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In situ TEM nanoindentation of nanoparticles

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ABSTRACT

The deformation behavior of nanoparticles continues to be an exciting area for materials research. Typically, nanoparticles show a conspicuous lack of dislocations, even after significant deformation. Therefore, it has been suggested that dislocations cannot exist or/do not play a role on the deformation of nanoparticles. In situ TEM nanoindentation is a critical tool for addressing this issue because it allows for the deformation to be monitored in real time. In this article, we discuss some of the experimental needs and challenges for performing in situ nanoindentation TEM experiments on nanoparticles. In addition, we show both diffraction contrast and phase contrast in situ TEM nanoindentation experiments on silver nanoparticles with diameters below 50 nm. Evidence of the presence of dislocations was observed during deformation, but upon unloading dislocations disappeared.

1. Introduction

Currently, single-crystal nanoparticles play an increasingly important role in a wide variety of fields including pharmaceuticals, advanced materials, catalysts for fuel cells, energetic materials, as well as environmental detection and monitoring. They can be end products themselves, as in the case of quantum dots or pharmaceutical drugs, or they can be incorporated into separate end products, such as in polymer matrix nanocomposites or carbon supported high surface area catalysts in fuel cells. Because the properties of nanoparticles are crucial to their performance and/or the performance of products containing nanoparticles, it is very important to have a fundamental understanding of the deformation mechanisms occurring in nanoparticles, including the type and density of crystal defects participating in these deformation processes. In fact, crystal defects such as surface steps, can affect the catalytic and thermal properties of individual nanoparticles, whereas dislocations and twins can affect the mechanical properties, radiation resistance and energy release exhibited by individual nanoparticles.

The literature on the deformation of individual nanoparticles is relatively scarce. Gerberich et al. (2003, 2005, 2006) have employed scanning-probe-microscopy, while Deneen et al. (2006, 2007) used in situ transmission electron microscopy to study the deformation of silicon nanoparticles. The experiments were done on particles around 200 nm in diameter. Both elastic and plastic deformation, as well as fracture was observed. Mook et al. (2007) analyzed these results and concluded that for nanoparticles in compression, scale effects exist for the modulus of elasticity and fracture toughness. Namely, the modulus increases with increasing mean pressure, while fracture toughness increases for smaller particle sizes. Mordehai et al. (2011) performed in situ STM nanoindentation experiments on gold nanoparticles ranging from 80 nm to 180 nm and reported a decrease in strength of the particles with decreasing particle size due to the efficiency of free surfaces in draining dislocations. Unfortunately, they were unable to find direct evidence of dislocations due to the limitations of STM.

Contrary to the starvation of dislocations in individual nanoparticles, twins have been often observed. Wu et al. (2000) reported a high frequency of twins in Pb and Ge nanoparticles, while Slouf et al. (2006) working on colloidal solutions found the presence of twins in Au nanoparticles. Again, in both cases, perfect dislocations were not observed. In another study, Armstrong et al. (2009) observed SnO_2 nanoparticles of different sizes, which were milled for various times. For larger particle sizes, shear bands, stacking faults, twins and cracking were observed, while for smaller nanoparticles these defects became less pronounced and at some size completely absent. Nevertheless, an increasingly plastic behavior was observed for smaller particle sizes, which seems to indicate instability of defects, such as dislocations.

In situ TEM nanoindentation experiments have been also performed in polycrystalline CdS hollow spheres ranging in diameter from 200 to 450 nm (Shan et al., 2008), as well as on clusters of silicon particles around 50 nm in size (Lockwood and Inkson, 2009). The former work reported that these particles can achieve both a high compression to failure and withstand very high shear stresses with respect to their ideal strength, while Lockwood and

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Inkson (2009) observed rotation of the nanoparticles, followed by cracking at the interface between two nanoparticles. However, the nanoindentation of polycrystalline particles and/or clusters of nanoparticles should be distinguished from individual nanoparticles, as single crystalline nanoparticles do not contain grain boundaries and thus deformation should be dominated by free surfaces. In fact, MD simulations performed by Zhang et al. (2011) and Mordehai et al. (2011) claim that below a critical nanoparticle size, dislocations become unstable due to the presence of free surfaces.

