Physics 0551 Lecture #6

Title: Experimental Methods for Diffraction Studies

The following discussion focuses on methods primarily used for x-ray diffraction. However, these techniques are *universal* in their application.

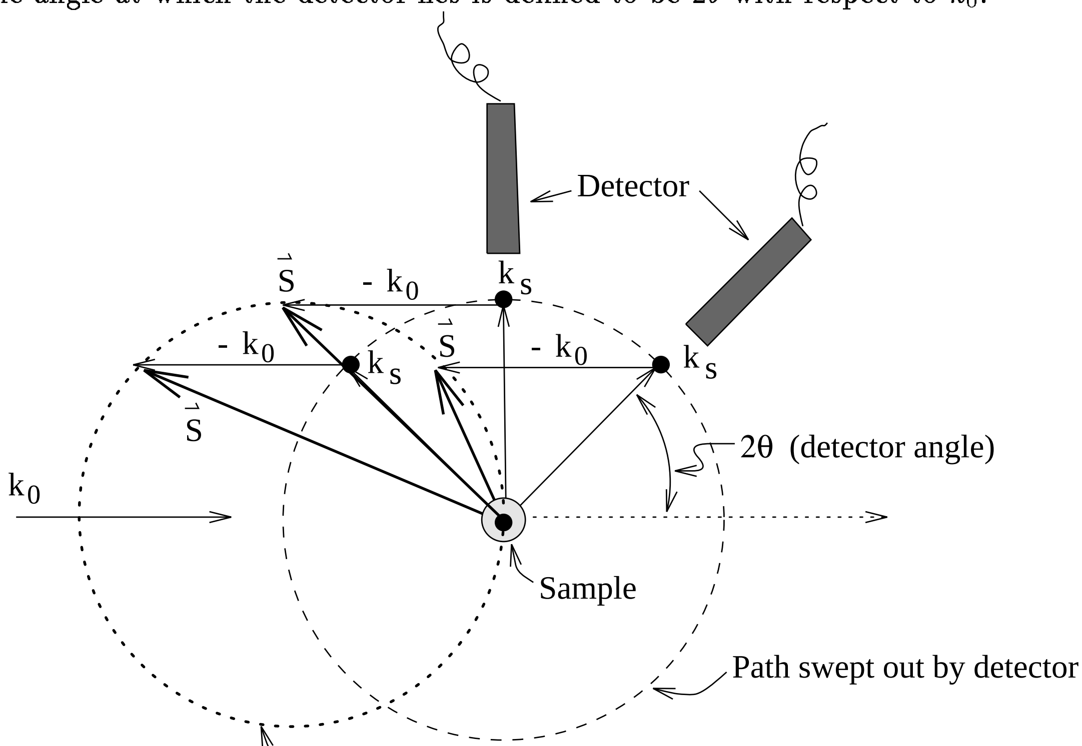
In order to perform an experiment on a real sample, it is fist necessary to study the significance of placing a point detector in real-space. Assume the following:

1 - An incident x-ray beam as depicted below

2 - A sample S located at the origin which scatters the x-ray beam only weakly. (No multiple scattering).

3 - A detector with a point-like aperture which can rotate in the plane of the paper and with its rotation axis through the sample.

4 - The angle at which the detector lies is defined to be 2θ with respect to $\hat{k}_0$.



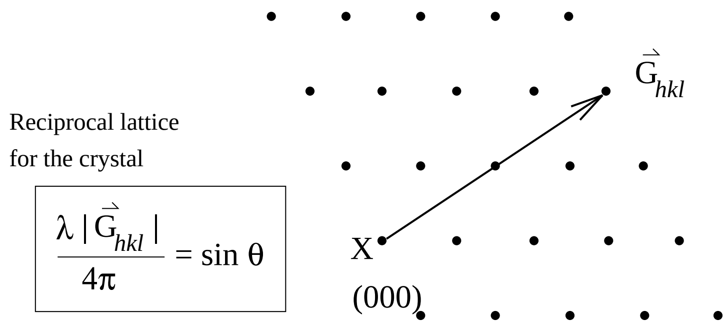
Notice that all diffraction vectors lie on a circle of diameter $2|\vec{k}_0|$ with lengths that vary from 0 to $2|\vec{k}_0|$. By choosing an angle 2θ, **both** the direction and magnitude of $\vec{k}_d$ are determined.

