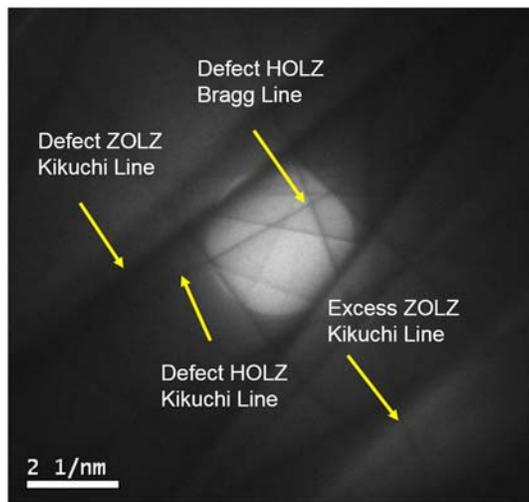


18. Using CBED

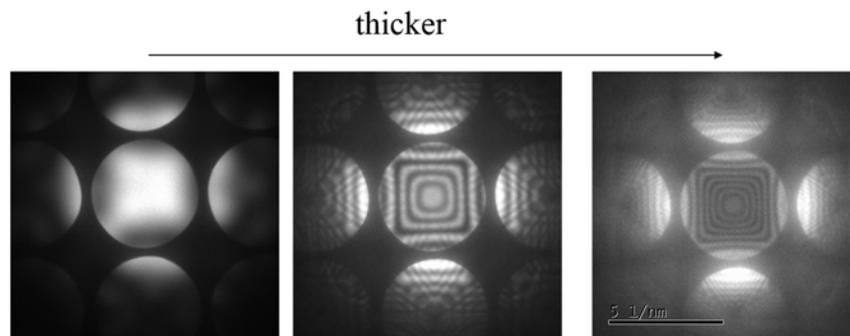
CBED features

Common features in a CBED pattern can be seen in the example below. Excess and defect ZOLZ Kikuchi lines are fairly strong and broad. (Defect) HOLZ (Bragg) lines and defect HOLZ Kikuchi lines are relatively sharp.



Influence of thickness

The K-M fringe oscillation frequency generally increases with thickness. Beyond some thickness, though, there is so much diffuse scattering, they get a bit hard to see.



Intensity oscillation in a CBED disk (I)

We can get a sense of where the K-M fringes arise from our analysis of the variation in intensity with excitation error in a two beam condition. There we found

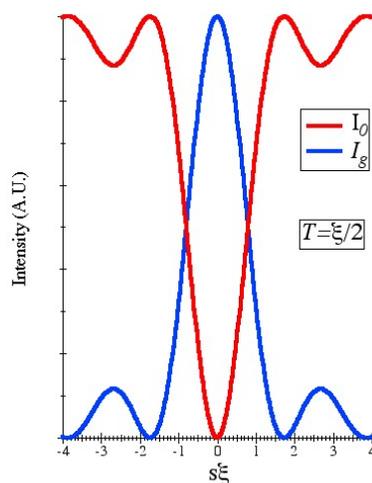
$$I_g = \left[\frac{\sin(\pi s_{eff} T)}{s_{eff} \xi} \right]^2$$

where

$$s_{eff} \equiv \sqrt{s^2 + \left(\frac{1}{\xi} \right)^2}$$

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The intensity profile depends on excitation error s , extinction distance ξ , and thickness T . This should be related to the intensity in the DF disk, where a line through the center of the disk is at the Bragg condition.



According to the same model, the BF intensity should be

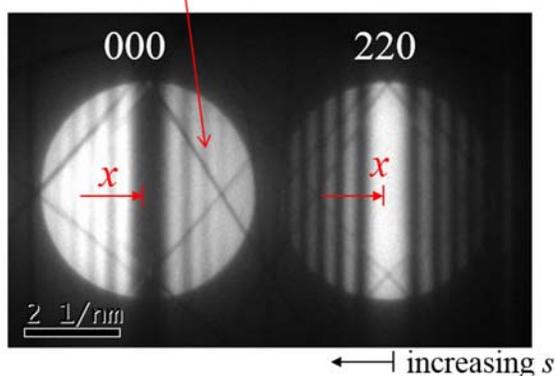
$$I_0 = 1 - \left[\frac{\sin(\pi s_{eff} T)}{s_{eff} \xi} \right]^2$$

Let's see if this holds.