In this context, to understand the mechanical properties of individual nanoparticles, it is critical to perform in situ TEM nanoindentation experiments. This approach will allow (1) real-time characterization of the microstructure and defect behavior, such as dislocation nucleation and motion, (2) high spatial resolution ranging from the mesoscale to the atomic scale and (3) the possibility of correlating load–displacement curves with the evolution of the microstructure. In this paper, we will first discuss some of the experimental requirements and challenges for performing in situ nanoindentation TEM experiments on nanoparticles. Subsequently, we will show some results from in situ nanoindentation of silver nanoparticles using diffraction contrast and phase contrast TEM. Finally, we will point out some future avenues for enhancing the capabilities of in situ TEM nanoindentation of nanoparticles.

2. Experimental requirements and challenges for in situ TEM nanoindentation of nanoparticles

In situ TEM nanoindentation of nanoparticles allows us to observe and record deformation events at various scales as they occur in real time. Besides a transmission electron microscope, the two most important components for these in situ TEM experiments are: (i) a specimen holder capable of applying a force on the nanoparticle (Fig. 1) and (ii) a camera capable of recording the events in real time. The choice of these components will depend on the experimental requirements. For example, some experiments are better performed with a wide field of view, others require high magnification, yet others would benefit from acquiring load–displacements curves. To achieve useful results, the experimentalist must select an appropriate microscope and imaging technique as well a suitable specimen holder and recording device.

Starting with the selection of the microscope, the key issue for in situ TEM nanoindentation is the choice of the pole-piece gap associated with the instrument. The pole-piece gap is the distance between the magnetic pole pieces, which compose the magnetic electron lenses, and within which the in situ holder is located. In conventional TEMs, ultra-high resolution pole-pieces (URPs) used for high resolution imaging have a typical gap of 2 mm, which is too narrow for several of the nanoindentation holders available commercially. Often, nanoindenters that fit in URPs do not have load measurement capabilities. On the other hand, TEMs with analytical (ARP) and cryogenic pole-pieces (CRP) can accommodate any in situ nanoindentation holder but the overall image resolution is degraded. More recently, with the development of aberration-corrected TEMs, several of the manufacturers have installed ARP pole-pieces in these instruments, thus allowing both high-resolution imaging and in situ nanoindentation to be performed with any in situ nanoindentation holder.

The imaging mode of the in situ nanoindentation experiment must also be selected. In situ TEM nanoindentation experiments can be carried out in either diffraction contrast mode or phase contrast conditions. Typically, deformation experiments are performed in diffraction contrast since dislocation observation and analysis is facilitated under this imaging mode and the resolution requirements are lower. However, diffraction contrast imaging of in situ nanoindentation of nanoparticles can be very challenging. This is because there are numerous difficulties in establishing a two-beam condition in an in situ nanoparticle nanoindentation experiment. First, because of imperfections in adjusting the eucentric height of the sample, tilting causes image shifts that are very large compared to the field of view. As a result it is common to lose track of the area of interest during tilting. Additionally, it is difficult to generate a Kikuchi map from a single nanoparticle, making accurate tilting very time consuming and difficult. Finally, the experimental setup of in situ nanoindentation experiments tends to be extremely sensitive to vibrations. The vibration can cause the area of interest to move unpredictably or lead to violent collisions between the sample and the probe. For these reasons traditional dislocation analysis in diffraction contrast in situ nanocompression experiments is very challenging. Without an accurate knowledge of the diffracting conditions used for imaging, the diffraction contrast produced during nanoindentation is ambiguous. This problem is likely to become less critical in the future with the development of smart automated specimen holders, which can tilt the specimen to a specific zone axis, while maintaining the nanoparticle within the same field of view.

