Focal-Series Reconstruction of Nanoparticle Exit-Surface Electron Wave

Michael A. O'Keefe*, E. Chris Nelson* and Lawrence F. Allardº

*Materials Science Division, LBNL B2R200, 1 Cyclotron Road, Berkeley, CA 94720, USA
ºHTML, ORNL, Building 4515, MS6064, 1 Bethel Valley Road, Oak Ridge, TN 37831-6064, USA

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Transmission electron microscopy (TEM) at sub-Ångstrom resolution is important for nano-technology. As researchers design and build artificially-structured nano-materials such as semi-conductor devices, ceramic coatings, and nanomachines that operate at the atomic level, the requirement for "seeing" what has been built becomes more crucial. Identifying atom positions requires appropriate resolution, i.e., the ability to separate distinct objects in images [1]. Heavy (metal) atoms are routinely imaged in TEM specimens at resolutions from 2Å to 1.5Å. Better resolution (near 1Å) is required to "see" lighter atoms such as carbon [2], nitrogen [3] and even lithium [4]. The one-Ångstrom microscope (OÅM) project [5] exceeds Scherzer resolution [6] to reach better than one-Ångstrom at 300keV by using a combination of a modified CM300FEG-UT with computer software [7,8] able to correct C_S and generate sub-Ångstrom images from experimental image series. The OÅM has demonstrated that a resolution of 0.78Å is possible with this technique [9].

The OÅM was designed to achieve sub-Ångstrom resolution in order to allow imaging of light atoms in the presence of heavy metal atoms as well as having the capability to image atom positions in and around defects in crystalline specimens. Because the OÅM uses reconstruction from focal series to produce the electron wave at the specimen exit surface, there has been concern that the method might be difficult to apply to non-periodic specimens such as nanoparticles. We have used the OÅM to image and apply focal-series reconstruction (FSR) of the exit-surface wave (ESW) to a 70Å particle of gold supported on amorphous carbon (fig.1).

We recorded 20 images at focus values ranging from –2600Å to –2144Å. These images were input to the TrueImage™ software from FEI [7,8] and the ESW computed. The phase of the complex ESW shows the positions of the atom columns in the specimen as white dots (fig.2), and its diffractogram shows it contains information to 1.23Å (fig.3). When the effect of the amorphous support was reduced by high-pass filtering, atom positions clearly showed a five-fold twinning (fig.4) that was not visible in the original image.

The result demonstrates that through-focal reconstruction of the ESW does not need large crystal expanses to work properly. Although [110] Au structures may not need sub-Å resolution to show all the useful structural details of the particle in this orientation, it is clear that focal reconstruction of the ESW can certainly improve original data that is much more difficult to interpret directly. We expect this technique to prove even more useful when applied to nanoparticles containing finer spacings than the 2.35Å separation of the 111 planes in the present gold nanoparticle [10].

