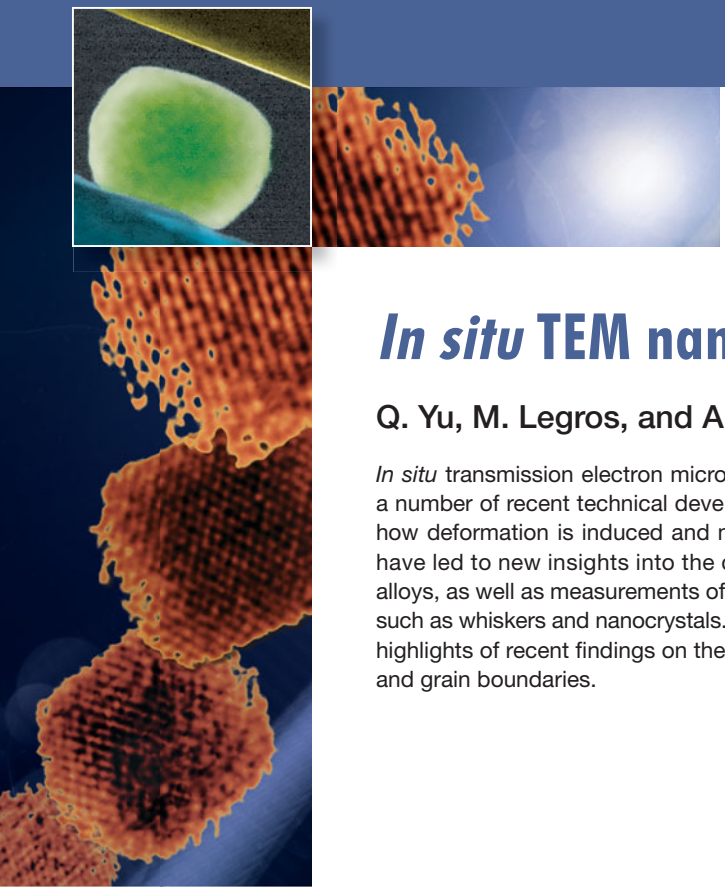


In situ TEM nanomechanics

Q. Yu, M. Legros, and A.M. Minor

In situ transmission electron microscopy (TEM) nanomechanical testing has benefited from a number of recent technical developments related to both how deformation is imaged and how deformation is induced and measured inside a TEM instrument. These developments have led to new insights into the deformation mechanisms of a wide range of metals and alloys, as well as measurements of the unusual mechanical properties of small-scale objects such as whiskers and nanocrystals. Herein, we describe this recent progress through selected highlights of recent findings on the dynamic behavior of defects such as dislocations, twins, and grain boundaries.

Introduction

The mechanical properties of any material are highly correlated with its defect structure. Starting in the late 1950s,[1] *in situ* straining stages for use in transmission electron microscopes that could provide dynamic observations of dislocation motion in metals were developed. In the following years, research teams fabricated various transmission electron microscopy (TEM) straining holders, including some that could operate at low or high temperature. Since then, *in situ* TEM straining tests have been performed to study the behavior of dislocations and their interactions with other defects such as twins, grain boundaries (GBs), and interfaces.

After early load sensor integration developments,[2,3] *in situ* TEM mechanical testing with quantitative load measurement expanded toward commercialization in the beginning of this century, including mechanical probing modes such as nanoindentation.[4] These developments are particularly useful for studying size effects in materials, whereby mechanical properties and microstructure can be tuned by the physical dimensions (internal such as grain size or external such as film thickness) of materials.[5–7] At the micrometer or nanometer scale, the sample dimensions can be of the same order as the defects that carry the deformation, such as the length of a dislocation line or the thickness or spacing of twins. When this occurs, strong size effects can change the deformation mechanisms and the corresponding mechanical properties. Quantitative *in situ* TEM mechanical testing is therefore an ideal tool for studying the origin and principles of these size effects. This review provides an overview of available *in situ* TEM techniques and recent studies performed on dislocation behavior, dislocation/boundary interactions, deformation twinning, and GB-mediated plasticity. We also provide a critical discussion of the effects of size on different deformation mechanisms and the correlated mechanical properties of materials. Some of these mechanisms are summarized in **Figure 1**.

