Lab 7

X-ray Diffraction

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Brief Introduction

At the turn to the 20th century there was debate over the nature of X-rays, which had been recently discovered by Wilhelm Roentgen in the 1890s. One experiment that was done was to pass X-rays through crystalline materials, which produced a pattern of spots on a piece of film placed behind the crystal. This was explained as diffraction (an interference phenomenon) by Sir William Henry Bragg, lending support to the idea of X-rays as waves. The diffraction of X-rays by crystals ultimately paved the way for determination of molecular structures.

Here you will do some X-ray diffraction experiments on crystalline materials as a way of extending the 1-D and 2-D diffraction experiments you did last week to three dimensions.

Questions.

1. In your notebook, sketch the diffraction pattern expected from a 2-D grid with equal spacing in both directions. You made such a measurement last week.

2. Now suppose you had many such grids in various orientations rotated about the incident beam of light, and all these grids diffracted simultaneously. Sketch the resulting diffraction pattern.

3. Now suppose you plot the intensity of the result from #2 vs theta, the diffraction angle. Sketch the resulting 1-D diffraction pattern.

4. If the grid spacing became larger, how would the diffraction pattern change?

Diffraction from a 3-dimensional crystal is similar to your example in 2-D above. In the crystal, each atom acts as a scattering center. Because the crystal is in 3-dimensions, the scattering geometry is usually defined a bit differently from the 1-D case you're familiar with. This is illustrated below.

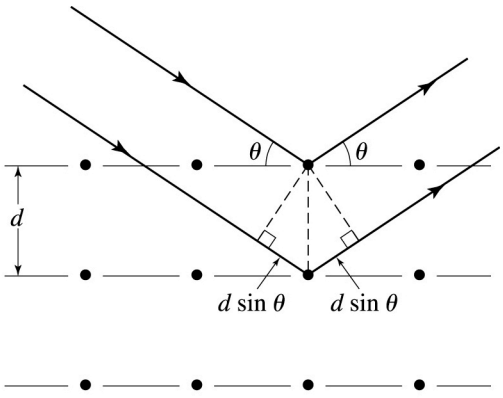

Figure 2.8 copyright 2009 University Science Books

Each dot represents an atom or molecule, and scatters the incoming light. We will get constructive interference (and the crystal will 'diffract') when:

$$2d\sin\theta = n\lambda$$

This is called Bragg's law. The angle theta is defined somewhat differently from the 1-D case. Another difference between the 1-D and 3-D case is that the lattice usually needs to be rotated for the geometry to be the correct geometry needed to get constructive interference.

Although the 3D case is more complicated than for 1-D and 2-D, the basic facts are the same. As with the 1-D and 2-D cases:

a. The spacing of the diffraction peaks is related inversely to the slit spacing (*d*). Here the slit spacing refers to the spacing between the atoms in the crystal.

b. The 'envelope' which defines the pattern of the diffraction peak intensities comes from the particulars of the atoms doing the scattering. In the 1-D case, this was the width of the slit. In the crystal, this refers to the arrangement of electrons in the atom.

To tie things again to last week, the diffraction pattern may be thought of as a mask placed over the Fourier transform of the atom or molecule. Therefore, by measuring the diffraction pattern we are essentially sampling the Fourier transform of the molecule in discrete locations. By doing the reverse Fourier transform of the diffraction pattern we can recover the structure of the molecule. This is the primary way that structures of molecules are determined.

Experiments

Diffraction from metallic crystals

Many metals crystallize in a face-centered cubic arrangement. This means the metal atoms are arranged on a cube, with one atom at each vertex, plus another one in the center of each face. These metals include copper, gold, silver, aluminum, platinum.

After being trained on the X-ray diffraction instrument, measure the diffraction patterns from several metal samples – copper, aluminum, zinc, lead and brass.

Compare the patterns of copper and aluminum. What are the similarities and differences and how are they related to the structure of the crystal? Use the "Lattice information" button to estimate the lattice spacing in each crystal. Use a 'User defined' Crystal and Lattice. Your lattice is an F-centered lattice. a,b, and c correspond to the slit spacings in the three dimensions, and alpha, beta and gamma correspond to the angles between these slits. For the cubic crystal, a=b=c and alpha=beta=gamma=90 degrees. Enter various values of a=b=c to make the predicted peaks line up with the actual peaks. What do you conclude about the relative spacing of copper and aluminum in their respective crystals? What might this say about the radii of those atoms?

Zinc crystallizes in a different arrangement from copper and aluminum - a hexagonal arrangement. Measure the diffraction pattern from zinc and compare to the previous two. What are the similarities and the differences? Repeat for lead.

Measure the diffraction pattern of brass, and compare to the other metals. Brass is usually composed primarily of copper and zinc, sometimes with a third element present. Try to develop a model for the composition of this sample of brass.

Diffraction as an identification technique

The combination of lattice parameters with intensity pattern can be a powerful method for identifying materials. By comparing the diffraction pattern of an unknown sample to a database of known diffraction patterns, often a material can be identified. Find a crystalline material whose identity is unknown to you. Take a diffraction measurement of this material. Use the program Jade to try to identify the material.