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REVIEW

In situ experimental mechanics of nanomaterials at the atomic scale

Lihua Wang[1], Ze Zhang[1,2] and Xiaodong Han[1]

Sub-micron and nanostructured materials exhibit high strength, ultra-large elasticity and unusual plastic deformation behaviors. These properties are important for their applications as building blocks for the fabrication of nano- and micro-devices as well as for their use as components for composite materials, high-strength structural and novel functional materials. These nano-related deformation and mechanical behaviors, which are derived from possible size and dimensional effects and the low density of defects, are considerably different from their conventional bulk counterparts. The atomic-scale understanding of the microstructural evolution process of nanomaterials when they are subjected to external stress is crucial for understanding these 'unusual' phenomena and is important for designing new materials, novel structures and applications. This review presents the recent developments in the methods, techniques, instrumentation and scientific progress for atomic-scale *in situ* deformation dynamics on nanomaterials, including nanowires, nanotubes, nanocrystals, nanofilms and polycrystalline nanomaterials. The unusual dislocation initiation, partial-full dislocation transition, crystalline–amorphous transitions and fracture phenomena related to the experimental mechanics of the nanomaterials are reviewed. Current limitations and future aspects using *in situ* high-resolution transmission electron microscopy of nanomaterials are also discussed. A new research field of *in situ* experimental mechanics at the atomic scale is thus expected.
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INTRODUCTION

Recent studies on nanostructured materials, including nanowires (NWs),[1,2] nanotubes (NTs),[1,3] nanocrystals (NC),[4] micro/nano-pillars (NPs)[5–7] and nanocrystalline,[8,9] have revealed a variety of 'unusual deformation' phenomena compared with their bulk counterparts, such as high strength, nano-piezoelectric effects and unusual plastic deformation behaviors. Nanostructured materials can apparently sustain a larger dynamic range of elastic and plastic strains than conventional materials. The results from these studies indicate that the fundamental dislocation processes that initiate and sustain plastic flow and fracture in nanoscale materials are considerably different than in their conventional bulk counterparts. These 'unusual' phenomena not only allow these materials to possess excellent mechanical properties but also enable the tuning of their band structures and related novel electronic, magnetic, optical, photonic and catalytic properties. Revealing the atomic-scale deformation mechanisms of nanomaterials (NM) and controlling their elastic and plastic properties are useful for realizing the desired mechanical, physical and chemical properties through the application of stress or strain.

Although extensive studies have been conducted to investigate the mechanical properties of NM,[10] the majority of the atomic mechanisms are based on computer simulations, which may suffer from inaccuracies due to the empirical or semi-empirical interatomic potentials, grain boundary (GB) structures and high strain rates.[11,12] This article focuses on recent *in situ* atomic-scale experimental studies on the deformation behaviors of NM. We briefly introduce some important techniques and methods that have been used for the time-resolved visualization of nano-mechanics that utilize *in situ* microscopy, specifically, the techniques used for gaining an atomic-scale understanding of the deformation behaviors of NM. The size effects that lead to the ultra-large elasticity in these materials will be discussed. The atomic-scale *in situ* transmission electron microscopic (TEM) investigations on the elastic–plastic transition and the plastic deformation mechanisms of NM will be reviewed.

EXPERIMENTAL TECHNIQUES BY *IN SITU* MICROSCOPY

The early testing devices for NWs and NTs were based on the atomic force microscopy (AFM) technique.[1–3] These devices enabled the direct determination of force as a function of displacement and revealed the unusual mechanical properties of NM.[13–15] However, these techniques cannot normally reveal the actual deformation mechanisms that involve dislocation activities in the NM because of

[1]Institute of Microstructure and Properties of Advanced Materials, Beijing University of Technology, Beijing, China and [2]Department of Materials Science and Engineering and State Key Laboratory of Silicon Materials, Hangzhou, China
Correspondence: Professor Z Zhang or Professor X Han, Institute of Microstructure and Properties of Advanced Materials, Beijing University of Technology, Beijing 100124, China.
E-mail: zezhang@zju.edu.cn or xdhan@bjut.edu.cn
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the difficulties in interpreting the displacement–force correlations to details of the dislocation initiation and interaction activities.[16]

TEM is one of the most powerful and effective techniques that has nanoscale and atomic-level resolution capabilities along with the ability to obtain crystallographic and chemical information. *In situ* TEM experiments have been used since the 1960s.[17] During the past decade, the use of TEM for investigating *in situ* mechanics and the related physics of materials has been one of the most interesting research fields due to the unexpected and unusual mechanical and physical size-effects in materials with micron/nano dimensions or volumes.[4–7,9,10,13–18]

The simplest approach for performing *in situ* deformation experiments in a TEM utilizes a conventional TEM single-tilt straining stage, which can tilt the strained/stressed sample along one axis. With this stage, the total elongation of the sample can be determined by monitoring the elongation from subsequent TEM images. In most cases, the cross-sectional area of the sample being strained is unknown. These aspects prevent a correct stress measurement. Using the Gatan straining holder, the microstructural evolution of nanocrystalline thin films and single crystals[19–21] have been recorded *in situ* during the deformation process in the TEM.

Quantifying the stress–strain relationship is an important requirement in regular mechanical property studies. Based on nanoindentation techniques, AFM and scanning probe microscopic tips have been developed that can be used in a TEM.[22] The samples are normally mounted on a piezo-driven support within the stage. Load sensors with high resolution are available for different regions of interest. The displacement is either measured capacitively or is deduced from the applied piezo-motion. The NanoFactory TEM–STM (scanning tunneling microscopy)/AFM holders[22] and the Hysitron picoindenter[6,7] are the most commonly used devices for deformation testing with the ability to quantify the stress–strain relationships. With stress versus strain curves, the TEM–STM holder could also apply a specific bias and measure the current response of the NMs. Several other in-lab-developed techniques and devices have also been used in studies of the deformation mechanisms and strained physics of NM.[23–29]

The micro-electromechanical system (MEMS) is another important approach for the *in situ* microscopy mechanics by TEM.[8,30] These devices are based on Si technology, and film deposition, lithography and etching techniques are used to design actuators and force sensors on a chip. One of the first successful MEMS devices for *in situ* TEM deformation studies of metallic lines was developed by Saif's group.[30] The sample was co-fabricated with the MEMS structure and suspended as a line on the device. During loading, the gap distance between the sensing beams changes proportionally to their stiffness, while the stiffness of the sensing beams is measured using the nanoindentation technique.