Intensity oscillation in a CBED disk (I)

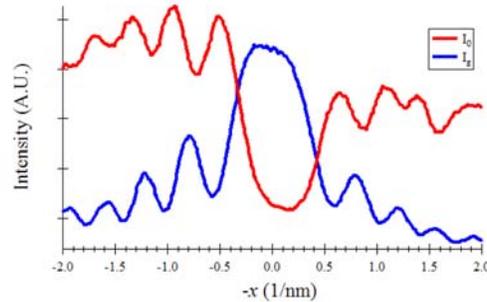
The oscillations in near a 220 two-beam condition in the 000 (BF) and 220 (DF) disks can be seen below.

bright-field disk shows
"anomalous absorption"



Using Kikuchi diffraction, we found that $s = g \cdot x/k$, where the quantity x was measured outward from a diffraction spot to the excess Kikuchi line. The excess line indicates where the crystal would satisfy the Bragg condition if the beam tilt were changed. In CBED, for a two-beam condition, this corresponds to the bright, central band through the disk, perpendicular to g . So, to again relate s to x , we need to

measure from the center of the band. So s changes from positive to negative as we move outward radially through the disk.



The only major difference with the preceding theory is that half of the BF disk looks darker than the other half. This is due to absorption effects, sometimes called “anomalous absorption” which we will now investigate.

Absorption

Absorption in this context, refers to incoherent and inelastic scattering that attenuate the coherent electrons. Mathematically, it manifests itself as an imaginary term in the crystal potential $\Phi(\mathbf{r})$. Say for example, we just have a uniform potential that is complex:

$$\Phi(\mathbf{r}) = \Phi_0 + i\Phi'_0$$

Where Φ_0 and Φ'_0 are both real. Then the crystal structure function is

$$U(\mathbf{r}) = U_0 + iU'_0$$

Where U_0 and U'_0 are also both real. Now the electron wave equation is

$$[\nabla^2 + 4\pi^2(k^2 + U_0 + iU'_0)]\psi(\mathbf{r}) = 0$$

The solution is now an attenuated plane wave

$$\psi(\mathbf{r}) = e^{2\pi i(\mathbf{K} + i\mathbf{K}') \cdot \mathbf{r}} = e^{2\pi i\mathbf{K} \cdot \mathbf{r}} e^{-2\pi\mathbf{K}' \cdot \mathbf{r}}$$

If we put this back in to the wave equation

$$\nabla^2\psi(\mathbf{r}) = (K^2 - K'^2 + 2iKK')\psi(\mathbf{r})$$

We get conditions on K and K'

$$K^2 - K'^2 = k^2 + U_0 \text{ and } 2KK' = U'_0$$

Then we can figure out K and K' , etc.

Absorption in a crystal

In a crystal, the potential with absorption could be written $\Phi(\mathbf{r}) + i\Phi'(\mathbf{r})$, and the crystal structure function is $U(\mathbf{r}) + iU'(\mathbf{r})$. Now the eigenvalue operator for our wave function is $\tilde{A} + i\tilde{A}'$, where \tilde{A} and \tilde{A}' are Hermitian matrices. As you recall, the diagonal elements of \tilde{A} are s_g , and the off-diagonal

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elements are $1/2\xi_{g_2-g_1}$. The elements of \tilde{A}' are $1/2\xi'_{g_2-g_1}$. (Notice that the diagonal elements of \tilde{A}' are all $1/2\xi'_0$.) We should now have eigenvalues that are complex:

$$(\tilde{A} + i\tilde{A}')|\psi^{(j)}\rangle = (\gamma^{(j)} + i\gamma'^{(j)})|\psi^{(j)}\rangle$$

