NANO 703/703L
Lab: TEM Diffraction Calibration
Due: Following Lab Session

BACKGROUND

We previously demonstrated that the magnification in the plane of the CCD is proportional to that on the viewing screen. The calibration constant *C* is equivalent to the size of a CCD pixel projected upward onto the plane of the viewing screen. This constant is valid whenever the final image diverges from the crossover below the projector lens.

In diffraction mode, the excitation of the intermediate lens is reduced, so that the projector lens (*PL*) projects the diffraction pattern formed in the back focal plane of the objective lens (*OL*), rather than the sample image. Comparable excitation of the projector lens is used in imaging and diffraction mode, so *C* remains unchanged, but can be expressed with units appropriate for diffraction.

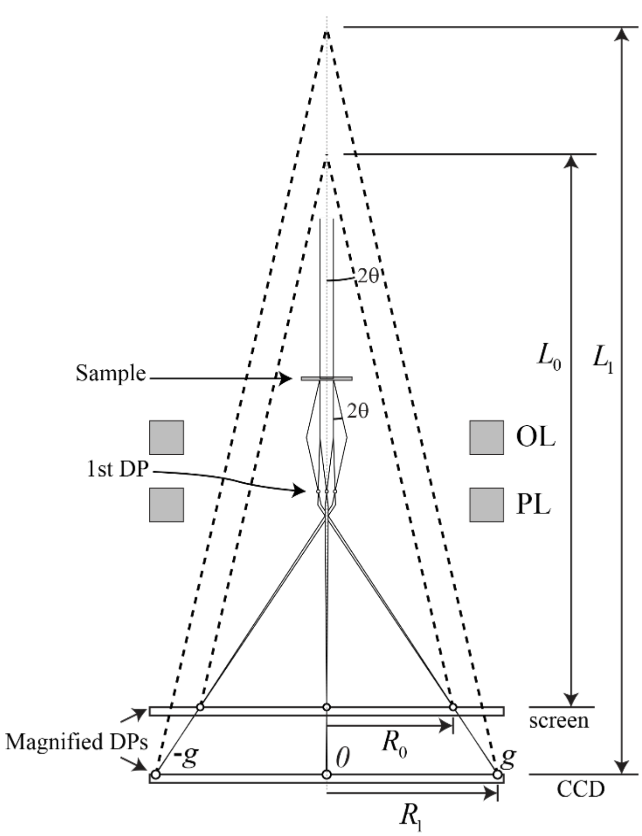


Fig.: Simplified ray diagram showing the camera lengths for the screen and CCD.

Diffraction causes electrons to deviate from the direction of the incident, or direct, beam, labeled *0*. A particular set of lattice planes, with spacing *d*, identified by its Miller indices $(hk\ell)$, using an electron wavelength λ, gives rise to a diffracted beam (reflection) *g* at an angle $2\theta_B$ from the incident beam direction. Bragg's Law states:

$$2\sin\theta_B = \frac{\lambda}{d}$$

In a traditional diffraction experiment, conducted without the use of electron lenses, a pattern is observed at a camera length *L* from the sample, producing diffracted beams that impinge on film or a detector at a radius *R* from the direct beam. The diffraction pattern scales with *L*, which allows a simple geometric analysis of the diffraction pattern.

In the TEM, notice that the vertical position of the sample does not move when we adjust the camera length. Rather, the lenses magnify the pattern by expanding the angles made by the various diffracted beams from the optic axis. Thus, *L* refers to the point from which the diffraction spots appear to diverge, but does not represent a physical distance in the TEM. In fact, *L* can vary from as little as 20 cm to as large as 100 m.

The electron wavelength in vacuum is a function of the incident beam energy. In high-energy TEM, λ is almost always much less than the *d*-spacing of the observed reflections. Thus, we can write

$$2\sin\theta_B \approx 2\tan\theta_B \approx \tan(2\theta_B) = \frac{R}{L}$$

We have derived a useful relationship:

$$Rd = \lambda L \qquad (1)$$

Scattering from a single crystal produces a discrete set of diffracted beams. Using parallel illumination, this gives rise to sharp spots in the diffraction pattern. The symmetry of the pattern is directly related to the crystal orientation.

