crystals. This overview reviews size-dependent tensile behaviors of metallic nanowires and demonstrates that surface dislocation nucleation becomes a dominating mechanism when the sample sizes are reduced to tens of nanometers or below.

…describe this work to a layperson?

Metallic nanowires are high aspect-ratio one-dimensional nanomaterials. The ability to achieve the full potentials of these technologies is limited by how they will behave at relevant length scales, in particular, their mechanical performance and reliability. The in-situ characterization tools reviewed in this paper can evaluate critical mechanical properties and directly observe deformation and fracture processes in real time.

INTRODUCTION

Due to their interesting electrical, chemical, magnetic, optical and mechanical properties, one-dimensional (1-D) nanoscale materials and structures, such as nanowires, nanobelts, nanorods, and nanotubes, have been extensively investigated in the past two decades. They are widely considered as ideal candidates to be used in miniaturized devices such as sensors/detectors, actuators, electronic/optoelectronic devices, solar-cells/power generators, and carriers of drugs. In particular, metallic nanowires (NWs) and nanorods (NRs), with diameters ranging from a few to hundreds of nanometers, have stimulated great interest recently as important building blocks for future nanoscale electronic and electromechanical devices in various applications.[1] More importantly, current micro/nano lithography technologies are becoming less and less efficient when the critical feature size is approaching the limit of sub-10 nm. Ultrathin metallic nano-wires,[2] such as gold, silver, tellurium, palladium, and platinum nanowires with diameters less than ~10 nm, have been chemically produced and are expected to potentially satisfy the stringent nanoelectronics requirements. For example, ultrathin gold nanowire has been widely considered as a promising candidate for next-generation interconnects and as active components in future nanoscale devices,[3] owing to its excellent electrical and mechanical properties as well as desired chemical inertness. However, the ability to achieve the full potential of aforementioned 1-D metallic nanostructures in these fascinating applications is ultimately limited by how these one-dimensional building blocks will behave at relevant length scales, in particular, their mechanical performance and reliability.

Mechanical properties of materials deviate largely from their bulk counterparts when characteristic dimensions become sufficiently small.[4,5] Size-dependence in mechanical properties for metals at micron scales has been well documented[5–8] in recent years. Size dependent plasticity and fracture behaviors of 1-D metallic materials, especially at the nanometer length scale, have recently generated great interests[8–10] because of their important effects on assembly, performance, and reliability of functional nano-electronic and nano-electromechanical-systems (NEMS) devices. Size-dependent mechanical study of 1-D metallic nanostructures also possess exciting potential for revealing the fundamental mechanisms responsible for physical origins of size effects in many important processes

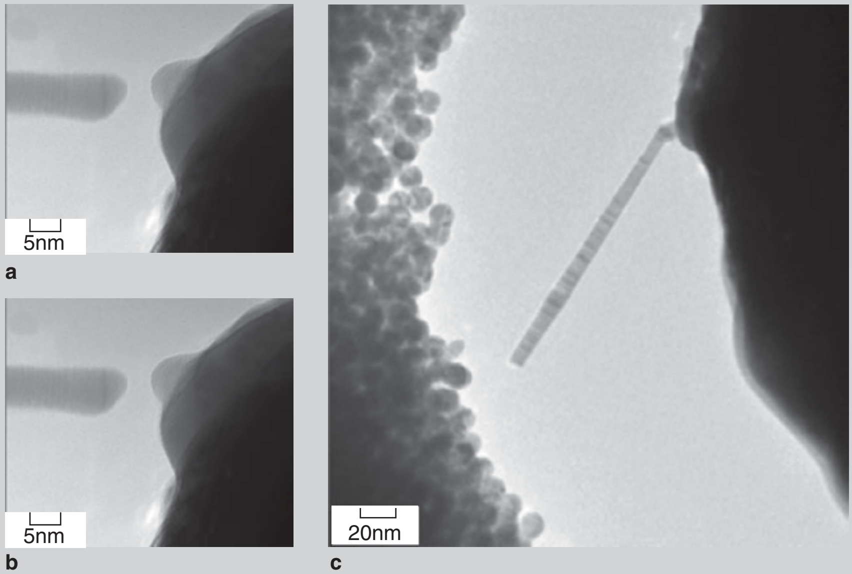

Figure 1. "Cold welding" assisted sample clamping techniques for ultrathin gold nanowires:[29] (a) a probe with a nano-sized gold asperity approaching toward an ultrathin gold nanowire; (b) cold welding occurred and made the strong contact; (c) the ultrathin nanowire was then successfully attached to the probe.

such as deformation, fracture and fatigue, as the length scale approaches atomic spacing.[10] Therefore, this review will focus on research activities that systematically probe mechanical behaviors of 1-D metallic structures at nanometer length scale (with diameters from a few nanometers to hundreds of nanometers), using advanced in-situ quantitative characterization methodologies.[11] These systematic quantitative studies capable of observing and following the initiation and evolution of mechanical processes also provide valuable insights into discoveries of how atomic structures could be manipulated in a predicable manner to enable development of new materials and novel functional structures.