The aforementioned commercial stages or MEMS devices can provide quantitative stress–strain data along with revealing the microstructural evolution process of the strained materials. Numerous investigations[31–34] have provided valuable quantitative insight into the dislocation mechanisms of NM using *in situ* TEM observations. However, these devices only have single-tilt capabilities, which makes reliable investigations of linear and planar defects inconvenient because a 'double-tilt' ability is generally required to obtain the desired 'two-beam' condition or the ideal crystallographic orientation. Direct atomic-scale investigations using these devices are difficult. Therefore, for reliable defect investigations in crystalline materials, developing microscopy mechanic devices with double-tilt capabilities is necessary.

With the use of a conventional double-tilting stage and qualitative irradiation of multiwalled carbon nanotubes (MWCNTs),[27] ultra-high pressures can accumulate in the cores of the MWCNTs. With this method, Sun *et al.*[27,35] investigated the deformation dynamics of NCs through the controlled irradiation of MWCNTs. Han *et al.*[25,26,36–38] developed an alternative TEM grid technique that can bend or conduct axial tensile experiments on individual NWs using a pre-broken colloidal thin film on the TEM grid. The above two methods do not require mechanical tensile attachments, and the specimen could be tilted along a pair of two orthogonal directions with large angles. Therefore, with a proper observing direction, the deformation process in individual NWs or NCs can be recorded *in situ* at the atomic scale. However, the strain rate and deformation mode is normally uncontrollable.

Controllable *in situ* straining experiments without sacrificing the double-tilt capability in the TEM was developed by exploiting the differences in the thermal expansion coefficients between different materials. This method has been used to investigate plasticity in thin films[39,40] on substrates, and it requires a conventional heating stage. The mismatch in the thermal expansion coefficients between the film and the substrate will cause the film to experience tensile or compressive stress. With this method, the strain applied by the substrate on the thin film is very small, and the film–substrate diffusion or chemical reactions during heating will complicate the interpretation of the results.

Based on the thermal bimetallic technique that has been used in SEM analyses,[28] Han *et al.*[38] developed a novel *in situ* controllable tensile testing device for TEM measurements, which can slowly and gently deform the NWs, NTs, NPs and nanocrystalline thin films. Their method can even measure regular TEM samples assisted by focused ion-beam fabrication while retaining the double-tilt capability for performing high-resolution TEM (HRTEM) observations and regular 'two-beam' dark field imaging investigations.[29] The strain rate is also controllable in the range from 10^{-2} to $10^{-5}\,s^{-1}$. The TEM extensor is composed of two thermally actuated bimetallic strips. The conventional TEM heating stage, now with the fabricated bimetallic strips, can therefore serve as a double-tilt, displacement-controlled tensile stage. With this device, investigations of the plastic deformation behavior of nanocrystalline materials,[29,41,42] NCs[43–45] and metallic glasses[46,47] can be conducted and observed *in situ* at the atomic scale.

SIZE EFFECT OF ULTRA-LARGE ELASTICITY IN NWS AND NCS

It has been hypothesized that the ideal elastic strain of metallic crystals is on the order of 8%.[10] Small samples always sustain ultra-high elastic strain before yielding.[2,4–8] This behavior is in contrast to conventional bulk metals, which can only deform on the order of 0.2% elastic strain. The majority of the applied strain of bulk metallic materials is performed by inelastic strain, and the strength of the material is primarily increased through work hardening.

The ultra-large elasticity in small-sized samples was discovered as early as 1924 by G. F. Taylor,[48] who observed that antimony wires with a diameter of 30 μm can be repeatedly bent without breaking, although bulk antimony is very brittle. In 1949, Bragg and Lomer[49] reported an experimental investigation on the *in situ* deformation of an extended raft of bubbles floating on a soap solution and observed that the bubble crystal can be elastically deformed to approximately 10%. Later, Herring and Galt[50] observed that tin whiskers with a diameter of 1.8 μm can sustain 2–3% elastic strain, though bulk tin can only sustain approximately 0.01% elastic strain. Subsequent mechanical tests performed on a variety of whiskers,[10,39] NWs,[5,6]

NC[51] and NTs[1,3] have revealed that these micro- and nanoscale components can, in fact, sustain large elastic strains. In the past 10 years, a wide range of metallic micro- and nanoscale pillars have been used to investigate the deformation mechanism of small-sized samples.[52–55] The majority of the results convincingly indicate that the yield and flow stresses increase when the sizes of the samples decrease, which is a phenomenon known as 'smaller is stronger.' This remarkable trend has been comprehensively reviewed by Zhu and Li,[10] Dehm,[15,39] Uchic *et al.*,[52] Kraft *et al.*,[53] and Greer *et al.*[55]

For bulk materials, we normally only measure the yield strength for the motion of pre-existing dislocations. Pristine crystals of the micro- and nanoscale samples can be produced to minimize the number of defects that inevitably exist in their bulk counterparts. This procedure offers the opportunity to observe large elastic strains close to the theoretical limit.[10] This prediction was directly observed by Yue *et al.*[43] Using the bimetallic tensile technique, the process of lengthening the atomic bonds was captured and an elastic strain that approached the theoretical elastic limit was observed in Cu NWs. During the deformation of a Cu NW with a diameter of approximately 5.8 nm along the [001] direction, the lattice experienced an approximate elastic strain of 7.2% along the [001] direction, which was directly observed. After the NW was fractured, the lattice returned to its initial value.

DIRECT ATOMIC MECHANISMS OF THE SIZE EFFECTS ON THE UNUSUAL PLASTICITY OF SINGLE CRYSTALLINE FCC (FACE CENTER CUBIC) METALS

The ultra-high strength achieved in NCs implies that their deformation behaviors are considerably different from their bulk counterparts. The uniaxial compression methodology was first introduced by Uchic *et al.*[5] Greer *et al.*[56] extended this method to the nanoscale region; they observed that single crystalline Au NPs exhibited unprecedented strengths that were nearly 50 times greater than their bulk counterparts. More recently, the understanding of elasticity and plasticity in small volumes has been further enriched through tensile experiments performed by Mompioua *et al.*,[32] Kiener *et al.*[57] and Dehm *et al.*[58] Because the deformation mechanism of these micro- NPs have been comprehensively reviewed by Zhu and Li,[10] Dehm,[15,39,58] Uchic *et al.*[52] Kraft *et al.*,[53] Legros *et al.*[59] and Greer *et al.*,[55] we only provide a brief summary here. Next, we focus on the atomic-scale *in situ* TEM investigations on NMs that are <100 nm.