Now allow the detector to move out of the plane of the paper. Thus the detector will sweep out of sphere of diameter $2|\vec{k}_0|$! This sphere is called the Ewald sphere and represents all of the accessible diffraction vectors. By choosing the wavelength of the incident beam, the size of the Ewald sphere can be adjusted. There is an inverse relationship between the size of the Ewald sphere and the incident beam wavelength.

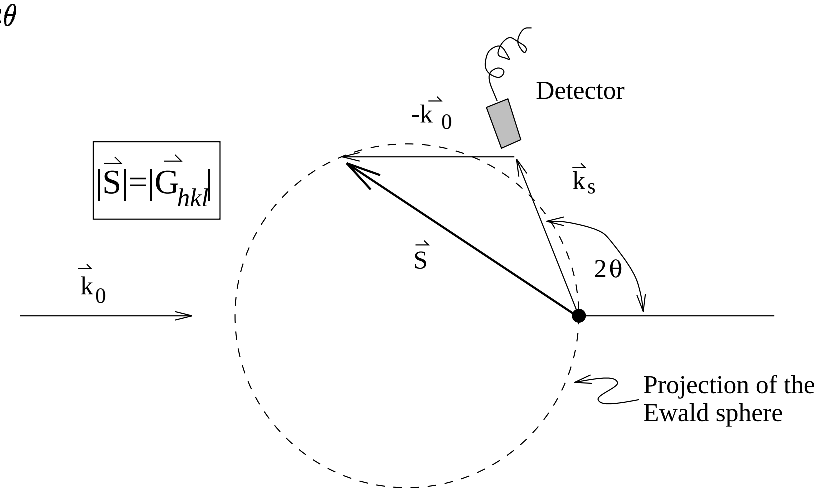
In order to observe the diffraction peak for a particular $\vec{G}_{hk\ell}$ vector in a single crystal sample, there are two essential steps.

1 - Adjust 2θ so that $|\vec{s}| = |\vec{G}_{hk\ell}|$

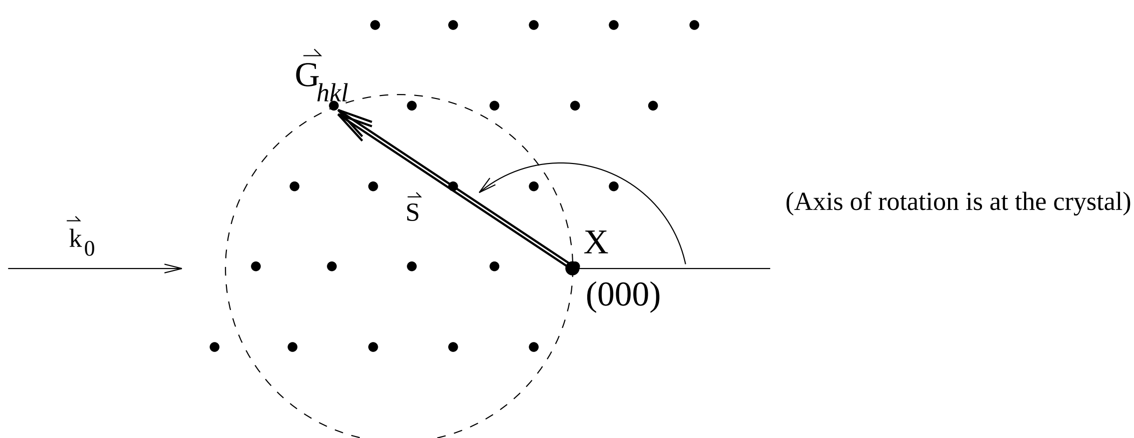
2 - Rotate the crystal so that $\hat{s}||\hat{G}$