Then our Bloch waves will have a damping term

$$\psi^{(j)}(\mathbf{r}) \rightarrow \left\{ \sum_{\mathbf{g}} C_{\mathbf{g}}^{(j)} e^{2\pi i \mathbf{g} \cdot \mathbf{r}} \right\} e^{2\pi i \mathbf{k} \cdot \mathbf{r}} e^{2\pi i \gamma^{(j)} z} e^{-2\pi \gamma'^{(j)} z}$$

The only problem is, this is a difficult problem to solve. Since $\tilde{A} + i\tilde{A}'$ is non-Hermitian, it can be very hard to diagonalize.

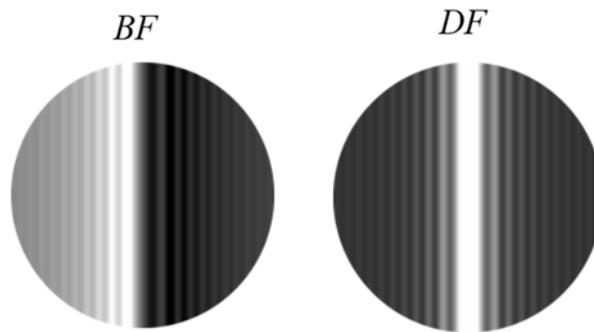
Treat absorption as a perturbation

The trick is to treat absorption as a perturbation. We typically expect $\Phi' \ll \Phi$. A common assumption is that $\Phi'_g \approx (0.10)\Phi_g$, that will at least give us a qualitative sense. In that case, we just solve the Bloch waves first, while ignoring absorption. Then we apply first-order perturbation theory to estimate the imaginary (damping) terms.

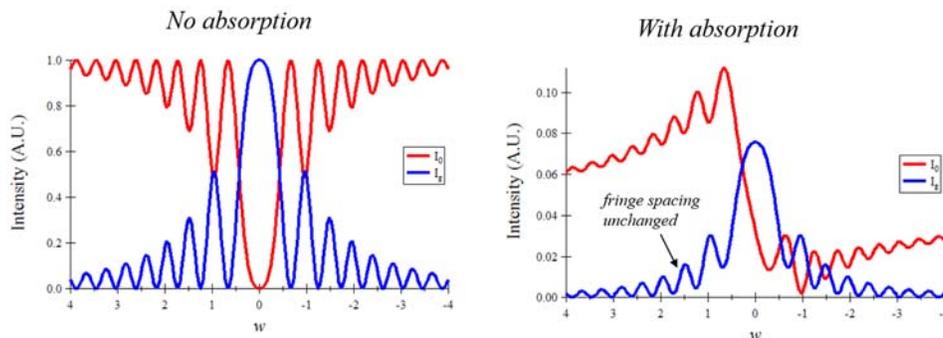
$$\gamma'^{(j)} \approx \langle \psi^{(j)} | \tilde{A}' | \psi^{(j)} \rangle = \sum_{\mathbf{g}, \mathbf{g}'} [C_{\mathbf{g}}^{(j)}]^* \cdot \left(\frac{1}{2\xi'_{\mathbf{g}-\mathbf{g}'}} \right) \cdot C_{\mathbf{g}'}^{(j)}$$

Two-beam simulation

A two-beam simulation using these assumptions is shown below. It gets the main features of the experimental CBED pattern.



Fortunately, the period of oscillation in the DF disk is not changed by absorption. So as long as we don't care about the absolute intensity, we can still back out s , ξ , and T .



