

Review Article

In Situ Atomistic Deformation Mechanisms Study of Nanowires

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“Smaller is stronger,” sub-, micro-, and nanomaterials exhibit high strength, ultralarge elasticity and unusual plastic and fracture behaviors which originate from their size effect and the low density of defects, different from their conventional bulk counterparts. To understand the structural evolution process under external stress at atomic scale is crucial for us to reveal the essence of these “unusual” phenomena and is momentous in the design of new materials. Our review presents the recent developments in the methods, techniques, instrumentation, and scientific progress of atomic scale *in situ* deformation dynamics on single crystalline nanowires. The super-large elasticity, plastic deformation mechanism transmission, and unusual fracture behavior related to the experimental mechanics of nanomaterials are reviewed. *In situ* experimental mechanics at the atomic scale open a new research field which is important not only to the microscopic methodology but also to the practice.

1. Introduction

Unlike conventional materials, when the size is decreased to nanoscale, the materials such as nanowires (NWs) [1], nanopillars [2–4], nanotubes [5–7], and nanocrystals [8, 9] perform various unique properties, particularly in mechanical performance [10]. For example, not limited to the elastic strain of only a fraction of 0.2% as the bulk materials, an ultralarge elastic limit (~2%) has also been observed in nanocrystalline (nc) or nanotwinned materials [11–14]. Recent studies show that nanostructured materials can sustain ultrahigh elastic strain before yielding [1, 2, 4, 15–17] due to the change of the origin of the plastic deformation from inner dislocation sliding to dislocation nucleation on sample surfaces [18]. Theoretical results even predicated that the elastic limit can reach 8% for single crystalline copper NWs [19], which has been proved by experimental result [20]. For the study of plasticity, the size effect on the dislocation behavior has also been well studied [21–24]. Although extensive works have been done to study the mechanical properties of nanomaterial, however, due to the limit of experiments at atomic scale, most of the atomic mechanisms were revealed by simulations; there would be a lot of discrepancies and even wrong predications due to the empirical or semiempirical potentials and boundary

conditions used during the simulation process. Although some research groups and commercial companies have paid much more attention to developing new *in situ* experimental technique [2–4, 7, 25–30], studying the mechanical properties of nanomaterials at atomic scale is still a challenge for us today. Here, we introduce several *in situ* atomic scale experimental techniques and relevant research results. The elastic-plastic and also the fracture behaviors of single crystalline copper NWs have been studied systematically, which is important for us to understand the unusual mechanical properties of nanomaterials at atomic scale.

2. Methods

The nanomechanics can be dated back to 1997. Wong et al. used the AFM tip to measure the strength and the toughness of nanorod and nanotube [5], after which more and more researchers plunged into this new field [6]. Although a lot of excellent works have been done and deepened our understanding of the mechanical properties of nanomaterials dramatically, these techniques and studies cannot normally reveal the actual deformation mechanisms due to the deficiency of atomic scale information, such as the dislocation initiation sites, the interaction activities, and the dislocation types. So, some researchers began to use Transmission Electron

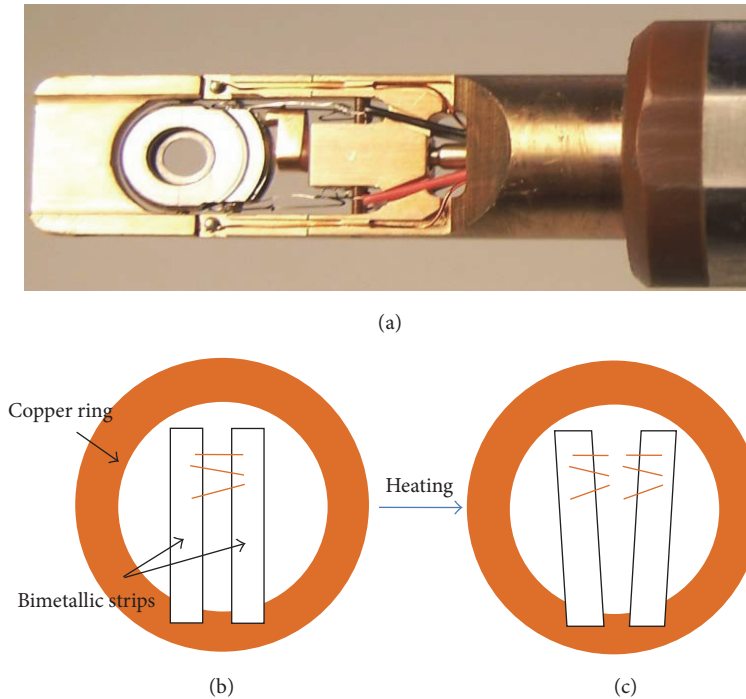


FIGURE 1: (a) shows the Gatan 652 double tilt heating holder; (b) and (c) are the sketch maps showing how the thermal bimetallic strips driven TEM device appears to work.

Microscope (TEM) which can provide atomic-level resolution to study the mechanical properties of nanomaterials [31]. TEM demonstrated its powerful strength in studying the mechanical properties of nanomaterials in the following decades [2–4, 6–8, 17, 26–32]. Several research groups and companies are very professional in this field. For example, Chen et al. [25] and Bai et al. [7] developed their own mechanical measurement instruments, respectively, and did a lot of excellent works. Hystrion developed a new mechanical measurement system, named PI-85 used in SEM and PI-95 used in TEM to study the size effect on the mechanical properties of micro- and nanomaterials. Professor Greer and Professor Shan did a lot of excellent works based on these instruments [3, 4, 28]. A Nanoindenter XP was used by Professor Bei to study the mechanical properties of Mo-alloy single crystal micropillars [33–35]; the effects from overstrain and focus ion beam milling were scientifically studied. Gatan Company developed a single tilt straining holder. Microstructural evolution such as dislocation nucleation and escape has been *in situ* recorded in TEM [36] to exhibit *in situ* deformation progress of nanocrystalline thin films and single crystals [29]. Furthermore, NanoFactory, which has been purchased by FEI, embedded AFM cantilever into a piezo-driven holder (TEM-STM) to quantify the force loaded to the sample to study the relationship between the stress and the structure evolution [30]. With the development of semiconductor technology, MEMS has also been used in special TEM holder to help the mechanical study of nanomaterials [26, 27].

