Reveal the size effect on the plasticity of ultra-small sized Ag Nanowires with *in situ* atomic-scale microscopy

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Abstract

Revealing the atomic-scale deformation mechanisms of metallic nanowires (NWs) is important for their practical application. However, there are few reports providing direct atomic-scale experimental elucidation on those metallic NWs. Here, we conduct serial *in situ* deformation tests on silver (Ag) nanowires with diameters of 3-11 nm. The *in situ* atomic-scale observations reveal a transition in the deformation

mechanism with a decrease in the diameter of Ag NWs. For the $\bar{[554]}$ and $[001]$ oriented NWs with diameters of ~ 11 nm, the plastic deformation is dominated by full dislocation that involves leading and trailing partial dislocations, whereas the full or extended dislocations are rarely observed in the NWs with diameters in the range of ~ 5 -8 nm, and their plastic deformation is governed by SF generation and annihilation.

Moreover, for the $\bar{[111]}$ oriented NWs, 60° mixed and pure edge dislocations are frequently observed when the diameter is approximately 5 nm and the plastic deformation is accommodated by relative slip between two adjacent $\{111\}$ planes for NWs with diameters below ~ 3 nm. These results indicate that the plastic deformation not only depends on the size of NWs but also can be significantly impacted by the loading orientation.

Key words: silver nanowire; size effect; in situ; plasticity; atomic-scale.

1.Introduction

Metallic Nanowires (NWs) have attracted a great deal of interest not only because of their potential in nanotechnology applications but also for their fundamental scientific interest[1, 2]. However, NWs are typically used under external stress and/or strain to achieve novel properties and functions for practical applications[3, 4]. A dynamic atomic-scale elucidation of deformation mechanisms of NWs under external stress and/or strain is therefore fundamentally important for developing new materials with desired properties[1, 2]. *In situ* nanomechanical testing is a powerful means to study the deformation mechanisms of NWs through real time imaging. In recent years, many experimental studies have been conducted to investigate the atomic-scale deformation dynamics of metallic NWs such as Au[5-9] and Cu[10]. Based on these experimental observations, it has been speculated that there is a transition in the deformation model from full dislocations to partial dislocations with the decrease of the grain size [5-8, 11, 12]. Therefore, it is expected that the partial dislocations should dominate the plastic deformation in metallic NWs with diameters at the nanometer level.

There are very few direct atomic-scale experimental reports on the deformation behavior of metallic NWs with ultralow SF energy, such as Ag. The atomic-scale understanding of deformation mechanisms of Ag NWs so far has relied on previous molecular dynamics (MD) simulations[13-18]. Most of these MD simulations have predicted that the deformation is controlled by partial dislocations for the low stacking fault energy metals. MD predictions can provide important insights into the deformation mechanisms of NWs; however, the validity of these simulation results is still debatable under the experimental conditions. In addition, most of the previous studies have only considered the size effect on the deformation mechanism, although the loading orientation and stacking-fault (SF) energy also play a non-negligible role in determining the deformation mechanism. Thus, the atomic-scale observation of dynamic deformation process of Ag NWs under different loading orientations is necessary for understanding the deformation mechanisms of metallic NWs with ultralow SF energy.

2. Experiment details

In this study, we conducted a series of *in-situ* tensile tests on Ag NWs inside a high-resolution transmission electron microscopy (HRTEM) using an in-lab developed deformation device. The deformation mechanism of Ag nanowires with diameters in the range of 3-11 nm was investigated at the atomic scale in real time. Our *in situ* atomic-scale observations show a clear transition in deformation mode as the size of the Ag NWs decreased. However, the transition not only depends on the NWs but also can be significantly impacted by the loading orientation and the SF energy.

The Ag NWs with different diameters were obtained using our recently developed method[19, 20]. By pulling the nanocrystalline thin film (prepared by the magnetron sputtering method), many ligaments that connect the opening cracks are formed, which are often made of a single grain with freshly opened surfaces. Each such ligament can be treated as a single-crystalline NW. The uniaxial tensile tests of

Ag NWs were conducted inside a JEOL-2010F TEM (200 kV) at strain rates from $\sim 10^{-3} \text{ s}^{-1}$ to $\sim 10^{-4} \text{ s}^{-1}$ and at temperatures (below $\sim 60^\circ\text{C}$) far below the melting point of Ag, using an in-lab developed TEM tensile device[19, 20].