In situ TEM straining techniques

Various tools for mechanical testing by a transmission electron microscope that exist today[8] are briefly detailed in this section. They span from simple motorized grips to elaborate mechanical testing units capable of returning values for load and displacement.

In classical tensile holders for TEM, samples are attached to a pair of jaws, and one side is slowly pulled by an electric motor located in a housing unit outside the TEM instrument. A rigid shaft ensures strain transmission at speeds ranging from 10 nm/s to 10 μm/s. This simple technique[9] has various advantages because of its high versatility. Virtually any thin or thinned sample can be strained as soon as it is attached to

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DOI: 10.1557/mrs.2014.306

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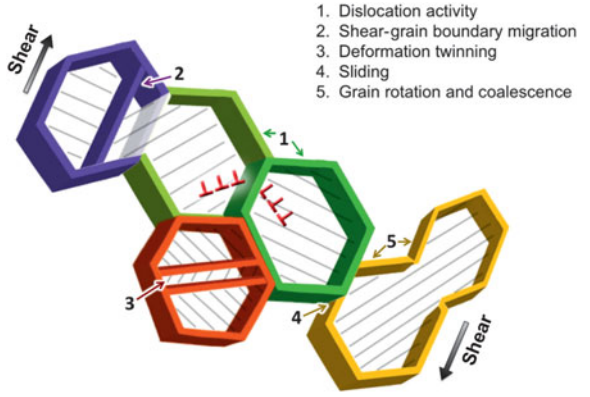


Figure 1. Schematic of various deformation mechanisms that can be studied by *in situ* transmission electron microscopy mechanical testing.

a stretchable grid.[10] Options for heating (up to 1200°C with a miniaturized furnace as shown in **Figure 2**a) or cooling (down to liquid He or, more often, liquid N_2 temperatures) can be easily added. The very rigid strain transmission allows for the deformation of thin films[12,13] or brittle alloys.[14,15] Incorporating a load cell[16] or double-tilt capability[17] can soften strain transmission and therefore be detrimental for controlling crack propagation in samples whose brittleness can be increased by smaller dimensions.

Other custom-made holders based on tensile actuation such as bending[18] and pure shear[19,20] holders can be found in the literature (for full reviews, see References 8 and 21). Heating holders can also be used to strain metallic films on hard substrates provided that the difference in coefficient of thermal expansion between the two materials is sufficiently high.[18] Both plan-view and cross-sectional configurations have enabled insights into observations of metal–interface deformation mechanisms.[22,23]

Nanoindentation holders are derived from the instrumented indentation technique that consists of measuring the force and displacement of a hard tip forced into a flat surface. This technique is used for measuring the hardness; the elastic modulus; and under certain conditions, the yield stress of a material.[24] The force and displacement are usually measured through a change in capacitance between a reference and a movable electrostatic plate attached to the tip.[25–27] In the 1990s, several holders were developed based on the accurate positioning capabilities of piezoelectric stacks and tubes,[28–32] followed by nanoindentation tips holders initially used to deform metals[33–38] and access plasticity mechanisms.[39,40] A few companies (e.g., Hysitron and Nanofactory Instruments AB, although the latter ceased commercial activities in 2013 and was purchased by FEI) further implemented these holders with sensors to read applied forces.[41–43] Maximum loads accessible with such instruments are typically lower than 10 mN, and the noise floor is typically below 0.5 μN. Atomic force microscopy holders have also been developed for smaller force ranges.[44]

To be indented, a TEM sample must have a specific geometry,[45] such as a free half-space (Figure 2b) in front of the electron-transparent zone. The use of cylindrical nanopillars with a constant section deformed by flat punch tips is the easiest way to measure stress from load values.[46–48] Standard samples are currently shaped as nanopillars, wedges, or H-bars, as shown in Figure 2b.

Other tests are neither pure compression nor real indentation tests for which known approximations can apply.[25] Finite element calculations therefore are needed to quantitatively determine the stress transmitted to the sample[49–51] in tests such as wedge/wedge indentations (wedge indenters facilitate contact and prevent slipping). The contact of the tip with the sample generates strain gradients that also disrupt imaging conditions during the deformation. These disruptions can be partly overcome by capturing dynamic sequences in dark field,[47] by correcting the bending as the test progresses, or by using a damping region.[51]

Some holders are based on microelectromechanical systems (MEMS) that can be either passive (mounted on straining or nanoindenter holders) or active (with electrical input and output). Push-to-pull (or PtP) devices are passive MEMS devices that consist of a microfabricated Si platform that is elastically deformable in compression (using nanoindentation holders) and a pair of facing pads to which the sample to be pulled is attached.