Because of the difficulties aligning and interpreting diffraction contrast in situ nanoindentation experimenst, phase contrast in situ nanocompression experiments are an attractive alternative. In the case of phase contrast in situ TEM nanoindentation experiments, the difficulty lies in aligning the crystal along a particular beam direction. This problem can often be solved by selecting a nanoparticle that is already in a good orientation prior to the onset of the deformation experiment. Another problem in phase contrast imaging is image interpretation, which often requires the assistance of computer simulations. However, computer simulations of how a dislocation affects the phase contrast of a nanoparticle are often complex, because the strain produced by the dislocation and the shape of the nanoparticle must be very well defined. These considerations are often not easily handled by available software. Finally, phase contrast nanocompression experiments often have to sacrifice load sensing capabilities due to the space restrictions of the URP.

For the selection of the in situ nanoindentation holder, the first consideration goes into assessing which pole-piece gap is installed in the TEM, as it determines the type of holders that will fit the instrument and the image resolution. Secondly, it is important to decide whether to use a single-tilt or double tilt holder. The latter are more expensive but sometimes critical in providing the ability

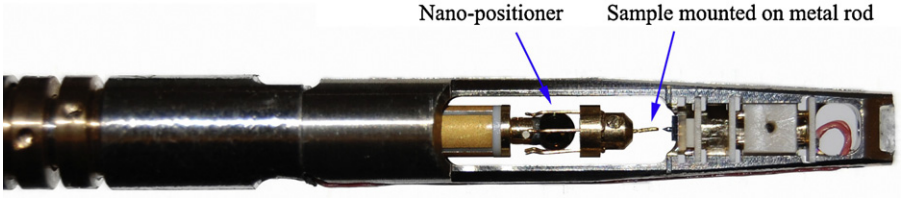

Fig. 1. In situ TEM nanoindentation holder.

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to reach specific zone axes. However, as mentioned above, tilting experiments on nanoparticles are challenging.

Another key factor is whether the objective of the experiment is to acquire a more mechanistic view of the deformation mechanisms involved during nanoindentation, or/and to obtain quantitative information. In the former, the holder can be simply a movable tungsten or diamond tip with no capability for measuring stress and/or strain. The advantage in this case is a significant reduction in the purchasing cost of the holder. Additionally, these simple holders are able to more readily fit into TEM's equipped with URPs. On the other hand, if quantitative data is desired, a more sophisticated nanoindentation holder is required. These are equipped with transducers for electrostatic actuation and capacitive displacement sensing. In this fashion, quantitative force–displacement curves can be acquired in situ. However, in the case of in situ TEM nanoindentation of nanoparticles, it is often difficult to identify the area of contact between the tip and the nanoparticle. Additionally, accurate determination of the 3-D geometries of both the indenter and the sample are possible only for the most well defined systems. As a result, the conversion from force to stress can be highly inaccurate and thus should be considered carefully.

Furthermore, a limitation with the current in situ nanoidentation holders is their inability to impose high strain rates on the sample. This is especially important in the case of larger nanoparticles (~100 nm), as the typical elongation rates for the holders range from 0.1 to 0.01 μm/s. Therefore, certain deformation events are very difficult, if not impossible, to capture. Another general challenge in working with any of the nanoindentation holders is controlling the position of the tip. Although these holders have sub-Ångstrom resolution in *X*, *Y* and *Z* positioning, due to the use of piezoelectric materials, aligning the tip along the direction parallel to the electron beam is not a trivial task. As TEM observations are always performed in projection, *Z* positioning of the tip is normally done with the image wobbler on, such that the sample and the nanoindentation tip can be made coplanar and aligned. Finally, it is critical to decide on the recording system. Wide-angle cameras in the 35 mm port are often lens-coupled CCD cameras with a fast read out. These are ideal for recording large fields of view and faster events where a large number of frames per second must be acquired. However, they lack resolution. As an alternative, high resolution fiber-coupled CCD cameras can be used. These have relatively slow read outs and narrow areas of view, making them fit for high magnification and slower dynamic experiments. More recently, advanced direct detection cameras have been developed (Li et al., 2006). These are likely to create a major leap in in situ TEM observations, as they are able to acquire 400 frames per second instead of the typical 30 frames second for regular CCD cameras. As a result, deformation events, such as dislocation nucleation and motion will be more likely to be captured in real time.

