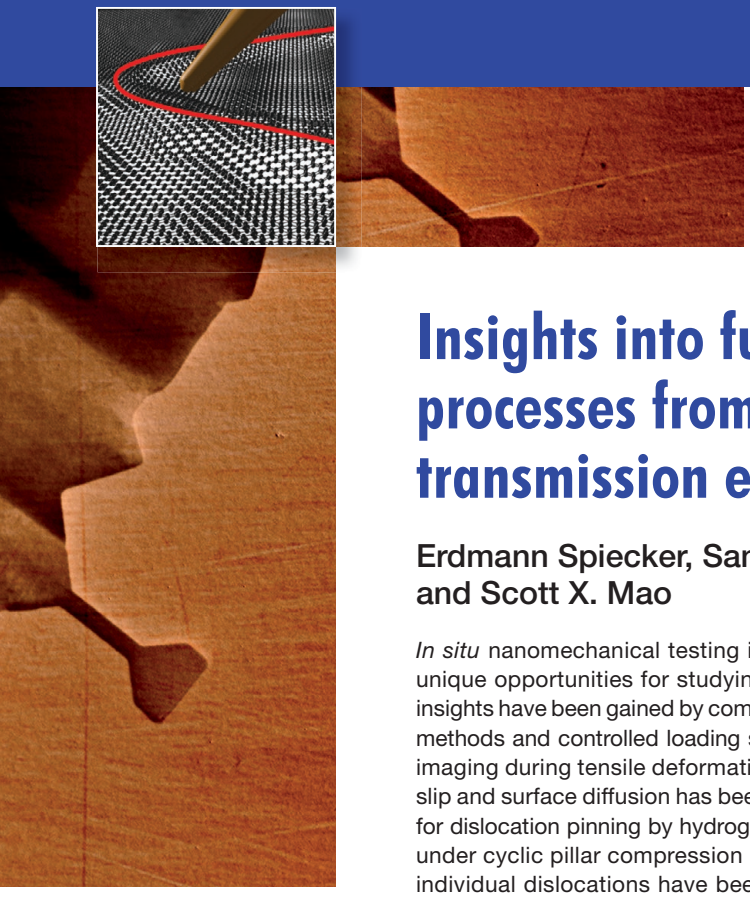


Insights into fundamental deformation processes from advanced *in situ* transmission electron microscopy

Erdmann Spiecker, Sang Ho Oh, Zhi-Wei Shan, Yuichi Ikuhara, and Scott X. Mao

In situ nanomechanical testing in (scanning) transmission electron microscopy provides unique opportunities for studying fundamental deformation processes in materials. New insights have been gained by combining advanced imaging techniques with novel preparation methods and controlled loading scenarios. For instance, by applying *in situ* high-resolution imaging during tensile deformation of metallic nanostructures, the interplay of dislocation slip and surface diffusion has been identified as the key enabler of superplasticity. Evidence for dislocation pinning by hydrogen defect complexes has been provided by *in situ* imaging under cyclic pillar compression in a tunable gas environment. And, for the very first time, individual dislocations have been moved around *in situ* in two-dimensional materials by combining micromanipulation and imaging in a scanning electron microscope.

Introduction

Relating the macroscopic deformation behavior of a material to its microscopic defect processes is one of the key challenges of materials science. Transmission electron microscopy (TEM) has contributed continuously and extensively to this endeavor, starting with the investigation of crystal dislocations more than 60 years ago.[1] Already in these early days, first *in situ* mechanical testing stages were developed to observe dislocations at work under applied load.[2,3] Since then, a large variety of sample holders and devices for *in situ* nanomechanical testing inside the TEM have been explored, ranging from classical *in situ* straining stages to nanoindentation holders and microelectromechanical systems-based actuators.[4,5] Progress has also been made in the preparation of highly versatile samples for nanomechanical testing. In particular, modern focused ion beam (FIB) instruments enable site-specific preparation of samples with increased surface quality,[6–9] which can be further improved by post-treatments.[10,11] Finally, TEM and scanning transmission electron microscopy (STEM) technologies have seen major developments in recent years with regard to resolution, stability, detection efficiency, and data processing, all of which are available for *in situ* mechanical testing studies.