However, due to the introduction of complex extra accessories in TEM, it sacrificed the “Y” tilt which is crucial

to ensure the observation of the sample along specified zone axis to get the atomic information. Since 2008, serious efforts have been made by Professor Han’s group, colloidal thin film contraction method [37–41]; thermal bimetallic strips driven TEM device have been developed continuously [20, 22, 24, 42–44]. Colloidal thin film contraction method is to make several preset cracks on a commercial TEM colloidal thin film grid; electron beam irradiation provides the force loaded to the NWs with two ends bridged on the crack of the colloidal thin film to do the tensile and bending tests [22, 37–41]. Because there is no extra device introduced into TEM, the sample holder is the standard double tilts TEM holder, so it is easy to tilt the sample to a low index zone axis to observe the sample which ensures the *in situ* test at atomic scale. Thermal bimetallic strips driven TEM device is another new novel method [20, 23, 24, 42–44] which is effective in conducting the *in situ* tensile tests of materials. As shown in Figure 1, the main part is two bimetallic strips adhered on a TEM ring, with a Gatan 652 double tilt heating TEM holder (Figure 1(a)); these two bimetallic strips provide planar loading force to the sample bridged on the two bimetallic strips (Figures 1(b) and 1(c)); this smart device has the same size with the commercial TEM grid, so there is no sacrifice for the double tilt capability as the colloidal thin film contraction method but with a controllable strain rate. Recently a new TEM holder is invented which can not only do the *in situ* deformation experiments at atomic scale but also quantify the force loaded to the sample. It is a completely new system to study the mechanical properties of nanomaterials which will give us more evolution information during the deformation process [45].

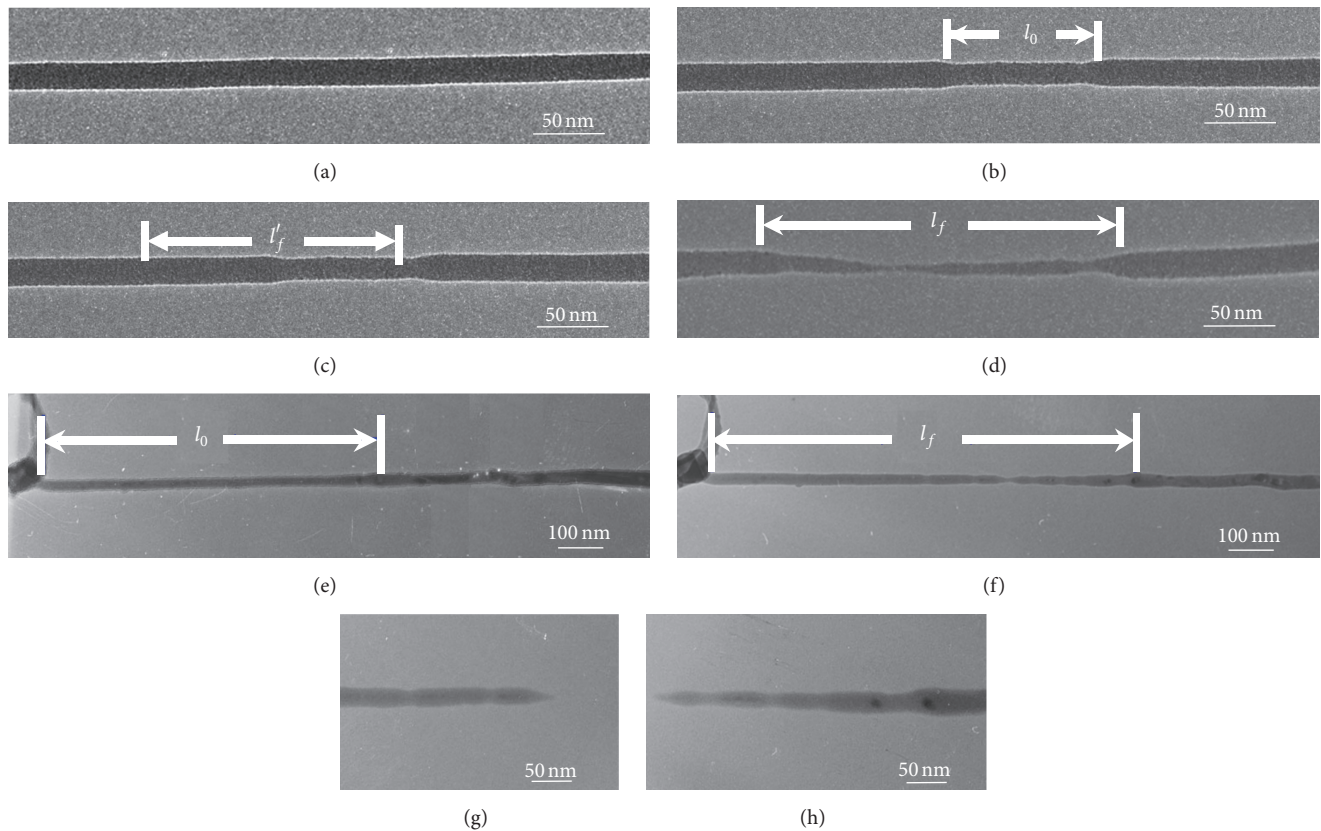


FIGURE 2: ((a)–(d)) A Si NW during the *in situ* application of axial tension. The arrows indicate the corresponding positions of the extended NW throughout the procedure. ((e)–(h)) Another example of an extended Si NW with buckling character. The axial elongation of the Si single crystalline NWs can be revealed by comparing the corresponding l_0 and l_f [41].