1. Set 2θ



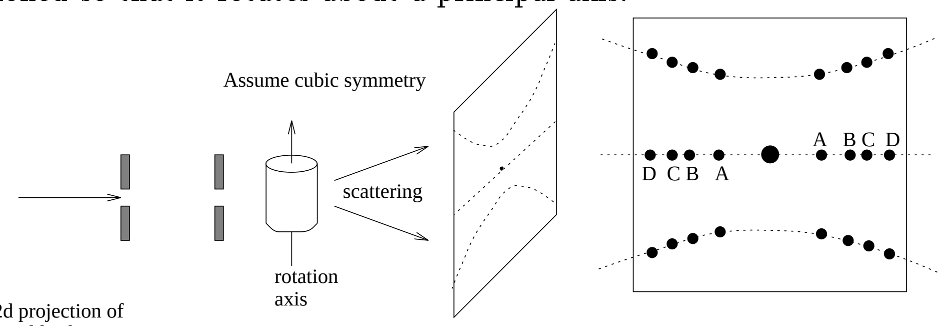
2. Rotate crystal to bring $\hat{s}||\hat{G}_{hk\ell}$

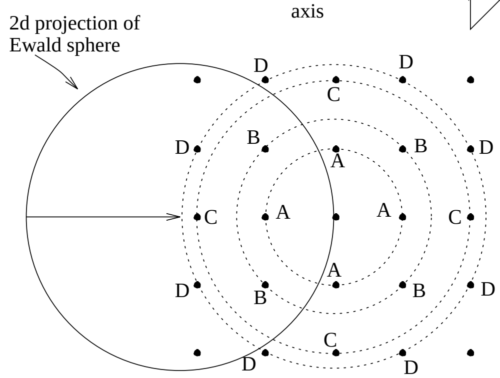


Notice that by rotating the crystal different $\vec{G}$'s will intercept the Ewald sphere. When this occurs, it is then possible to observe this reflection. (Provided of course that the detector is at the correct angle.)

We can replace the point detector with a piece of film. In this way, large portions of the Ewald sphere can be viewed simultaneously.

Rotating Crystal Method:
A crystal is positioned so that it rotates about a principal axis.

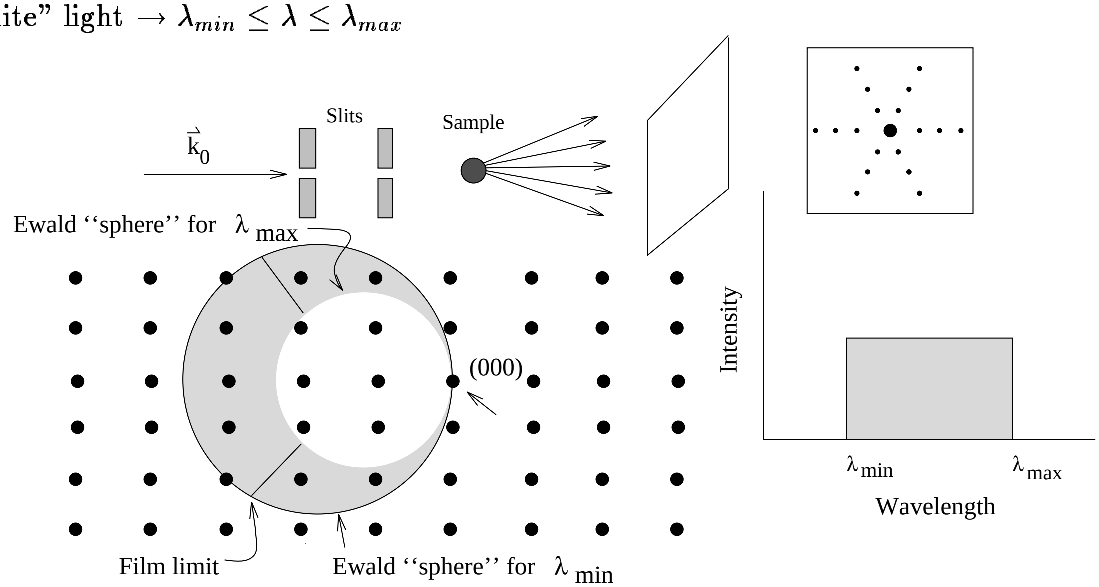




Peak	Multiplicity
A	4
B	4
C	4
D	8

Laue method

This method is very different in comparison to the previous one. In this case a collimated beam of "white" light is incident on a stationary crystal.
"White" light $\rightarrow \lambda_{min} \leq \lambda \leq \lambda_{max}$