Several theories have been proposed to explain this size-dependent strengthening behavior. Two prominent deformation mechanisms have been proposed, which include dislocation starvation[60] and the single-arm source theory.[61] In the former, the plasticity results from the surface nucleation of dislocations once all the pre-existing mobile dislocations have been annihilated at the free pillar surface, and this process was observed *in situ* by Minor and colleagues[6,33] and Kiener and Minor.[7] In the latter, the creation of dislocations is caused by the operation of single-arm sources; the increase in strength arises from the progressively harder operation of dislocation sources with a reduction in the pillar diameter. These concepts contrast the classical plasticity model, in which the dislocations multiply, and therefore, the overall dislocation density increases, which results in work hardening. Because the diameters of FIB-fabricated NPs are >100 nm, obtaining atomic-scale images of these NPs is difficult. Therefore, the atomic-scale deformation mechanisms of these NPs are highly reliant on molecular dynamics (MD) simulations.

As the size of the NCs continues to decrease (<100 nm), it appears that the size effect has an obvious effect on the types of dislocations, which changed from full dislocations ($d\sim>200$ nm) to partial dislocations. Seo *et al.*[62] reported that defect-free Au NWs ($d\sim100$ nm) exhibit superplasticity upon tensile deformation, which is associated with twin boundaries and the propagation of partial dislocations. Sedlmayr *et al.*[63] observed partial/twinning-mediated plasticity in Au nanowhiskers with diameters between 40 to 200 nm. Partial dislocation emission was also observed in the sub-20-nm-sized gold NWs.[64] Yue *et al.*[44] quantitatively revealed an obvious effect of the sample dimensions on the plasticity mechanisms using *in situ* tensile tests of Cu single crystalline NWs with diameters between 1000 to 70 nm in a HRTEM. When the size of the single-crystal NW size was reduced to $<\sim150$ nm, the normal full dislocation slip was overwhelmed by partial dislocation-mediated plasticity. In fact, the impact of size-dependent dislocation mechanisms has also been observed in thin film.[65]

For NWs <20 nm, the AFM/STM tip-based technique is one of the most powerful approaches for probing the deformation properties. By dipping the STM (TEM–STM holder) probe tips into Au, Au NWs with diameters of a few nanometers can be obtained because of its high adherence ability. This method was first used by Agraite *et al.*[22] Following this report, the deformation behaviors of Au NWs with various diameters (even <10 nm) have been investigated. Figure 1 presents a typical *in situ* atomic-scale observation, in which the partial dislocations emitted from free surfaces dominated the deformation of the $d\sim10$-nm-sized gold NWs.[66] Figures 1a–c present sequential HRTEM images that illustrate the entire dislocation process, including the nucleation of a leading partial dislocation from a surface step (Figure 1a), the stacking fault (SF; as indicated by an arrow in the inset of Figure 1b) and the trailing partial dislocation, which eliminates the SF (Figure 1c).

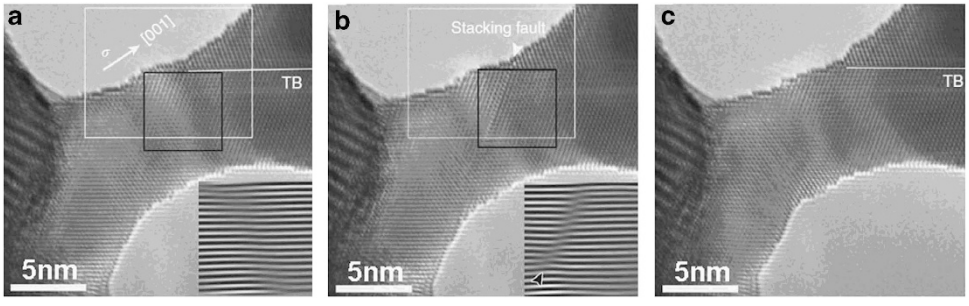

Figure 1 Sequential high-resolution transmission electron microscopic images revealing the emission of a dislocation from a free surface. (**a**) No dislocations were observed. (**b**) A leading partial dislocation emission and resulting stacking faults were observed. (**c**) A trailing partial emission eliminates the stacking fault. Figure from Zheng *et al.*[66] Copyright 2010 Nature Publishing Group.

When further reducing the size of the NC to approximately 6 nm, the *in situ* atomic-scale observation indicates that lattice slips were the dominant plastic events.[67] For the 3-nm-sized NC, Kizuka[68] observed that only lattice slip occurs. The lattice slip on the {111} planes was also observed by Matsuda and Kizuka[69] in Pd NWs. The fcc–bcc phase transition was observed *in situ* and in real-time in a $d \sim 1.8$-nm-sized Au junction.[70]

In 1998, Ohnishi *et al.*[71] reported a suspended, single chain of gold atoms. No dislocations, lattice sliding or phase transitions were observed in this small-sized NW of approximately 1 nm. The single atom chain was formed by breaking the atomic bonds one by one. Figures 2a–f present TEM images of approximately 1 nm gold NWs formed between the substrate and the tip during the withdrawal of the tip. The dark lines in the gold NW represent rows of gold atoms that span the distance from the substrate to the tip. Figure 2g presents typical HRTEM images of a single chain of gold atoms. Bettini *et al.*[72] observed the *in situ* formation of single atomic chains of Ag and the Au–Ag alloy.

It is clear that the deformation mechanisms change as the crystal size decreases. As shown in Figure 3, when the diameter of the NC is greater than approximately 200 nm, full dislocations dominate the plasticity, whereas for smaller NCs, partial dislocations are prevalent (200–10 nm). As the diameter continued to decrease, new deformation phenomena, such as lattice slip on the {111} planes, phase transitions and fracturing of the bonding atoms, were observed.

DIRECT ATOMIC MECHANISMS OF THE SIZE EFFECT ON THE UNUSUAL PLASTICITY IN SEMICONDUCTOR NWS

Bulk semiconducting and ceramic materials are normally brittle and fracture upon any mechanical deformation for shape changes at room temperature (RT). However, when the size of the material decreases to a small scale, the defect-free structure normally makes the NM survive at high fracture stresses,[10,25,26,51,73] which could eventually provide the materials with the ability to overcome the critical resolved shear stresses and nucleate ductile dislocations or make these ductile-featured dislocations mobile. The fracture and deformation behaviors of NMs can be significantly different from that of their bulk counterparts. In 2007, Han *et al.*[26] directly observed the unusually large bending strain plasticity in ceramic SiC NWs close to RT. The approximate bending strain was calculated using the traditional

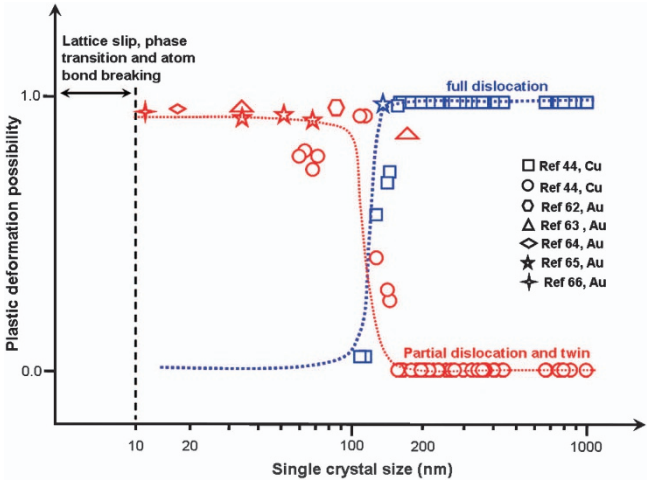

Figure 3 Sample size dependence of two plasticity mechanisms: relative contribution to the overall plastic strain experienced by the sample region under observation, from full dislocation slip (blue symbols) versus partial dislocation (red symbols)-mediated processes.