3. Large Strain Elasticity and Plasticity of Semiconductor NWs

Today, semiconductor NWs, such as silicon NWs [49–51], GaAs NWs [52, 53], and InAs NWs [54, 55], have been widely used for fabricating various electronic and optoelectronic nanodevices due to their excellent properties. Although recent results show that retained elastic-plastic strains in NWs significantly affect their electronic properties [56], there is only limited information on the mechanical property of the Si NWs due to the difficulty of carrying out *in situ* tensile or bending measurements on individual NWs. Several experimental approaches have been developed to study the mechanical properties of NWs and nanotubes (NTs) based on atomic force microscopy (AFM) at nanometer-scale spatial resolution [1, 5, 6]. However, the failure in revealing the atomic scale structural evolution information limited the application of AFM measurements in the *in situ* elastic-plastic-fracture study. With the size decreased to nanoscale, recent works show that elastic strain of silicon nanowires [51] can reach about 10%, which is about 100 times larger than that of their bulk counterpart; a few samples even reached ~16% tensile strain, with estimated fracture stress up to ~20 GPa. Besides, the elastic modulus of amorphous Al_2O_3 increases significantly when the thickness of the layer is smaller

than 5 nm [57], which is attributed to the reconstruction of the bonding at the surface of the material, coupling with the increase of the surface-to-volume ratio with nanoscale dimensions. With the bimetallic method [20, 23, 42–44], the origin of high elastic strain (13%) in amorphous silica nanowires has been successfully revealed, attributed to the bond's elastic elongation and the change of bond angle for amorphous nanomaterials [42].

With colloidal thin film contraction method developed by Professor Han's group [37–41], *in situ* TEM observation of the elastic-plastic-fracture processes of a single Si NW was recorded at atomic resolution. Large strain plasticity (LSP) of silica NWs has been first reported which subverts the brittle nature recognition of silicon in its bulk form [41]. Figures 2(a)–2(d) show a series of images of a single crystal Si NW that was extended by the force created by the shrink of the broken colloidal thin film. A clear plastic deformation was observed at the center of the NW; such LSP was also observed in SiC NWs [37], another brittle material in its bulk form. Both of these two results show that the LSP is attributed to a brittle-ductile transition which originates from a dislocation-initiated amorphization. MD simulation revealed a transition in the onset of silicon plasticity which depended on the temperature and stress magnitude [58]. For the case of high temperature and low stress, partial dislocation loops are

nucleated in the $\{111\}$ glide set planes. However, at low temperature and very high stress, perfect dislocation loops are formed in the other set of $\{111\}$ planes called shuffle. The following study of the mechanical property of pure amorphous brittle materials, silica, confirmed that when the size decreased to nanoscale, the materials will perform LSP, originating from the broken rotation and reformation of Si-O bond [39]. Furthermore, the strain rate effect [43] has been successfully studied with the bimetallic method [20, 23, 42–44], shedding light on the deformation behavior of amorphous materials.

4. Elastic Strain Limit of Single Crystalline Metallic NWs

Conventional materials yield at an elastic strain of only a fraction of 1% [59]. This apparent ending of the elastic regime is governed by the activation of dislocations (and/or their sources) that are inevitably preexisting inside the material. Based on the Hall-Petch effect [60, 61], researchers strengthen materials via inner grain nanocrystallization [11–14, 62, 63] or surface nanocrystallization [64] and so forth. An ultralarge elastic limit ($\sim 2\%$) has also been observed in nanocrystalline (NC) [59] or nanotwinned materials [11, 12, 14, 65]. During the transition of the origin of the plastic deformation from inner dislocation sliding to dislocation nucleation on sample surfaces [18, 19, 66], small volume crystals always sustain ultrahigh elastic strain before yielding [15–17, 32]. It has been presumed that the ideal elastic strain of metallic crystals can approach 8% [18]. After Taylor observed that antimony wires with a diameter of $30\ \mu\text{m}$ can be repeatedly bent without breaking [67], subsequent mechanical tests on a variety of whiskers [10, 68], micro- and nanopillars [2–4, 16, 33–35, 69–74], NWs [1, 34–38, 66], nanocrystal [8, 9], and nanotubes (NTs) [5–7, 31] have revealed that these micro- and nanoscale components can sustain large elastic strains and the yield and flow stresses increase when the size decreases. When the size was decreased, the interior dislocations density will decrease dramatically; with the development of sample preparation method, perfect NWs with nearly no interior dislocations can be prepared [75], the materials can be defined as defect-scarce nanostructures [66], new dislocations have to be nucleated on free surface or stress concentration region which requires high stress which is approaching the theoretical stress, and the elastic strain is approaching the theoretical elastic limit. Chen et al. [66] measured the surface dislocation nucleation strengths in high-quality $\langle 110 \rangle$ Pd nanowhiskers subjected to uniaxial tension directly and found the nucleation strengths were weakly size- and strain rate-dependent, and a strong temperature dependence was first uncovered.