The development of *in situ* TEM/STEM nanomechanics has been documented in a number of review articles.[4,12–14] Here, we describe further progress by presenting selected recent work in which advanced *in situ* TEM/STEM studies have provided direct insights into fundamental deformation and defect processes in different classes of materials, ranging from metallic nanostructures to nanotwinned biological materials, from well-defined ceramic bicrystals to ultimately thin two-dimensional (2D) materials. Advanced *in situ* aspects range from atomic-resolution studies during mechanical straining, to highly controlled sample geometries and loading scenarios, to cyclic loading combined with environmental TEM, to micromechanical defect manipulation in low-voltage STEM.

Interplay of dislocation slip and surface diffusion

In nano-sized materials, the significant occurence of surfaces and interfaces as well as short diffusion lengths dramatically

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facilitate mass transport, thereby leading to fundamentally new deformation mechanisms.[15,16] Recent advances in *in situ* high-resolution TEM (HRTEM) have successfully addressed the technical challenges for tracking atomic diffusion under loading, providing atomistic insights into diffusion-mediated deformation.[17–19]

Zhong et al.[18] recently discovered unusual superplastic behavior in single-crystalline Ag nanocrystals at room-temperature (**Figure 1**a–b), which is in stark contrast to the common plastic instability of defects-scarce metallic nanocrystals. The superplastic deformation results from an interplay between crystal slip and surface diffusion. As shown in Figure 1d, emission of one dislocation (yellow arrow) generates an atomic step (red arrow) that subsequently migrates toward faceted surface areas. This can be understood from the locally increased chemical potential associated with the step that activates surface diffusion to restore the low chemical potential in the smooth surface (Figure 1e), inducing a self-healing mechanism that redistributes the surface dislocation sources over the gauge length and eliminates the tendency for necking.

