

X-Ray Project, Part II

PHYS 310

Introduction

The title of this project probably sounds to you as though this is just about a technique. And it is, to a certain extent. But really, this is several different projects, all of which can be executed on a really cool device that we have from LD Didactic that can not only produce x-ray sources but which can also use these x-ray sources to study (1) electronic structure of a range of atomic elements, (2) the crystal structure of many solids, and (3) the Compton Effect in which a single photon scatters off an elementary particle. (These are actually three separate projects – you will not have time to do all three of these.) But there is also a lot of interesting physics involved in understanding the production of x-rays. And x-ray devices are used so often in science and technology that it is important for y'all to have some familiarity in principles and techniques involving x-rays.

X-rays are commonly produced when beams of electrons slam into a material. Several scientists inadvertently produced x-rays throughout the 18th Century as a by-product of experiments that they were doing with electron beams and cathode-ray tubes, and some even noticed that they could be used for “seeing through” matter. They were first formally identified¹ by Wilhelm Röntgen² in 1895 who realized that he had evidence for a beam of something that was clearly not visible light. He dubbed them “X-rays” because he did not know what they actually were. It was later determined that x-rays are a form of electromagnetic radiation with a wavelength (frequency) that is smaller (larger) than that for visible light (or ultraviolet radiation). Wavelengths (frequencies) range from around 10 pm to 10 nm (3×10^{19} to 3×10^{16} Hz) for x-rays, corresponding to photon energies ($E = hf$) that range from ~ 100 eV to 100 keV. In large enough doses, x-rays can pose physical hazards since these energies exceed the binding energies in biologically-relevant molecules and can therefore cause mutations that can lead to cancer. On the other hand, when used judiciously, x-rays are incredibly useful for medical diagnostics since they can penetrate the body and can be used to form precise (and 3-D) maps of internal structures in the body, especially when used in conjunction with radiocontrast agents that effectively absorb x-rays.

X-ray diffraction is used *extensively* in a wide range of fields in science and engineering to determine the atomic and molecular structure of various materials. X-ray scattering off a crystal effectively performs a Fourier transform of the crystal structure (often referred to as *reciprocal space*). As an example, x-ray diffraction was used to determine that DNA is a double-helix. To this day, physicists, biologists, chemists and geologists use x-ray scattering as a primary diagnostic tool for a range of materials in physics, engineering, geology, chemistry and biology. In fact, there are national laboratories that provide x-ray sources that scientists can use for these studies. (As your instructor about *synchrotron* x-ray sources – another way to produce particularly strong beams of x-rays.)

Background and Theory

We will not go over the theory in detail in this write-up, because there are numerous sources, several of which we have in PDF form. The excerpt from “Elements of X-ray Diffraction,” 2nd Edition (B. D. Cullity)¹

¹ “Elements of X-ray Diffraction,” 2nd Edition, B. D. Cullity, Addison-Wesley (1978).

² Röntgen was awarded the first ever Nobel Prize in physics for his “discovery” of x-rays.

provides a good overview of the production and absorption of x-rays, including a discussion of *bremstrahlung* (braking radiation) and of K_α and K_β emission spectra due to transitions in atomic electrons down into the lowest energy level (the *K-shell*). The excerpt from Ashcroft & Mermin's *Solid State Physics*³ covers the basics of microscopic crystalline structure and how that structure can be revealed by x-ray diffraction. And there are several "LD Physics Leaflets" that cover a range of both physics principles and the experimental techniques used to test and elucidate those principles using this device.

Safety

The x-ray device has several built-in safety features which pretty much guarantee that you will not be exposed to harmful levels of x-ray radiation. The second task below includes a discussion of safety features.

Tasks for Group #2

1. Skim the handout titled "Properties of X-rays" (from *Elements of X-ray Diffraction*¹). Presumably, you are already familiar with the important parts of this handout because the previous group presented on this stuff. The bottom line is that you should be aware that the x-ray source produces a spectrum of x-ray radiation composed of a combination of (a) *bremstrahlung* (braking radiation) that produces a broad spectrum; and (b) some discrete emission lines (at well-defined wavelengths) from the K_α and K_β transitions.

In this project (Part II of x-rays), we are interested in the discrete emission lines and *not* the broad *bremstrahlung* spectrum. I.e., we want a well-defined x-ray wavelength.

2. The new part of the theory (not discussed by the previous group) is the discussion about using Bragg scattering for crystallography. We have a handout – excerpts from Ashcroft & Mermin's *Solid State Physics* – that goes into details about different aspects of crystal structure in solid materials. There is quite a lot in this handout, but the key things that you will need to understand for this project are:
 - a. From chapter 4, you need to understand the basic idea of repeating crystal structures (a *Bravais lattice*), the concept of a *unit cell*, and some of the different types of crystal structure, particularly simple cubic (SC), body-centered cubic (BCC) and face-centered cubic (FCC) Bravais lattices. The portion of the excerpt from Chapter 4 also discusses the sodium chloride and cesium chloride structures, which are very relevant to this project because you will be doing Bragg scattering on both of these.

There is a lot of discussion of other types of lattices, especially close-packed lattices. These are all important, but you don't need to study that in detail. You should know that

³ "Solid State Physics," Ashcroft & Mermin, Saunders (1976).

there are a wide range of different types of lattices and, in fact, there are a lot of scientists that devote their entire careers toward using x-ray diffraction to study the structure of a variety of materials.