SIZE-EFFECT STUDY OF METALLIC NANOWIRES

Before designing and developing methodologies for systematic mechanical testing of 1-D metallic nanostructures, it is necessary to review and understand the three major factors affecting size effects in mechanical behavior of metals: microstructural constraint, geometry constraint, and loading configuration. Among these, the microstructural constraint is due to grain size effects described by the famous "Hall–Petch" relationship (the smaller the grain size, the stronger the materials) or the inversed "Hall–Petch" relationship for grain size of a few nanometers which have been extensively studied for bulk nanocrystalline materials.[12] Twin boundary effects[13] and phase transformation effects[14] are also important mechanisms in this category. The geometry constraint is normally due to substrate/thin film interface and/or free surface effects, such as sample dimensions effects (wire diameters, film thicknesses, indenter penetrating depth, etc.). It has recently been suggested that a similar "Hall–Petch" type of relationship (with slope of –0.6 to –0.8 for face-centered cubic (f.c.c.) metals as compared to –0.5 of the regular "Hall–Petch" slope)[15] also exists when one plots the normalized yield strength against the wire/pillar diameter. A significant decrease in this slope has been predicted for nanowires with diameters below a few tens of nanometers due to the transition from collective dislocation dynamics to surface dislocation nucleation controlled plasticity by atomic simulations.[16] Finally, the proven effects of loading configuration or imposed strain gradient (i.e., indentation loading, bending, or torsion, etc.) have been shown for metallic samples at meso- and micron-scales.[17–20] To get a clear understanding of their individual contributions and therefore unambiguous underlying mechanisms, experiments need to be carefully designed to decouple the influences of the three factors whenever it is possible.

To address the first two factors, microstructural constraint and geometry constraint, systematic characterization of size-dependent mechanical behaviors should be performed on free-standing metallic nanowire samples with well-defined dimensions (e.g., length and aspect ratio) and controlled crystalline structures (usually single crystals with known orientation is preferred). Despite numerous nanowire growth/synthesis techniques that had been developed in the past several decades, to fabricate metallic nanowires with diameters less than 20 nm in large quantity and with high quality was still a significant challenge. Earlier approaches such as the template-based synthetic method,[21] would normally produce nanowires with larger diameters and suboptimal microstructures. It was not until the very recent chemical breakthrough that has allowed the production of ultrathin single-crystalline metallic nanowires, often in solution, with a large quantity and superior quality.[2] These solution-based chemical synthesis methods can often produce single-crystalline metallic nanowires with diameters less than 10 nm, for example, ultrathin gold nanowires with diameters ranging from 3–9 nm with uniform <111> crystalline orientation.[3]

On the other hand, loading configuration plays a more important role in quantitative mechanical testing of nanowires with increasingly smaller sample sizes. In the past two decades, significant progress has been made for quantitative testing 1-D nanomaterials under various loading geometries, such as bending,[22] buckling,[23] and compression.[24] These loading configurations, oftentimes accompanied by complicated stress fields in the sample, may hinder the effective mechanical assessment of ultrathin nanowires with high aspect ratios. Therefore, tensile testing geometry becomes an ideal option. However, the efficient and relatively easy-to-interpret tensile testing method, for measuring intrinsic mechanical properties of exceedingly small nanowire samples inside scanning electron microscopy (SEM) and transmission elecgtron microscopy (TEM), poses significant challenges due to difficulties associated with sample clamping, alignment, and accurate measurements of load and displacement.

SAMPLE POSITIONING AND CLAMPING CHALLENGES

Sample positioning, in this case,

refers to the placement of a nanowire sample at the desired location with nano/micrometer precision before actual testing can take place. Sample clamping refers to the solid bonding at both ends of the nanowire samples in tensile loading configuration. Manipulating and positioning individual nanowire specimen can be realized by using a micromanipulator under an optical microscope (for nanowires with hundreds of nanometers) or a nanomanipulator inside an SEM (for nanowires with tens of nanometers). The fact that the specimens must be freestanding, firmly clamped at both ends, and well aligned in the tensile loading direction makes sample positioning and clamping quite a challenging task. While numerous progress has been made in sample harvesting, manipulation, and gripping for micro and nanoscale tensile testing,[8] there remain some issues which will affect the fidelity of measurements, as commented in a recent *JOM* paper.[25] Specifically, for testing nanowire samples using a dedicated platform, several special circumstances need to be carefully considered during sample clamping processes: sample coating/contamination issues when using focus ion beam (FIB) deposition process for sample clamping;[25] sample alignment, and bonding strength when glue was used;[26] charging issue for sample clamped without a conductive path while tested under high vacuum conditions probed by electron beams.[27]

For high-aspect-ratio ultrathin nanowire (with sub-20 nm diameters) samples, achieving the preferred uniaxial tensile loading presents even bigger challenges in terms of sample handling and clamping due to their exceedingly small dimensions. Typical sample positioning and clamping procedures involving FIB induced deposition may introduce significant surface contamination (i.e., the volume of the coating layer will be comparable to the sample itself for ultrathin nanowires)[25] and local heating induced spot welding technique could potentially damage the initial sample structures and morphologies.[28] These drawbacks have been successfully overcome by a recently discovered "cold welding" technique,[29] which allows near perfect bonding formed between ultrathin nanowires and substrates[30] by gentle mechanical contact alone (Figure 1), without any local heating or deposition needed.[29] The robust picking-up and clamping procedure assisted by a "cold welding" process can be performed with high efficiency and repeatability,[27] allowing systematic tensile study of ultrathin gold nanowires.