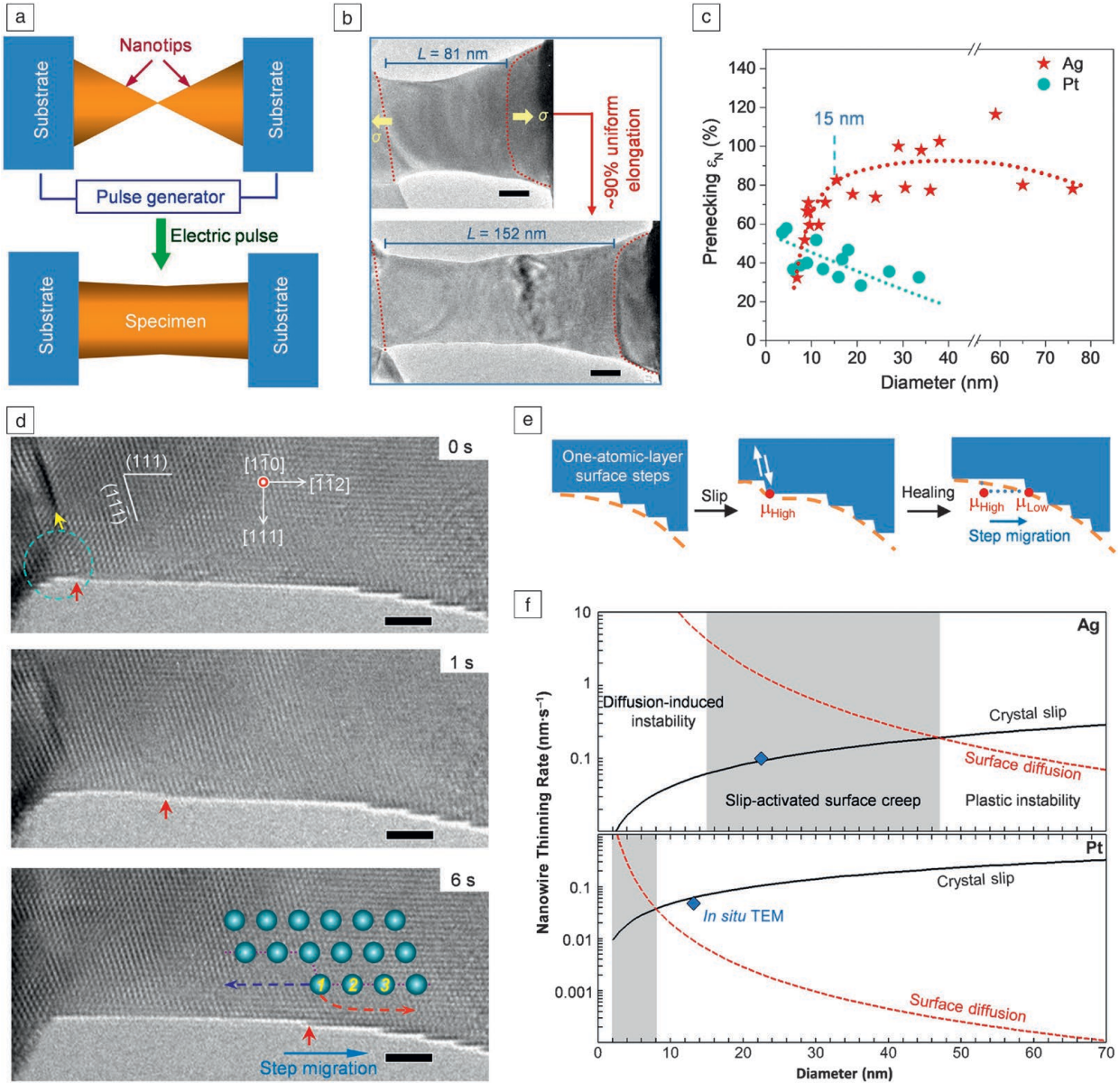


Figure 1. *In situ* high-resolution transmission electron microscope observation of slip-activated surface creep in metal nanocrystals.[18] (a) Schematic of specimen preparation. (b) Superplastic behavior of Ag nanocrystal. Scale bar = 20 nm. (c) Normalized elongation before necking as a function of diameter of Ag and Pt nanocrystals. (d) Crystal slip and subsequent surface-diffusion-assisted migration of an atomic step. Scale bar = 2 nm. (e) Illustration of slip-activated surface creep. (f) Diameter-dependent thinning rates of plastically strained nanocrystals based on crystal slip and strain-dependent surface diffusion. Slip-activated surface creep (shaded area) occurs at different diameters for Ag and Pt explaining the different mechanical behaviors in (c). Note: TEM, transmission electron microscope. Note: μ, chemical potential in (e).

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The slip-activated surface creep regime varies significantly among metals with different surface diffusion energy barriers and homologous temperatures, resulting in profound differences in their mechanical behaviors (Figure 1c). These findings provide guidelines for simultaneously maximizing ductility and strength in nanoscale materials.

Nanotwin-governed toughening mechanism

While nanotwinned metals have been extensively studied in recent years,[12,20–24] much less is known about the effects of nanotwins on the mechanical properties of ceramic materials. In the example presented here, advanced *in situ* TEM nanomechanics was combined with finite element modeling (FEM) to unravel the microscopic mechanism underlying the high fracture toughness of a conch shell composed of nanotwinned aragonite ($CaCO_3$).[25] An *in situ* TEM technique has been developed that enables real-time observation of crack propagation and quantitative assessment of fracture toughness by measuring the crack tip opening displacement (CTOD). Using this technique, distinct differences between nanotwinned and single-crystalline aragonite have been observed (**Figure 2**). In nanotwinned aragonite (conch, Figure 2a–c), the main crack is temporarily arrested by multiple twin boundaries, accompanied by the nucleation of nanocracks ahead of the tip of the main crack.[25,26] The crack tip appears blunt and is surrounded by amorphous (Figure 2c) and nanocrystalline phases, indicating considerable energy dissipation during crack propagation. In contrast, the main crack in single-crystalline aragonite remains sharp and propagates rapidly along the (110) cleavage plane (Figure 2d–f). Quantitative evaluation of the *in situ* data reveals a much increased CTOD for nanotwinned aragonite (conch, Figure 2g), corresponding to a one order of magnitude larger fracture energy compared to single-crystalline aragonite (Figure 2h), as calculated using FEM. These results demonstrate that preexisting growth twins play an essential role in enhancing the fracture toughness of brittle ceramic materials.[26]