3. Results and discussion

Figure 1 shows the atomic-resolution TEM images illustrating the tensile process of an Ag NW. The NW is $[110]$ direction oriented and the tensile-loading direction is $[\bar{5}54]$ (as indicated by the red arrow). No dislocation is observed before the elastic strain limit of the single-crystalline Ag NW with a diameter of $\sim 11 \text{ nm}$, as seen in Figure 1a. A similar trend was observed for many other individual Ag NWs with diameters of $\sim 10 \text{ nm}$. Figure 1b is a typical HRTEM image captured during the plastic deformation showing that two dislocations, namely “1” and “2” nucleated at the top and bottom surfaces of the NW, respectively. The detailed analysis indicated that “1” and “2” are extended 60° full dislocation and 60° full dislocation, respectively. The extended dislocation is formed by the emission of a leading partial dislocation followed by a trailing partial dislocation on the same plane with a Burgers vector of $\mathbf{b} = \frac{1}{2}[\bar{1}01]$, indicated by the red arrow in Figure 1b. The two partial dislocations are separated by a SF, and the width of the SF is determined by the local stress state. The inset shows the enlarged HRTEM image from the red-framed region of Figure 1b, in which the local Burgers circuit is drawn to determine the Burgers vector of dislocation “2”; the extra plane is seen for the (111) plane. This configuration represents a typical 60° full dislocation. These nucleated extended dislocations can travel across the NWs and eventually can escape from the surface of NWs. As shown in figure 1c, the extended dislocation “1” escaped during further straining, and full dislocation “2” dissociated into an extended dislocation. Similarly, dislocation “2” also escaped with the increasing strain. Meanwhile, a full dislocation “3” with $\mathbf{b} = \frac{1}{2}[\bar{1}01]$ and a SF resulting from a partial dislocation are observed in the NW, as seen in figure 1d. This result is consistent with the previous experimental observations that the plastic deformation is controlled by the slip of partial dislocations involving leading and trailing partial dislocations.

In addition to the leading and trailing partial dislocations resulting in the full dislocation events, SF resulting from a single partial dislocation is also observed. Figure 2 presents a series of *in situ* HRTEM images showing the dislocation dynamics involving the nucleation of partial dislocations in a silver nanowire with a diameter of $\sim 8 \text{ nm}$ along the $[110]$ axis. No dislocation is detected in the NW before its elastic limit, as seen in Figure 2a. During continuous straining, three SFs, “1”, “2”, and “3”, are observed due to the nucleation and glide of partial dislocations (Figure 2b). Figure 2c shows that the SF “1” is eliminated by a trailing partial dislocation. Another two partial dislocations are nucleated from the surface of NW upon loading and then propagated on a $\{111\}$ slip plane leaving behind two SFs (“4” and “5” as seen in Figure 2d). The extensive elongation of the NW led to the elimination of SFs “2” and “4” (Figures 2d-f), which is similar to the SF “1” observed in Figure 2c. This SF

generation and annihilation caused by the nucleation of leading and trailing partial dislocations is prevalent during the tensile test. In this study, we found that the plasticity is dominated by a leading partial dislocation nucleating first from the NW surface. After this leading partial dislocation escaped from the another surface of NW, a trailing partial dislocation is nucleated from the same location of the leading partial dislocation, thereby eliminating the SF and preventing the formation of extended dislocation (see [Supplementary movies 1](#)). This indicates that the NW size can significantly affect the plastic deformation of Ag NWs.

[Figure 3](#) illustrates two *in situ* HRTEM observations of partial dislocation dynamics during tensile loading of a NW with a diameter of 6.5 nm along the [001] direction. Before the elastic limit, no dislocation is detected in the NW as seen in [Figure 3a](#). During continuous straining, four SFs “1”, “2”, “3”, and “4” are observed ([Figure 3b](#)) due to the nucleation and glide of partial dislocations in the NW. Most of the SFs are eliminated after further pulling, similar to SF “1”, as observed in [Figure 2c](#). During the deformation, the plasticity is dominated by the nucleation and propagation of the first leading partial dislocation leaving behind a SF, followed by a trailing partial dislocation eliminating the SF without forming the extended dislocation.

In addition to the size effect on the dislocation configuration, we found that the tensile-loading direction can also affect the deformation mechanisms of Ag NWs. [Figures 4a-e](#) show a series of TEM images illustrating the tensile-loading process of an Ag NW with a diameter of ~5 nm along $\bar{[111]}$. No dislocation is observed in the NW at relatively low strain rates, as seen in [Figure 4a](#). In [Figure 4b](#), a full dislocation “1” is nucleated at the upper surface of the NW. The detailed analysis indicated that the full dislocation is a 60° mixed dislocation with a Burgers vector (b) of $\mathbf{b} = \frac{1}{2}[101]$.