IN-SITU TENSILE TESTING

In addition to non-ideal loading configurations such as bending and buckling, earlier quantitative mechanical testing of metallic nanostructures were primarily carried out ex situ (i.e., no real time monitoring of microstructure/morphology evolutions during testing). Atomic force microscopy deflection techniques, which did not permit a one-to-one correlation between mechanical data and internal structural evolution, were used to measure mechanical properties of Au nanowires.[31] Several in-situ tensile testing techniques had recently been developed for characterizing 1-D metallic nano-and micro-structures[8] with diameters from hundreds of nanometers to a few microns, inside an SEM. For example, G. Richter et al. performed in-situ tensile tests of single crystal Cu nanowhiskers (Figure 2a–d) with diameters from 75–300 nm in a focused ion beam-scanning electron microscope (FIB-SEM). The corresponding quantitative measurements revealed strength close to the theoretical value

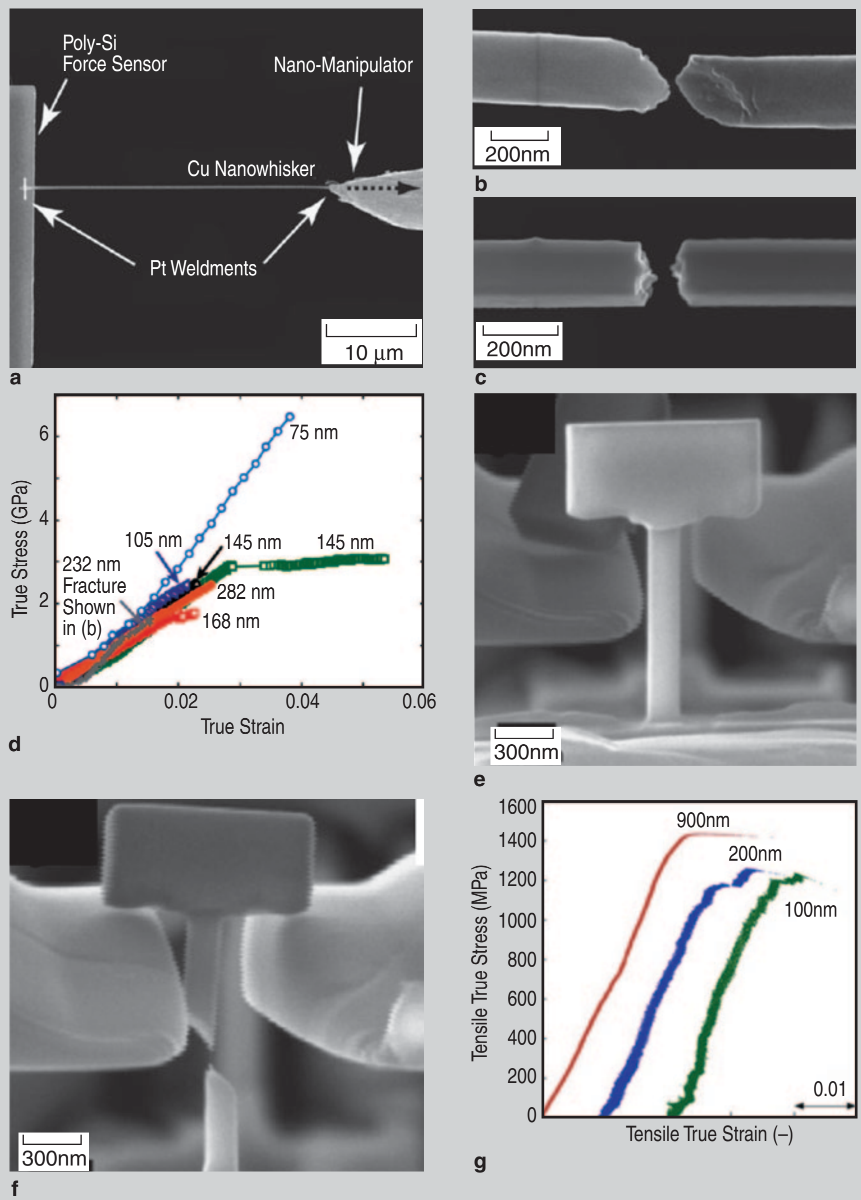

Figure 2. In-situ quantitative tensile testing of larger-diameter 1-D metallic nanostructures: (a–d) for a single crystal Cu nanowhisker;[34] (e-g) for a nanocrystalline Ni–4.4% W nanopillars.[32]