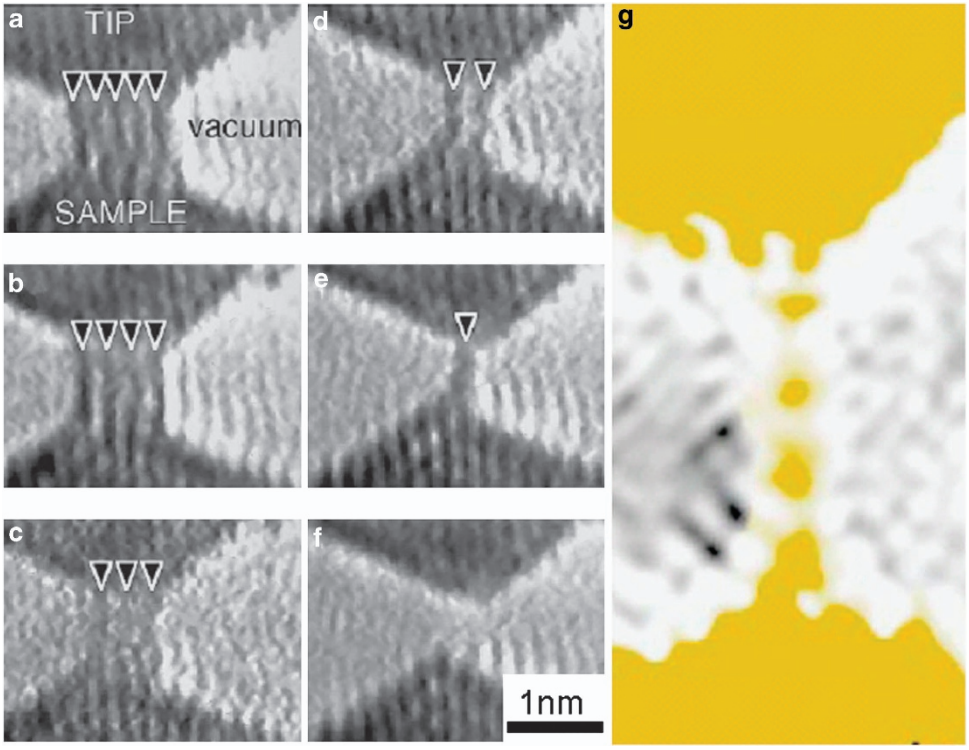

Figure 2 A gold bridge formed between the gold tip (top) and gold substrate (bottom), thinned from (**a–e**) and ruptured at (**f**). Dark lines indicated by arrows are rows of gold atoms. (**g**) transmission electron microscopicimage of a single chain of atoms. Figure from Ohnishi *et al.*[71] Copyright 1998 Nature Publishing Group.

formula $\varepsilon_{strain} = r/(r+R)\%$,[74] where R is the bending curvature and r is the radius of the Si NW. In these studies, the bending strain in the figures or text represented the maximum strain in the NW. *In situ* atomic-scale observations revealed that the plasticity of the SiC NWs was accompanied by a process of increased dislocation density during the early stages, followed by an obvious lattice distortion and then amorphization in the most strained region of the NW. Using the thermal bimetallic technique,[28] superplastic elongation ability was observed in the SiC NWs. This RT plasticity was also observed in Si NWs by conducting axial tensile[25] and bending tests[36] in the TEM. *In situ* atomic-scale TEM images revealed a considerable density of dislocations, and a crystalline–amorphous (*c–a*) transition is responsible for the ultra-large plasticity character of the Si NW. Following this report, large plasticity and the *c–a* transition have also been observed in other semiconductor NWs. Smith *et al.*[75] observed that Ge NWs become amorphous at the point of maximum strain of 17%. Asthana *et al.*[76] observed the *c–a* transition in the highly compressed region of the [0001] ZnO NWs after a number of loading and unloading cycles. Most recently, Tang *et al.*[77] revealed that under tension, Si NWs elastically deformed until an abrupt brittle fracture. Under a larger bending strain of $>20\%$, plastic deformation occurred because of dislocations.

Uniaxial compression tests were performed on Si pillars in the size range of 1–200 nm.[78] When the diameter of the pillar falls below a critical value (between 310 and 400 nm), it exhibits ductility. The brittle-ductile transition in GaAs has also been observed at RT.[79] *In situ* TEM compressive experiments on the Si NWs and particles have also been conducted.[80–82] Highly ductile features, plastic dislocations and strong strain hardening were directly observed. These results apparently indicated that for the semiconducting or ceramic materials, the RT brittle–ductile transition can be realized by reducing the size of the materials.

Atomic-scale imaging is an important technique for revealing localized or incipient phase transition phenomena in the deformation dynamics of crystalline materials. Wang *et al.*,[83] for the first time, directly observed an atomic mechanism for the crystalline–amorphous *(c–a)* transition through a dislocation reaction in ultra-large strained (up to 14%) Si NWs. The direct dynamic atomic-scale observations revealed that partial and full dislocation nucleation, motion and interaction and the *c–a* transition were responsible for absorbing the ultra-large strain during the bending of the Si NWs. Full dislocations were nucleated and then formed a Lomer dislocation by reaction. The continuous straining on the Lomer dislocations induced a *c–a* transition in the Si NWs. These results provide a direct explanation for the ultra-large straining ability and the *c–a* transition mechanism for those semiconductor and ceramic nanostructures.[25,26,36,75–77]

DIRECT ATOMIC MECHANISMS OF PLASTIC DEFORMATION IN NANOCRYSTALLINE MATERIALS

The plastic deformation behaviors of bulk polycrystalline metals are well understood.[84] However, the precise nature of the plastic deformation mechanism in nanocrystalline materials is still not fully certain. As proposed by MD simulations, when the grain size (diameter d) is less than approximately 15 nm, the dislocation activities subside, which may completely give way to the GB-mediated plasticity.[11,12] This phenomenon is often referred to as the inverse Hall–Petch effect. However, the existence of this inverse Hall-Petch effect is still under debate.[85]