3. Diffraction contrast in situ TEM nanoindentation

Diffraction contrast in situ TEM nanoindentation experiments are attractive because they can be performed in TEMs with larger pole-piece gaps, which then allow thicker holders. This permits more instrumentation options, including the possibility of generating stress–strain curves during in situ deformation (Minor et al., 2006). Additionally, diffraction contrast imaging is somewhat less sensitive to changes in focus than phase contrast TEM, which can aid interpretation of the in situ experiments.

Fig. 2 shows such a diffraction contrast in situ TEM nanoindentation experiment. A FEI Tecnai X-twin 200 kV TEM equipped with an analytical pole piece was used. The use of the analytical pole piece enabled the nanoindentation holder from Nanofactory Instruments to fit the pole-piece gap. For the experiment, silver nanoparticles were affixed to a metal wire by dry dipping and placed opposite to a sharp mobile diamond tip, which acted as the nanoindenter.

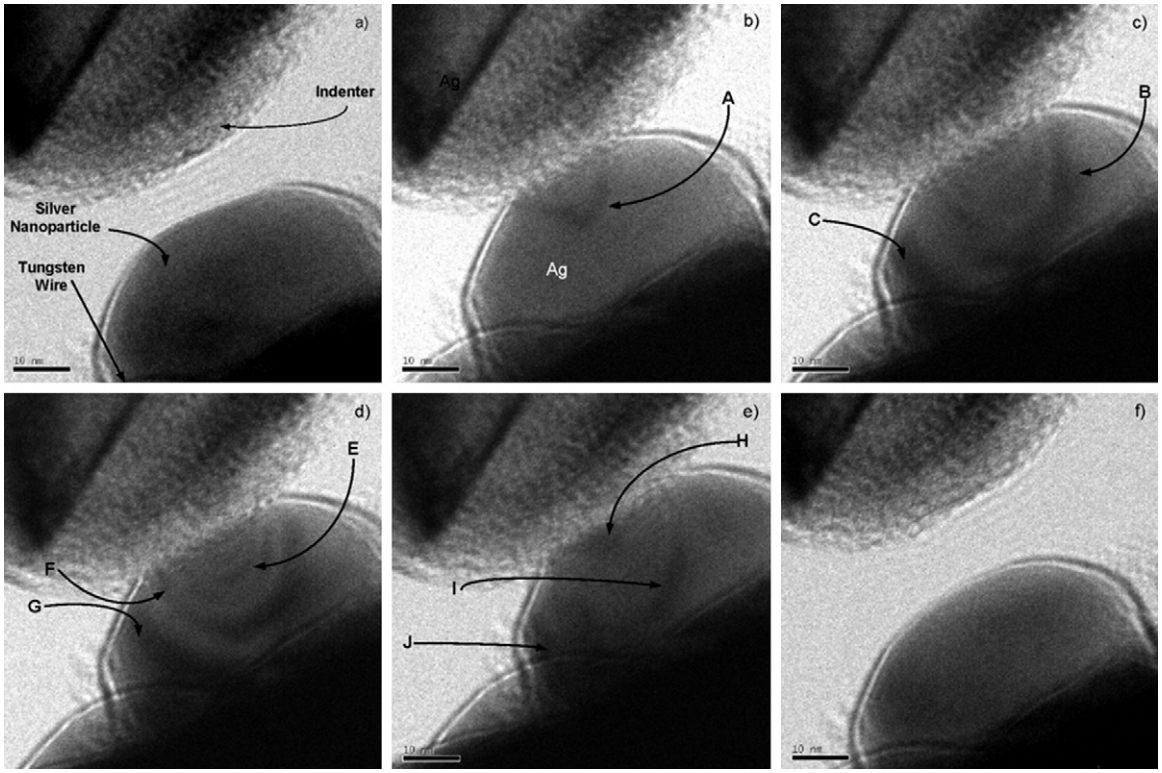

Fig. 2. TEM images taken from the diffraction contrast in situ nanoidentation experiment. (a) shows the probe and the nanoparticle before the nanoindentation experiment. (b)–(e) were taken during deformation, where contrast bands A–H are shown. (f) shows the nanoparticle and probe after deformation.