Interaction of dislocations with grain boundaries

The deformation of polycrystalline materials crucially depends on the interaction of dislocations with grain boundaries (GBs). *In situ* TEM nanoindentation techniques[27–30] provide unique opportunities to dynamically observe such interactions, including dislocation pileup and transmission. For example, a well-defined $SrTiO_3$ bicrystal with low-angle tilt GB was employed as a model system to directly observe the interaction of moving lattice dislocations with GB dislocations.[30] A TEM nanoindentation holder with double-tilt capability was used, which enables accurate orientation of the crystalline sample for optimized TEM contrast while the indentation is precisely controlled by a piezo-actuator. **Figure 3**a shows a dark-field (DF) TEM image of the initial GB with the specimen tilted to reveal the edge dislocation array on the GB plane. The geometric arrangement of the specimen, indenter and defects is illustrated in Figure 3b. During indentation, multiple screw dislocations are formed on a single slip plane and successively transmitted through the GB.[30] Upon transmission, each dislocation intersection produces a kink in the moving screw dislocation and a jog in the intersected GB edge dislocation (Figure 3d). The jogs accumulate to "super jogs," which can be directly observed (Figure 3c). The estimated super-jog length corresponds well to the product of the Burgers vector and the number of screw dislocations crossing the GB on the same glide plane. Such detailed insight into dislocation–GB interactions can only be obtained by advanced *in situ* TEM nanoindentation combined with detailed *ex situ* contrast analysis.

