Interpreting Images from a Transmission Electron Microscope

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Abstract

As the technology of Electron Microscopy advances and spreads, images of single atoms and nano-sized structures will become more available. If the specimen is approximately amorphous and homogenous and the image contains a region with a hole, it may be possible to get first-order approximations of thickness and a range of Z-values for individual heavy atoms contained in the specimen by using simulation and simple scattering calculations.

Background

In some cases, Transmission Electron Microscope (TEM) images can be thought of as scattering experiments. A beam of electrons is accelerated towards a specimen and detectors record the amount of current at a given time. This current is represented by a pixel's intensity in the image and is affected by many physical processes. In the model described here, diffraction effects are ignored and the process that scatters the electrons is due to Coulomb repulsion.

To give a basic understanding of how the images are recorded, Figure 1 shows a representation of the detector geometry.

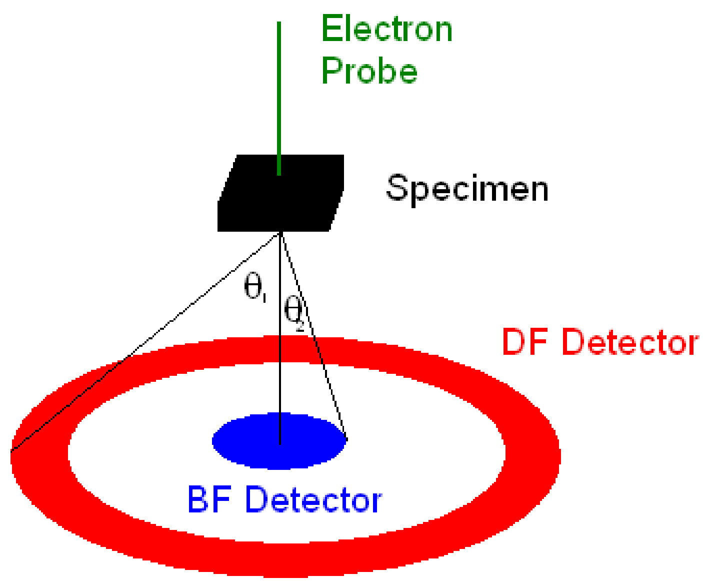


Figure 1

The beam is scanned across the specimen, called Scanning TEM (or STEM), and the detector current is recorded as a pixel intensity. If there is a small cluster of protons in the specimen, more electrons will be scattered out of the range of the Bright Field (BF) detector and into the Dark Field (DF) detector's range. In this way, the two independent images are complimentary. Further, since the images were taken simultaneously, one can assume that the same location on the images corresponds to the same region on the specimen.

Description of the Model

In the BF detector, less specimen leads to greater pixel intensity. If there is a region on the image where there is nothing for the beam to interact with, the value at this region can serve as a calibration of the incident beam intensity. A simple model to describe this situation is given by a mean free path equation:

$$I_{BF}/I_{BFo} = \mathrm{Exp}(-t/\tau_{BF}) \qquad \text{(Eqn. 1)}$$

where I_{BF} is the measured intensity, I_{BFo} the incident beam intensity, t the specimen thickness, and τ_{BF} the mean free path length. A mean free path length represents the average distance the electron has to travel through the specimen before it undergoes an event that scatters it outside the detector. As the thickness increases, the measured intensity I decreases, which agrees with intuition. This equation will be used to calculate the thickness of the specimen.

For the case of the DF detector, a similar model is given:

$$I_{DF}/I_{DFo} = 1 - \mathrm{Exp}(-t/\tau_{DF}). \qquad \text{(Eqn. 2)}$$

The variables represent similar quantities, but are different in value since the detectors are made of different materials and τ is dependent on the angle of scattering. The value for thickness in a certain region, however, must be equal regardless of the detector. This provides a connection between the two images.

After thickness data is calculated, the dependence of scattering on the Z value of the atom might be useful in helping identify the atom. With help of simulation, the amount of scattering is found to go as $Z^{1.7}$. By defining the quantity, f_H, which is a constant that gives scattered intensity per proton and comes from simulation, one can calculate the predicted scattering of a single atom with the equation:

$$I_{DFatom}/I_{DFo} = f_Z = f_H\, Z^{1.7} \qquad \text{(Eqn. 3)}$$

This can be solved to give an approximation for Z.