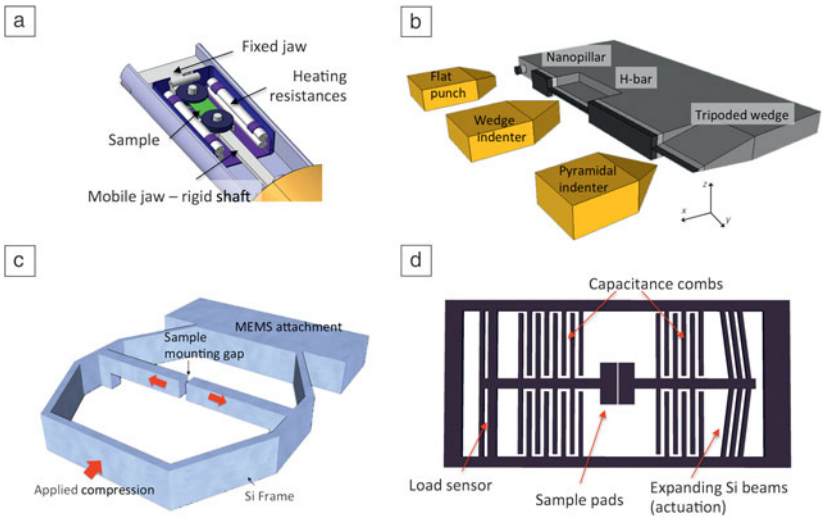


Figure 2. Examples of *in situ* transmission electron microscopy mechanical testing techniques: (a) Tip of a classical straining holder with high-temperature option, (b) sketch of the various types of straining configurations used in nanoindentation holders, (c) theta (θ) geometry for a push-to-pull microelectromechanical systems (MEMS) device, and (d) an electrically driven MEMS device for *in situ* measurement of load and strain. Adapted from Reference 11. © 2011 American Institute of Physics.

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In Figure 2c, for the θ- shaped example (derived from References 52 and 53), the sample is located in the middle of the MEMS device, and compression on the outside of the structure provides tension in the central gap. In addition to the θ shape, other PtP geometries[54] can also maintain stable fields of view during actuation.

Stress–strain curves obtained with these techniques need to be corrected by removing the stiffness of the empty PtP. The dimensions and corresponding stiffness of the PtP can be adapted to the load range investigated, depending on the type of sample tested (fibers, nanowires, films). To avoid micromanipulation (harvesting, welding) of fragile samples,[55] the co-fabrication of self-supported nanocrystalline films with an elastic testing frame is also possible, either on simple structures[12] or on elaborate MEMS devices (Figure 2d), where the relative displacement of Si beams with known stiffness serves to calculate the applied load.[11,56,57] Relative displacements in the MEMS structure can be measured by TEM, but capacitance readouts can allow for direct access to the displacement of Si combs and, thus, to strain and load. Joining of linear actuators based on the thermal expansion of Si completes the design of these micromechanical testing machines that can be plugged into an electrical holder to perform monotonic or fatigue testing.[11,58–60] Finally, electrostatically actuated MEMS devices working in very low force ranges (1–100 nN) can be used to study nanojunctions or friction near the atomic level.[61–63]

Observations of grain boundary behavior

Grain boundary and dislocation interactions

Preventing dislocation motion by grain refinement has long been a practical way to engineer mechanical properties.[64–66] Therefore, understanding the interactions of dislocations with GBs is of fundamental importance in tailoring the strength of polycrystalline materials. GBs and interfaces serve not only as barriers for dislocation motion, but also as sources for generating multiple dislocations or even as dislocation sinks.[67] Traditional *ex situ* structure and property characterization gives only an ensemble viewpoint of the interaction between dislocations and GBs. On the contrary, *in situ* TEM mechanical testing offers a tool that can visualize the individual dynamic interactions between dislocations and other defects.