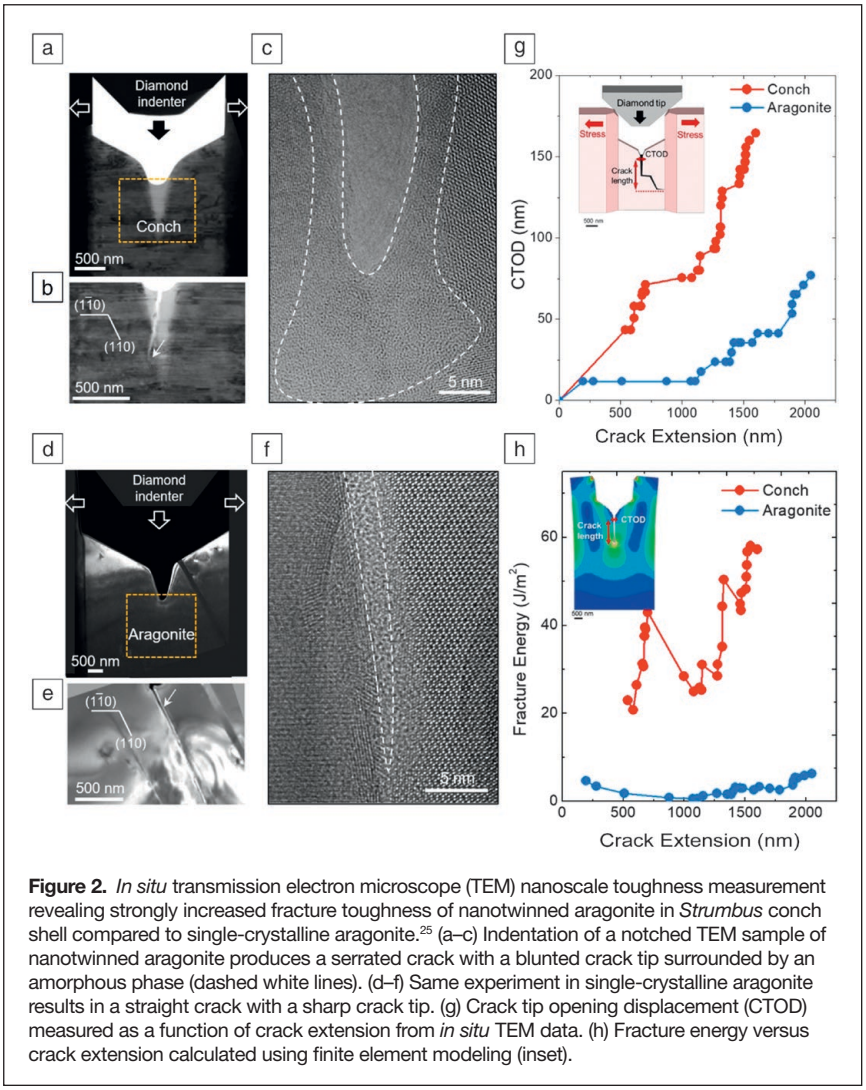


Figure 2. *In situ* transmission electron microscope (TEM) nanoscale toughness measurement revealing strongly increased fracture toughness of nanotwinned aragonite in *Strumbus* conch shell compared to single-crystalline aragonite.[25] (a–c) Indentation of a notched TEM sample of nanotwinned aragonite produces a serrated crack with a blunted crack tip surrounded by an amorphous phase (dashed white lines). (d–f) Same experiment in single-crystalline aragonite results in a straight crack with a sharp crack tip. (g) Crack tip opening displacement (CTOD) measured as a function of crack extension from *in situ* TEM data. (h) Fracture energy versus crack extension calculated using finite element modeling (inset).

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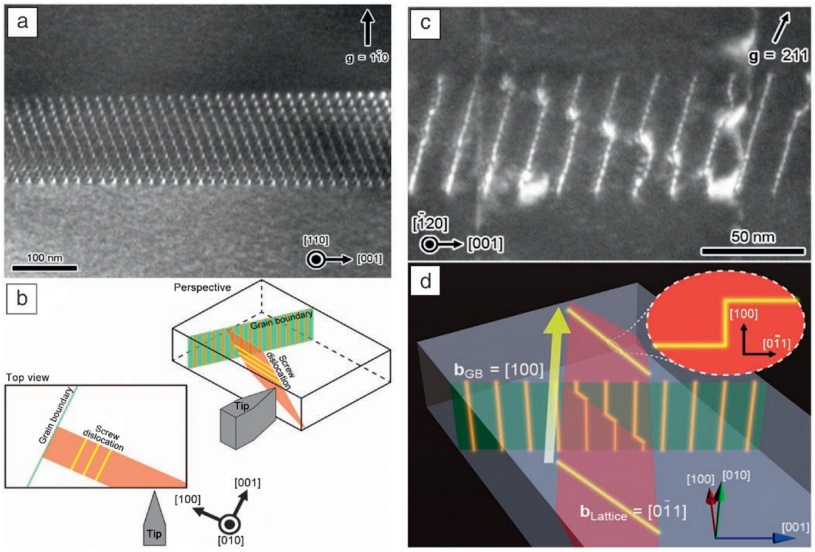


Figure 3. Interaction of screw dislocations with a low-angle tilt grain boundary (GB) in $SrTiO_3$.[30] (a) Dark-field transmission electron microscope (DF)-TEM image of initial GB, comprising an array of edge dislocations. (b) Geometric arrangement of specimen, indenter and defects. (c) DF-TEM image of the GB after *in situ* indentation revealing super jogs in the GB edge dislocations. (d) Schematic illustration of the process. Upon transmission of the GB by multiple screw dislocations, super jogs are formed in the GB edge dislocations, while kinks appear in the screw dislocations. Note: b_{GB}, Burgers vector of grain boundary dislocation; $b_{Lattice}$, Burgers vector of lattice dislocation.

Dislocation locking by point defect complexes

Hydrogen embrittlement has been a major concern in industry for more than a century.[31] Still, the atomic mechanism by which hydrogen affects the mechanical behavior of metals remains controversial.[32–34] Due to its high diffusivity, hydrogen is thought to be a weak inhibitor or even promoter of dislocation movement, which is supported by *in situ* TEM studies[35] and simulations.[36,37]

