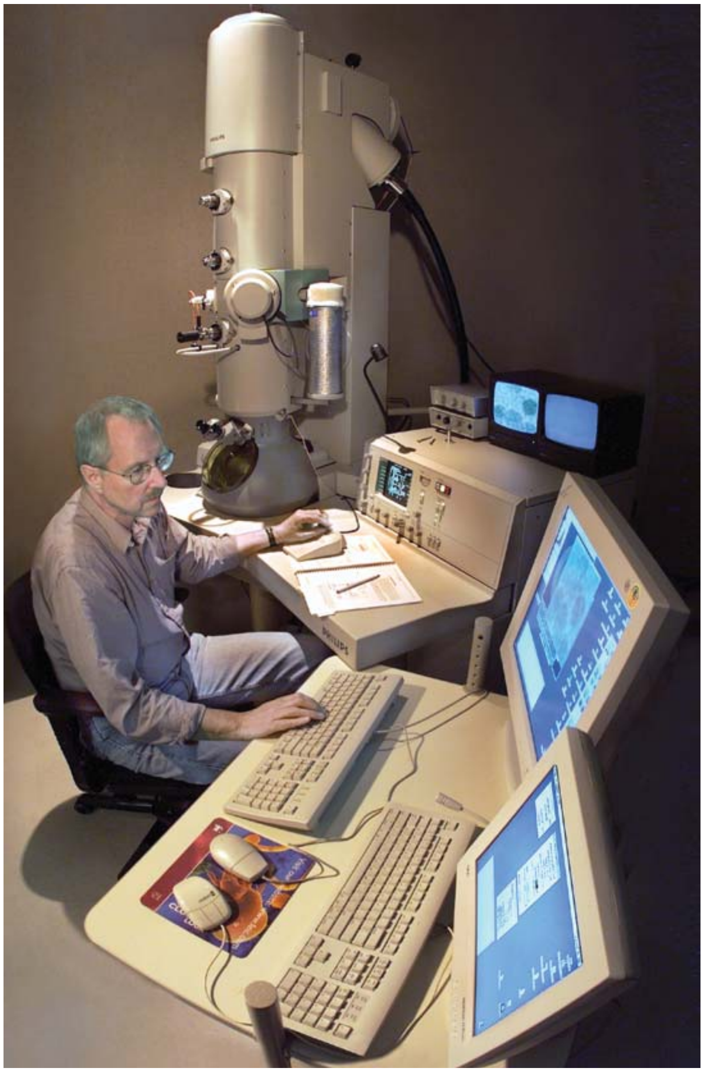
Sub-Ångstrom Resolution with a Mid-Voltage TEM

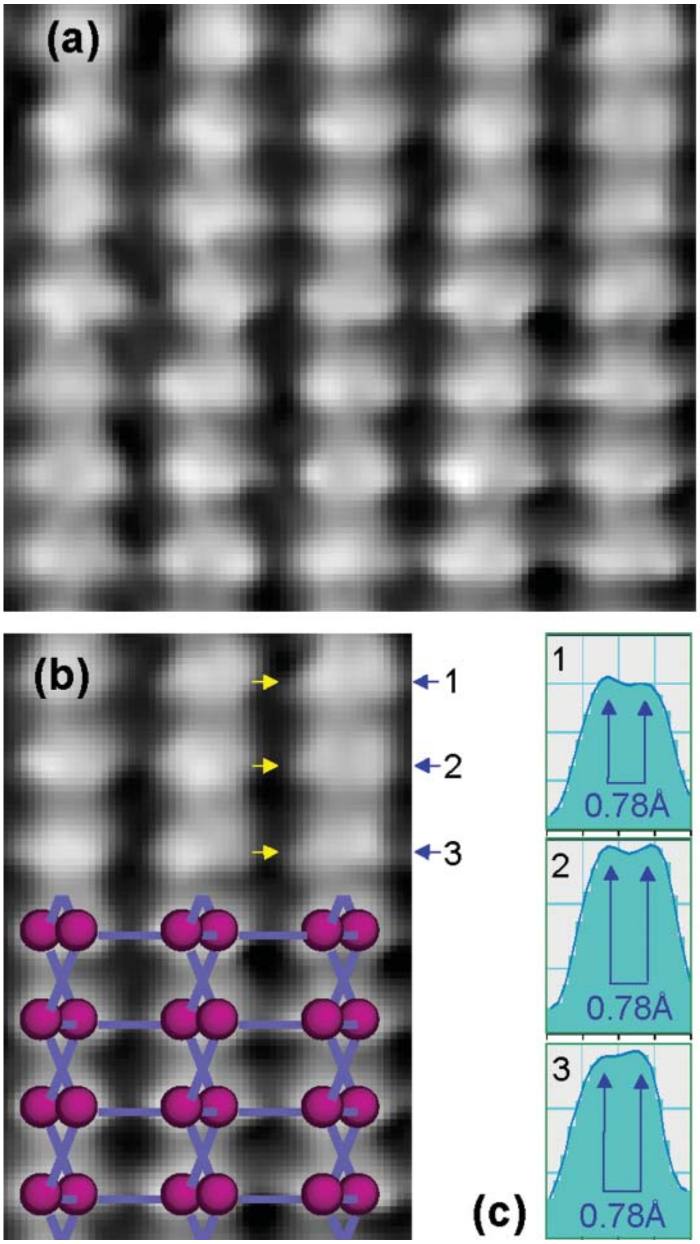
Michael A. O'Keefe, Crispin J.D. Hetherington‡ & E. Chris Nelson**
**NCEM & Materials Sciences Division, LBNL, Berkeley*
‡Current address: Department of Materials, Oxford University, UK
maokeefe@lbl.gov

The One-Ångstrom Microscope (OÅM) project was established at the NCEM to produce images at sub-Ångstrom resolution (O'Keefe, 1993). The project was implemented using a Philips CM300FEG/UT with hardware modifications designed to correct objective lens three-fold astigmatism and extend information transfer to 0.8 Ångstrom (O'Keefe *et al.*, 2001a). A Gatan image filter (GIF™) was used to bring the image magnification to more than three million times at the CCD camera to provide adequate real-space sampling.

