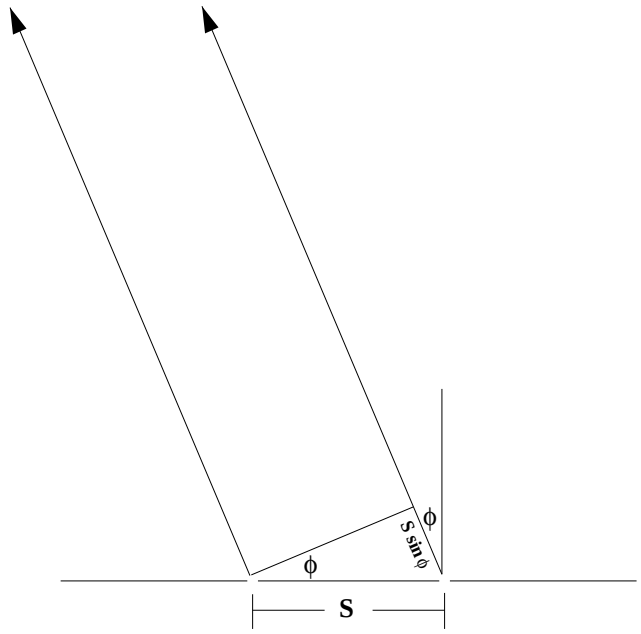
Lecture Notes - Mineralogy - Diffraction

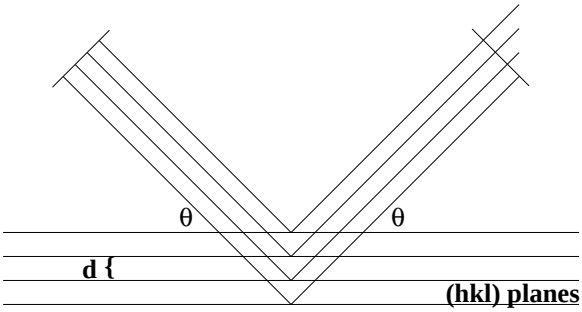
- Two of the most fundamental aspects of a mineral, its space group and unit cell, can be determined from x-ray diffraction experiments. To understand these experiments, which we will do in lab, we must explore the physics of diffraction. **Diffraction**, generally defined as a departure of a ray from the path expected from reflection and refraction, was first observed for light early in the nineteenth century. Sets of narrow slits and ruled gratings were observed to produce **diffraction patterns** when the spacing of the slits is similar to the wavelength of light used. Because all of the slits in a diffraction grating are illuminated by the same source of light, the set of slits may be con-sidered to be a set of light sources all **in phase** with one another. Light rays traveling perpendicular to the diffraction grating will remain in phase. Light rays traveling at an angle ϕ to the perpendicular will not be in phase, except for special angles such that $\mathbf{S} \sin \phi = \mathbf{n} \lambda$, where **S** is the spacing of the slits, λ is the wavelength of light, and **n** is an integer. We may used this expression to determine λ for a laser or **S** for a diffraction grating from a measurement of the spacing of the diffraction pattern.

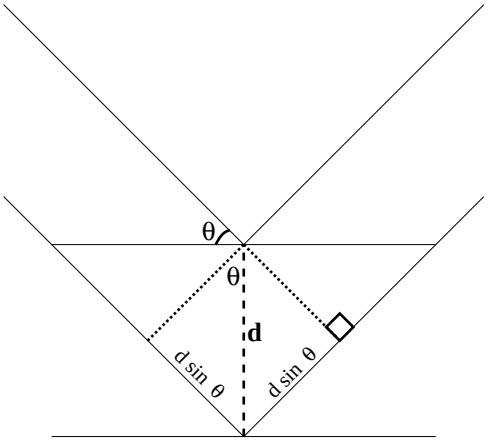


- Diffraction of x-rays by crystals is possible because the spacing of planes of atoms in crystals is similar to the wavelength of x-rays. The atoms in crystals behave like little x-ray sources as they scatter incident x-rays. Although an x-ray diffraction experiment can be designed to be very similar to an optical diffraction grating experiment (a Laue experiment), most experiments involve x-ray “reflections." For the diffracted x-rays to be **in phase**, the geometry of the experiment must satisfy **Bragg’s law**:

$$\mathbf{n} \lambda = \mathbf{2}\, \mathbf{d_{(hkl)}} \sin \theta$$

$\mathbf{d_{(hkl)}}$ is the spacing between the parallel planes of atoms with Miller index (hkl), θ is the complement of the usual angle of incidence, λ is the wavelength of the x-