Benefiting from the newly *in situ* deformation methods [20, 23, 42–45], Yue et al. [20] and Wang et al. [76] have observed super elasticity close to or beyond the theoretical limit. Using the bimetallic tensile technique [20, 23, 42–45], *in situ* tensile test of Cu NWs with different sizes has been conducted. Figure 3 shows the size effect on the elastic strain limit of Cu NWs via direct measurements during tensile testing. The elastic strain achievable in Cu NWs has been found to increase with the decreasing sample diameter, with

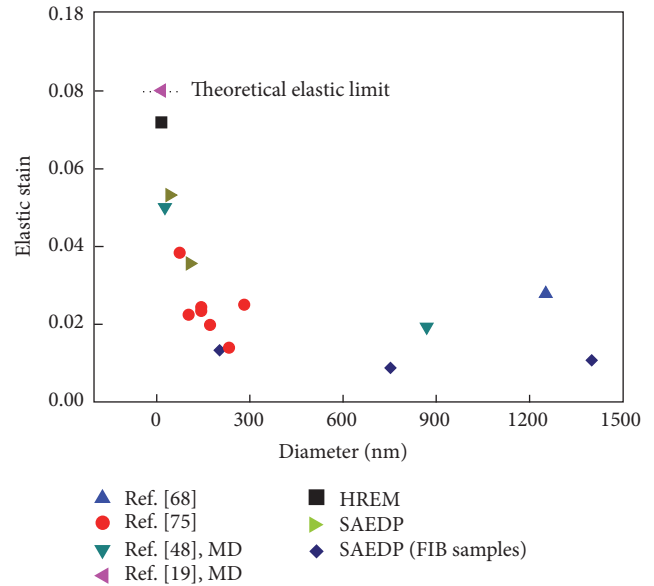


FIGURE 3: Size dependence of the elastic strain limit of single crystalline Cu NWs and fibers [20].

an approximate elastic strain of 7.2% observed directly during the deformation of a Cu NW with a diameter of $\sim 5.8\ \text{nm}$. The process of lengthening the atomic bonds at atomic scale has been captured, and the calculated strain approached the theoretical elastic limit predicted by MD simulation [19]. Besides, with colloidal thin film contraction method [22, 24, 37–41], Wang et al. [76] observed a continuous and gradual lattice deformation in bending nickel NWs to a reversible shear strain as high as 34.6%. This complete continuous lattice straining, four times that of the theoretical elastic strain limit for unconstrained loading, was attributed to transitions from face-centred cubic lattice to body-centred tetragonal structure and to reoriented face-centred cubic structure which is different with the traditional tensile strains.

5. Pseudoelasticity through Phase Transformations and Reorientation of Single Crystalline NWs

Structural transformation of nanomaterial was observed from FCC Au NWs by Diao et al. [77]. Phase transformation happened accompanied with more than 30% contraction strain when the size was reduced to $1.83\ \text{nm} \times 1.83\ \text{nm}$; the structure transformed from a FCC to a BCT structure, attributed to surface induced reorientation. Such surface induced analogous phenomena have been observed widely in many metallic NWs, such as Au [74, 78, 79], NiAl [80], Ag [81], Pd [82], Fe [83], Cu-Zr [84, 85], and Cu [48, 86]. Recently, Seo et al. [46] reported a superplasticity of defect-free Au NWs on tensile deformation and demonstrated a size effect of the Au, Pd, and AuPd NWs on the twin propagation stress without reduction of plasticity [87]. Figure 4 shows the entire tensile process of the Au NW. Seo et al. described the whole tensile deformation using three distinctive stages as shown in the stress-strain curve: Region 1 is the superelastic