Observations of GBs acting as dislocation sources have been reported for various materials, including molybdenum and copper.[68–70] It has also been observed that the incorporation of dislocations into the boundary is highly related to misorientation between adjacent grains.[71,72] As an example of the use of *in situ* TEM to study dislocation interactions with different grain misorientations, Ohmura et al.[73] performed *in situ* TEM nanoindentation on Fe–0.4 wt% C tempered martensitic steel and studied the dynamic evolution of the dislocation pileup structure with both a high-angle GB and a low-angle GB (**Figure 3**). They observed that in the case of a low-angle GB, the dislocations induced by the indenter piled up against the boundary, as shown in Figure 3a (right side). A high density of dislocations was then suddenly emitted on the far side of the GB into the adjacent grain as a critical stress was reached during nanoindentation. Figure 3b shows the case of the high-angle GB, for which numerous dislocations were simply absorbed into the boundary, with no indication of pileup or strain transmission.

As illustrated by the preceding examples, *in situ* TEM experiments provide a window into the dynamic interactions between dislocations and GBs with known structural features. Taken together, dislocation–GB interactions are complex processes, and the before and after conditions for a given interaction provides valuable insights into dislocation plasticity mechanisms. Evaluating the strength of a GB as an obstacle requires careful analysis of grain orientations and dislocation character.[74] Simple criteria for dislocation crossing can be found in ideal cases such as dislocation–nanotwin interactions,[75] but they can be difficult to predict *a priori* for any general boundary. The interaction between dislocations and GBs is also sensitive to the chemical composition, which not only changes the boundary energy, but also increases barriers to dislocation motion.[76] Because most earlier *in situ* TEM studies on this subject were largely phenomenological,

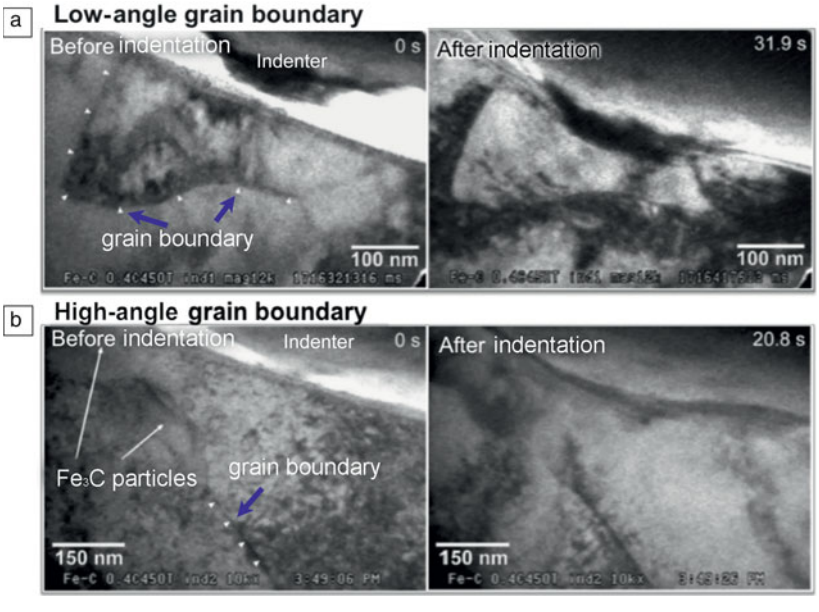


Figure 3. Interactions of a dislocation with (a) low-angle and (b) high-angle grain boundaries. A dislocation pileup was observed in the case of a low-angle grain boundary. The high-angle grain boundary absorbed dislocations. Reproduced with permission from Reference 73. © 1953 Elsevier.

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more systematic *in situ* TEM tests with quantitative stress measurements are required to investigate the details of dislocation–boundary interactions.

