Chapter 3
Basic Crystallography and Electron Diffraction from Crystals

Lecture 15

CHEM 793, 2008 Fall
Announcement

Midterm Exam: Oct. 22, Wednesday, 2:30 – 4:30
Close note, and bring your calculator
Outline

• Kikuchi Line and its indexing
• Double diffraction
• CBED pattern (convergent beam electron diffraction)
• Conventional high energy electron diffraction relies on elastic scattering. However, in a thick enough specimen, inelastic scattering will also take place. Inelastically scattered electrons travel in all directions but their distribution peaks in a forward direction, as shown in Figure (a). Note that in reality, the scattering is occurring in 3 dimensions.
• More electrons are scattered forward than sideways. This contributes a grey background around the central spot of the diffraction pattern, as shown in Figure (b).
(1). Electrons which have been inelastically scattered can subsequently be diffracted, but only if they are now traveling at the Bragg angle, $\theta_B$ to a set of planes, \{hkl\}.  

(2). Two sets of electrons will be able to do this - those at (+ $\theta_B$) and those at (- $\theta_B$), as seen in Fig. (a).

(3). This diffraction results in intensity changes in the background. Because there are more electrons at A than B (since electrons passing through A are closer to the incident direction than those through B) one bright line is developed (the excess line) together with one dark line (the deficit line).

(4). Because the electrons are inelastically scattered in all directions, the diffracted electrons will form a cone, called Kossel cone, not a beam. Hence we observe Kikuchi lines - not Kikuchi spots, as seen in Fig. (b).

(5) The spacing of the pair of Kikuchi lines is the same as the spacing of the diffracted spots from the same plane. However, the position of the lines is very sensitively controlled by the orientation of the specimen and Kikuchi lines are often used to set the orientation of a crystal in the TEM to an accuracy of 0.01 degrees.
(6) Kikuchi (1928) described this phenomenon before the development of the TEM; it can occur in any crystalline specimen. (7). Kikuchi lines are useful for precise determination of specimen in a TEM. When we tilt the specimen, we tilt its reciprocal lattice. Tilts of the reciprocal lattice with respect to a stationary Ewald Sphere do not cause any substantial changes in the positions of the diffraction spots, but the individual spots grow or fade in intensity. (8). The positions the Kikuchi lines are extremely sensitive to the tilt of the specimen. During a tilt, the Kikuchi lines move as if they are affixed to the bottom of the crystal. With a long camera length typical for diffraction work, there is significant movement of the Kikuchi lines on the viewing screen.
This simulation shows Kikuchi lines moving in relation to the diffracted spots when the crystal is tilted through small angles.
Kikuchi line images
Some Kikuchi line micrographs for silicon are shown.

(a) The Kikuchi lines pass straight through the transmitted and diffracted spots. The diffracting planes are therefore tilted at exactly the Bragg angle to the optic axis.

- The ideal specimen thickness will be such that we can see both the spot pattern and the Kikuchi lines as seen in Fig. (a). This is one of the few situations when thinner is not necessarily better. In most cases, the specimen is the thinner and the better.
(b) The crystal has now been titled **slightly away** from the Bragg angle, so that the Kikuchi lines no longer pass through the transmitted and diffracted spots.
(c). The crystal is tilted so that **more than one set of planes** are diffracting. Each set of diffracting planes has its own pair of Kikuchi lines.
Indexing Kikuchi Lines

The separation between the two Kikuchi lines is the same as the separation between the (hkl) diffraction spot.

(a) Only Diffraction Spot Pattern

(b) Diffraction Spot Pattern with Kikuchi Lines

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Indexing Kikuchi Lines

The separation between the two Kikuchi lines is the same as the separation between the (hkl) diffraction spot and the (000) spot.

We can index the Kikuchi lines by measuring their separations in much the same ways as we index diffraction spots. Consider two different pairs of Kikuchi lines from the planes (h1k1l1) and (h2k2l2). The separations between their pairs of excess and deficit lines, p1 and p2, are in the ratio:

\[ \frac{p_1}{p_2} = \frac{\sqrt{h_1^2 + k_1^2 + l_1^2}}{\sqrt{h_2^2 + k_2^2 + l_2^2}} \]

Figure shows ratios of \(\sqrt{32}\) and \(\sqrt{8}\) for indexed (440) and (220)

Diffraction Spot Pattern with Kikuchi Lines
Indexing Kikuchi Lines

The angles between intersecting Kikuchi line pairs are the same as the angles between their corresponding diffraction spots, at least so long as the Kikuchi line are not too far from the center of the view screen. These angles are helpful for indexing Kikuchi lines in the same way that the angles between pairs of diffraction spots are useful for indexing diffraction patterns. For example the angle, $\Phi$, between the (220) and (400) Kikuchi line in left figure is:

$$\Phi = \arccos \left( \frac{1}{\sqrt{8}} [220] \cdot \frac{1}{\sqrt{16}} [400] \right)$$

$$= 45^\circ$$

Diffraction Spot Pattern with Kikuchi Lines

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Indexing Kikuchi Lines and constructing Kikuchi lines

For a crystal oriented precisely on a zone axis, we can generate an indexed Kikuchi line pattern from its indexed diffraction. Each (hkl) Kikuchi line is drawn perpendicularly to the line between the (000) and (hkl) diffraction spots, bisecting this line. The procedure is shown in the figure.

Kikuchi line, (-400), bisects the line between (000) and (-400)

Kikuchi line, (400), bisects the line between (000) and (400)

Diffraction Spot Pattern with Kikuchi Lines
Specimen Orientation and Deviation parameter (s)
The positions the Kikuchi lines are extremely sensitive to the tilt of the specimen. During a tilt, the Kikuchi lines moves as if they are affixed to the bottom of the crystal. With a long camera length typical for diffraction work, there is significant movement of the Kikuchi lines on the viewing screen. The Kikuchi lines can be used to determine the sign and magnitude of the deviation parameter, s, which quantifies how accurately the Laue condition is satisfied.

\[ s = \frac{g^2 x}{k r} = \frac{x}{r} g^2 \lambda \]

- x is the distance between the diffraction spots and its corresponding bright Kikuchi line (E-line)
- r is the distance between the (000) and (hkl) diffraction spots.
- \( \lambda \) is the wavelength
- the unit of s is Å\(^{-1}\) or nm\(^{-1}\)
The sign of $s$

- $s$ points from the Ewald sphere to the reciprocal lattice point.

- For Kikuchi line, $s=0$, when the Kikuchi line runs exactly through its corresponding diffraction spots.

- $s<0$ if the excess line lies inside its corresponding diffraction spot $g$. In this case the reciprocal lattice point lies outside the Ewald sphere.

- $s>0$ if the excess line lies outside its corresponding diffraction spot $g$. In this case the reciprocal lattice point lies inside the Ewald sphere.
Δk = g + s

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g + s + g

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Δk = g + s

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s_g = 0

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s_g < 0

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s_g > 0

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\( x < 0 \)