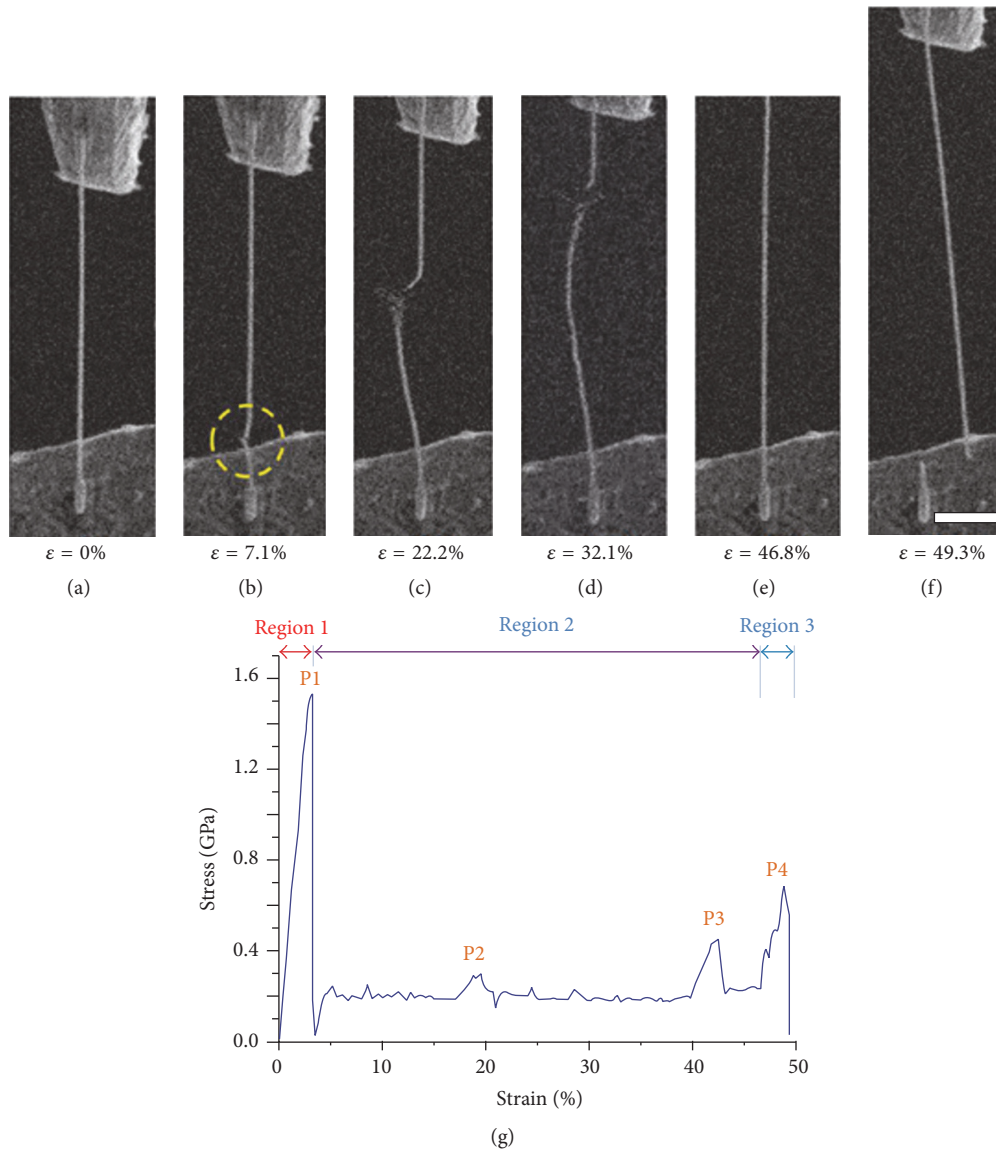


FIGURE 4: ((a)–(f)) SEM images show the entire tensile process until a final fracture of single Au NW; (g) is the corresponding stress-strain curve of a $\langle 110 \rangle$ Au NW for the whole tensile process. ϵ in ((a)–(f)) represents total strain in each tensile stage [46].

and super strong region, Region 2 is the superplastic region which will be a recoverable strain (or a rubber-like pseudoelastic) indicated by MD simulations [48], and Region 3 is the tensile process of a reoriented $\langle 100 \rangle$ NW. Guo et al. [47] also observed a superelasticity of individual VO_2 NWs which is arising from the M1-M2 structural phase transition. Figure 5 shows how the M2 phase nucleates, grows, and propagates. A plateau was detected in the stress-strain curve, indicating the superelasticity of the NW. Figures 6(b)–6(d) are the TEM images which show the phase transformation from M1 to M2.

6. Atomic Plastic Mechanisms of the Size Effects on Single Crystalline Metallic NWs

The plasticity of metallic NWs in response to mechanical stresses is another important aspect to be considered

in designing nanoelectromechanical system (NEMS) and microelectromechanical system (MEMS), in which metallic NWs serve as building blocks. Yu et al. discovered the high strong size effect on deformation twinning [88]. The stress required for deformation twinning of single crystal titanium alloy increases drastically until the sample size is reduced to one micrometer, below which the deformation twinning is entirely replaced by ordinary dislocation plasticity. It seems that there is an obvious size effect on the types of dislocations, which changed between full dislocations and partial dislocations [21, 23]. For FCC metals, such as Cu, slip of full (or extended) dislocations attributes to the plastic deformation with bulk size. High strain rate may cause deformation twinning (DT) which usually happened at very low temperature [89–93]. However, when the characteristic physical size of these FCC metals becomes small, it is of great interest to

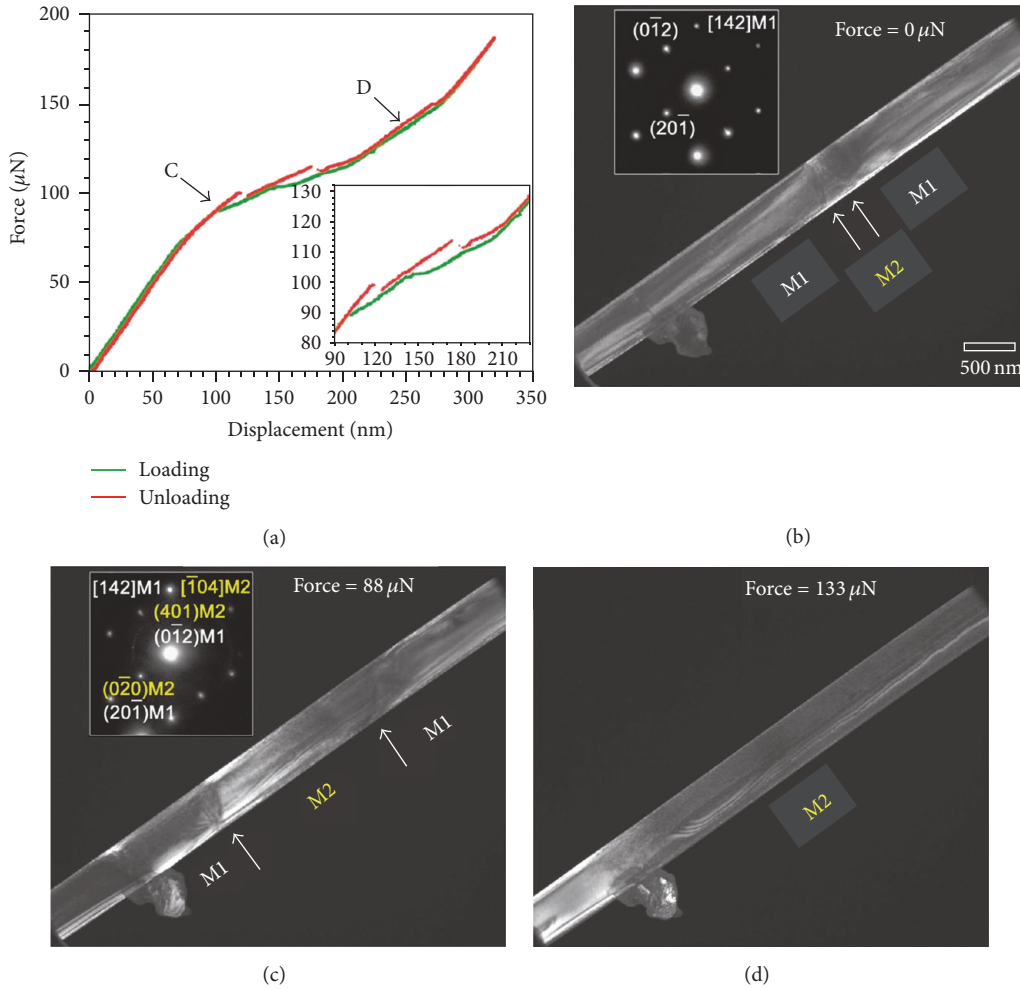


FIGURE 5: (a) Force versus displacement curve of a VO_2 NW on the PTP device. (b) Dark field TEM images of the unstrained VO_2 NW. (c) Sample at loading of 88 μN and (d) sample at loading of 133 μN [47].