- b. From chapter 5, the main thing that you need is the idea of multiple lattice planes and the use of *Miller indices* to denote the different planes. I'll be honest – I found the description in this section to be hard to follow. I prefer an approach for Miller indices discussed in *Introductory Solid State Physics, 2nd Edition*, H. P. Myers. That book describes a method for determining Miller indices associated with particular planes. This can be done easily for a 2-D lattice. See Fig. 2 from the *Physics Leaflet* P 7.1.2.4 (“Debye-Scherrer Scan: determining the lattice-plane spacings of polycrystalline powder samples”) to clarify the following approach for determining Miller indices for a particular set of planes:
 - i. Draw a plane (line) through the origin, and then draw the next one over (it doesn't matter which way).
 - ii. Determine where this line it intersects the axes as a fraction of the fundamental lattice vectors in each direction; e.g., 1, 1/2, 1/3, ... of the lattice spacing.
 - iii. Invert and take absolute value to get the indices.

Note: the index is also the number of planes that intersects per unit in that direction.

- c. From chapter 6, the most important thing is the Bragg formulation. (We won't do anything with the Laue or Ewald approaches.) The geometrical structure factor is also important, as it determines which sets of planes show up (and their relative intensities) in Bragg scattering of powder samples for different types of lattices.
3. Familiarize yourself with the x-ray device by following the instructions in the “X-Ray Production: General Overview of Apparatus” handout. By the end of this handout, you should be able to determine the spectrum of whatever x-rays are being produced by the particular anode. **Use this approach to determine a spectrum for the copper anode, both without and with a nickel filter over the detector.** (Do a scan from 5 degrees to 30 degrees, 0.1 degree spacing, and 5 seconds per sample. Use 35 kV for the potential difference and 1.00 mA for the filament current.)

Presumably, you should find that the nickel filter knocks out most of the K_{β} emission line. The result is that you have an x-ray source with emission with a well-defined wavelength from the K_{α} emission. (There is also, of course, the bremsstrahlung radiation, but it is the peak that we will use for the Bragg scattering.)

4. To get a complete picture of the crystal structure, we will use *powder* diffraction, also known as Debye-Scherrer method. Read the “Physics Leaflet” P 7.1.2.4 (“Debye-Scherrer Scan: determining the lattice-plane spacings of polycrystalline powder samples”). Follow the method described in this leaflet to do a scan of a powder sample of NaCl. There might already be a sample that has been prepared. If not, the leaflet describes how to grind a salt sample and

mount it in a slide.

It might be of interest to do two scans, one “quick” (i.e., an hour) scan from 10 – 60 degrees (35 kV, 1.00 mA, spacing 0.1 degree, 5 sec per sample) and then an overnight scan over the same range but with 140 or 150 second per sample. **Question:** why is it important to take data over a longer period of time? And why do you think that many scientists who do crystallography take their samples to national synchrotron x-ray sources?

5. Analyze the results from your overnight scan. You should be able to identify several peaks in the spectrum, with each peak corresponding to a different set of planes. Use the Bragg relation $n\lambda = 2d\sin\theta$ (given the wavelength that you should have determined from step 3) to determine the spacing of each set of planes. And then try to identify which set of planes corresponds to which peak and see if you can figure out the structure of the NaCl unit cell from this information.

Hints: the spacing is related to the Miller indices and the length a_0 of the unit cell via

$$d_{hkl} = \frac{a_0}{\sqrt{h^2 + k^2 + l^2}}$$

It can be helpful to define $Z = h^2 + k^2 + l^2$. Recognizing that Z must be an integer, if you can figure out the unit cell spacing a_0 from the data (often the d for the first or second peak), then calculating $(a_0/d)^2$ for each peak will get you a sequence of integers (or ratios close to integers). You can then figure out the Miller indices (hkl) from the value of Z for each peak. When doing this, the angle for whatever peak corresponds to a_0 is the critical one and, in fact, you might find that if you don't get a good pattern of integers, a slight tweaking of the peak angle can make a big difference.

Note: the thing that enables you to figure out the structure of the unit cell is peak intensities for the different combinations of Miller indices. A simple cubic (SC) – which is also sometimes called *cubic primitive* – lattice will produce peaks for every set of planes. A face-centered cubic (FCC) lattice will display peaks *only* for Miller indices where each one is even (e.g., 200, 220, 420) or each is odd (e.g., 111, 311, 331). And a body-centered cubic lattice (BCC) will display peaks only for Miller indices that add up to an even total (e.g., 110, 200, 211, 220).

And it can be challenging to distinguish between a lattice with unit cell length a_0 versus a lattice with a unit cell length $a_0/2$. In fact, the NaCl crystal pattern falls into that category. The key way to distinguish – look for the (111) peak (which can be very hard to see in a NaCl powder spectrum).

When you finish, you should be able to say that NaCl forms one of two different types of crystal lattice structures (and you can determine a_0). And if you can identify the (111) peak, then you will be able to determine the lattice structure and a_0 without any alternative.

6. Repeat steps 4 and 5 with cesium chloride, which has a different type of structure than NaCl. It is a little more difficult to get a good spectrum with CsCl (the overnight run is absolutely crucial), but the analysis of the peaks is easier than for NaCl.

A final comment: What you are doing in this project is just the tip of a very large iceberg. We are looking at the simplest of structures – cubic with either SC, FCC or BCC structures. But crystal lattices can be rectangular with different lattice constants in different directions, non-orthogonal, hexagonal, helical, etc. And the unit cells can be composed of a variety of different atoms in different locations that can have a significant effect on the relative intensities of the different peaks and the locations of the peaks if photographed on a film (instead of scanning a single detector).