Imaging Materials at Atomic Resolution in Transmission Electron Microscope

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Background

Humans have used lenses of various types to magnify objects since the middle ages, but microscopy in science really took off in the 17th century with the publication of Hooke’s Micrographia. The first microscopes, along with most in the world today, were optical, using visible light to form magnified images of objects. The numerous groundbreaking discoveries in physics of the late 19th and early 20th century paved the way for a new approach: electron microscopy, wherein samples are illuminated with beams of high-energy electrons, rather than with visible light.1 The resolving power of a microscope of any kind is defined as the minimum separation of two points that the microscope can image as distinct objects. For traditional optical ‘scopes, the resolving power is diffraction-limited by the wavelength (λ) of light used to illuminate the sample, and the numerical aperture (NA) of the objective lenses. In 1839, building on the work of George Airy and Ernst Abbe, the English physicist John William Strutt (AKA: 3rd Baron Rayleigh) proposed the Rayleigh Criterion to explain and calculate the resolution limit (R) of a diffraction-limited optical system (1). The shorter the λ, and the larger the NA, the smaller the R value (and therefore the greater the resolving power). The NA value in optical microscopes is limited to a maximum of 1.0 for dry objectives, and can go as high as 1.5 using oil immersion. But because the shortest wavelength of visible light is 400nm, even with oil immersion the R value will be larger than 200 nm. This was the approximate limit of optical microscopy before the advent of the electron microscope (although more recently developed techniques have subverted this limitation).2

The resolving power of electron microscopes is likewise limited by the wavelength of the illumination source, but because that source is a beam of fast-moving electrons, the wavelength in question is much, much smaller than that of visible light. For a particle like an electron, we can find the deBroglie wavelength using the formula (2).3 Scanning Electron Microscopes (SEM) use accelerating voltages from 5-30 kV, allowing far more resolution than traditional optical ‘scopes. In a Transmission Electron Microscope (TEM), such as the one we used (the MagLab’s JEM-ARM200F), the electrons are given even more energy (200 keV) attaining higher speeds, shorter wavelengths, and the smallest R values (3). Imaging a sample in the TEM requires electrons to pass through it. Detectors also collect and integrate those signals as well to improve resolution. This requires the sample material to be extremely thin. If the sample is more than approximately 500 nm thick, most of the incident electrons will be absorbed, rather than transmitted through the material to create an image.

Adding Scanning functionality to the microscope allows the electron beam to sweep across the surface of the sample, acquiring even more information. Known as Scanning Transmission Electron Microscopy (STEM), this requires the addition of scanning coils (similar to those found in an old CRT monitor) to direct the beam, and integrates even more electron detectors.5

Our main task for the summer was to create a technical document detailing the steps required to prepare thin samples for the TEM. We made several TEM samples from bulk Si and SrTiO3. The full version of that document spans some 20 pages of instructions and pictures. Below is a condensed version of this procedure.

1. Select a 3 mm specimen (Si or SrTiO3), trim it as needed, then install it into a disk polisher for the first side polishing with a silicon carbide paper. After initial polishing and flattening of the sample surface, remove the wax with acetone.
2. Affix the sample to a 3 mm copper ring using M-bond 610 glue under an optical microscope. Allow the glue to cure on a hot plate at 165˚C for over 2 hours.
3. Next, attach the sample with more melted wax to the tripod polisher and carefully level it on the flat plate of the grinder. Use the 15 µm, 9 µm and 1 µm diamond lapping films successively to polish material away.
4. While polishing to thin the sample, use the inverted optical microscope periodically to measure its thickness in different areas to ensure it remains level. Continue removing material until it reaches just 20 µm thick. Then, use acetone to dissolve the wax, freeing it from the tripod polisher.
5. Finally, install the sample in the ion mill for final thinning. Use both beams with energy set at 5.0 keV at a 7° angle to bombard the surface of the sample with Argon ions from above and below. The amount of time required depends on the material and its precise shape and thickness, but generally we begin with 1 hour of initial milling, followed by shorter periods as necessary. In between milling sessions, examine the sample through the mill’s video microscope until a little hole appears, indicating the sample is thin enough to be imaged by the TEM.

Results

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References


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