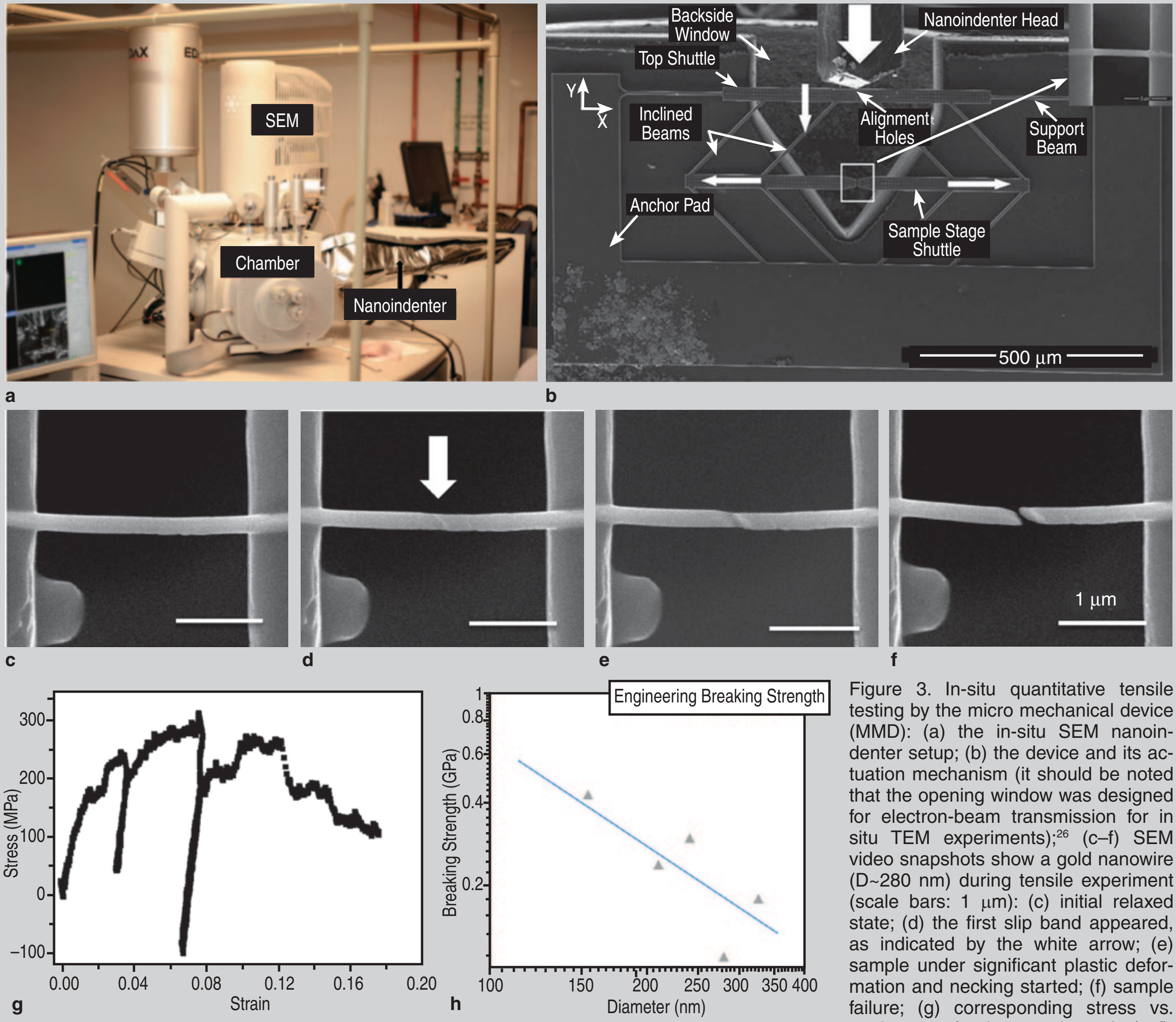

Figure 3. In-situ quantitative tensile testing by the micro mechanical device (MMD): (a) the in-situ SEM nanoindenter setup; (b) the device and its actuation mechanism (it should be noted that the opening window was designed for electron-beam transmission for in situ TEM experiments);[26] (c–f) SEM video snapshots show a gold nanowire (D~280 nm) during tensile experiment (scale bars: 1 μm): (c) initial relaxed state; (d) the first slip band appeared, as indicated by the white arrow; (e) sample under significant plastic deformation and necking started; (f) sample failure; (g) corresponding stress vs. strain curve for the test shown in (c–f); (h) a summary of five successful tests of gold nanowires with various diameters, clearly showing size effect.[27]

and rationalized that the properties of nanomaterials could be engineered by controlling initial defect and flaw densities.[32] In addition, the result shown in Figure 2d again demonstrated the typical "smaller is stronger" size effect as reported in many earlier papers.[5–10,31] Interestingly, in a recent paper by Greer group, using their custom-made in-situ mechanical testing platform SEMentor,[33] unusual size-induced weakening effect had exhibited for 60 nm-grained Ni-4.4% W polycrystalline nanopillars[34] (i.e., "smaller is weaker"). It was reported that grain-boundary-mediated deformation processes activated by the free surfaces at much larger grain sizes should be responsible for the lower attained strength.

Another way to perform quantitative in-situ tensile tests on 1-D nanostructure is to develop microelectromechanical systems (MEMS)-based testing platforms which allow a wider range of samples to be tested and could be small enough to fit into a TEM chamber. In the past decade, significant progress has been made by developing various MEMS devices to perform in-situ tensile experiments on metallic nanowires and thin films, carbon nanotubes, and biological fibrils.[35] However, most of these MEMS platforms relied on quite complicated setups that involved electro- or thermo-mechanical coupling effect and capacitance-based displacement/load sensing. Therefore, their implementations and adaptions to different testing environments could be both challenging and expensive. We have recently designed and developed a simple micro mechanical device (MMD),[26,36,37] which was based on a simple pure mechanical "push-pull" actuation mechanism. A quantitative nanoindenter was used to actuate the device and also to measure the load and displacement independently (Figure 3a). The simple design can significantly minimize the sources of errors and reduce the cost for the device fabrication. Its actuation involves the usage of an in-situ nanoindenter that applies a load on the top shuttle of the device in the vertical direction (along the y axis; see Figure 3b). Four sets of inclined symmetrical beams transform the motion of the top shuttle into a two-dimensional translation of the sample stage shuttles, resulted in the uniaxial tension on the nanowire sample. In Figure 3c–f, a single crystalline gold nanowire with diameter of ~200 nm was tested in tension. The sample failed in a very typical ductile mode, and shear bands were clearly developed in the plastic deformation stage (Figure 3d). Load applied on the sample and the sample elongation could either be derived from nanoindenter load and displacement data using conversion factors obtained from finite element simulations[36] or by a force reduction method.[37] The load and displacement resolution of the devices are dictated by that of the nanoindenter and can be in the order of a few tens of nano-newtons and a few nanometers, respectively.[26] Corresponding stress versus strain curve was plotted in Figure 3g, which included three loading-unloading sections for extracting sample's elastic modulus.