For example, many previous *in situ* TEM studies have observed GB-mediated plasticity. In 1995, Ke *et al.*[20] observed that grain rotation is the dominant plastic deformation mechanism based on *in situ* HRTEM measurements. Shan *et al.*[86] *in situ* observed grain rotation in nanocrystalline Ni using dark-field TEM images. The GB migration in Al was observed[31,87] based on the contrast changes in the TEM images. However, there are also many TEM observations that contrast with the above MD simulations and TEM observations, including *in situ* ones, which suggest that the dislocations are highly active even in the approximately 10-nm-sized grains.[29,41,42,88]

Recently, Wang *et al.*[41] developed a new bimetallic technique that can perform *in situ* axial tensile deformation on NM at the atomic scale (Figure 4a). With this method, the direct atomic-scale observation revealed that an array of inter-grain dislocations can also induce grain rotation in nanocrystalline gold.[42] A high activity of dislocation behaviors were also observed in nanocrystalline Pt with grain sizes less than approximately 10 nm.[29,41] As shown in Figure 4b, a Lomer dislocation was observed in the grain, which was formed by dislocation reactions. With further loading, the destruction and the reformation of the Lomer lock in the grain was also observed, as shown in Figures 4c and d. In addition to the formation of Lomer dislocations, the dislocation annihilation and storage of full dislocations in the $d \sim 10$ nm were also detected.[41] These dislocation activities were also observed by HRTEM examinations in Ni,[89] Al[90] and Cu nanocrystalline.

It is still uncertain whether the inverse Hall–Petch effect really exists.[85] However, a considerable number of *in situ* and *ex situ* TEM observations exhibit the same trend: the grain size has an obvious effect on the type of dislocations and the deformation twins. Wang *et al.*[29] provided direct experimental evidence for the transition from full dislocation to partial dislocation with decreasing *grain size*. For grains larger than $d \sim 10$ nm, full dislocation activities are the dominant deformation mode. For the smaller grains ($d \sim < 10$ nm), partial dislocations that generate SFs are prevalent. Li *et al.*[89] presented an *in situ* atomic-scale observation of reversible SFs in small-sized nanocrystalline Ni and deformation twinning in Ni and Cu was also observed *in situ* at the atomic scale.[91] Partial dislocation or deformation twinning have also frequently been observed in other small-sized FCC metals by post-mortem examination,[29,89–92] even for those metals with high SFs or twinning energies, such as Al,[90,92] Pt[29] and Ni.[89,91,92] This size effect on the dislocation type is very similar to that observed for single crystals. Figure 5 summarizes the size-dependent plastic deformation mechanisms of Pt nanocrystalline samples; for grain sizes above a critical value, full dislocations will dominate the plastic deformation, whereas below this critical value, partial dislocations will gradually control the plastic deformation. As the grain size continues to decrease, it will transition to GB-mediated plastic deformation (theoretical inverse Hall–Petch effect), although this issue remains uncertain. For different materials, the transition region is different.

Twin-structured crystalline materials always exhibit high strength and high ductility.[93] For twin-structure crystalline copper (with submicron-sized grains), Lu *et al.*[93] revealed that the strength increases with decreasing twin thickness, which reaches a maximum at 15 nm followed by a softening at smaller values. A strong twin thickness effect on the dislocation behaviors was also proposed based on post-mortem observation: dislocation–dislocation interaction hardening in coarse twins, and dislocation–twin boundary (TB) interaction hardening in fine twins. This claim was confirmed by Wang *et al.*[94] The authors' *in situ* atomic-scale observations revealed that the extended dislocations can hardly cross the TB in Cu with growth twins. Wang[95] and Li[19] investigated the stability of growth twins in Cu using *in situ* nanoindentation.