Grain boundary motion

When the mean free path of dislocations is further impeded by GBs, which occurs in nanocrystals, a very large increase in mechanical resistance is observed.[77] At the smallest scales (usually in the range of several to tens of nanometers), *in situ* TEM has shown that metallic polycrystals deform through alternative, GB-based plasticity mechanisms that can occur at lower temperatures and more rapidly than anticipated from macroscopic values. This is the case for stress-assisted grain growth[78] and rotation,[13,79] for which diffusion coefficients seem to be close to those of the liquid phase, even at room or moderate temperatures.[44]

Among these mechanisms, shear-migration coupling has recently been the focus of many theoretical and experimental studies. In contrast to dislocation-based plasticity, the shear produced by a moving GB can result in different values, characterized by a coupling factor parameter called β. Recent results obtained by *in situ* TEM of ultrafine-grained aluminum show that many deformation modes are activated, including shear-migration coupling.[80,81] The coupling factor β can be measured experimentally using image correlation analysis, as in **Figure 4**a–b, where nanoparticles on the surface serve as fixed references. The returned values can be compared with the predictions of models such as that of Cahn and Mishin,[81] and although numerous statistical data are still missing, β sometimes appears smaller than predicted. One reason for this discrepancy could lie in the atomic-scale mechanisms that guide the migration of GBs. The Cahn and Mishin model assumes the motion of intrinsic GB dislocations (therefore directly related to the GB misorientation), whereas Rae and Smith insist on the role of step propagation.[82] When no stress is involved, GBs can move without inducing any strain (β = 0), as in Figure 4c–d. High-resolution imaging of bicrystals shows that steps decorate GBs and that the motion of imperfect steps could result in the shear-associated migration of GBs.[83] Recent simulations showed that step dislocations (disconnections) are probably the basic mechanism leading to GB migration.[84] These disconnections (marked D in Figure 4a) are found in non-ideal, far-from-equilibrium GBs, largely present in nanocrystals, and can be created from interactions between lattice dislocations and GBs.[83,85] They are also evidenced in grain rotation.[86]

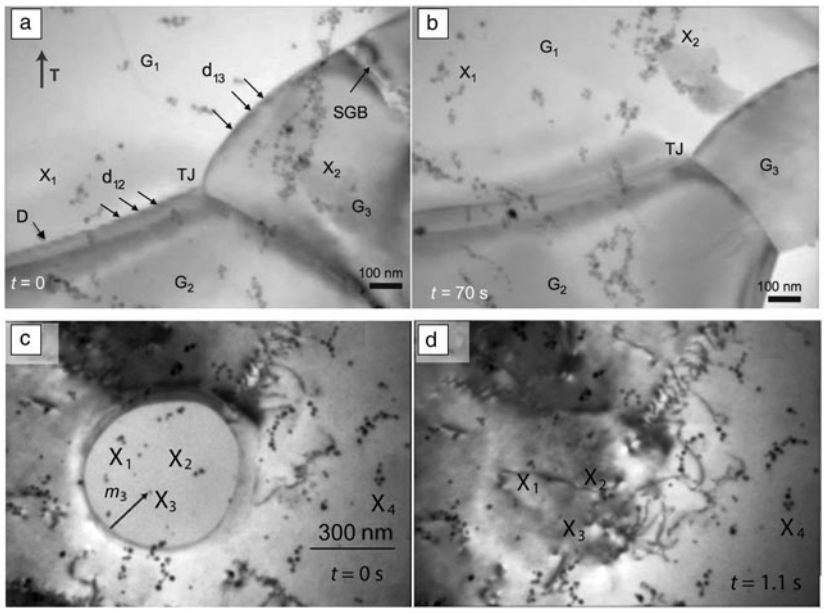


Figure 4. Stress-related grain boundary (GB) motion. $X_{1–4}$ represent fixed markers (surface nanoparticles). (a–b) Stress-induced GB motion in ultrafine Al at 350°C. GBs denoted d_{12} and d_{13} move toward the lower right of the image. (c–d) Grain annihilation in Au at 300°C. In the absence of stress, this GB motion does not engender any strain. (a–b) Reproduced with permission from Reference 83. © 2013 Taylor & Francis. (c–d) Reproduced with permission from Reference 86. © 2012 Elsevier. Note: D, step dislocations (disconnections); G_1, grain 1; G_2, grain 2; G_3, grain 3; TJ, triple junction; SGB, subgrain boundary; *t*, time; m_3, migration distance to X_3; **T**, straining axis.