By using this new platform, systematic tensile studies have been performed on gold and nickel nanowires with diameters ranging from 70 nm–300 nm,[26,27] and both exhibited strong size effect. In addition, by plotting the nanowire diameter versus breaking strength in a logarithm scale, as illustrated in Figure 3h, a fitting parameter of ~0.598 was obtained. This is very close to the value of 0.61 for single crystal gold pillars reported earlier,[10] indicating the deformation mechanism for gold nanowires at this length scale (~hundreds of nanometers in diameter) might still be similar to that of micro and submicron pillar samples. Finally, it might be noted that, our device was also designed with the consideration for in-situ TEM experiments similar to some other MEMS device designs.[35,38,39] The needed device modifications included the reduced overall dimension and weight of the device, as well as the added open-window area directly underneath the sample stages for electron beam transmission. Aided by a high resolution TEM-nanoindenter holder (NanoFactory™ Instruments, Sweden), individual nickel nanowires with diameter around 300 nm were successfully tested inside a FEI™ Tecnai G^2 F30 TEM, while the stress versus strain data were simultaneously collected.[40] The real time TEM images and diffraction analysis provided useful insights into internal structural evolutions that are critical for uncovering the origin of the mechanical size effect in metals.

IN-SITU TENSILE TESTING: ULTRATHIN METALLIC NANOWIRES

In the past, mechanical characterization of metallic nanostructures with sub-10 nm diameter was mostly performed on samples prepared by in-situ formation of nano-sized junction via mechanically controllable break junction technique (MCBJT, as shown in Figure 4)[41] or by nanosized tip-substrate[42] / tip-tip[43] contact methods (Figure 5). Combining these sample preparation methods with commercially available

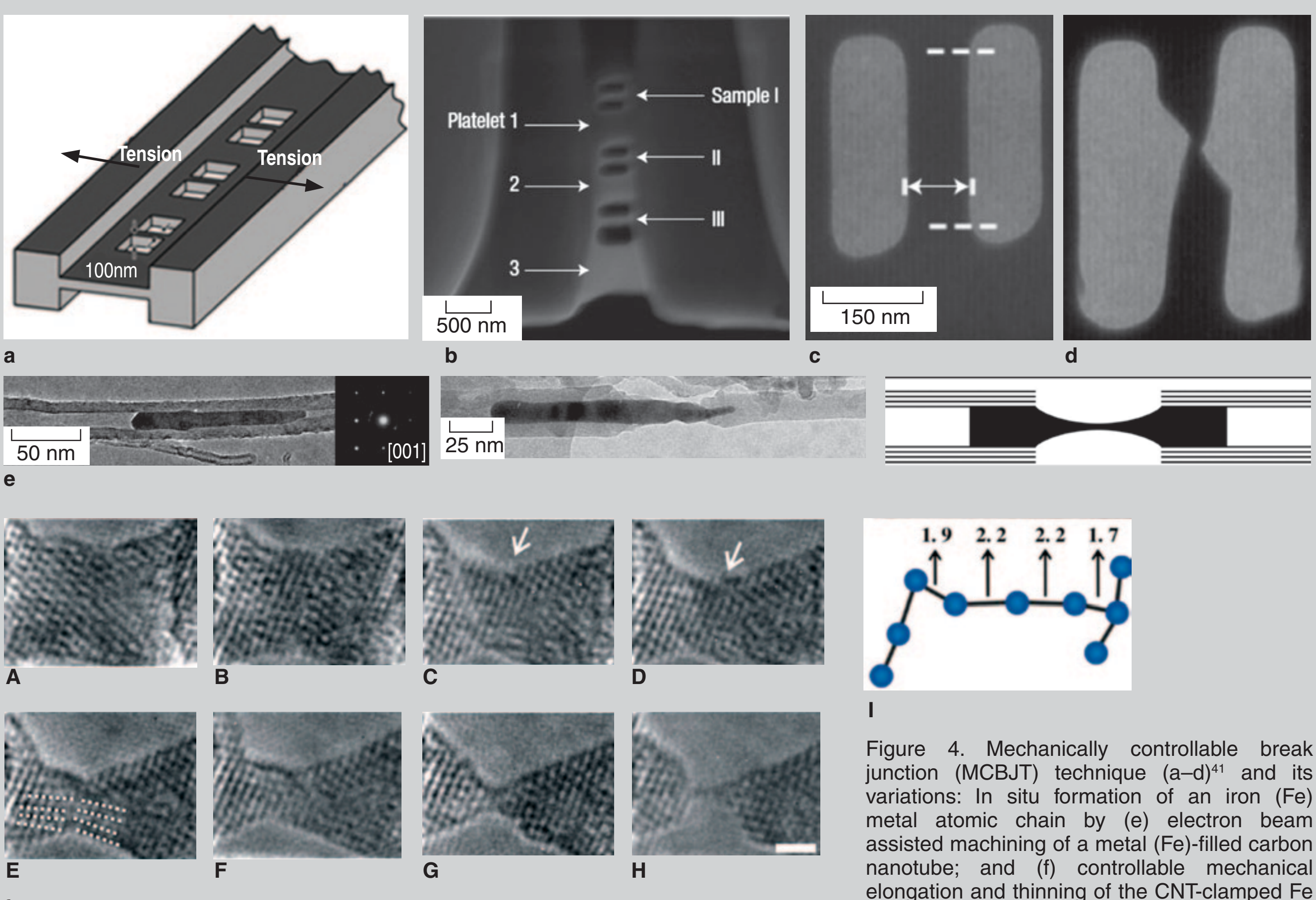

Figure 4. Mechanically controllable break junction (MCBJT) technique (a–d)[41] and its variations: In situ formation of an iron (Fe) metal atomic chain by (e) electron beam assisted machining of a metal (Fe)-filled carbon nanotube; and (f) controllable mechanical elongation and thinning of the CNT-clamped Fe nanowire junction (diameter <6 nm), due to the internal strain existing in the TEM sample.[44]

