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Three-Dimensional Imaging of Dislocations and Defects in Materials at Atomic Resolution Using Electron Tomography

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Dislocations and defects strongly influence many of the properties of materials, ranging from the strength of metals and alloys to the efficiency of light-emitting diodes and laser diodes (1). Presently there are several experimental methods to visualize dislocations. Transmission electron microscopy (TEM) has long been used to image dislocations in materials (2,3). A TEM image, however, represents a 2D projection of a 3D object. Using weak-beam dark-field and scanning TEM (STEM), electron tomography has been used to image 3D dislocations at a resolution of ~5 nm (4). Recently, we achieved 3D imaging of dislocations and defects in materials at atomic resolution with electron tomography (5,6). Electron tomography was originally developed in 1968, and has been primarily applied to determine the 3D structure of biological systems (7). Over the last decade, electron tomography has been increasingly applied in materials science and nanoscience through the use of STEM (4). Previously, the highest resolution achieved by STEM tomography was around 1 nm in three dimensions (4). However, improving the 3D resolution from ~1 nm to the atomic level remained a challenging task, which requires new tomographic reconstruction algorithms, better projection alignment methods, state-of-the-art STEM instruments, and more robust data-acquisition procedures.

Over the past few years, we have made important progress in achieving atomic resolution STEM tomography. We have developed a novel tomographic method, termed equally sloped tomography (EST) (8-10), which allows the 3D image reconstruction of a tilt series with a limited number projections and a “missing wedge” (*i.e.* specimens cannot usually be tilted beyond ±79°). We have also developed a center of mass method that can be used to align the projections of a tilt series with atomic level accuracy (5). Using an FEI Titan 80-300 S/TEM, we determined the 3D structure of a ~10 nm Au nanoparticle at 2.4 Å resolution (5). Individual atoms are observed in some regions of the particle and several grains are identified in three dimensions. The 3D surface morphology and internal lattice structure revealed are consistent with a distorted icosahedral multiply twinned particle. More recently, in combination of 3D Fourier filtering and STEM tomography, we observed nearly all the atoms in a multiply-twinned Pt nanoparticle (6). We discovered the existence of atomic steps at 3D twin boundaries of the Pt nanoparticle and, for the first time, imaged the 3D core structure of edge and screw dislocations in materials at atomic resolution. These dislocations and the atomic steps at the twin boundaries are hidden in conventional 2D projections, and appear to be a significant stress-relief mechanism. The ability to image 3D disordered structures such as dislocations at atomic resolution is expected to find application in materials sciences, nanoscience, solid state physics and chemistry.

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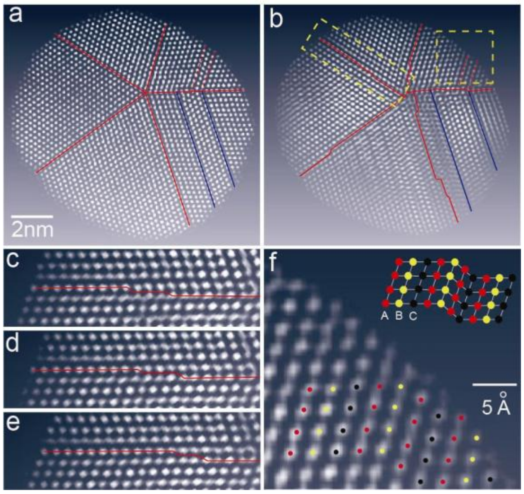

Figure 1. Grain boundary comparisons between a 2D experimental projection and several 2.6 Å thick internal slices of a Pt nanoparticle. **a**, Experimental projection in the XY plane suggesting twin boundaries (red lines) are flat. Blue lines show two subgrain boundaries. **b**, A 2.6 Å thick internal slice indicating the existence of atomic steps at the twin boundaries (red lines). The subgrain boundaries (blue lines) are widened by two lattice spacings relative to those in (**a**). **c**, Zoomed view of a twin boundary in (**b**). **d**, and **e**, a 2.6 Å thick slice above and below the slice of (**c**), revealing that the atomic steps vary in consecutive atomic layers. **f**, Zoomed view of a stacking fault in the 2.6 Å thick internal slice, which is in good agreement with the classical model for an fcc extrinsic stacking fault (inset). (Ref. 6)

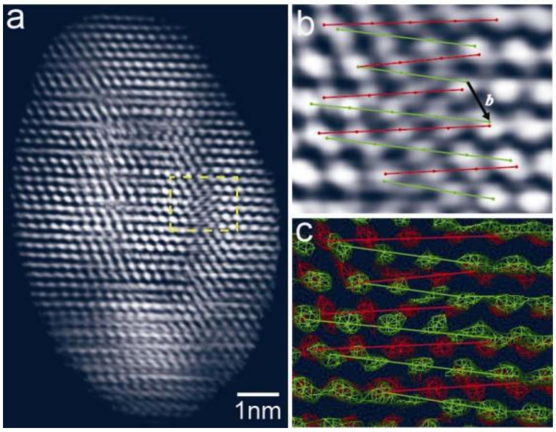

Figure 2. Observation of the 3D core structure of a screw dislocation in the Pt nanoparticle at atomic resolution. **a**, Volume renderings of a 5.3 Å thick slice (two atomic layers) in the direction. **b**, Zoomed view of a screw dislocation showing the zigzag pattern, a characteristic feature of a screw dislocation. **c**, Surface renderings of the screw dislocation where the atoms in green are on the top layer and those in red in the bottom layer. The zigzag pattern is more clearly visualized, the Burgers vector (***b***) of the screw dislocation was determined to be $1/2[01\bar{1}]$. (Ref. 6)