X-Ray Diffraction Lab

Debye Scherrer Camera and Hull-Davey Charts

Objective: To become familiar with use of the Debye Scherrer Camera and to learn to use Hull-Davey Charts to index tetragonal and hexagonal crystal structures.

Download Data (Excel Files):
Copper (FCC)
Germanium Single Crystal (Diamond Cubic)
Molybdenum (BCC)
Indium(BC Tetragonal)
Brass Key (FCC Compare to Copper)
Germanium Ground to Powder(Compare with Germanium Single Crystal above, Diamond Cubic)
Second Run of Moly(BCC)
Zinc (HCP in Table 10.2)

Background: Debye Scherrer Camera: Cullity pp. 96 (Chapter 3), pp. 161-187 (Chapter 6), pp. 233 (Chapter 8). Hull-Davey Graphical Method: Cullity chapter 10, pp. 324.

Indexing of unknown crystal structures involves some form of trial and error procedure. The higher the number of observed reflections the better are your chances to narrow down the field to a single crystal structure. For high-symmetry, cubic systems the job of indexing is fairly simple. Figure 10-2 shows prototype Debye-Scherrer patterns for polycrystalline (powder) simple, body-centered, face-centered and diamond cubic systems. The planes which correspond to each reflection are noted. Changes in the lattice spacings and crystal composition at lattice sites will effect the intensity and absolute values for the angles of diffraction but the relative "finger-print" for these patterns will not change! This means that a Debye-Scherrer or Diffractometer trace for a cubic system can be indexed by glancing at the pattern and comparing it to the calculated patterns shown in figure 10-2.

For lower-symmetry crystals indexing becomes a bit of a nightmare. There are several approaches to ease the burden of indexing these lower symmetry systems. The main issue is that for systems where the lattice distances are not all the same, the relative positions of the diffraction lines can swap places and move around. For instance, in a tetragonal system the c/a ratio governs this. In Hexagonal and orthorhombic (polyethylene) systems it becomes even more complex. As you might guess, a simple approach would be to make prototype plots such as the indexed patterns in figure 10-2 but with an x-axis which reflects the c/a ratio for tetragonal (for example). This is basically what is done in a Hull-Davey Chart such as in Figure 10.3. The pattern is converted to an x/y plot where the y axis is the c/a ratio and the x-axis is function of the diffraction angle (and via application of Bragg's law for these specific conditions, the hkl index of the planes.) Once you convert your diffraction pattern in 2θ to the reduced parameter this is marked on a sheet of paper and the reduced markings are matched up with the Hull-Davey plot. This graphical matching yields the c/a ratio and indexing of all reflections. If a proper correspondence can not be found then the crystal is probably not tetragonal. Hull-Davey plots are available for Hexagonal and other low symmetry systems. They can also be constructed in a graphing program for the specific problem you are interested in.

The Debye-Scherrer camera is a simple photographic camera which is most useful for polycrystalline (powder) wire samples although it can be used for small, non-wire samples. This is a cylindrical camera which covers an equator on the sphere of reflection (appendix 1). We will use conventional x-ray film in this camera so will use the dark room for film loading and development. The developed images will be read on film readers.

Itemized Things to do:

1. *Make a guess as to the type of crystal for each pattern or diffractometer trace using figure 10.2.*
2. *Calculate the d-spacing for each reflection using Bragg's Law (7 reflections are sufficient)*
3. *Calculate lattice parameter using Appendix 3.*
4. *If your guess in 1 was right then the lattice parameters will be fixed in value.*
5. *For tetragonal you will need to use the Hull/Davey chart to index.*