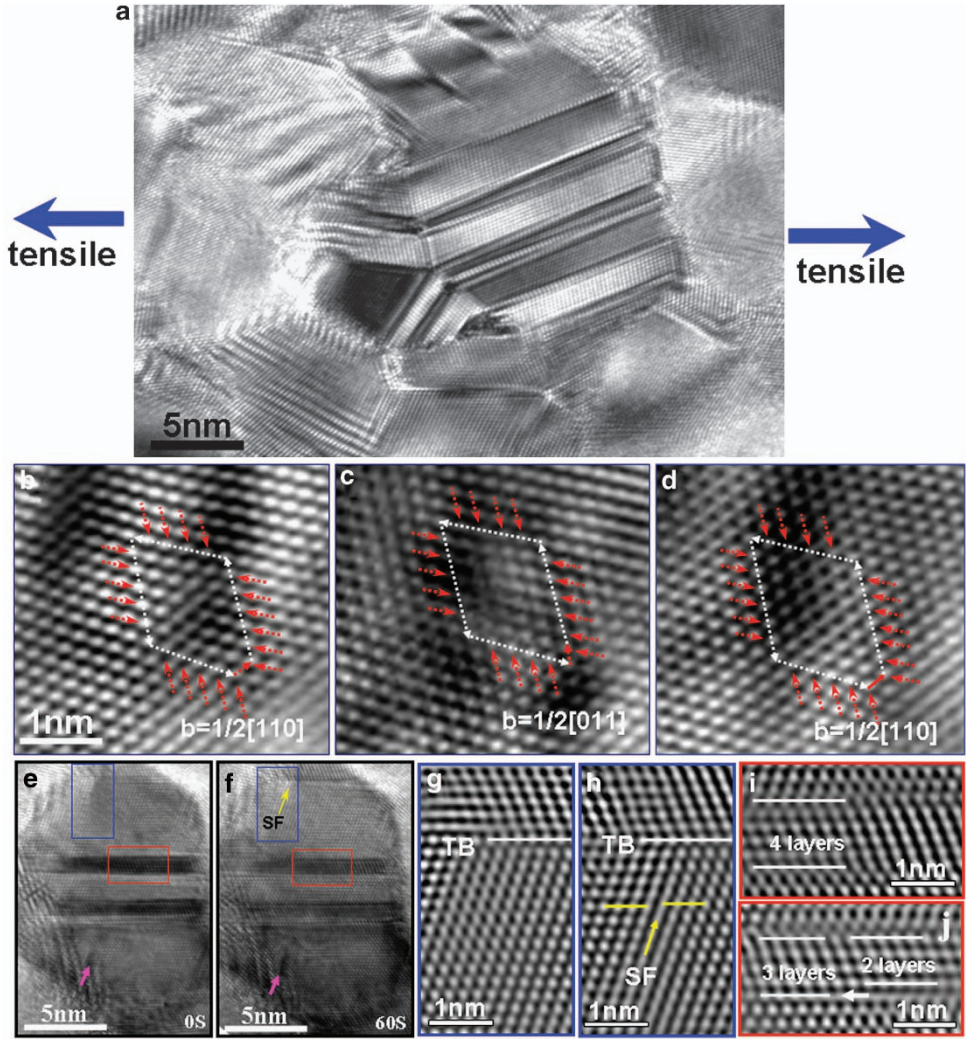

Figure 4 (**a**) A schematic illustration of the dynamic processes of the nanocrystal can be recorded *in situ* during tensile loading with the bimetallic techniques. (**b**–**d**) Enlarged high-resolution transmission electron microscopic (HRTEM) image of the same region that was recorded at a different stage of deformation. (**b**) Lomer dislocations are observed in the grain. (**c**) Under further loading, the Lomer dislocations undergo a destruction process; only 60° of full dislocations were observed. (**d**) We further discovered the re-formation of Lomer locks in the grain. (**e**, **f**) Two consecutive HRTEM images. (**g**, **h**) Partial dislocation nucleated and glided incline toward the twin boundary (TB) in the thicker twin. (**i**, **j**) Partial dislocation nucleated and glided incline parallel to the TB in the thinner twin. Figure from Wang *et al*.[29] Copyright 2010 The American Physical Society. SF, stacking fault.

For twin-structured nanocrystalline (grain size <100 nm) materials, the understanding of the atomic mechanism of deformation dynamics is primarily based on MD simulations. Li *et al*.[96] reported an 'inverse Hall–Petch' effect on the thickness of a twin plate. There is a critical twin thickness (λc) for a given grain size, and for the twin thickness $\lambda > \lambda c$, partial dislocations intersecting with the TB dominate the plastic deformation, which results in strengthening. When $\lambda < \lambda c$, partial dislocations glide along the TB, which results in softening. Experimentally, Yue *et al*.[97] directly observed this cross-over in nanocrystalline Cu thin films. As an example, Figures 4e and f present two consecutive HRTEM images collected during the loading. Figures 4g and h are the enlarged HRTEM images collected from the blue-framed region in Figures 4e and f. A partial dislocation resulted in a nucleated and glided incline toward the TB in the thicker twin lamellae (approximately 5 nm). For thinner twin lamella (approximately 1.2 nm), the partial dislocation emission and gliding along the TB resulted in a decrease of the thickness from four atomic layers (Figure 4i) to three and two atomic layers (Figure 4j).

OTHERS: ATOMIC MECHANISMS OF THE DEFORMATION CHARACTERISTICS OF NTS AND HIGH-PRESSURE EXPERIMENTS ON NCS

NTs are a special class of NM because their thickness is typically one or a few atomic layers. The measurements of the Young's modulus of carbon NTs in TEM were firstly conducted by Treacy *et al*.[98] and by Poncharal *et al*.[99] Later, many other methods have been used to investigate the plastic deformation mechanisms of carbon NTs. In 2002, Demczyk *et al*.[100] conducted *in situ* pulling and bending tests on an individual MWCNT in a TEM. Utilizing the MEMS in the TEM, Zhu *et al*.[8] measured the fracture strength of MWCNTs, which is 15.84 GPa. Atomic-scale images revealed that the crystalline structure of the MWCNTs (graphite sheets) transformed to amorphous carbon during plastic loading. In 2003, Troiani *et al*.[101] obtained single-walled carbon nanotubes (SWCNT) from an amorphous C film by the combined effect of irradiation and axial strain. *In situ* HRTEM observations revealed that ductile NTs developed either a junction or a linear chain of C atoms before failure.

Huang *et al.*[102] discovered that at high temperatures, SWCNT can undergo superplastic deformation. Figure 6a shows a SWCNT with an initial length of 24 nm. At tensile failure, the SWCNT was 91 nm long, which represented a tensile elongation of 280%; its diameter was reduced from 12 to 0.8 nm, as shown in Figure 6d. Kinks frequently form during tensile straining (Figures 6b–d), and they propagate along the tube and then accumulate (Figure 6d) or disappear at the ends. The NT immediately narrows after the kink passes. The super-elongation is due to the high activity of the nucleation and motion of the kink and the atomic diffusion that occurs at high temperatures (2000 °C). The authors' tensile tests at RT revealed that all of the NTs failed at a strain of $<15\%$.

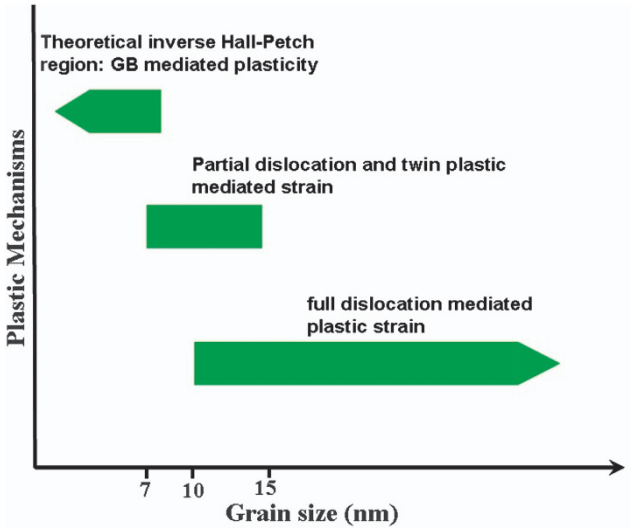

Figure 5 Grain size effect on the plastic deformation mechanisms in Pt nanocrystalline samples. As the grain size decreases, there is a transition from full dislocation to the partial dislocation. When the grain size is less than approximately 7 nm, grain boundary (GB) will mediate the plastic deformation.

Because of the special structure, buckling was always observed in the compressive side of the NTs, especially under a bending stress. The atomic-scale structure of this buckling in bent carbon NTs was observed using *ex situ* HRTEM.[99] Recently, the buckling and fracture modes of thick (diameter >20 nm) MWCNTs under compressive stress were examined using *in situ* TEM by Zhao *et al.*[103] The buckling behavior of MWCNTs under compression falls into two categories; the first is non-axial buckling and subsequently complex Yoshimura patterns can be induced on the compressive side of the MWCNTs. The second is axial buckling followed by catastrophic failure. Furthermore, the shell-by-shell fracture mode and planar fracture mode of MWCNTs have been directly observed.