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The first step of the nanoindentation experiment was to bring the sample and the indenter into mutual focus (Fig. 2a). Subsequently, the indenter was translated towards the sample until contact was made (Fig. 2b). Immediately after, a contrast band (labeled A) appeared in the nanoparticle. As previously mentioned, due to the ambiguities of diffraction contrast imaging, the origin of the contrast band is unclear without a more detailed analysis, which will be discussed later. The strain on the nanoparticle was incrementally increased (Fig. 2b–e) and several contrast bands (B–H) were observed. It is difficult to determine whether the contrast bands moved between the images or if each image has unique contrast bands because of the rapid motion of the contrast bands during straining. After the straining experiment, the probe was removed from the sample until there was no contact (Fig. 2f). Under these conditions the contrast bands were no longer observed in the nanoparticle.

Correctly determining the origin of the contrast bands is critical to the interpretation of any diffraction contrast experiment. During nanoindentation, contrast bands can be caused by four different mechanisms: thickness fringes, stress contours, bend contours, and dislocations. The first three are artifacts are not associated with defects, and are based solely on the interaction of lattice strains and the electron beam in a (near) pristine crystal. These artifacts could in principle be ruled out by setting the appropriate conditions in the TEM. However, the delicate nature of the experimental setup in many in situ nanoindentation experiments makes tilting very challenging. Therefore, images of dislocations under various *g*-conditions are difficult to acquire because tilting causes far too much mechanical vibration in the instrument, which commonly translates the sample away from the experimental area.

Without the accurate tilting capability and to determine the origin of the contrast bands shown in Fig. 2, each of the possible artifacts was investigated. Consider first the effect caused by the thickness fringes, which are due to changes in crystal thickness. The fraction of electrons scattered to any reflection *g* is a function of the thickness of the sample. In diffraction contrast mode, this effect can lead to bright–dark contrast variations that alternate as the thickness of the samples changes. This modulation in intensity is characterized by the extinction length, which is 35 nm for the {1 1 1} type planes of silver at 200 kV (shortest extinction length for allowed reflections in silver). This means that a distance of 35 nm should be observed between every contrast band. However, Fig. 2c–e shows three or more coexistent contrast bands within the nanoparticle, which would then require a total thickness of around 105 nm. Because the particle tested was only 50 nm in diameter, it is not possible for the contrast bands to be due to thickness fringes.

Stress contours caused by variations in interplanar spacing due to deformation of the lattice planes are another artifact that could potentially explain the contrast bands. For example, if a crystal is being compressed normal to a {1 1 1} type plane, the corresponding {1 1 1} type reflection can become strongly excited. This can happen under relatively small strains if the (1 1 1) plane is near the Bragg condition. However, to produce a second contrast band it is necessary to excite the next co-linear reflection. This would require an elastic strain of approximately 50%, which is not possible for a metal. Because the nanoparticle contains three contrast bands in Fig. 2d and e, and the generation of more than one stress contour is not possible in a metal, it is safe to conclude that the contrast bands are not due to stress contours.

Bend contours, which occur when elastic bending of the lattice planes changes the diffraction conditions, is another elastic artifact that could explain the contrast bands. In fact, the type of contrast and shape shown by the bands in Fig. 2 is very similar to the bands reported in a previous nanoindentation experiment performed in a silicon film (Minor et al., 2005), which were described as bend contours. However, a simple classical two-point bending calculation shows that an applied stress of ~0.5 GPa is required to cause enough deflection for three bend contours to be observed simultaneously and the stress would need to be applied to the nanoparticle in a direction parallel to the support. In this regard, an estimate of the stress due to the Van der Waals force between the nanoparticle and the support gives a value of ~5 MPa. This quantity is far too small to sustain the ~0.5 GPa stress that would be required to produce three bend contours. In other words, the nanoparticle should slide along the support before bending. In fact, when we attempted to cantilever nanoparticles intentionally during in situ nanoindentation, we observed that the nanoparticles tended to slide freely across the support. Thus, bend contours are certainly a possible explanation for the appearance of the contrast bands, but they cannot be confirmed at this time.