Requirements to Apply the Model

To apply this model, certain requirements must be fulfilled. (1) It is important that the images be taken simultaneously so that the calculated thickness from the BF image corresponds to the same point on the specimen in the DF image. Without this connection, I_{DFo} cannot be calculated. (2) The specimen has to be approximately amorphous so that diffraction effects are minimized. The wavelike behavior of the electrons is neglected, so the specimen must approximate this simplification. (3) There must be a "hole" in the specimen so that the incident intensity for the BF detector can be calibrated. This also serves the purpose of approximating the noise level in the DF detector since this region should ideally be completely dark in the image. (4) The specimen has to primarily consist of a homogenous, low-Z material, such as carbon, to provide consistent contrast with the heavier atoms.

Applying the Analysis to Approximate Thickness

Assuming our specimen meets the requirements, the next step is to obtain a mean free path. Using the Kirkland[1] routines, STEM images were simulated once a list of atom positions, Z-values, and microscope parameters were given. I approximated amorphous carbon by randomly assigning the atoms an (x,y,z) coordinate with the requirement that no atom was within 0.2 nm of another. I approximated the carbon density as about 1.7 g/cc and included ten Zirconium atoms to show contrast of high-Z material. I then simulated the ion-milling thinning process by removing all atoms above a certain line, giving an overall "V" shape. Figure 2 shows the simulated dark field and bright field images, respectively.

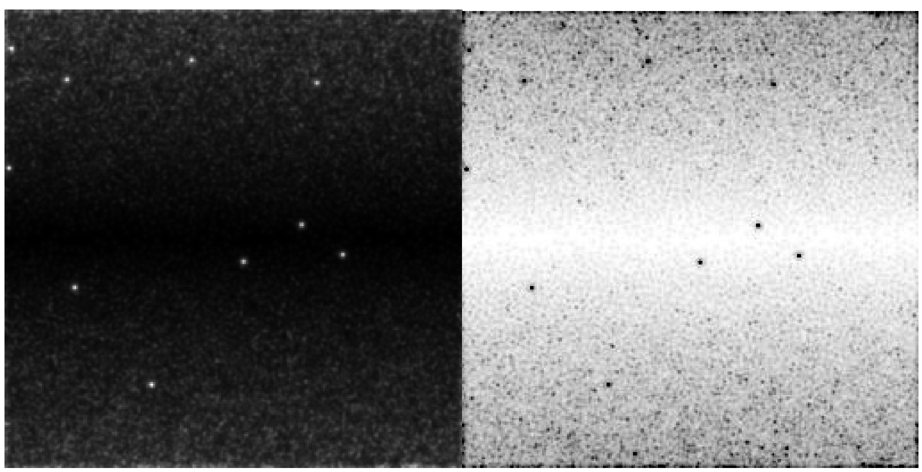


Figure 2

Since the box dimensions are defined and the slope from the simulated milling process is known, the approximate thickness at any given region is known. The incident intensity is normalized to one for each image, so detector efficiency is not an issue. Using Eqn. (1) and (2), an expression for the mean free paths can be found and several data points can be taken.

Using the ImageJ software, one can draw a horizontal line at a given thickness and measure the average pixel value. Using this method, the following results were obtained:

	tau_DF	tau_BF
Avg	1.46E-04	1.53E-06
Stdev	6.02E-06	1.53E-07

Table 1

` Once an approximate mean free path length has been found for the specimen, it can be used to calculate thickness in an experimental BF image, like Figure 3.