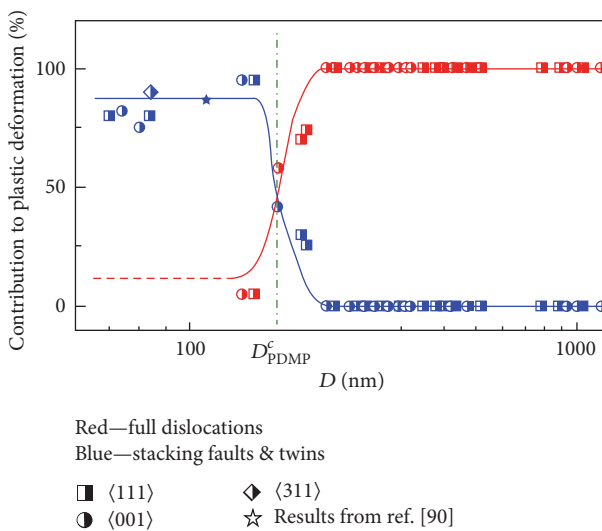


FIGURE 6: Sample size dependence of two plasticity mechanisms: relative contribution to the overall plastic strain experienced by the sample region under observation, from full dislocation slip (red symbols) versus partial dislocation (blue symbols) mediated processes [23].

note that such well-documented mechanical behavior may change. For example, an interesting observation in earlier study reveals that the general trend of diminishing DT breaks down when the grain size in a polycrystalline metal is reduced into ultrafine-grained (UFG) or NC regimes. Stacking faults and deformation twins, originating from partial dislocations, become more detectable and even preferable than ordinary full dislocation slip [21, 23, 94–99]. With the STM-TEM holder produced by NanoFactory, Zheng et al. studied the deformation behaviors of Au NWs with various diameters (even <10 nm) [30]. *In situ* atomic scale observation indicated that partial dislocations emitted from free surfaces dominated the deformation of the sub-10 nm-sized gold NWs. Furthermore, lattice slips would dominate the plastic events with the size below ~ 6 nm [100–102]. Seo et al. reported that defect-free Au NWs (~ 100 nm) exhibit superplasticity upon tensile deformation, attributed to the propagation of twin boundaries [87]. Partial/twinning was found to dominate the plasticity in Au nanowhiskers with diameters below 200 nm [103, 104]. With the thermal bimetallic strips tensile device [20, 23, 42–45], Yue et al. [23] quantitatively revealed an obvious effect of the sample dimensions on the plasticity