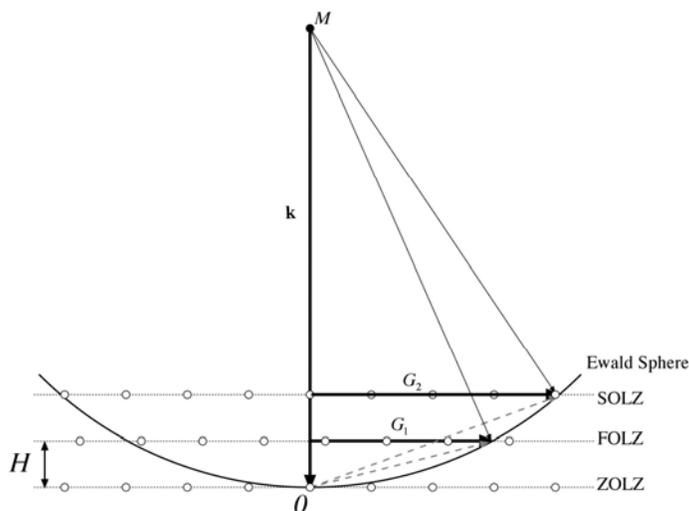
HOLZ ring radius

A simple construction can be used to find the reciprocal-space radius G_N of the n^{th} HOLZ ring. If the displacement from the ZOLZ is nH , we must have

$$k = \sqrt{(k - NH)^2 + G_N^2}$$

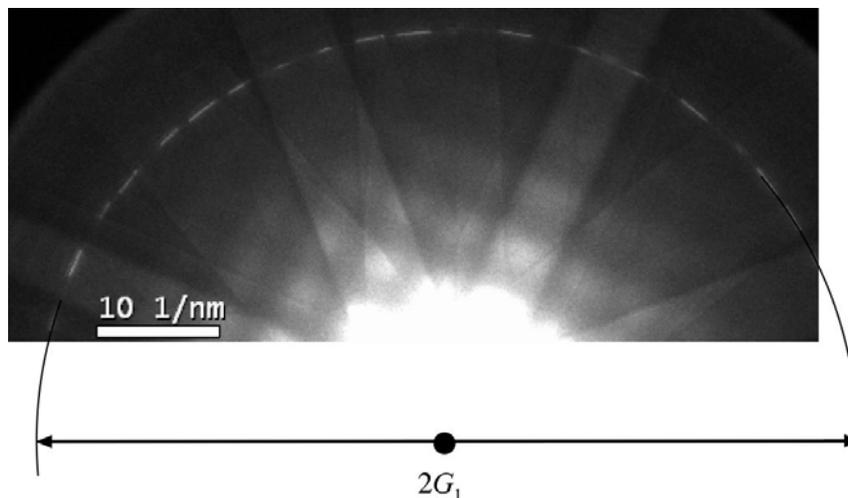
We can solve for the radius G_N and approximate

$$G_N = NH \sqrt{\frac{2k}{NH} - 1} \approx \sqrt{2kNH}$$



Find H from HOLZ ring diameter

We may measure a HOLZ ring radius G_N and want to calculate H . First measure the diameter $2G_N$, as shown below for a pattern from Si taken on the $[001]$ zone at 125 KeV.

**Find H**

Now solve for H :

$$NH = \frac{1}{\lambda} - \sqrt{\frac{1}{\lambda^2} - G_N^2} \left[\approx \frac{\lambda(G_N)^2}{2} \right]$$

Here are the numbers in this example.

$$2G_N = 67.9 \text{ nm}^{-1} \rightarrow G_N = 34.0 \text{ nm}^{-1} \rightarrow NH = 1.89 \text{ nm}^{-1}$$