The pure-edge dislocations also frequently nucleate after further pulling, as noted by the red-framed region of [Figure 4b](#) and [Figure 4c](#). [Figure 4f](#) and [Figure 4g](#) are enlarged HRTEM images corresponding to the red-framed regions of [Figure 4b](#) and [Figure 4c](#), respectively. Local Burgers circuits are drawn to determine the Burgers vectors and the extra planes of atoms are seen for both the $(\bar{1}11)$ and the $(1\bar{1}1)$ planes ([Figure 4e](#)). This configuration represents a Lomer dislocation (LD)[\[21, 22\]](#) with $\mathbf{b} = \frac{1}{2}\bar{[110]}$. This is the first time *in situ* atomic-scale observations have revealed the formation of Lomer-Cottrell lock dislocation in Ag NWs. Interestingly, SF or extended dislocation resulting from the partial dislocations is rarely observed during tensile loading. This full dislocation dominated deformation mechanism is different from the previous theoretical and experimental studies, which predicted that the partial dislocation is the dominant plasticity in nanoscale metals with low SF energy.

The diameter of the NW decreased due to extensive tensile elongation. The plastic deformation is controlled by the discrete cooperative slip events on two conjugate {111} planes for the NWs with diameters of ~ 3 nm, resulting in a number

of enlarged steps on the surface of the NW as seen in Figure 4e. These surface steps are several atomic planes apart exhibiting a saw-tooth morphology compared to that in Figure 4d and correspond to the quantized plastic deformation (see Supplementary movies 2). The relative slip between two adjacent $\{111\}$ planes can lead to the inhomogeneous deformation, which resulted in a rapid reduction of cross-section area (Figure 4e). During the plastic deformation, a SF or extended dislocation resulting from a partial dislocation is rarely observed.

These results show that there is an obvious size effect on the plastic deformation of Ag NWs, which is schematically illustrated in Figure 5. For $[\bar{5}54]$ and $[001]$ oriented NWs with diameters above ~ 11 nm, the plastic deformation is governed by full or extended dislocation, which involves leading and trailing partial dislocations (Figure 5a). In contrast, the full or extended dislocation is rarely observed for the NWs with diameters in the range of ~ 5 -8 nm, which shows the SF generation and annihilation as the dominant deformation mechanism, as illustrated in Figure 5b. Moreover, the loading direction can also significantly affect the deformation mechanisms of Ag NWs. The plastic deformation is dominated by the full dislocation that involves mixed and pure edge dislocations (Figure 5c) for the $[\bar{1}11]$ oriented NWs with diameters above ~ 5 nm, whereas the plastic deformation is accommodated by the relative slip between two adjacent $\{111\}$ planes for the NWs with diameters below ~ 3 nm (Figure 5d).

The *in situ* observations show a distinctive size effect on the deformation mechanisms of $[\bar{5}54]$ and $[001]$ oriented Ag NWs, in which there is a transition from extended dislocations to partial dislocations. This transition is similar to the NC case, in which dislocations switch from full to partial as the grain size decreases below a critical value [19, 23]. For Ag NW with a SF energy of ~ 18 mJ/m² [24, 25] and a shear modulus of 56.7 GPa [26], the critical grain size is ~ 50 nm. By considering the NW diameter equivalent to the grain size, it is reasonable observed that the plastic deformation is governed by leading and trailing partial dislocations in these small-size NWs. **In large-sized NWs, the trailing partial dislocation is always nucleated before the leading partial dislocation escaped from the surface of NW. Thus, extended or full dislocations are observed. For relatively small-sized NWs, leading partial dislocation can easily escaped from the surface of NW before the trailing partial dislocation nucleation, thereby partial dislocations are frequently observed. The observed extended and partial dislocations confirmed that the MD simulations are useful in understanding the plasticity in metallic NWs despite mild differences in the strain rate.**

Additionally, the schmid factors of the full and partial dislocations for the NWs with various loading directions are also calculated. For the NWs with $[\bar{5}54]$, $[001]$ and $[\bar{1}11]$ loading direction, the calculated maximum schmid factors for the

full dislocations is 0.334, 0.408 and 0.272, respectively, which are relatively smaller than the partial dislocations with maximum schmid factors of 0.386, 0.471 and 0.314 respectively. According to this calculation, it is reasonable that the plastic deformation is governed by partial dislocations in these small-sized NWs. However, the *in situ* atomic-scale observation indicate that the full dislocations are prevalent even in a ~5 nm sized Ag NW with loading direction of $\bar{[111]}$. This observation is contradict with the schmid factor calculation and previous MD simulations prediction[15, 27-35], which may be attribute to the different surface energy and morphology of $\bar{[111]}$ oriented NWs [6]. This might need more investigation. Here, the observation of full dislocation and the plasticity transition from full dislocation to relative slip between the $\{111\}$ planes indicate that the plastic deformation can be significantly impacted not only by the size but also by the loading direction, as observed in the case of the $\bar{[111]}$ oriented Ag NWs. These observations may also be valid for the metals with similar SF energy curve to Ag such as Cu, Au[26, 36-41].