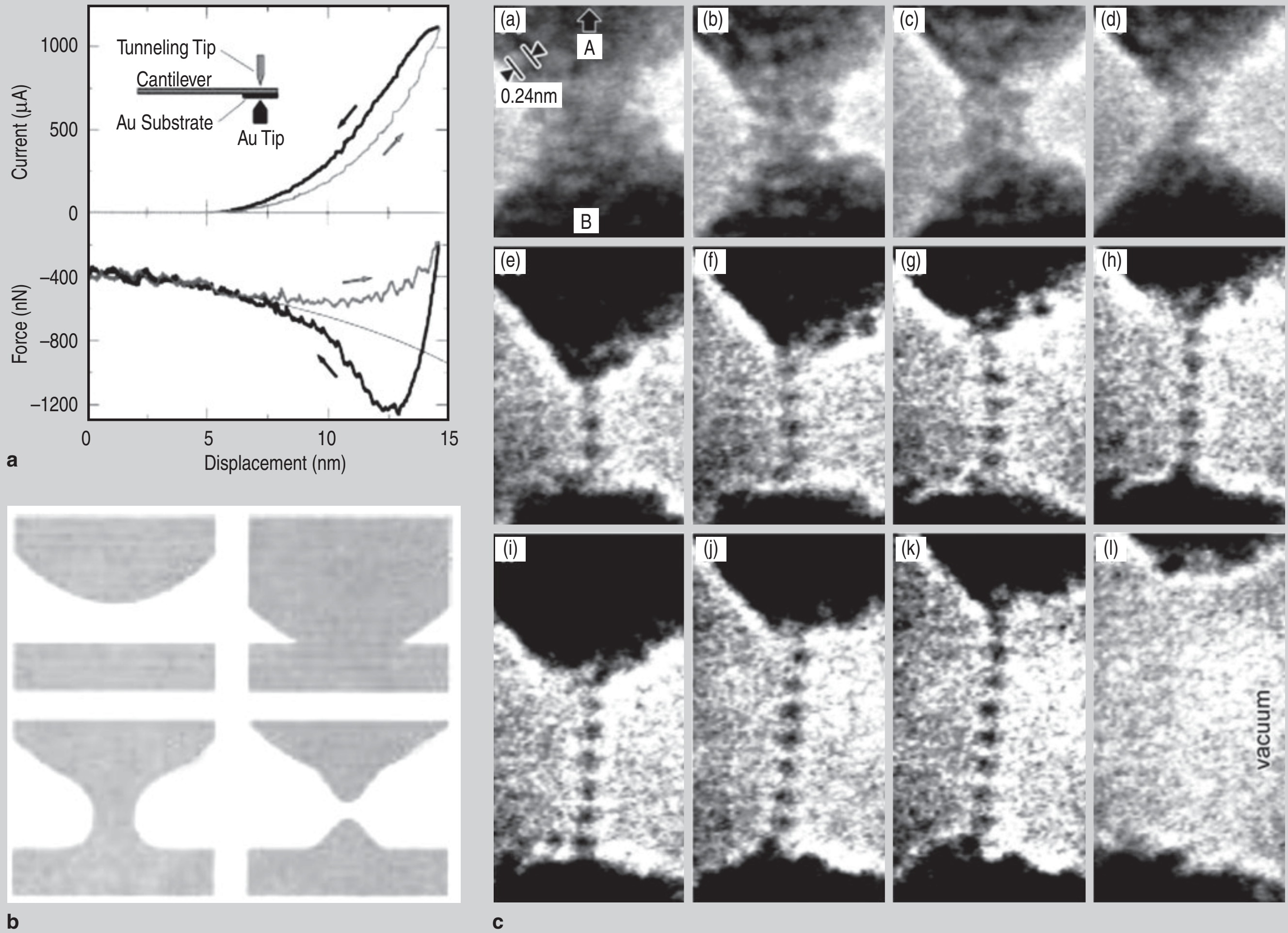

Figure 5. Tip-substrate contact method for in-situ gold nanojunction formation (a) and a schematic illustration of the concept (b), the setup also allows simultaneous force vs. displacement (as well as current) measurements;[42] (c) in situ straining of the gold junction with ~5 nm width until a single atom chain formed.[45]

qualitative in-situ mechanical straining stages inside a TEM provided an early overview of the mechanical deformation process of metals with ultra small dimension. In particular, deformation and fracture of gold atomic chains (Figures 4 and 5) were extensively studied. Several shortcomings of these earlier studies were the poorly controlled crystalline structure and orientation of the quasinanowire samples and non-uniform sample diameters (ranging from a few Armstrong to a few microns). These drawbacks hindered quantitative characterization and understanding of deformation and fracture mechanisms for metals at the nano to atomic length scales.

The recent development of novel instrumented TEM holders with quantitative capability for force and displacement (as well as electrical) measurements have been quite successful. Coupled with the advancement of chemically fabricated ultrathin metallic nanowires and the improvement of sample handling techniques, in-situ quantitative tensile tests of sub-20 nm nanowires become possible. By using a TEM-AFM sample holder (NanoFactory™), we reported one of the first in-situ quantitative tensile tests of gold nanowires with diameter less than 10 nm.[29] Rather than pulling randomly formed nanosized gold nano-junctions, we successfully clamped individual prefabricated free standing Au nanowires onto the AFM cantilever which acted as the force sensor (Figure 6), and performed quantitative in-situ tensile tests directly inside a high resolution TEM (Figure 6b–c). For quantitative tensile tests, an AFM cantilever beam with known spring constant (e.g., k = 4.8 N/m) was deflected by an individual nanowire sample pulled by the piezo tube under displacement control mode. Sample elongation and the change in diameter were monitored directly via real time TEM imaging. The applied force was then calculated by the recorded deflection of the AFM cantilever. This process can be further improved by adopting the latest TEM-AFM holder that is equipped with a highly sensitive MEMS-AFM sensor[46] providing fast force data acquisition.