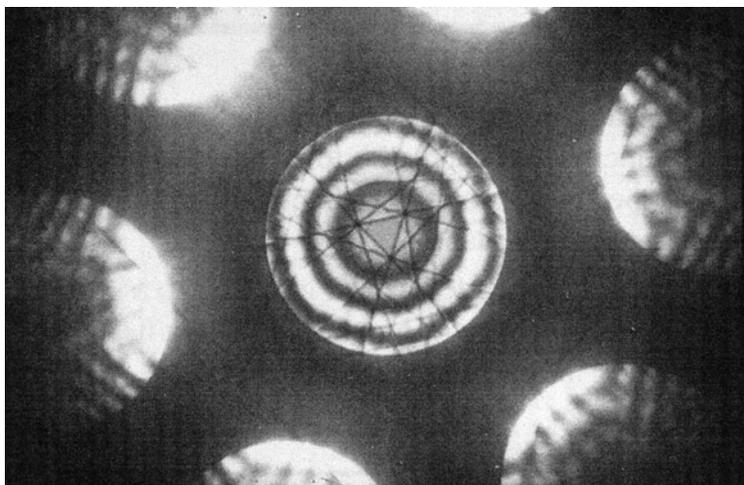
We can relate H to the lattice parameter in the z direction. We found earlier that $H = 1/|\mathbf{r}_{uvw}|$ for any structure. For cubic [001], $|\mathbf{r}_{uvw}| = 1/a$. For fcc, we know the FOLZ has $N = 1$, so $H = 1/a$.

Putting the numbers in, I get $a = 0.53 \text{ nm}$. The accepted value for Si is $a = 0.54 \text{ nm}$.

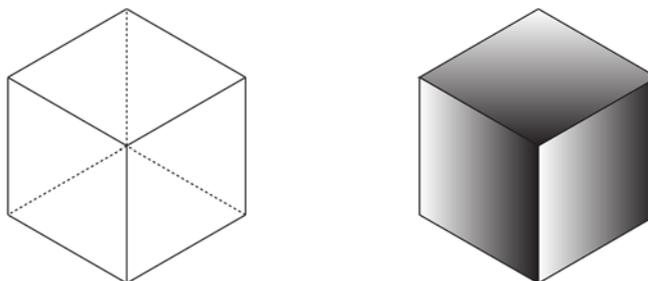
Symmetry in CBED patterns

The symmetry of CBED patterns can be used to solve directly the crystal structure of a material, particularly to classify the crystal by its crystallographic space group. The symmetry of the whole-pattern is called the “whole-pattern” symmetry. It could be either a K or a K-M pattern.

The details in K-M patterns are also useful. The details within the BF disk provide the BF symmetry. Those in a reflection can be used to find the DF symmetry.