Xie et al.[38] recently observed a different scenario (**Figure 4**) by careful design of a cyclic pillar compression experiment inside an environmental TEM (ETEM). Starting with an aluminum micropillar containing a few dislocations, cyclic loading with maximum stress of 350 MPa was programmed to directly compare the dislocation behavior with and without hydrogen. The experiment revealed that hydrogen can convert initially mobile dislocations into sessile dislocations under the same or even two times higher stress (Figure 4d–e). Upon degassing, the locked dislocations could be reactivated under cyclic loading to move in a stick-slip manner.[38] However, relocking the dislocations required a much longer waiting time (~10^3 s) than expected from hydrogen interstitial diffusion (Figure 4g–i). Atomistic simulations revealed that both the strong locking and slow relocking can be attributed to superabundant hydrogenated vacancies (HVs) (Figure 4f). HVs show much slower diffusion than interstitial hydrogen. Moreover, HVs change the core structure of an edge dislocation causing jogs, which require much higher shear stress to move. For metals with high vacancy concentration exposed to hydrogen, HVs could thus be a key plastic flow localization agent.

In situ manipulation of individual dislocations in 2D materials

Even though 2D materials are only one or a few atomic layers thick, they may contain dislocations.[39–43] Among the various 2D materials, bilayer graphene is the thinnest material that can host dislocations with extended dislocation lines.[42,43] Such basal dislocations are trapped between the two sheets of carbon atoms and cannot escape to the surface. Strain relaxation results in topographic ripples along the dislocation lines.[43]

Schweizer et al. recently devised a method to directly manipulate individual dislocations *in situ* in bilayer graphene for the very first time by building upon the ultrathin nature of bilayer graphene and the unique properties of basal dislocations.[44] The method is based on a small tip mounted on a micromanipulator that can drag around dislocation lines inside an SEM (**Figure 5**a) while they are imaged using low-voltage STEM. *In situ* manipulation was used to study fundamental properties of dislocations, such as line tension and node formation (Figure 5b) as well as interaction with free edges (Figure 5c). Furthermore, a switching reaction was found, based on the recombination of dislocation lines. Upon dislocation reaction, the interconnectivity of bilayer areas with identical stacking order could be changed demonstrating a "topological switch" (Figure 5d).[44] Since basal dislocations act as barriers for charge transport in bilayer graphene[45,46] such switching reactions may be utilized as fundamental building blocks in future dislocation-based devices.

Challenges and future prospects

The previously discussed examples illustrate the versatility, by which advanced *in situ* nanomechanical experiments in TEM/STEM can provide new insights into deformation processes down to the atomic scale. Considering the extremely rapid development of TEM/STEM technology in the past 10–15 years, the exploration of state-of-the-art methodologies such as highly stable aberration-corrected TEM/STEM for *in situ* nanomechanics is still in its infancy. Liu et al.[19] recently used *in situ* aberration-corrected HRTEM in tandem with a direct detection camera to monitor the formation and migration of dislocation slip-induced surface steps during tensile testing of nanoporous gold. Making use of the strongly reduced contrast delocalization in aberration-corrected HRTEM, a first attempt was made to measure elastic strain at surface steps directly from *in situ* data in order to elucidate the interplay of strain, surface diffusion, and step migration.