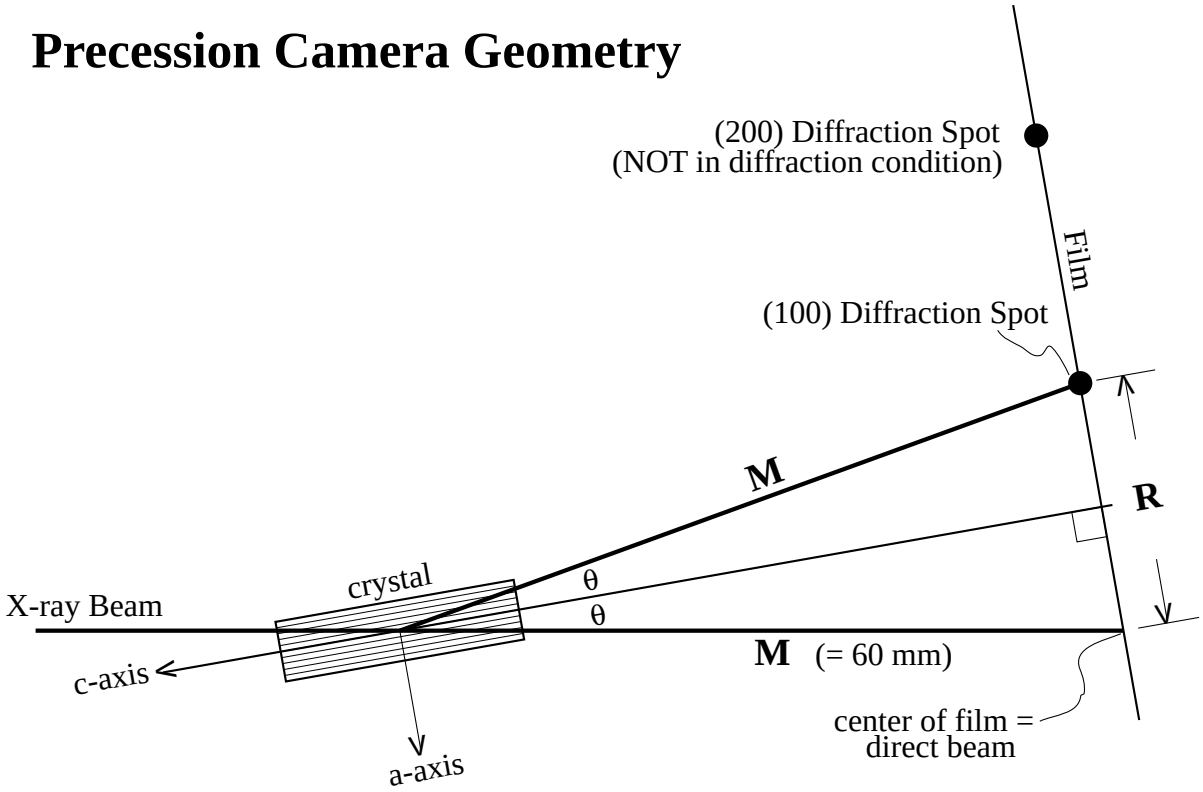


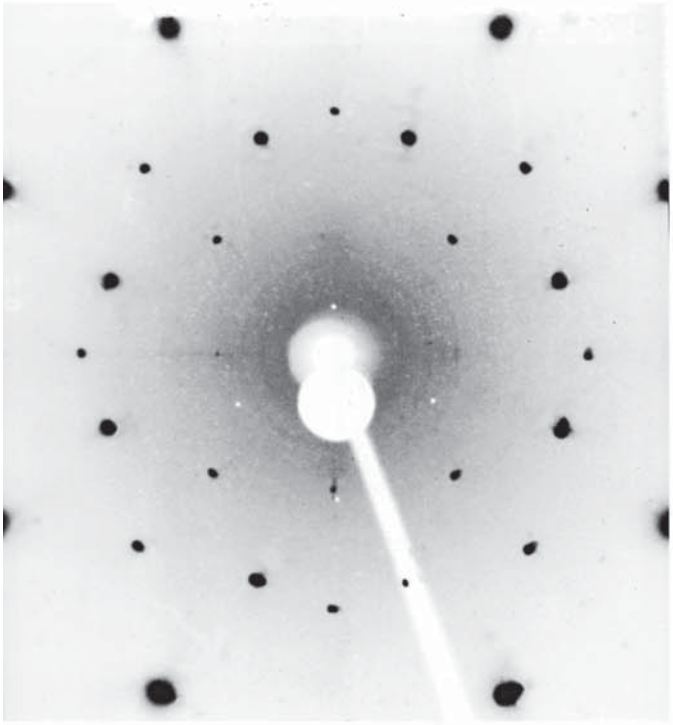
rays, and **n** is an integer. Note that each set of planes (hkl) may produce more than one diffracted ray, each with different values of **n**.