4. Conclusion

In conclusion, extensive *in situ* experiments were conducted to obtain the atomic-scale tensile processes of Ag NWs with different diameters and orientations during the tensile-loading process using HRTEM. *In situ* investigation has provided the following important insights into the deformation mechanisms in ultra-thin Ag NWs: (1) the dynamic atomic-scale plastic deformation mechanism of Ag NWs with diameters below ~10 nm, which is rarely investigated experimentally; (2) the size effect on the dislocation mechanisms in Ag NWs is different from that in metals with high SF energy; (3) the plastic deformation can be significantly impacted not only by the size but also by the SF energy and loading direction; (4) the plasticity transition from extended dislocation to the SF generation and annihilation as the diameter decreased for the $\bar{[554]}$ and $[001]$ oriented NWs; and (5) the switching of the deformation mechanisms directly from full dislocation to relative slip between the $\{111\}$ planes for the $\bar{[111]}$ oriented NWs. These results will advance the fundamental understanding of the deformation mechanisms in metallic NWs, particularly the ultra-thin metallic NWs and will also provide useful guidelines for the design and manufacturing of novel nanometals.

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Figure captions:

Figure 1. *In situ* atomic-scale observations of the extended dislocations resulting from leading and trailing partial dislocations for the $[5\bar{5}4]$ oriented NWs: (a) HRTEM image of an Ag NW with a diameter of ~ 11 nm with no dislocation detected before the elastic limit; (b) A typical HRTEM image that captured the plastic deformation and the nucleation of two dislocations. The inset is an enlarged HRTEM image corresponding to the red-framed region; (c) The extended dislocation “1” escaped and full dislocation “2” dissociated into an extended dislocation; and (d) Similarly, dislocation “2” also escaped with increasing strain and a third full dislocation “3” along with a SF resulting from a partial dislocation are observed.

Figure 2. Sequential *in situ* HRTEM images showing the partial dislocations dynamics for the $[5\bar{5}4]$ oriented Ag NW with a diameter of ~ 8 nm: (a) Before the elastic limit, no dislocation is detected in the NW; (b) With continuous straining, three SFs resulting from the partial dislocations are observed; (c) SF “1” is eliminated by a trailing partial dislocation emission; (d) Another two partial dislocations are nucleated from the surface of NW (viz., “4” and “5”) after further pulling; and (d-f) SFs “2” and “4” are eliminated similar to SF “1” (c).

Figure 3. *In situ* observation of partial dislocation dynamics during tensile loading of the $[001]$ oriented Ag NWs: (a) Before the elastic limit, no dislocation is detected; (b) Upon further straining, four SFs “1”, “2”, “3”, and “4” resulting from the nucleation of partial dislocations are observed.

Figure 4. A series of TEM images shows the dislocation behavior of an Ag NW with a 5 nm diameter along the loading direction of $[\bar{1}11]$: (a) No dislocation is detected in the NW at relatively low strain; (b) A 60° full dislocation “1” with $\mathbf{b} = \frac{1}{2} [101]$ nucleated at the upper surface of the NW; (c, d) Nucleation of pure-edge dislocations is frequently observed as noted by the red-framed region; (f, g) Enlarged HRTEM images corresponding to the red-framed region of (b, c) with a Burgers circuit to identify the dislocation representing a Lomer dislocation with $\mathbf{b} = \frac{1}{2} [\bar{1}10]$; and (e) For the NWs with the diameters of ~ 3 nm, the discrete cooperative slip on two conjugate $\{111\}$ planes led to a number of enlarged steps on the surface of the NWs.

Figure 5 is a schematic diagram showing the deformation mechanism of Ag NWs: (a-b) In the case of $[5\bar{5}4]$ or $[001]$ oriented NWs, the plastic deformation is governed by full or extended dislocations for the NWs with diameters of ~ 11 nm; the SF generation and annihilation dominated the plastic deformation for the NWs with diameters in the range of $\sim 5-8$ nm; and (c-d) In the case of $[\bar{1}11]$ oriented NWs, the plastic deformation is dominated by full dislocation when the diameter of NW is

above ~5 nm; the plastic deformation is accommodated by relative slip between two adjacent {111} planes for the NWs with diameters below ~ 3 nm.

Figure1

Figure2

Figure3

Figure4

Figure5