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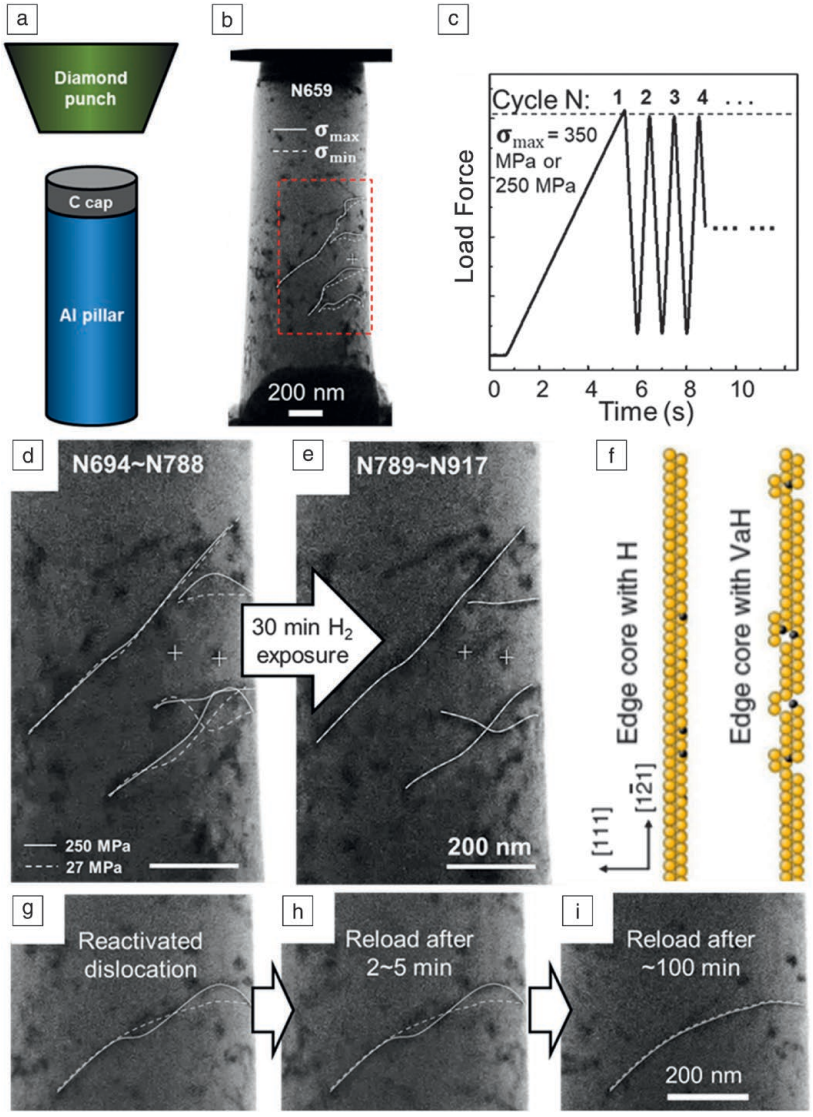


Figure 4. *In situ* observation of the pinning effect of hydrogenated vacancies on dislocation mobility.[38] (a, b) Nanomechanical setup implemented in an environmental transmission electron microscope and aluminum pillar sample showing the observed area (dashed box). (c) Programmed cyclic compression. (d) Dislocation positions at peak and valley stress (solid/dashed lines) before hydrogen exposure. (e) All marked dislocations ceased motion after hydrogen charging. (f) Schematic showing edge dislocation with interstitial hydrogen (left) and hydrogenated vacancies (right). (g–i) A reactivated dislocation was recharged with hydrogen after a different waiting time, showing strong pinning after ~100 min.

In general, precise knowledge of local strain and stress states during *in situ* nanomechanical testing is highly desirable and key to quantitative understanding of defect processes, such as dislocation nucleation and propagation. In TEM/STEM experiments, this information can hardly be extracted from load-displacement curves because of delicate sample geometries and limitations in mechanical stability and force resolution. A promising alternative is the direct mapping of strain during *in situ* imaging. Here, we only mention nanobeam electron diffraction strain mapping (or four-dimensional STEM [4D-STEM], combining a 2D nanodiffraction pattern acquired at each point of a 2D scan area), which extracts strain from local diffraction patterns acquired at each probe position using a pixelated detector.[47,48] Even though the technique is much slower than regular TEM or STEM imaging, time resolution can be improved using state-of-the-art direct detection camera technology.[47–50] Pekin et al. recently demonstrated the possibilities of this multidimensional *in situ* imaging approach by mapping strain evolution during planar slip in stainless steel under *in situ* deformation.[50]

The direct dislocation manipulation demonstrated for bilayer graphene can be extended to other 2D materials such as few-layer MoS_2 (or other transition metal dichalcogenides), which will provide new insights into fundamental defect processes in this emerging class of functional materials. Precise measurement of manipulation forces in conjunction with simulations may provide direct access to fundamental defect characteristics such as lattice friction (or Peierls' potential) and stacking-fault energies. Finally, a combination of mechanical manipulation with other *in situ* techniques such as electrical probing is highly promising in understanding the functional properties of individual defects.