Observations of size-related dislocation slip

Intrinsically, the mechanical properties of a metal can be directly related to the physical dimensions of a single defect. For instance, the yield strength of a single crystal is correlated with the critical resolved shear stress $\tau_{CRSS} = Gb/L$, where L is the length of the dislocation, G the shear modulus and b the Burgers vector. This phenomenon becomes quite important as the physical dimensions of the material decrease to the same order as the size of the dislocations. By combining nanofabrication techniques and quantitative *in situ* TEM nanomechanical testing, the effect of size on dislocation behavior and its corresponding impact on the mechanical properties of small samples have been broadly studied in fcc-, bcc-, and hcp-structured materials.[87,88]

In fcc materials, such as Ni and Cu, both *in situ* TEM nanocompression and nanotension experimental results suggest that dislocation multiplication can be constrained by crystal size; dislocations can easily slip out of the sample through the crystal surfaces without any multiplication events. Shan et al. obtained direct evidence for such exhaustion phenomena in Ni nanopillars.[89] Kiener and Minor[90] carried out quantitative *in situ* TEM tension experiments on Cu and showed that a decrease in dislocation density was associated with hardening of the material, as shown in **Figure 5**a. They also emphasized the role of sources to control the flow stress and, in some cases, to balance the loss of dislocations.[47,91]

In bcc materials, observations of dynamic dislocation activities were achieved by straining Mo fibers *in situ* in tension using a PtP MEMS device. As shown in Figure 5b, dislocation multiplication was observed before

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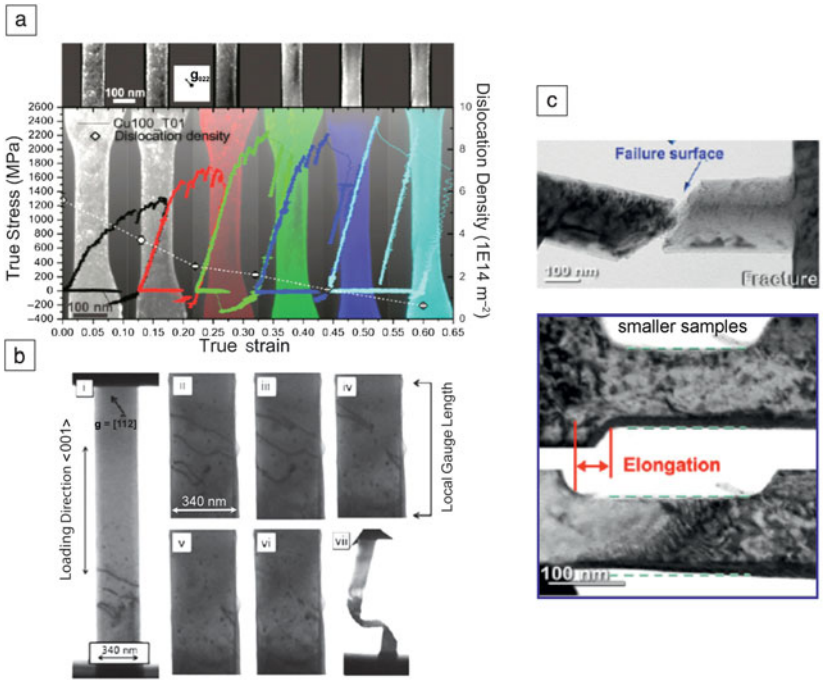


Figure 5. Dislocation behaviors in fcc, bcc, and hcp materials studied through *in situ* transmission electron microscopy mechanical testing. Dislocation starvation was observed in both (a) fcc[90] and (b) bcc materials. Generally speaking, dislocation starvation refers to the phenomenon in which the dislocation density dramatically decreases to an extremely low value. (a) The dislocation density (defects can be seen as bright features in the upper row of dark-field images), as denoted by the white diamond symbols, and the values on the right-hand axis of the graph reduce by an order of magnitude. Correspondingly, the deformation characteristics become more stochastic as the defect density decreases. (b) (i–vii) Representation of the trend of decreasing dislocation density in a bcc Mo fiber during tension. Dislocation multiplication was also observed in the bcc material before the escape of dislocations from the surface.[92] (c) Multiple slip has occurred in smaller hcp Mg samples, resulting in better ductility. Note: **g**, diffraction vector.

the dislocations were annihilated at the surface. The linear elastic stage of the mechanical curve is not affected until the annihilation of the dislocation source and the subsequent occurrence of a dramatic plastic instability. Similar to fcc materials, multiple slip systems might have been activated after the primary slip system was exhausted.