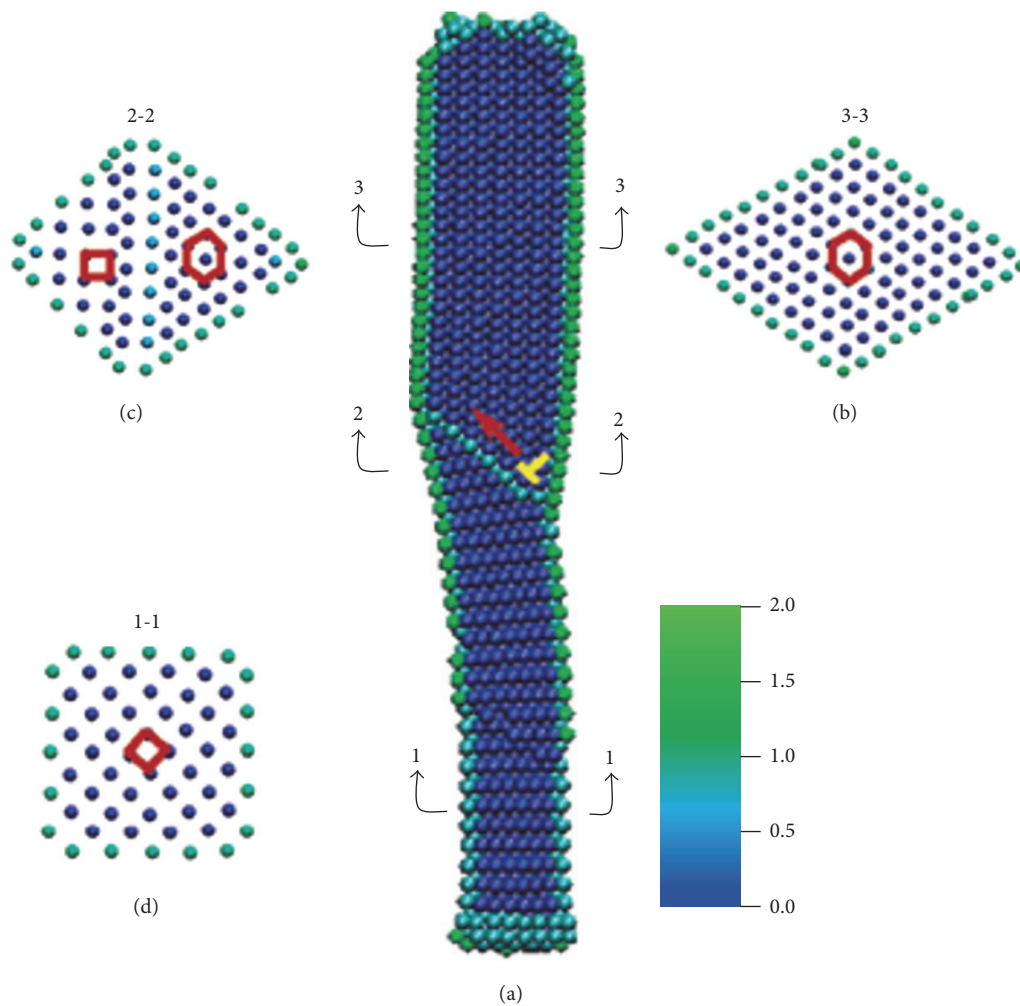


FIGURE 7: Lattice orientations on the cross sections of a $1.76 \times 1.76 \text{ nm}^2$ Cu nanowire at a strain of 0.24; (a) a sectional view along the wire axis and the $\langle 110 \rangle$ diagonal of the cross section, (b) elongated hexagonal lattice on the cross section in the unrotated domain with the $\langle 110 \rangle / \{111\}$ configuration, (c) a cross section in the transition region containing both the $\langle 001 \rangle / \{001\}$ and the $\langle 110 \rangle / \{111\}$ configurations, and (d) square lattice on the cross section in the rotated domain with the $\langle 001 \rangle / \{001\}$ configuration. Atoms are colored according to their centrosymmetry values [48].

mechanisms of Cu single crystalline NWs with diameters between 1000 and 70 nm in a HRTEM. Figure 6 shows the summary of the size effect on the plastic contribution of the partial dislocation mediated plasticity and the full dislocation. When the size was reduced to ~ 150 nm, the normal full dislocation slip was overwhelmed by partial dislocation mediated plasticity.

7. Rubber-Like Fracture Behaviors of Single Crystalline Cu NWs

An unusual kind of deformation behavior [105, 106] has been discovered since 1932 [107] which have puzzled the world over 60 years. Such so-called pseudoelastic rubber-like behavior happened in a lot of alloys (including Au-Cd, Au-Cu-Zn, Cu-Al-Ni, and Cu-Zn-Al) [108–110]; after aging for some time in a martensitic state, these alloys can be deformed like a soft and pseudoelastic rubber. Recently, such

rubber-like pseudoelastic behavior has also been discovered in single crystalline Cu NWs in atomistic simulations [48] on top of the experimental confirmation. Figure 7 shows the cross section intersecting the twin boundary, indicating the lattice transition between the two domains. Upon loading and unloading in single crystalline Cu NWs, this behavior is different from the classical austenite-to-martensite phase transformation reported before but a reversible crystallographic lattice reorientation driven by the high surface-stress-induced internal stresses due to high surface-to-volume ratios at the nanoscale level [48]. This phenomenon occurs only in NWs but does not occur in bulk materials.

With size and the dislocation density decreasing, a lot of fabulous phenomena have been exhibited to our field of vision. For small volume crystals with large surface area, such as whiskers [75, 111] and wires [1, 74] or pillars [33–35, 69–72, 112], in which the defect density can be made very low, tests have found that the apparent yield strength increases

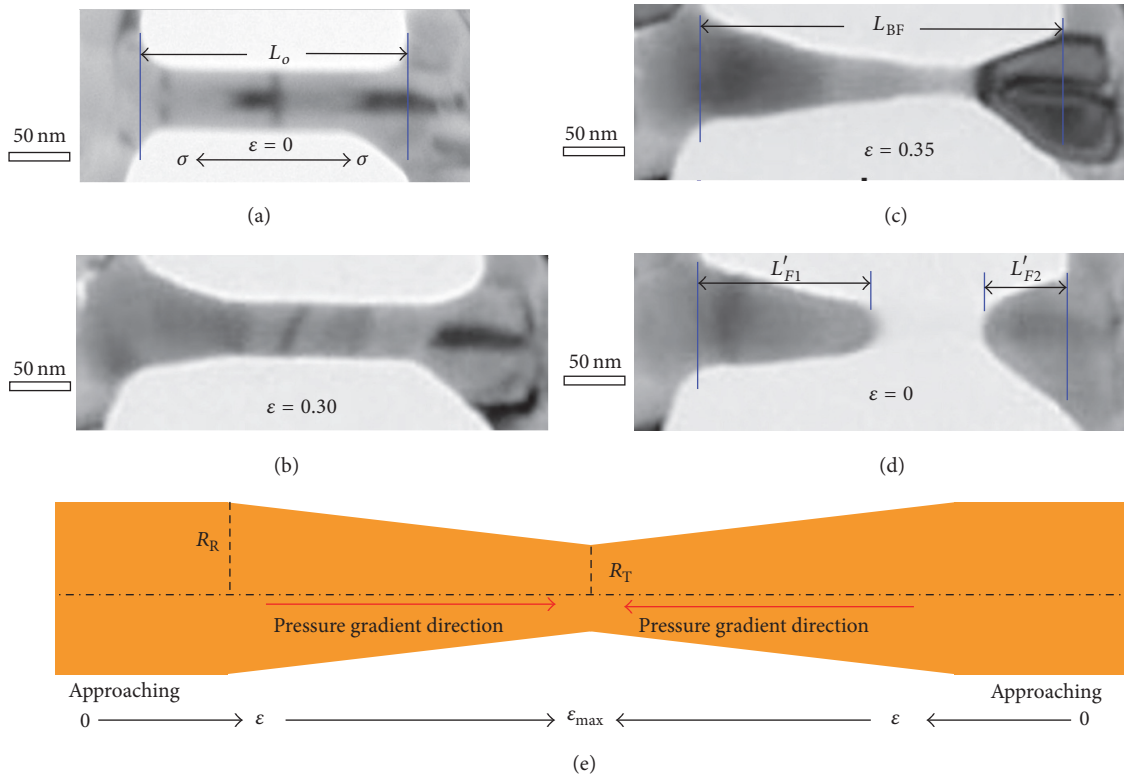


FIGURE 8: (a)–(d) An example of a single crystal Cu NW under uniaxial tensile test with a recoverable strain up to $\sim 35\%$; (e) a simple sketch map of the rubber-like behavior of the fractured single crystalline Cu nanowire tips [44].