The measured engineering breaking stress for the specific sample (Figure 6) was about 600±50 MPa that was much higher than that of a bulk crystal gold (~80 MPa).29 When considering the actual instantaneous diameter during the necking process, the measured true stress was about 7 GPa (Figure 6d), which is very close to the theoretical strength of gold crystal.[10] To fully reveal the underlying deformation mechanisms for sub-10 nm gold nanowires, researchers carried out qualitative tensile tests inside a high resolution transmission electron microscope (HRTEM). To facilitate high quality atomic imaging of the deforming region, shorter gold nanorods were normally used.[29,30] In-situ HRTEM tensile experiments done by H. Zheng et al. shows that it is the partial dislocations emitted from free surfaces that dominate the plastic deformation of sub-10 nm sized gold nanocrystal (Figure 7).[30] This is in sharp contrast with the traditional plastic deformation in bulk materials where plasticity is mediated by dislocation nucleation, multiplication and subsequent interaction. This observation demonstrates a good agreement with the results obtained from the corresponding molecule dynamics (MD) simulations in a qualitative manner.[30] The potential of bridging the gap between simulations and experiments seems to be quite promising with further developments of quantitative in-situ mechanical experiments of ultrathin nanowires and MD simulations with realistic time scales.

INSIGHTS GAINED

Finally, with the availability of ultrathin gold nanowires, the robust "cold-welding" sample clamping techniques as well as the in-situ TEM-AFM testing platform equipped with MEMS-based force sensor, systematic measurements of the mechanical strengths for <111>-oriented single crystalline ultrathin gold nanowires with different diameters (~5–15 nm) were performed.[47] Plotted together with the previous results from larger-diameter single crystal <111> gold nanowires (Figure 3), Figure 8a shows a comprehensive picture for size-dependent deformation behaviors of gold nanowires. A bi-linear relationship starts to emerge for the engineering tensile strength variations as a function of nanowire diameter. It was found that, while "smaller is stronger" still holds in general, the extent of increases in tensile strength for nanowires with decreasing diameter seemed to be more dramatic for larger diameter group (100–300 nm in diameters) than for ultrathin gold nanowires (D<15 nm). These trends agree well with earlier theoretical predictions based on results obtained from previously conducted pillar compression studies (Figure 8b),[10,16] experimentally confirming the existence of different deformation regimes for 1-D metallic nanostructures. Surface dislocation nucleation events predicted to dominate the deformation process in ultrathin nanowires have also been verified by in-situ qualitative HRTEM experiments of sub-10 nm gold nanocrystal and recent MD simulation result of ultrathin aluminum nanowires.[48]

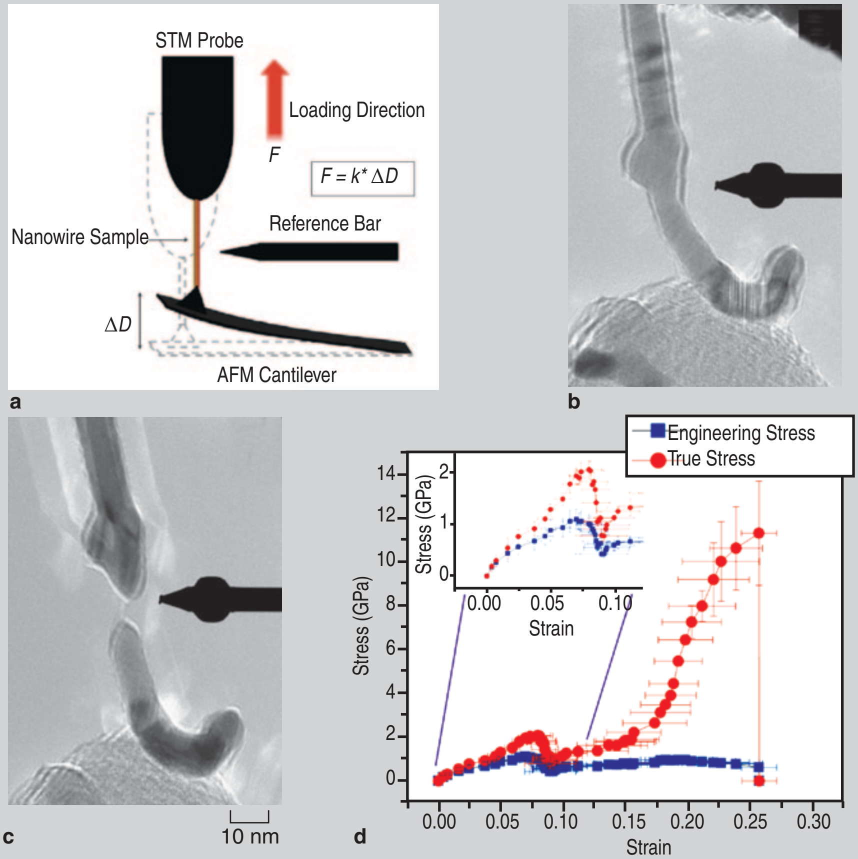

Figure 6. In-situ TEM quantitative tensile testing of an ultrathin gold nanowire with diameter of ~9 nm: (a) the experimental setup;[29] (b) before testing and (c) after breaking; (d) corresponding stress versus strain plots (for both engineering stress and true stress).[27] A stationary electron-beam-blocking bar was inserted as a reference marker.