We are now left with the possibility that the contrast bands are caused by the localized strain near the core of dislocations. However, as previously discussed, unless we have an accurate knowledge of the diffraction conditions used, there will be always an ambiguity in the interpretation of the images. For example, note the disappearance of the contrast bands once the nanoindenter is removed from the nanoparticle (Fig. 2f). This behavior is expected for any of the elastic type artifacts. However, dislocations can cause this phenomenon to occur as well. Indeed, an attractive force exists between dislocations and the nanoparticle's free surface due to a reduction in strain energy as the dislocation moves closer to the free surface. MD simulations performed by Zhang et al. (2011) and Mordehai et al. (2011) confirm that dislocations become unstable due to the presence of free surfaces. Therefore, both dislocations and elastic type artifacts in nanoparticles are expected to vanish when the strain is removed. As a result, unless the exact diffraction conditions are known, phase-contrast in situ TEM nanoindentation experiments are required.

4. Phase contrast in situ TEM nanoindentation

Phase contrast in situ nanoindentation experiments can avoid many of the ambiguities of the diffraction contrast experiments. Because phase contrast imaging can directly resolve atomic planes, in-depth discussions regarding contrast mechanisms for identifying dislocations are not necessary. Additionally, the mechanical limitations of in situ TEM nanoindentation experiments are less problematic for phase contrast imaging than diffraction contrast imaging because it is not necessary to change apertures or tilt the sample to identify dislocations and other defects, as long as an appropriate zone axis is selected in the first place. This can be simply done by choosing a nanoparticle that is already in a favorable orientation with respect to the electron beam.

Fig. 3 shows a phase contrast in situ TEM nanoindentation of a silver nanoparticle. This experiment was performed with a Nanofactory Instruments TEM-AFM holder, which is thin enough to fit an ultrahigh resolution pole-piece (URP). For this experiment an electropolished W probe was used instead of a diamond probe. From a mechanical perspective, the phase contrast nanoindentation experiment was almost identical to the diffraction contrast experiment. However the contrast mechanisms are completely differently.

Fig. 3a shows the silver nanoparticle before compression. The $(1\,1\,\bar{1})$ lattice fringes are in contrast in this image. The nanoparticle has no dislocations at this point, but a twin can be observed on the left side of the nanoparticle, as highlighted in Fig. 3a. This is confirmed by the presence of satellite diffraction spot shown in FFT (inset in Fig. 3a). Fig. 3b shows the nanoparticle at the beginning of deformation. A terminating lattice plane is clearly apparent, particularly in the magnified view shown in the inset. In phase contrast imaging, a terminating lattice plane is directly