significantly and correspondingly the elastic strain becomes much larger. This behavior is in contrast to conventional bulk metals, which will yield at a uniaxial tensile stress of tens of megapascals and an elastic strain of only a fraction of 1%. It has been confirmed that metallic crystals can perform ultralarge elastic strain from both the MD simulation result (8%) [19] and the experimental result (7.2%) [20]. However, it is still uncertain what would be the consequence when such huge elastic energies stored in the strained NWs are released in a short time, that is, the strain NWs' fracture behavior. Zheng et al. [30] observed that the Au nanocrystal exhibits a phase transformation from a face-centered cubic to a body-centered tetragonal structure after failure. Sun et al. [113] attributed such highly unusual Coble pseudoelasticity of Ag nanoparticles to surface diffusion. Based on the new homemade bimetallic technique that can perform *in situ* axial tensile deformation on nanomaterials (including single crystal [20, 23], nanofilm [20, 24, 44], and NWs [22, 42, 43]), *in situ* TEM tensile tests on copper NWs at a temperature close to room temperature [44], an astonishing crystalline-liquid-rubber-like behavior of the fractured single crystalline Cu NWs was revealed. From Figure 8, we can find that the retractable strain of the fractured crystalline NWs approaches over 35%. These abnormal behaviors originate from the fast release of the ultralarge elastic energy of the tensile NWs. The release of the ultralarge elastic energy has been estimated to generate a huge reverse stress as high as ~ 10 GPa. The consequent pressure gradient can increase the effective

diffusion coefficient [114–116]. The estimated concomitant temperature increase can reach as high as $0.6T_m$ of copper (T_m is the melting point) on the fractured tip of the NWs. Atomic diffusion process has been enhanced greatly due to the temperature increase.

8. Diffusion Dominated the Deformation of Nanomaterial

Based on the Hall-Petch relation [117, 118], decreasing the crystalline (grain) size can be used to strengthen materials, which is called “smaller is stronger” and the sample size effect on the strength and dislocation mechanisms has been studied intensively for these years [2–4, 15–17, 22, 23, 28, 32, 33, 88]. Deformation mechanism for crystalline materials at room temperature will change when the sample size was decreased. Yu et al. [88] reported a strong size effect on deformation twinning of Ti-5 at.% Al single crystal. They developed a “stimulated slip” model to explain such strong size dependence of deformation twinning. Yue et al. [23] found the full dislocation induced deformation will be replaced by partial dislocation mediated plasticity (PDMP) (including deformation twinning) when the sample size is below 150 nm. They also demonstrated this transition with quantitative contributions from PDMP and full dislocation. Recently, Tian et al. [119] find that such traditional plasticity mechanisms (dislocation slip and deformation twinning) will be replaced by diffusional deformation when the size of Sn

single crystal is decreased to 130 nm; “smaller is stronger” has also been changed to “smaller is weaker.” They found the yield strength of sample with size of 130 nm was 5 times lower than the yield strength of the 450 nm sample; this “smaller is weaker” trend is different from the “smaller is stronger” trend. And, a liquid drop-like fracture process which is mediated by the diffusional deformation has been revealed in Tian’s study.

Sun et al. [113] also observed a similar phenomenon from which they claimed that surface diffusion played the vital role in such highly unusual Coble pseudoelasticity of Ag nanoparticles. In their study, the Ag nanoparticle deformed like a liquid droplet but remained in its crystalline character; no dislocation activities were found during the process. Combined with MD simulation results, they found that the change of shape was controlled by single adatom movements, not by chain or island processes. Such Coble pseudoelasticity is different from the conventional pseudoelasticity driven by martensitic transformations [108–110, 120]; surface-diffusion-mediated pseudoelastic deformation happened in the sub-10 nm regime at room temperature, which is important to understand the shape change and shape stability of sub-10 nm material components.

9. Conclusion

With the development of *in situ* atomic scale experimental methods, the mechanical study of single crystalline NWs and other nanomaterials has achieved rapid and great progress, which benefits us greatly in understanding the essence of the unusually properties of nanomaterials. In this review, we summarized most of the *in situ* atomic scale technological advancements in instruments in the past decades. Our newly thermal bimetallic strips TEM device is highlighted and mainly introduced. Due to the fact that deformation mechanism of the micro-NPs has been comprehensively reviewed by other researchers, we only provide a brief summary about the size effect on the elastic, plastic, and even the fracture behaviors of single crystalline NWs. When the size is reduced to nanoscale, especially down to 100 nm, superelasticity, pseudoelasticity, superplasticity, cross-over of plasticity mechanism, and unusual fracture behavior have been demonstrated in this review which is helpful in understanding the mechanical properties not only for single crystalline metallic NWs but also for other nanomaterials such as thin films and nanoparticles.

Competing Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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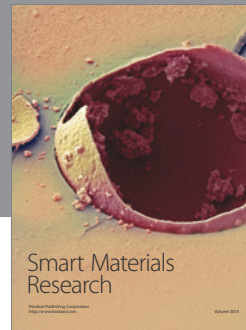
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