The activation of multiple slip systems in small samples facilitates arbitrary changes in shape. In materials with high deformation anisotropy such as hcp-structured materials, this effect was found, by *in situ* TEM nanomechanical testing, to be enabled through size reduction. For example, in single-crystal Mg, it was observed that multiple dislocation slip systems were activated once a critical crystal size (and hence a critical strength) was reached. As shown in Figure 5c, even in orientations where basal slip would dominate the deformation at a larger scale,[92] smaller crystals deformed at higher stresses, where the interaction between different slip systems significantly enhanced the strain hardening and the ductility of the material.

Observations of deformation twinning

Twinning deformation can involve the cooperation of different types of dislocations. Therefore, compared with perfect dislocation glide, plasticity based on twinning is more difficult to study, because it occurs as a collective mechanism, and its origin and evolution in space and time remain puzzling.[93] *In situ* TEM nanomechanical testing has recently provided new insights into deformation twinning by revealing the nucleation of twins and their propagation.

The nucleation and growth of deformation twins have been directly observed using *in situ* TEM mechanical testing in both Mg and Mg alloys. Yu et al.[94] and Ye et al.[95] reported the formation of deformation twins in submicron single-crystal Mg and a Mg alloy in compression. In both studies, twinning was observed to nucleate from a corner at the contact surface. Significant dislocation activity was observed during twin growth, specifically across the twin boundary. In Mg, $(10\bar{1}2)$ twins were also studied by *in situ* TEM nanotension experiments. Liu et al.[96] reported the formation of a $(10\bar{1}2)$ twin from the bottom of the sample by pulling a Mg single crystal along the [0001] direction and further compressing the sample along the $[1\bar{1}00]$ direction, as shown in **Figure 6**a. Yu et al. also performed *in situ* TEM nanotension tests on single-crystal Mg bars oriented along [0001], but instead of a single twin, they observed the formation of many nanotwins across the entire sample length (Figure 6b).[94] Similar nanotwinned structures were observed in *in situ* TEM nanobending tests as well, in which case the formation of the nanostructured twin occurred right in front of the crack tip. The discrepancy in the results between Yu et al. and Liu et al. demonstrates that subtle factors such as surface roughness and dilute compositional differences can often influence deformation in small-scale tests.

The critical stress for compression twin nucleation in pure Mg was found to be approximately 750 MPa in Yu et al.'s study.[94] The critical stress for the nucleation of an extension twin was about 800 MPa on average in Yu et al.'s tensile tests[94] and about 1.2 GPa in Ye et al.'s compression tests.[95] The same value varied between about 500 Mpa and 800 MPa in Liu et al.'s case.[96] This wide variation in quantitative data demonstrates the stochastic nature of small-scale mechanical testing, as well as the significant size effects associated with the twinning mechanism. In Yu et al.'s work,[94] significant strain hardening was obtained after the formation of a nanotwinned structure, presumably resulting from the high density of twin boundaries, which serve as barriers to further dislocation motion. The maximum flow stress then increased to about 1.2 GPa, ~400 MPa higher than that in Liu et al.'s study.[96]

In situ TEM nanomechanical testing provides direct insights into deformation twinning, the related mechanical properties,

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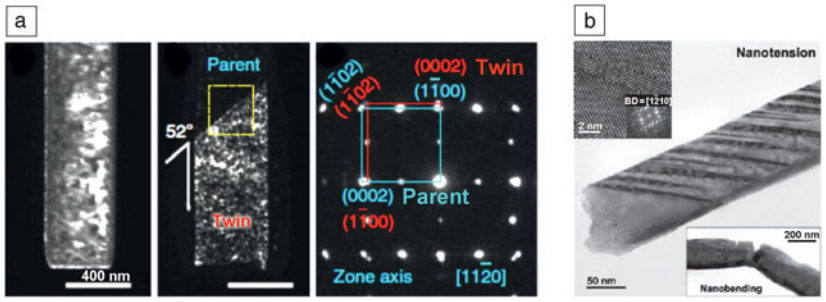


Figure 6. Deformation twinning behaviors studied through *in situ* transmission electron microscopy (TEM) mechanical testing. (a) Formation of a $\{10\bar{1}2\}$ twin in a nanocompression test on single-crystal Mg oriented along $[1\bar{1}00]$.[96] Dark-field TEM images showing the pillar (left) before and (right) after loading. (b) Formation of nanotwins in nanotensile and bending tests (lower right inset). The upper left inset image is a typical high-resolution TEM image showing the atomic structure of the nanotwins. Note: BD, beam direction. Reproduced with permission from Reference 93. © 2010 Nature Publishing Group.

and their relation to crystal size. However, the observation of very fast twin nucleation events remains a challenge. In addition to future improvements in sample preparation, higher time and spatial resolutions are needed to understand the complete picture of twin nucleation.