- X-rays are produced when electrons "boiled" from a filament are caused to strike a target of atoms by the force of a high voltage field. Decelleration of electrons as they approach atoms in the target creates a "white" background of x-rays called the Brehmstrallen radiation. Superimposed on this background are peaks of intense x-rays that have wavelengths that depend on the atoms involved. These peaks of characteristic wavelengths are produced when an atom looses an electron from an inner orbital, due to a collision from an accelerated electron, and then compensates by having an electron from an outer shell fill the partially vacant inner shell. The peaks are labelled **K**α, **K**β, **L**α, **etc**., depending on the specific energy levels involved. At Smith we have two x-ray generators, one equipped with a Mo tube for single crystal diffraction experiments, one equipped with a Cu tube for powder diffraction experiments. Filters may be used to block out (absorb) the **K**β x-rays from each tube so that λ can be considered to be **K**α.

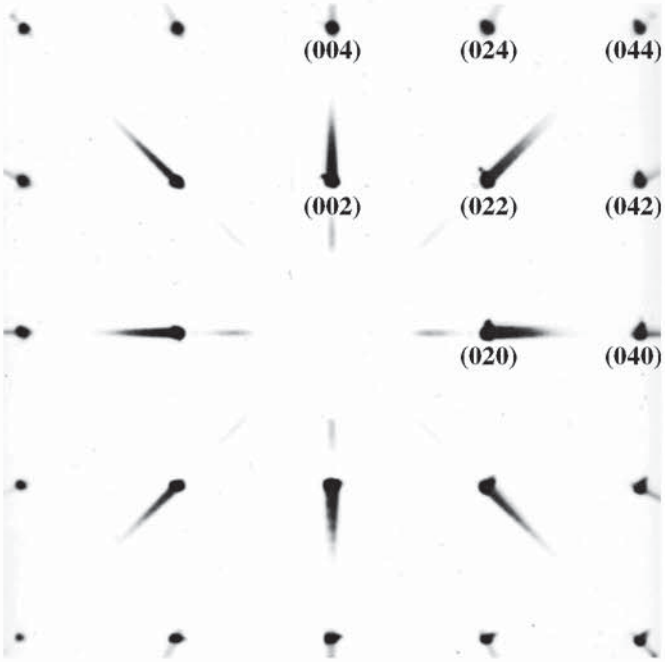
- To view diffraction from more than one set of planes (hkl), it is necessary to change both the angle θ and the orientation of the crystal. The **Buerger precession camera** is designed to do both, so that diffracted x-rays from all sets of planes that belong to a single zone are recorded on the film. The precession camera also moves the film that records the diffraction in a way that preserves the symmetry of the diffraction pattern. Neumann's principle requires that the symmetry recorded on the film include all of the symmetry elements of the point group of the crystal for the orientation chosen. The symmetry recorded on an appropriate set of precession photos may be used to determine the space group for the mineral.

- The distance **R** from the center of a precession photo to the "reflection" produced by a particular set of planes is proportional to the spacing $\mathbf{d_{(hkl)}}$ for those planes such that $\mathbf{d_{(hkl)} = M\lambda/R}$, where **M** is a magnification factor for the camera. Note that the distance **R** is **inversely proportional** to $\mathbf{d_{(hkl)}}$. For this reason, the set of all diffraction spots is called the **reciprocal lattice** for the crystal. The spacing of the reciprocal lattice is given by the reciprocal lattice vectors **a***, **b***, **c*** and angles α^*, β^*, γ^*. Direct lattice parameters may be easily recovered from the reciprocal lattice parameters. Note also that along any radial line, the same set of planes can produce several reflections for different values of **n** in the Bragg equation. Because these reflections appear to represent planes with spacings that are equal to $\mathbf{d_{(hkl)}/n}$, the spots are indexed as $\mathbf{d_{(nh\ nk\ nl)}}$ **.** For example, along the **b**-axis one observes reflections (010), (020), (030), etc.





Periclase Laue



Periclase Precession

Tourmaline