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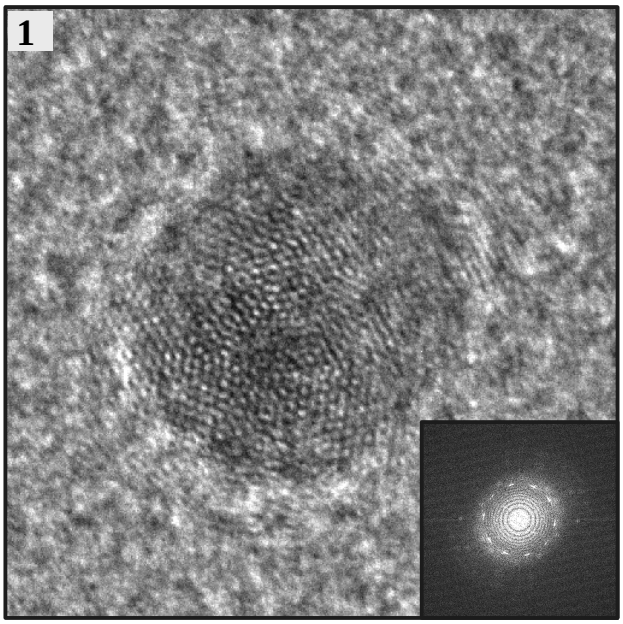
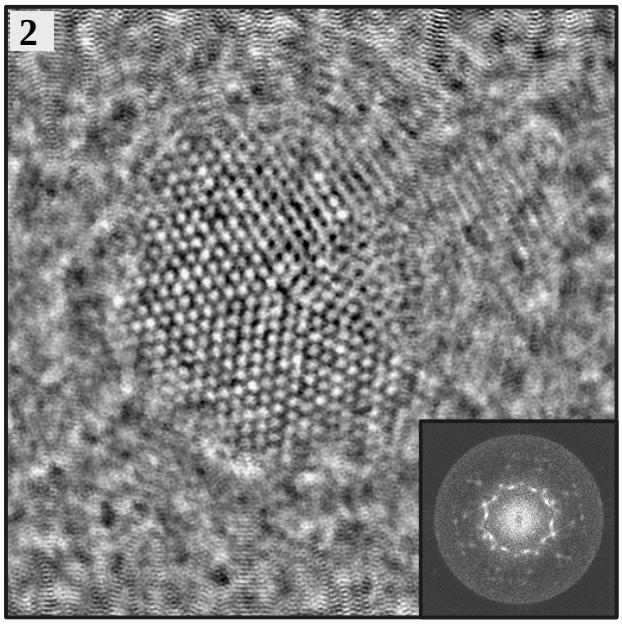
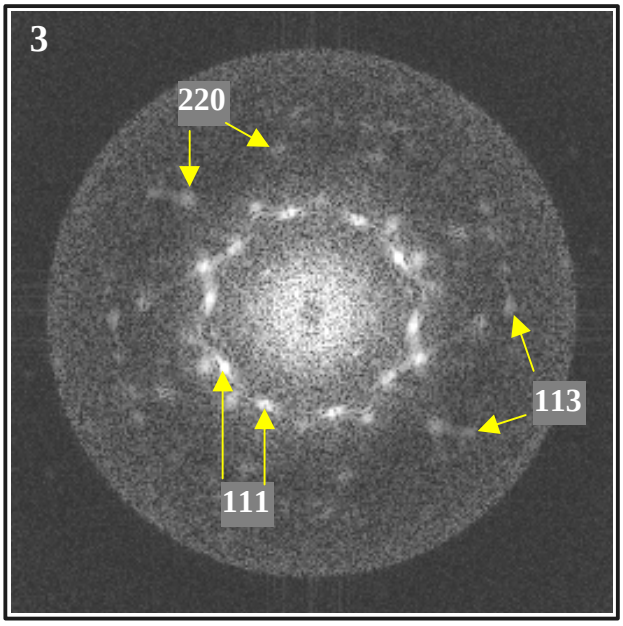
[10] Work supported by the Director, Office of Science -- through the Office of Basic Energy Sciences, Material Sciences Division, of the U.S. Department of Energy, under contract No. DE-AC03-76SF00098.







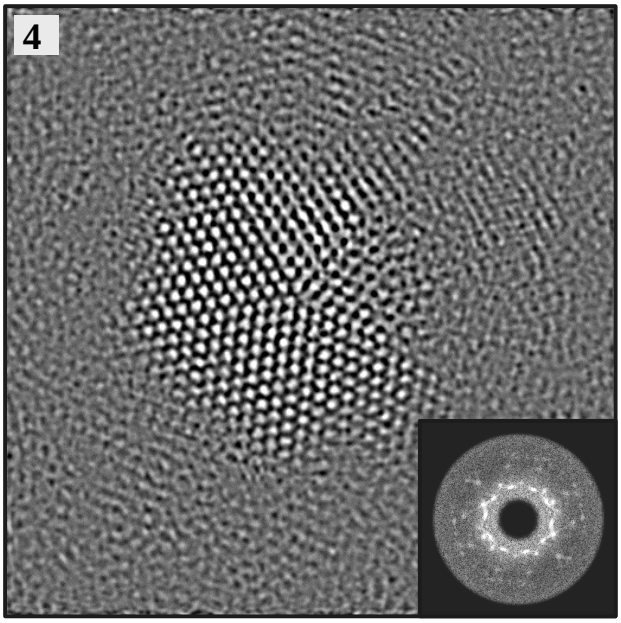


FIG. 1. Image of gold nanoparticle (approximately 70Å across) obtained on the OÅM. The inset diffractogram shows 111 spots at 2.35Å.

FIG. 2. Phase of the ESW reconstructed from 20 images shows atom column positions as white dots. The inset diffractogram shows the 111 spots with additional higher-order spots (see fig.3).

FIG. 3. Diffractogram from ESW phase shows strong 111 spots with weaker spots at 220 (1.44Å) and 311 (1.23Å). The 220 spots clearly demonstrate the (approximate) five-fold rotational symmetry. The cutoff at 0.9Å is due to the application of a 0.9Å limit during the reconstruction process.

FIG. 4. Annular high-pass filtering (inset) removes the low-frequency contributions coming from the amorphous carbon support to clearly reveal the five-fold twinning in the Au nanoparticle.