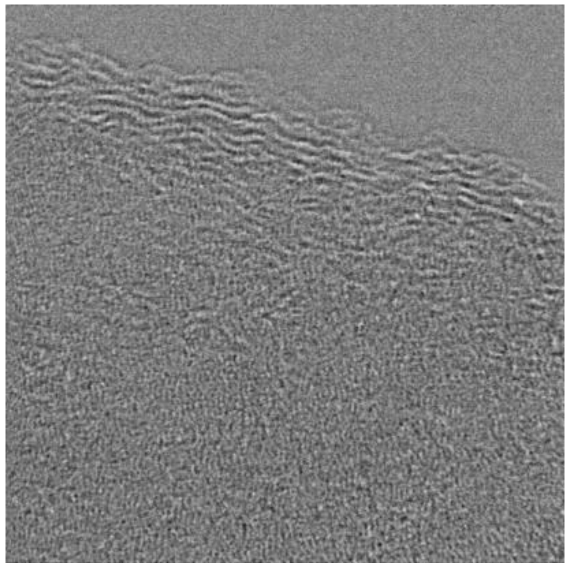


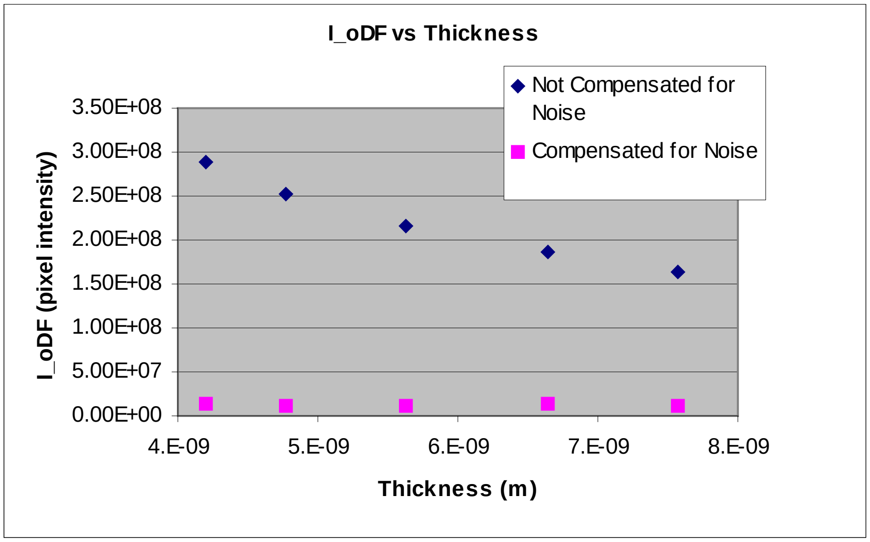
Figure 3

This BF image came from a carbon pre-solar grain from the meteorite Murchison (see Figure 5 for the DF image) and was taken at Oak Ridge National Labs. The STEM microscope used has sub-Angstrom resolution and is capable of resolving single atoms. The upper-right corner is a hole and is a measure of the incident beam intensity. The horizontal lines under the hole, which is likely the thinnest region, shows the "onion layers" of the graphite sheets. In this region, diffraction plays a substantial roll and can lead to pixel values that are higher than that of the hole. To help compensate for this problem, it is useful to blur the image. Applying this method to Figure 3, the thickness is largest at the bottom of the image at about 7 nm and decreases in the expected wedge-pattern to about 4 nm. Though the accuracy of this calculation is not currently known, the values are reasonable.

Calculating I_{DFo}

Since the BF and DF detectors cannot be assumed to have the same efficiency, and the incident beam intensity cannot be directly measured in the case of the DF detector, it must be calculated. To achieve this, Equation (2) can be used. I_{DF} is measured, τ_{DF} was found through simulation, and the thickness is inferred from the BF image.

When this was attempted, it would found that if a measure of average noise (found from average intensity of hole in DF image) was not subtracted from each intensity average, the I_{DFo} value was unstable. Graph 1 shows the results.



Graph 1

The noise-corrected data led to a value of 1.2E+07 within about 8%.

Calculating f_H

A simulated image was used to calibrate $f_{H,}$ which is the scale factor that describes the amount of scattering per Z. Using a simulated DF image of the same carbon specimen as before, but with a variation of heavy atom types (Fig. 4), Equation (1.3) can be used to get a value for f_H. Instead of using average intensities for this

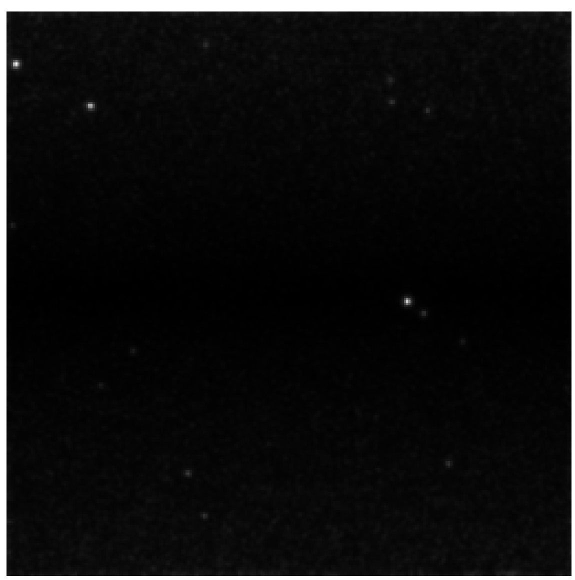


Figure 4

calculation, the maximum intensity in the region of the atom was used. To obtain the intensity due to the atom alone, an average value of intensity due to the carbon in this region was subtracted out. The remaining value is a measure of the amount of scattering due to the single atom. This analysis led to an f_H value of about 2.3E-07 within about 35%, which is our most unstable value.