For a powder sample, with many randomly oriented crystallites, continuous diffraction rings are observed, and the pattern has essentially no dependence on sample orientation. In either case, the diffracted spots or rings are labeled by their Miller indices as $hk\ell$ (without parentheses).

<u>Magnification Factor</u>

The geometric description we developed for imaging on the CCD is also valid for diffraction. Just as in imaging mode, the depth-of-focus of a diffraction pattern is quite large, so the focus of a pattern on the viewing screen is almost identical to that in the plane of the CCD, and the scale of the pattern on the CCD is proportional to that on the viewing screen.

For a diffracted beam incident at a screen radius $R_0$, the corresponding radius $R_1$ on the CCD is:

$$R_1 = F \cdot R_0$$

where $F$ is the same magnification factor previously measured in imaging mode.

Similarly, for an indicated camera length of $L_0$, the camera length on the CCD is $L_1$, where

$$L_1 = F \cdot L_0$$

Thus, the indicated camera length $L_0$ can be used to calibrate diffraction patterns on the CCD.

<u>Diffraction Constant</u>

The radius $R_1$ on the CCD of a diffraction ring will span a number of pixels *n*, such that

$$R_1 = n \cdot P$$

where *P* is the effective pixel size. We have

$$n \cdot P \cdot d = \lambda \cdot L_1 = \lambda \cdot F \cdot L_0$$

We can express this proportionality as:

$$n \cdot d = \left(\frac{1}{C}\right) \cdot \lambda \cdot L_0 \qquad (2)$$

The product $\lambda \cdot L_0$ is called the camera constant. Useful units for $1/C$ are:

$$\left[\frac{1}{C}\right] = \frac{\text{pix}}{\text{mm}}$$

Note that $C$ is the same quantity we determined by image calibration—the size of a CCD pixel projected onto the viewing screen.

For example, the *d*-spacing of lattice planes measured from a diffraction ring of radius *n* (in pix) at indicated camera length $L_0$ is:

$$d = \left(\frac{1}{C}\right) \cdot \frac{\lambda \cdot L_0}{n}$$

GOAL

In this experiment, we will use an evaporated aluminum standard, which has a broad distribution of of diffraction rings with known *d*-spacings provided by the manufacturer.

PROCEDURE

1) Create a table of measurements, as shown below:

| *E* (KeV) | $L_0$ (mm) | $1/d$ (nm$^{-1}$) | *n* (pix) | $\lambda$ (nm) | $\lambda \cdot L_0$ $(\text{nm} \cdot \text{mm})$ | $n \cdot d$ $(\text{pix} \cdot \text{nm})$ |
|---|---|---|---|---|---|---|
| . | . | . | . | . | . | . |

2) Repeat the following procedure for various microscope settings:

a) Select a beam energy *E* (in KeV). Compute $\lambda$ (in nm).

b) Select a camera length $L_0$ and record (in mm).

c) Measure $n$ (in pix) for various values of $1/d$ (in $\text{nm}^{-1}$). Record the results in the table.

<u>Tips:</u>
It is often difficult to determine the center of the diffraction rings. For best accuracy, one should measure the ring diameters 2*n* whenever possible.

At larger camera lengths, only a portion of a ring may be detectable in a single CCD exposure. In these cases, one can measure the difference in radius between two rings 1 and 2, taking the difference

$$\frac{1}{d} = \frac{1}{d_1} - \frac{1}{d_2},$$

where ring 2 has the smaller diameter.

d) Save the diffraction patterns and transfer to the network drive.

REPORT
Using the data in step 3, perform the following:
1) Plot $n \cdot d$ vs. $\lambda \cdot L_0$ and fit the curve to the function $y = a \cdot x$. Record the slope $a = 1/C$.

2) Record $1/C$ in units of $\text{pix}/\text{mm}$.

3) Evaluate *C*. Convert *C* to units of $\mu\text{m}/\text{pix}$. Compare to the value from the TEM Magnification Calibration Lab.

Submit a lab report describing the experimental procedure, which contains all raw data and result.