By conducting *in situ* bending tests in the TEM, Bai *et al.*[104] observed that multiwalled boron nitride nanotubes (BNNTs) exhibit two interesting phenomena. *In situ* HRTEM observation revealed that a severely distorted graphitic lattice recovered after the bending strain was released, and the measured I–V curves were suggestive of piezoelectric behavior in the deformed BNNTs, that is, normally electrically insulating multiwalled BNNTs may surprisingly transform to a semiconductor. Using a similar method, Golberg *et al.*[105] also observed that the multiwalled BNNTs can sustain large strains under bending deformation. The deformation of the BNNTs proceeded through the propagation of consecutive momentary kinks. These kinks were observed to be entirely reversible on reloading with no traces of residual plastic deformation. Low voltage aberration correction TEM makes the almost impossible task of imaging light atoms (boron, carbon, nitrogen or oxygen) without electron beam irradiation damage feasible. However, the deformation experiments of CNT are scarce,[106] and the majority of the works are related to investigations of the static structure or those conducted under electron beam irradiation.

HRTEM is the only method that is capable of directly viewing the atomic structure while providing information about the chemical structure; however, the disadvantage is that the observation must be conducted in a vacuum. It appears almost impossible to directly observe pressure-induced atomic motion. However, Sun *et al.*[27,35]

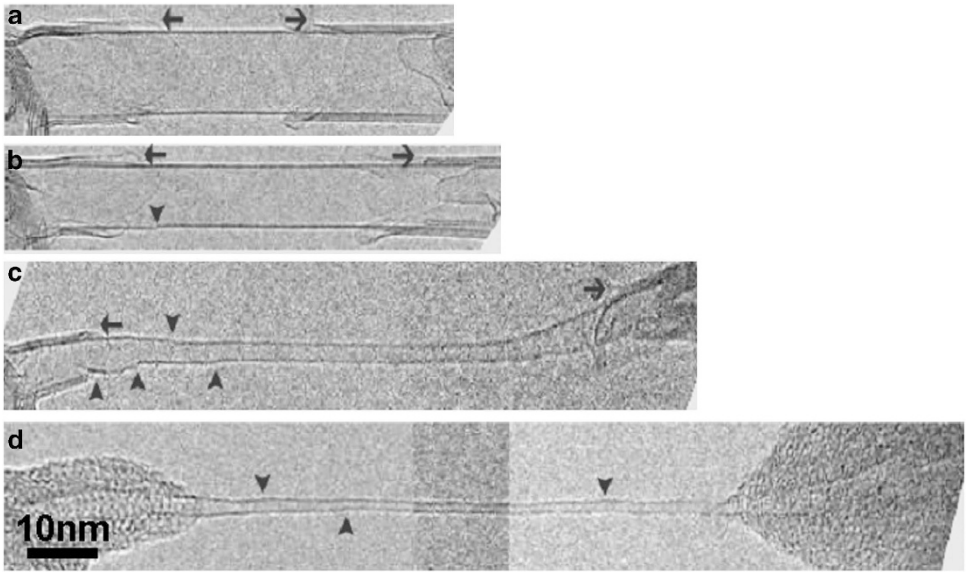

Figure 6 *In situ* tensile elongation of individual single-walled carbon nanotubes viewed via high-resolution transmission electron microscopy. (**a–d**) Tensile elongation of a single-walled carbon nanotube. Arrowheads mark kinks; arrows indicate features at the ends of the nanotube that are almost unaltered during elongation. Figure from Huang *et al.*[102] Copyright 2006 Nature Publishing Group.

developed a new *in situ* ultra-high pressure experimental method in the TEM. With this method, the atomic-scale behavior of Fe_3C NCs under ultra-high stress was observed in real time.[26] Up to 6% compressive strain was observed in the Fe_3C NCs. No visible defects, such as dislocations or twins, were observed during the deformation.

The ultra-high pressure experiment was also conducted using Au NCs,[107] and their TEM results provide evidence that the vacancy concentration in a nanoscale system can be less than in the bulk material. Figures 7a–d shows the gradual extrusion of Au NCs from a graphitic shell at 600 °C. Grain boundaries appeared at the bottleneck in the Au NCs where the deformation rate was the highest (Figures 7e and 7f). When the experimental temperature was $\leqslant$300 °C, twins were occasionally observed. The results indicated that the plastic behaviors are temperature dependent. This conclusion was confirmed by the experiment conducted with Pt at different temperatures[107] and the face-centered cubic phase NCs W and Mo with a typical size of 3–15 nm.[108] The authors claimed that the plastic deformation is governed by the activity of short-lived dislocations and that diffusion of these dislocations may also participate in the plastic deformation. Further experiments and modeling under these special conditions are required.

SUMMARY AND PERSPECTIVES

In situ atomic-scale experimental mechanics of NM is a rapidly growing field that is benefitting from technological advancements in instrumentation and improved specimen fabrication techniques, although stress–strain correlations are still absent in the majority of current studies. For nano-mechanics, *in situ* TEM experiments provide the possibility to directly observe the deformation mechanisms along with measuring stress–strain data at the appropriate length scale and dynamic observations, which are essential for understanding the deformation mechanisms of NMs. In the past decades, significant progress has been made regarding the deformation dynamics of NMs using quantified techniques, and the influence of material dimensions on the nucleation and multiplication of dislocations has, to some extent, been resolved. However, open questions remain regarding the dynamics of dislocation interactions in small volumes. With the emergence of atomic-scale experimental nano-mechanics, new opportunities and research fields may arise in this regime.

Furthermore, with the integration of equipment that allows for the quantification of strain–stress output for small-sized NM with HRTEM, Cs-corrected atomic-scale HRTEM imaging, individual