Calculating Z in an Experimental Image

Using the method of getting the maximum pixel value in the region of the atom and subtracting out the average background intensity, I_{DFatom} from Equation (3) is obtained. By solving this equation for Z, and using the f_H found from simulation, it is possible to obtain the approximate number of protons in the atom, which identifies the element. Since f_H was shown to vary significantly, the maximum and minimum values can be used to give a Z-value range. This analysis was done on Figure 5 and led to typical Z values between about 18-30. Though this method has not yet been checked on images with known elements, the fact that the predictions are between 6 and 92 shows potential.

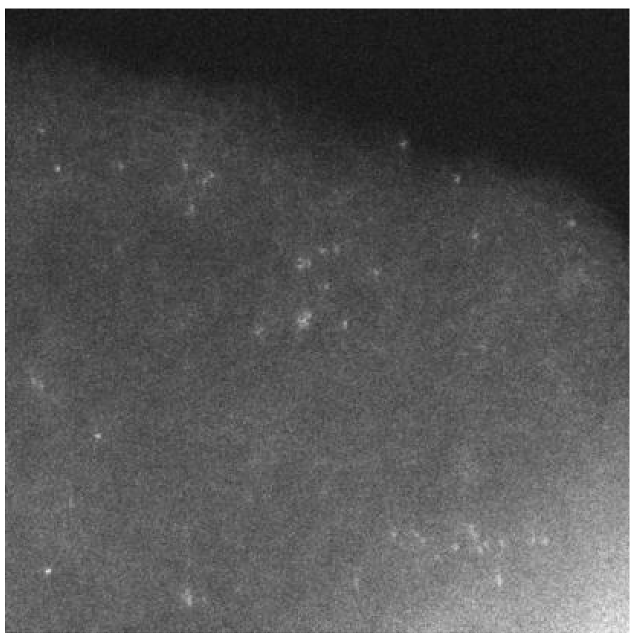


Figure 5

Obtaining τ_{BF} Without Use of Simulation

Ludwig Reimer's[2] work on modeling scattering phenomenon in the context of TEM work provides a way of calculating the mean free path length without the need to build a model specimen and simulate images. His equation for calculating a cross-section is

$$\sigma_{el} = Z^2 R^2 \lambda (1+E/E_o)^2 / [\pi a_H^2 (1+\alpha_o^2/\theta_o^2)] \qquad \text{(Eqn. 4)}$$

where σ_{el} is the elastic cross-section, R is a constant scattering factor, λ is the wavelength of the electron, E and E_o are the energy and rest energy of the electron, respectively, a_H is the Bohr radius, θ_o is a constant called the characteristic angle, and α_o is the half-angle of the exterior of the BF detector, which corresponds to θ_2 in Figure 1. The mean free path length can be found from this quantity by

$$\tau_{BF} = A/(N_A \rho \sigma_{el}) \qquad \text{(Eqn. 5)}$$

where A is the atomic weight of the material, ρ the density, and N_A Avogadro's number. By plugging in the known quantities and using Equations 4 and 5, the resulting expression is

$$\tau_{BF}\,(\alpha_o) = (0.346\ \mu\text{m})\,(1 + 8660\alpha_o^2). \qquad \text{(Eqn. 6)}$$

For the simulated image, the BF detector was defined to have an outside half-angle of 0.02 rad. Using Equation (6), τ_{BF} = 1.54 μm. The value found from simulation is about 1.53 μm.

Conclusions and Future Explorations

The reasonableness of the values given by this model justifies a more rigorous analysis of the accuracy of its predictions and the situations in which it could be legitimately applied. The method could be applied to images that contain internal standards of thickness or Z-values to help quantify its limitations. In the case of the specimen in Figure 5, the Z-value predictions could be compared to elemental abundances of the environment that the pre-solar grain was formed, for example.

To make the method more useful, an expression similar to Equation (6) that led to accurate values of an effective mean free path length for the more complex geometry of the DF detector would help eliminate the need to build a computer model of the specimen and run microscope-imaging simulations.

Acknowledgements

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References

1. Kirkland, E.J. *Advanced Computing in Electron Microscopy*. Plenum Press, New York and London (1988).
2. Reimer, L. *Transmission Electron Microscopy: Physics of Image Formation and Microanalysis.* Spring-Verlag, New York (1997).