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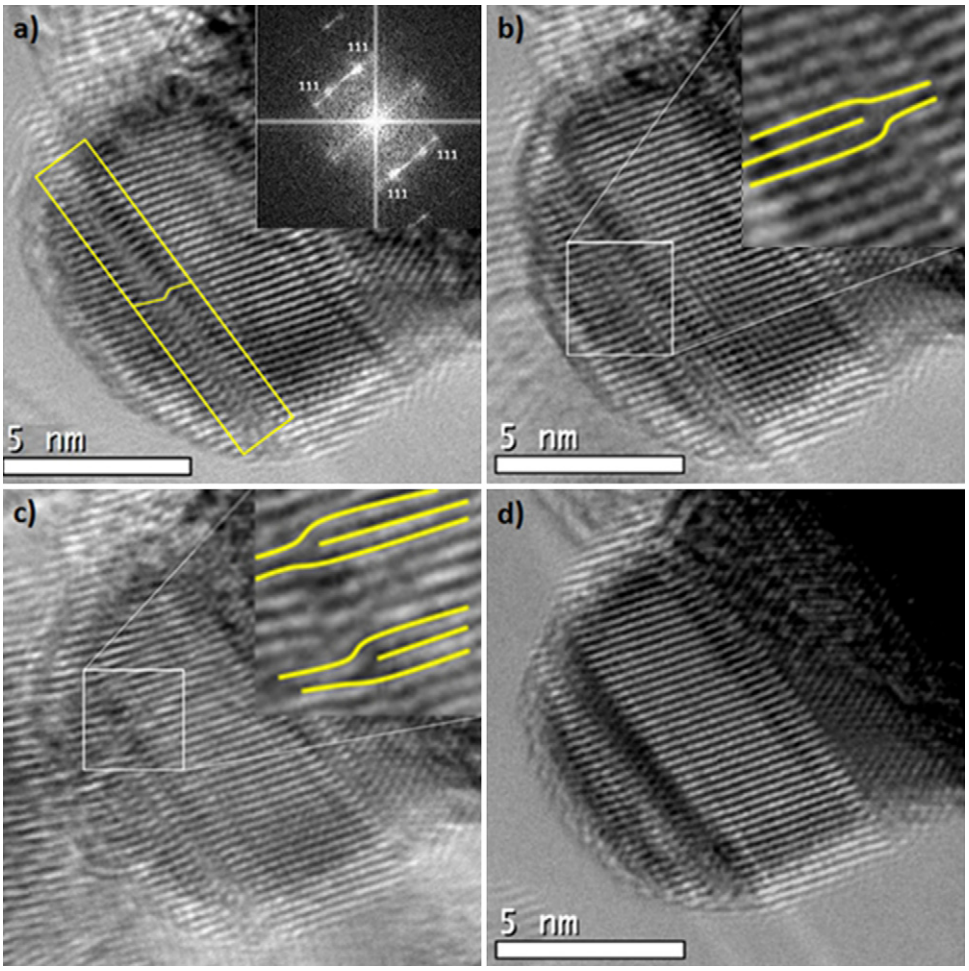

Fig. 3. (a) shows the nanoparticle before compression. A twin is highlighted in the image. (b) shows the beginning of the nanocompression experiment. An edge dislocation is highlighted in the inset. (c) shows the continuation of the nanocompression experiment. The inset shows two additional dislocations. (d) shows that no dislocation are observed after the indenter is removed.

interpretable as a dislocation. As the strain on the nanoparticle is increased, more dislocations are observed (Fig. 3c). Finally, when the indenter is removed dislocations can no longer be seen in the nanoparticles.

To ensure that the observed terminating planes were not artifacts caused by overlapping of the W probe and Ag nanoparticle, Fourier filtering using the {1 1 1} interplanar spacing of silver was performed (Fig. 4). In the filtered images, the fringes due to the indenter disappear, but the dislocations remain, indicating that the observed extra half planes are associated with the presence of dislocations.

Though the phase contrast in situ TEM nanoindentation experiment did not use load sensing capability, it is readily apparent that the general interpretation of whether dislocations are observed within nanoparticles is less complicated than the interpretation of the diffraction contrast images where exact tilting is required. Additionally, the lack of use load sensing capabilities is not a major drawback for in situ TEM nanoindentation of nanoparticles, as the exact contact area geometry is often difficult to define, which makes stress calculations very challenging. On the other hand, determining the Burgers vector of dislocations under phase contrast imaging is not trivial. Fig. 3 is a good example of this. The distortion due to the presence of the dislocation is observed on the $(1\,1\,\bar{1})$ planes, although the typical Burgers vector for a fcc metal, such as silver is $b = a/2\,[1\,1\,0]$. Therefore, the distortion seen on the $(1\,1\,\bar{1})$ planes is likely caused by the presence of a dislocation of type $a/2\,[1\,1\,0]$ on a {1 1 1} type plane not allowed by the zone axis under which the image was taken, and which is at an angle with the resolved $(1\,1\,\bar{1})$ lattice fringes. This is confirmed by the fact that the dislocations appear asymmetric in the TEM images.