Summary and outlook

Several examples of dislocation plasticity, twinning, and GB mechanisms observed by *in situ* TEM mechanical testing were described, where the mechanisms of interest are directly related to the mechanical properties. However, *in situ* TEM mechanical testing can also be used to interrogate properties and mechanisms in materials beyond defect interactions, such as phase transformations,[97,98] ultrafast electromechanical quenching,[99] and electron-beam irradiation effects[100] (see, for example, **Figure 7**). As *in situ* mechanical testing becomes more sophisticated, experiments will extend beyond the simple observation of mechanisms to measurements of key mechanical parameters.

Recent advances in TEM sample preparation and imaging techniques have the potential to make observations clearer and measurements more precise. For example, direct measurements of strain can now be accomplished using imaging techniques such as holographic strain measurement at small scales[101] or automated crystalline indexing tools.[55,102] Sample preparation will continue to improve, as a result of new sample handling and microfabrication technologies. Faster direct electron detectors working at the millisecond time scale can capture faster mechanisms[103] and/or collect more information.[104] Faster experiments can also enable clearer imaging of deformation, as stability during contact is always an issue. Of course, capturing ultrafast events such as dislocation nucleation in a perfect crystal might require ultrafast technologies[105] and remains a challenge.

In the future, a critical area for research will be the adaptation of *in situ* TEM mechanical testing to environmental conditions relevant to mechanical behavior. Whereas high-temperature straining has long been widely available, adapting quantitative techniques such as indentation to work at low or high temperatures has been more difficult. Beyond simply high-temperature environments, it is critical to study environmentally assisted deformation mechanisms such as stress-corrosion cracking. Many outstanding questions remain concerning the mechanisms by which interfacial oxides form in cracks and contribute to the dramatic reduction in fracture toughness. The ability to examine, with atomic resolution, the structure and chemistry of these films and the location of crack initiation using *in situ* TEM nanomechanical testing will lead to fundamentally new insights in this important field.

In summary, *in situ* mechanical testing in a transmission electron microscope is uniquely suited for investigating the origins of both strength and ductility in structural materials and has proven to be advantageous for directly observing and measuring plastic deformation due to dislocation plasticity and twinning. New technological developments in the field promise to enable greater insights into the mechanics of an even wider range of materials in a wider range of environments.

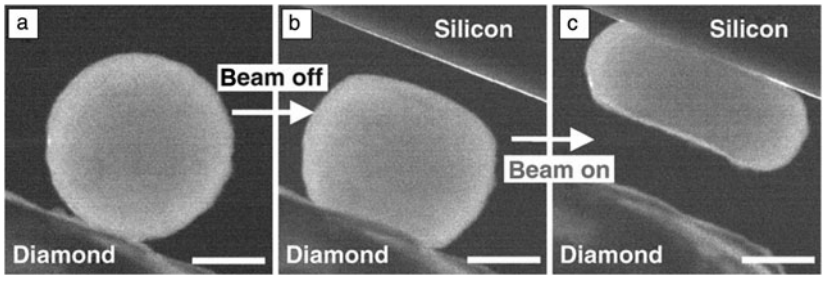


Figure 7. Example of the use of *in situ* nanomechanical testing to probe electron irradiation effects in silica nanoparticles. (a) The silica nanoparticle prior to compression. Two consecutive compression tests were performed: (b) first with the electron beam off and (c) then with it on. The silica particle was found to be much softer under electron-beam illumination. Scale marker is 200 nm. Reproduced with permission from Reference 100. © 2010 Nature Publishing Group.

Acknowledgments

A.M.M. was supported by the Molecular Foundry at Lawrence Berkeley National Laboratory, which is supported by the US Department of Energy under Contract DE-AC02-05CH11231. M.L. was supported by the French National Research Agency under the "Investissement d'Avenir" MIMETIS program (reference ANR-10-EQPX-38-01).

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