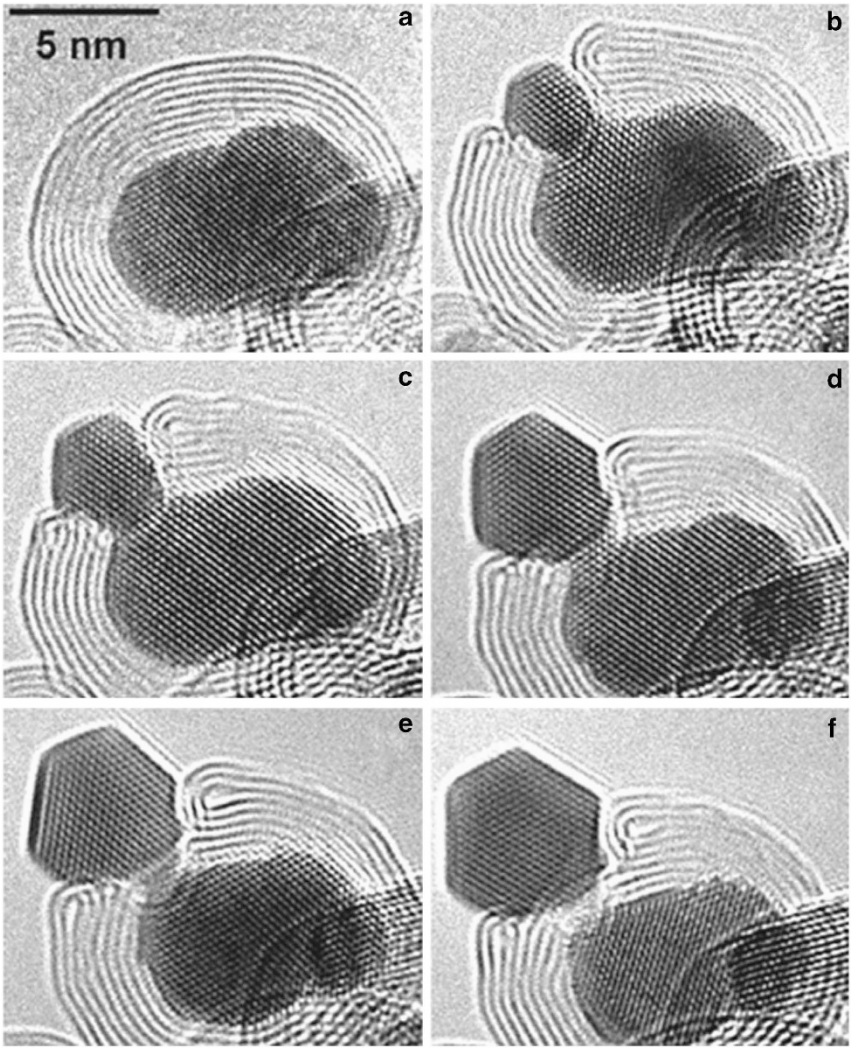

Figure 7 Extrusion of a gold crystal at 600 °C. No visible defects appear in the Au crystal. Irradiation times: (**a**) 0, (**b**) 240, (**c**) 300, (**d**) 480, (**e**) 600 and (**f**) 720 s. Beam current density: 200 A = cm^2. At 600 °C, the graphitic shells have a high structural perfection. Figure from Sun *et al.*[107] Copyright 2008 The American Physical Society.

atomic probing ability and real-time and atomic-scale chemical information detection, a golden age for conducting investigations of the experimental mechanics of materials at atomic scale can be expected, and the related research field will be very optimistic in the near future. However, care should be taken when conducting these *in situ* deformation dynamics studies using TEM due to the shower of electron beam irradiation. By taking advantage of the electron beam irradiation, unusual mechanical properties can be approached.[37,109,110] However, with an overdose of this radiation, significant irradiation effects will be integrated with the intrinsic physical properties of these materials. When conducting *in situ* experimental microscopy mechanics or physics investigations by assigning the fast electrons to interact with the observed objects, we have to obtain a balance between 'watching' and 'damaging' (overdosing).

Regarding atomic-scale nano-mechanics, to be specific, it is optimistic that several research fields can find solutions from the integrated technology of stress–strain correlations, atom-by-atom structural, chemical and even electronic property investigation with external stress/straining on the materials:

(1) For complex nanosystems that consist of multiple elements, atomic-scale imaging with accurate chemical distinction ability is necessary. Under stress/straining, the time-resolved and dynamic atomic-scale imaging ability for the strained materials provide new opportunities in nanoscience;

(2) The direct atomic mechanisms of GB sliding, diffusion and rotation for complex alloys (multiple elements and/or intermetallic compounds) are highly important for developing novel nanocrystalline materials;

(3) The interface structural evolution process includes the direct atomic-scale understanding of the twinning nucleation and/or propagation process in nanocrystalline materials;

(4) The interphase interface structural evolution process of those multiple-phased NMs when they are under stress/strain;

(5) With the ability of ultra-large elasticity, the lattice spacing of the NM can be significantly changed, and the band structure and the related physical and chemical properties can be tuned accordingly. The new fields of strained/stressed engineering are promising for NMs because the strain-induced polarity of NM creates novel piezoelectronics, giant magnetic-resistance effects, phase transitions, and so on. All of these needs not only require atomic-scale understanding but also Cs-corrected imaging to directly map the polarity, atomic-scale strain distributions with single atom resolution and accuracy (even for light atoms such as C, O and N atoms, and so on). New materials, novel functionalities and applications can thus be designed with atomic-scale precision. With, but not limited to the aforementioned possibilities, a golden age of 'nano-mechanics with an atomic scale' is promising.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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Professor Ze Zhang is a Member of Chinese Academic of Sciences. He is the Professor both in Institute of Microstructure and Property of Advanced Materials, Beijing University of Technology and Department of Materials Science and Engineering and State Key Laboratory of Silicon Materials. In 1987, he received a PhD degree in Chinese Academy of Sciences. From 1995 to 2001, he was a Professor and the Director of the Beijing Laboratory of Electron Microscopy, Chinese Academy of Sciences. From 1997 to 1999, and from 2012 to present, he is the President of the Asia Crystallography Associations of Societies. Professor Zhang discovered a new icosahedral phase with fivefold symmetry in a transition metal consisting of Ti–Ni–V alloy, and carried out a series of original and systematic study on the structure, defects and phase transformation of quasicrystal. Owing to his outstanding contributions in materials, he was elected as a Member of Chinese Academy of Sciences in 2001. His research interests also include the relationship of microstructure and physical properties of low-dimensional nanostructures, microstructures of defects in the complex metallic alloy phases, microstructure and growth mechanism of nanonostructures. Since 2002, he has been leading and chairing two national 973 programs for developing *in situ* microscopy techniques under external fields and related investigations.

Professor Xiaodong Han is the Director of Institute of Microstructure and Property of Advanced Materials, Beijing University of Technology. In 1996, he received a PhD degree in the Dalian University of Technology and Beijing Laboratory of Electron Microscopy. From 1997 to 1998, he conducted postdoctoral research in the City University of Hong Kong. From 1998 to 2001, he was a postdoc research scientist in the University of Pittsburgh. From 2001 to 2004, he worked for HKL Technology as a research scientist (now Oxford Instrument), where he focused on back-scattered electron diffraction by scanning electron microscope. From 2004 to present, he is a professor in the Beijing University of Technology. His research interests include structure–property correlations of materials using electron microscopy (transmission, scanning and scanning probe microscopy) and other advanced scientific equipments at the macro, nano and atomic scales. Since 2004, he has been focusing on developing atomic scale and multiple-scale *in situ* electron microscopy techniques for mechanics and nano-physics and related investigations on nanomaterial.

Dr Lihua Wang received PhD degree (2012) in the Beijing University of Technology, and worked as an assistant professor in the Institute of Microstructure and Property of Advanced Materials, Beijing University of Technology. His research currently focuses on atomic-scale nano-mechanics for FCC, BCC and Hexagonal metals.