5. Going forward

In the next few years, we are likely to see significant developments in in situ TEM nanoindentation of nanoparticles. First, with the advent of novel spherical and chromatic aberration-corrected TEMs, pole-piece gaps greater than 5 mm will be commonplace. This will allow in situ nanoindentation holders to become more sophisticated, possibly incorporating other means of stimulating the samples, such as heating, electrical field, magnetic field, liquids and gases. Furthermore, wider pole-piece gaps will allow more tilting capability, which is critical for certain types of experiments, particularly those involving diffraction contrast. Another key issue in tilting is the ability to accurately reach certain zone axes. In the near future, TEM goniometers will have the capability of automated tilting, while eucentric height correctors prevent the sample from straying away. As an alternative, diffraction-scanning transmission electron microscopy (D-STEM) (Ganesh et al., 2010) combined with precession microscopy (Vincent and Midgley, 1994) can be used to obtain an orientation map of an ensemble of nanoparticles, so that a specific nanoparticle with a particular orientation is quickly selected for observation, without

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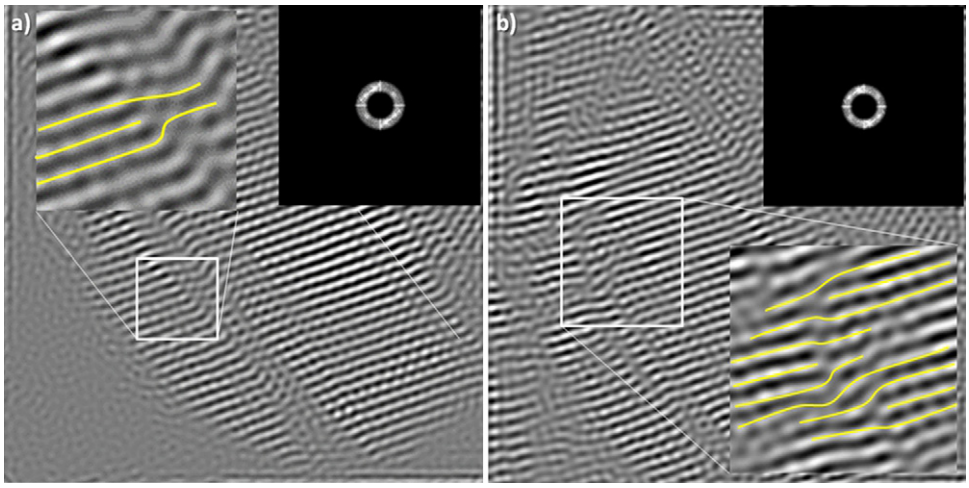

Fig. 4. Fourier filtering of Fig. 3b and c. The filtered images were made of only the periodic data corresponding to the interplanar spacing of the {1 1 1} type planes of silver. The dislocations are still clearly visible in the filtered images, while the lattice fringes from the tungsten probe are not resolved. This supports the conclusion that the observed extra half planes are associated with the presence of dislocations.

large amounts of tilting required. A crucial area for development in the field of in situ TEM nanoindentation is to be able to monitor the deformation processes within a wide range of strain rates. This would allow us to observe in real time dislocation nucleation events, as well as dislocation reactions. Several upcoming technologies are expected to have a strong impact in this area, namely advanced direct detection cameras capable of capturing 400 frames per second, dynamic transmission electron microscopy (D-TEM), which has the capability of nanoseconds time resolution, as well as the development of in situ holders with faster elongation rates.

6. Conclusions

In situ TEM nanoindentation is essential to fundamentally understand the mechanisms of deformation in individual nanoparticles. Both diffraction contrast and phase contrast imaging have advantages and disadvantages when used for in situ nanoindentation experiments. Provided accurate diffraction conditions can be achieved, diffraction contrast experiments can be very powerful in identifying the presence of dislocations, as well as their Burgers vector. However, as precise tilting is difficult, there is a myriad of artifacts than can resemble the contrast attributed to dislocations. In comparison, phase contrast imaging is relatively straightforward for identifying dislocations. Yet, the determination of Burgers vectors is not trivial.

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