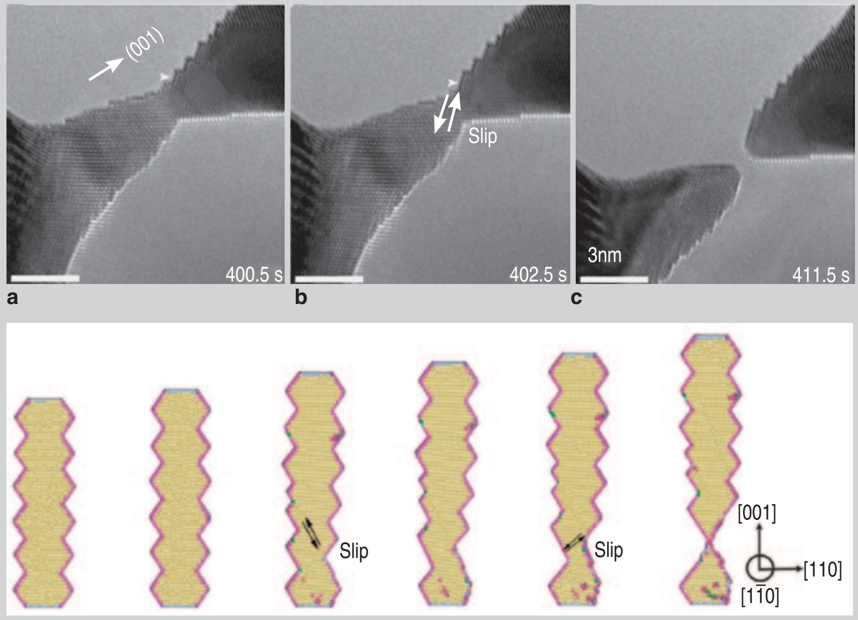

Figure 7. Qualitative in-situ HRTEM tensile testing of a sub-10 nm gold nano-crystal and corresponding MD simulation:[30] (a–b) the cooperative slip between two conjugate {111} planes, leading to the enlargement of the surface steps indicated by the arrowheads; (c) final fracture of the nanocrystal; (d–i) a side-profile view of the MD simulation of the necking process induced by slip (atoms are colored according to the coordination numbers).[30]

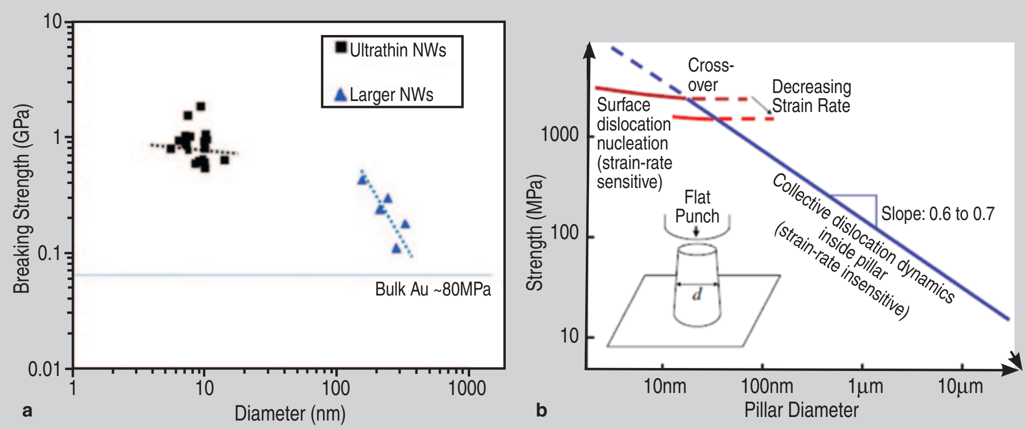

Figure 8. Size dependence of mechanical behavior of gold nanowires with diameter from a few nanometers to hundreds of nanometers:[27] (a) Engineering breaking strength versus diameter in logarithm scale): the black squares were data points for ultrathin gold nanowires tested inside a TEM and the blue triangles were data points from the larger diameter gold nanowires tested using micro mechanical devices inside a SEM; (b) Illustration of the surface effect on the rate-controlling process and the size dependence of yield strength in micro-and nanopillars of various diameter under compression.[16]

CONCLUSION

In order to have a complete understanding of size-dependent mechanical behaviors of metallic nanowires, it is necessary to test more Au nanowires in a broader diameter range. Additionally, current quantitative mechanical testing for larger-diameter metallic nanowires was mostly done inside SEM. By using the newly developed TEM-compatible MEMS testing stage, more insights from in-situ TEM tests of larger metallic nanowire sample are expected for a better understanding of deformation mechanism mediated by dislocation multiplications and interactions. However, sample orientations will be critical in order to observe important dislocation features. The important double tilting operation may raise new challenges for employing the MEMS-based platforms, which clearly needs further investigations.

Finally, in-situ electromechanical and thermal-mechanical characterizations of metallic nanowires could provide unique opportunities to study detailed mechanisms of these coupling effects on physical responses of metals at an unprecedented level. Due to the versatility of many developed quantitative in-situ testing methods, other multiphysics investigations of irradiation effects, electrochemical effects and optical effects on mechanical behaviors could also be carried out with these 1-D metallic nanostructures.

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