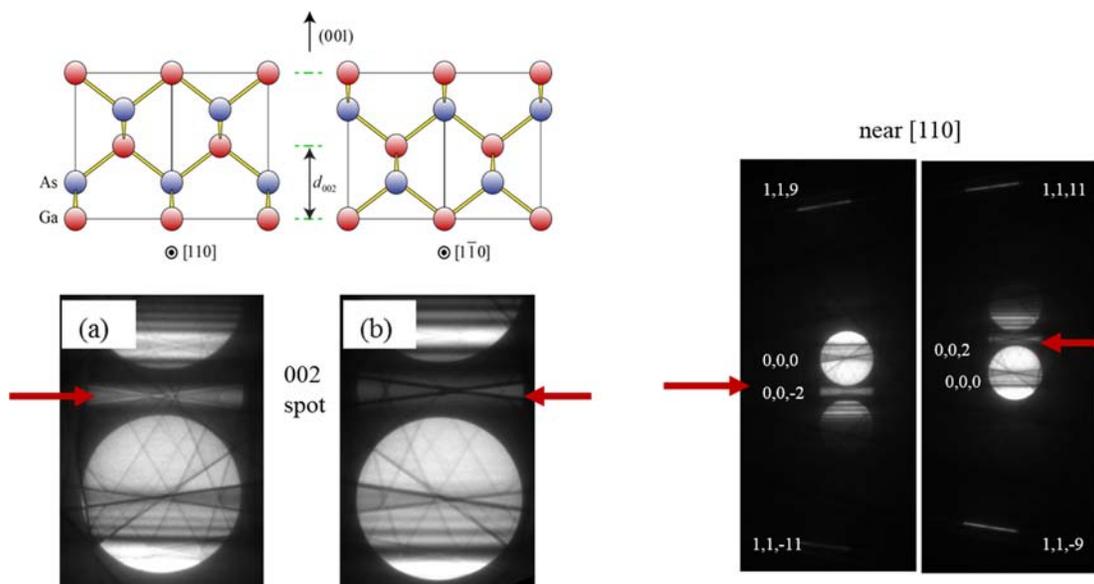


A popular example is the BF symmetry of Si in the $[111]$ orientation. Whereas the ZOLZ has six-fold symmetry, the BF disk features a central triangle of HOLZ lines with only three-fold symmetry. Imagine projecting a cube along its diagonal onto a plane. The edges of the projection would outline a hexagon and have six-fold symmetry. But if we look at the 3-D cube itself in the same direction, we see only three faces; the other three are on the opposite side. If we rotate the cube about this axis by 60° , the shadow is unchanged. But we have to rotate the cube by 120° for it to end up in an equivalent orientation to when we started. So, looking at the $[111]$ BF pattern, we now know that this zone is along a three-fold rotation symmetry axis, which is consistent with a cubic crystal, not six-fold, which would be consistent with a hexagonal crystal.



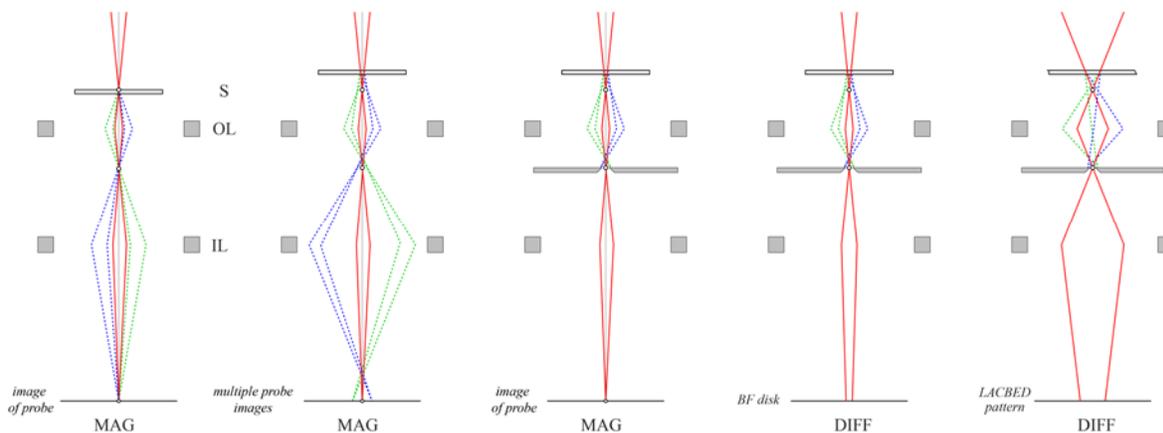
Polarity determination

Another use of CBED is determination of polarity of a non-centrosymmetric crystal. GaAs has the zincblende structure. The $\langle 110 \rangle$ faces normal to an $[001]$ surface with the bonds oriented in two possible ways. We can label these $[110]$ and $[1\bar{1}0]$. Setting up a four-beam condition that couples either $11\bar{9}$ and $(1,1,\bar{1})$ to $00\bar{2}$, or $11\bar{9}$ and $(1,1,1)$ to 002 , dynamical diffraction gives rise to either a bright cross or a dark cross in the location of the 002 spot depending on which axis we are looking along.



Large-angle CBED

The LACBED method was developed by Tanaka. In imaging (MAG) mode, start with the probe focused on the sample, like in CBED. Then raise the sample using the z control. The probe splits into several spots that look like a diffraction pattern. Put the diffraction aperture around the direct beam to block out the others. Then switch to diffraction mode. Nothing exciting yet, but then take out the condenser aperture. (Sometimes you need to remove a screw put in to prevent you from doing this.) You will see a CBED pattern that looks like the BF disk, only with enormous convergence angle α .



There is lots of information in the LACBED patterns, but it doesn't all come from one spot, as it does in normal CBED. That's because the probe is not focused on the sample. If you move the sample around, you will notice that it is a pattern superposed on an image. There are some tricks to get around this using computer automation, but none of them are easy as the method just described.