Conclusion

In summary, *in situ* nanomechanics in TEM/STEM provides unique opportunities for gaining deep insights into fundamental deformation and defect processes in materials down to the nanometer and atomic scales. Major progress has been made in the past years, as illustrated by the examples presented in this article. However, there remains a lot to explore and new technological advancements in the rapidly developing field of electron microscopy provide the perfect basis for major breakthroughs. Key prerequisites for future progress are expected to be advances in sample preparation and quality, improvements in mechanical force control and sensitivity, and full exploration of advanced TEM/STEM technologies, including direct detection cameras for improved time resolution and multidimensional *in situ* imaging.

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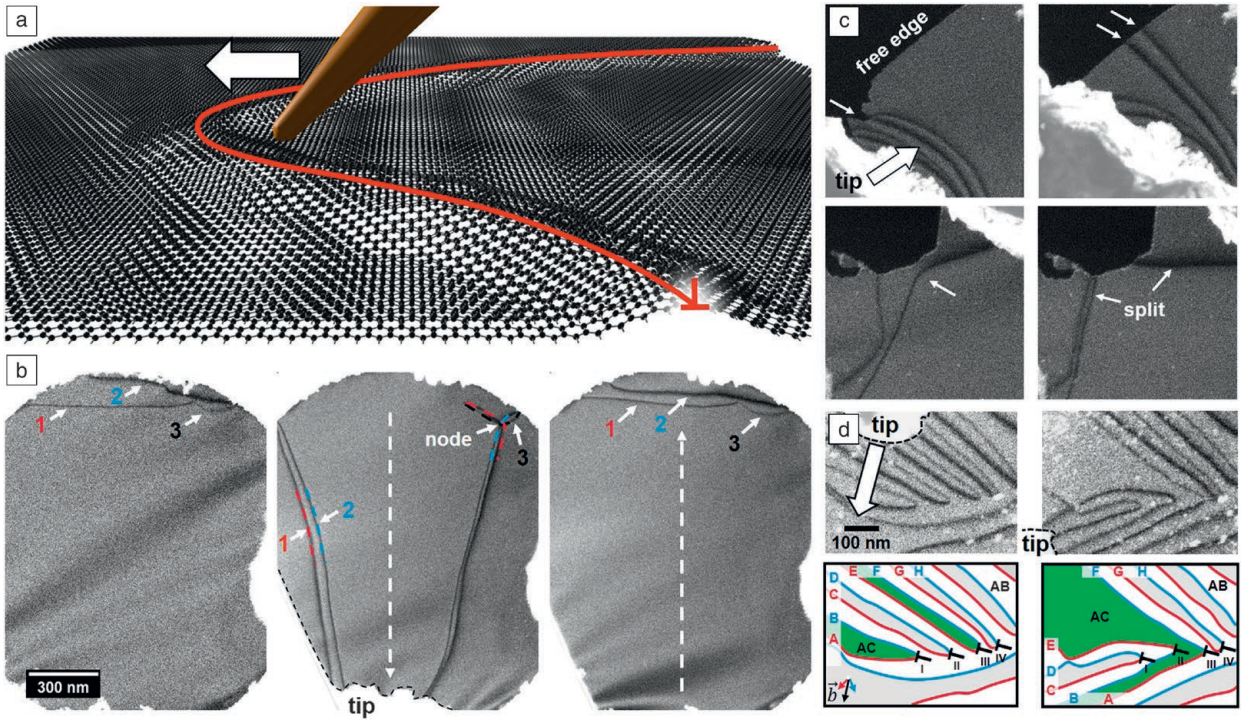


Figure 5. *In situ* dislocation manipulation in bilayer graphene.[44] (a) Artistic representation of the concept: a fine tip attached to a micro-manipulator can directly move around dislocations that are relaxed into topographic ripples. (b) Example showing the effect of line tension, which drives the dislocations back into their original state after manipulation. (1, 2, and 3 indicate the three partial dislocations with different Burgers vectors.) (c) Interaction of dislocations with free edges showing pinning at kinks and splitting of dislocation lines. (d) *In situ* manipulation can also be used to realize "topological switches," where regions of identical stacking order (green) are interconnected or separated.

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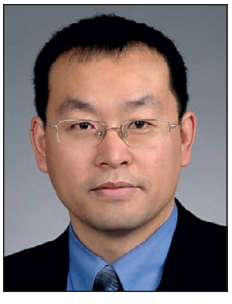
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