Phase-contrast imaging in the HRTEM produces images with peaks at atom positions by extracting the spatial distribution of the relative phase from the electron wave. Usually, the electron wave is imaged by direct interference of diffracted beams at optimum focus (Scherzer, 1949). Instead, the OÅM uses the FEI TrueImage™ focal-series reconstruction software (Coene et al., 1996; Thust et al., 1996) to derive the relative electron phase from a series of images taken over a range of focus chosen to minimize the effects of partial spatial coherence (O'Keefe, 2001). The result of this focal-series reconstruction is to produce an experimental reconstruction of the electron wave at the specimen exit surface with a resolution that extends to the microscope information limit (O'Keefe, 1992). Spread of focus, Δ, determines the TEM information limit, d_Δ. For rms (root mean square) variations of $\sigma(I)$ and $\sigma(E)$ in lens current I and beam energy E, spread of focus is given by $\Delta = C_C\sqrt{[(\sigma^2(E)/E^2 + 4\sigma^2(I)/I^2]}$ where C_C is the chromatic aberration coefficient. Information limit is $d_\Delta = \sqrt{(\pi\lambda\Delta/2)}$, where λ is the electron wavelength. The energy spread, $\sigma(E)$, measured for the CM300FEG-OÅM is 0.85eV rms for the normal gun extraction voltage of 4kV, and can be lowered to 0.77eV at 3.5kV extraction voltage (O'Keefe *et al.*, 2002). Combined with the fractional lens current ripple of 0.3 ppm rms, and the C_C of 1.45 mm, these energy spreads make the OÅM information limit 0.78Å at 4kV extraction voltage and 0.75Å at 3.5kV.



The CM300FEG OÅM Microscope installed in the OÅM lab.



(a) OÅM image reconstructed from a focal series and displayed at 60 million times magnification. (b) With superimposed model of silicon in [112] orientation. (c) Profiles of three atom pairs with 0.784Å scale bars.

Careful design of the OÅM laboratory minimized the deleterious effects of vibration, electromagnetic noise and temperature fluctuations (Turner *et al.*, 1997). In addition, it was found necessary to eliminate ground loops between the components of the microscope and its imaging filter. Specimen drift was minimized by setting the temperature of the cooling water exiting the objective lens coil so it matched the room temperature of 23°C and thus minimized temperature differences between the specimen, the specimen holder and the specimen stage. Low drift is essential to ensure sufficient overlap in the images of the focal series and provide enough common area for processing. Since the field of view on the CCD camera at 3.2 million times magnification is 150Å across, and acquisition of a 20-member focal series of images takes two and one half minutes, any drift rate greater than 15 Å/min would render the data set useless. To achieve maximum resolution, no talking or gross movements are allowed during the image series acquisition. In addition, we found that sudden atmospheric pressure changes have a significant effect—opening or closing an external building door located 50 feet away is enough to ruin an acquisition series by introducing an abrupt change in defocus. An airlock system would alleviate this problem.

Tests using a silicon specimen tilted into [112] orientation showed that the OÅM is capable of achieving resolutions down to 0.78Å (O'Keefe *et al.*, 2001b). To produce this resolution, a series of 20 images was recorded, starting at an underfocus of -3800Å and stepping towards overfocus in 11Å increments. The image drift was measured at 20.5Å over 148 seconds, or about 8.3Å/min. The reconstructed OÅM image shows white "blobs' representing pairs of silicon atoms arranged in "dumbbells" (a). A model of silicon in [112] orientation shows how the atoms are separated by 0.78Å in projection and demonstrates how they fit with the OÅM image (b). Resolution is the ability to determine how many objects (atoms in our case) a "blob" represents. Profiles of three of the "blobs" (arrowed in b) show that image peaks corresponding to the atom columns can just be distinguished (c), exceeding the Sparrow (1916) resolution criterion but not quite meeting that of Rayleigh (1874), which requires the dip between the peaks to be at 81% of the peak height. The "pixelation" visible in the image occurs because it is recorded with a sampling interval of 0.15Å, so the horizontal period of 3.136Å (the silicon lattice parameter of 5.432Å divided by √3) is about 21 pixels, and the resolved atoms are separated by only 5.23 pixels. Using a higher magnification to provide finer sampling would provide insufficient area for reconstruction.

The environment created by the OÅM laboratory has proven adequate to allow the OÅM to reach its theoretical resolution of 0.8Å and to give the NCEM the capability to image columns of the lighter atoms, all the way down to lithium (Shao-Horn *et al.*, 2003; O'Keefe & Shao-Horn, 2004). Environments that are improved even more, such as those provided by the AML and Triebenberg laboratories, should prove more than adequate to allow the upcoming sub-Ångstrom TEMs and STEMs to reach their full potential. ■

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