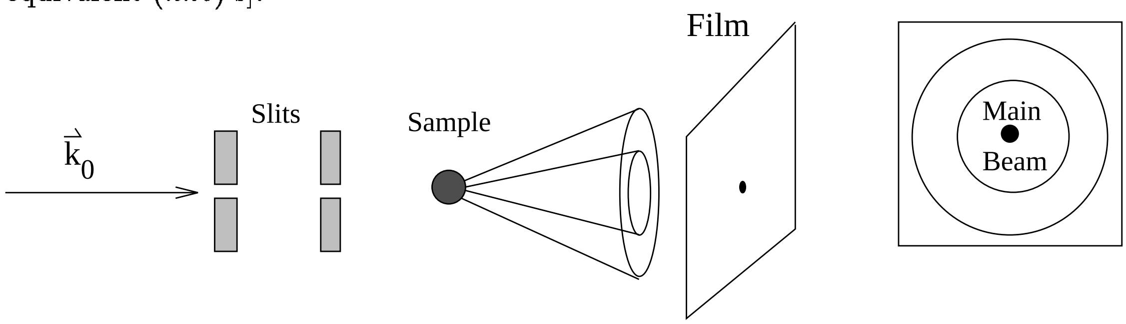


Shaded area represents the portion of the reciprocal lattice which can be seen if ''film'' surrounds the sample.

This method provides information as to symmetry and orientation.

Debye-Scherrer Powder Method

In this case a monochromatic beam is incident on a powdered sample. It is assumed that the powder contains all possible orientations of the crystal. Thus, diffraction occurs for all $|\vec{k}_d|$'s of a particular reciprocal lattice vector [i.e., $\vec{G}_{hk\ell}$ and symmetry equivalent $(hk\ell)$'s].



Since $|\vec{k}_0|$ is fixed, there is only one Ewald sphere. However all reciprocal lattice points within the sphere, $|\vec{k}_d| \leq 2|\vec{k}_i|$, can potentially be observed. The position of the rings gives information as to crystal symmetry and lattice spacing. Intensity gives information concerning the unit cell structure (i.e., the basis).

For example:

A simple cubic lattice with $a = 2$Å and $\lambda_{\text{photon}} = 1$Å

$$|a_1^*| = \frac{2\pi}{2\text{Å}} = 3.14\text{Å}^{-1} \qquad \frac{\lambda k}{4\pi} = \sin\theta$$

Diffraction peaks are at 3.14, $\sqrt{2}(3.14)$, $\sqrt{3}(3.14)$,.....Å^{-1}

	Multiplicity
$\|k\| = 3.14\text{Å}^{-1}$, $2\theta = 28.9°$, 6 superimposed peaks	
$(1\ 0\ 0)\ (0\ 1\ 0)\ (0\ 0\ 1)\ (\bar{1}\ 0\ 0)\ (0\ \bar{1}\ 0)\ (0\ 0\ \bar{1})$	6
$\|k\| = 4.44\text{Å}^{-1}$, $2\theta = 41.4°$, 12 superimposed peaks	
$(1\ 1\ 0)\ (1\ 0\ 1)\ (0\ 1\ 1)\ (\bar{1}\ 1\ 0)\ (1\ 0\ \bar{1})\ (0\ 1\ \bar{1})$	12
$(\bar{1}\ \bar{1}\ 0)\ (\bar{1}\ 0\ \bar{1})\ (0\ \bar{1}\ \bar{1})\ (1\ \bar{1}\ 0)\ (\bar{1}\ 0\ 1)\ (0\ \bar{1}\ 1)$	
$\|k\| = \sqrt{3}\pi\text{Å}^{-1} = 5.44\text{Å}^{-1}$, $2\theta = 51.3°$, 8 superimposed peaks	8
$(1\ 1\ 1)\ (1\ 1\ \bar{1})\ (1\ \bar{1}\ 1)\ (\bar{1}\ 1\ 1)\ (\bar{1}\ \bar{1}\ \bar{1})\ (\bar{1}\ \bar{1}\ 1)\ (\bar{1}\ 1\ \bar{1})\ (1\ \bar{1}\ \bar{1})$	