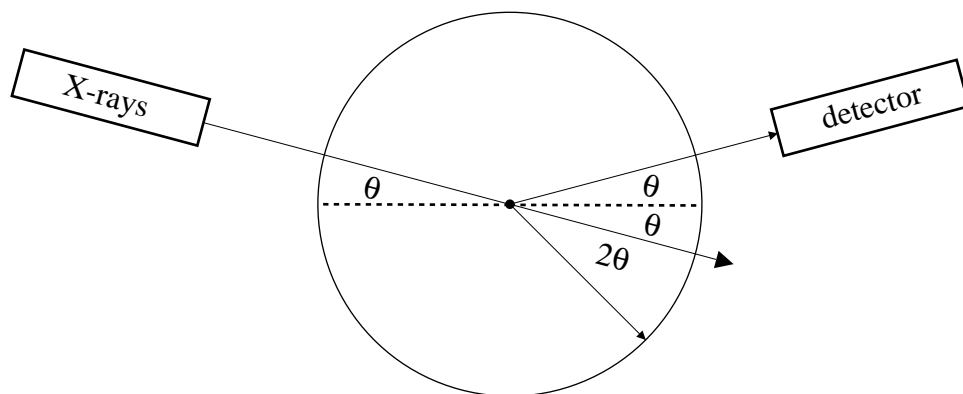


Lecture Notes - Powder Diffraction

- X-ray diffraction by mineral powders is one of the mineral identification and characterization techniques most used by geologists. Powder diffraction experiments require only a small quantity of a mineral. As little as 10 mg can yield good data, although 500 mg is the quantity required for common sample mounts. Sample preparation is extremely simple — no crystal faces or cleavages are needed for crystallographic orientation, because no sample orientation is required. Reliable, accurate results are obtained in a relatively short time (10 minutes to 2 hours).
- The principle behind the design of powder diffraction experiments is the random orientation of crystals in a mineral powder. If the powdered crystals are randomly oriented, then for all sets of planes (hkl) some of the crystals in the powder will be in the correct orientation (usually horizontal) with respect to the x-ray source to satisfy Bragg's law for the proper angle θ . In other words, at least a few of the mineral grains in the powder will diffract for each of the planes (hkl) during a scan through the angles θ . The more finely ground the powder, the more likely that all orientations are present in abundance. The ideal powder size is 5-10 μm .
- Two main types of powder diffraction experiments are possible at Smith: automated powder diffractometer experiments yielding (digital) computer output and Debye-Sherer experiments providing (analog) film output. In the Debye-Sherer camera, a strip of film is wrapped around the powder sample so that diffracted beams from a fixed x-ray source may be recorded for all values of θ simultaneously. The powder diffractometer moves both the x-ray source and an electronic detector through arcs



of θ values and sends to a computer periodic signals proportional to the averaged diffracted x-ray intensity. Both experiments provide the intensities for diffracted beams as a function of the diffraction angle θ (or 2θ).

- Mineral unknowns may be identified from powder diffraction data using the **ICDD Powder Diffraction File**. Both intensity and 2θ (or d_{hkl}) values are used in the search. Computer searches of the file (on CDROM) may lead to a unique match with a known powder diffraction pattern. However, because the chemical composition of most minerals is variable, because some aspects of a mineral's structure may depend on its history, and because any measurement contains some error, the observed diffraction data may not exactly match the standard data for a given mineral. This makes identification more challenging and chemical data or other physical property data may be needed to confirm a result for some samples.
- Once a mineral has been identified, the Powder Diffraction File data card may be used to **index** the observed diffraction peaks. Miller indices along with measured 2θ values (from which d_{hkl} can be calculated using Bragg's law) may be used to determine the unit cell parameters of the specimen. In principle, six measured d_{hkl} values are sufficient to determine the six unknown unit cell parameters. However, a more accurate result will be obtained if more d_{hkl} values are used to "over determine" the solution. Various computer programs are available to perform a least squares data reduction on the over-determined data set to give a "best fit" unit cell. If the unit cell of a standard is known, values for each d_{hkl} can be calculated and compared with observed d 's to see how closely the data match the standard unit cell.

Si O ₂ Silicon Oxide Quartz, Syn				
Rad:CuKα1	Lambda:1.540598	Filter:Monochromator crystal used	d sp:Diffractionmeter	
Cutoff:	Int:Diffractionmeter	I/Icor:3.6	Ref:Swanson, Fuyat., 18 61, (1981)	
Sys:Hexagonal		S.G.:P3221		
a:4.9133±0.0002	b:	c:5.4053±0.0004		
α:	β:	γ:	Z:3	mp
Ref2				
Dx:2.649	Dm:2.649	SSFOM: F30=76.6(0.0126,31)	Volume[CD]:113	
εα:	ηωβ:1.544	εγ:1.553	Sign:+	2V:
Ref3				
Color:Colorless				
Sample from the Glass Section at NBS, Gaithersburg, Maryland, USA, ground single-crystals of optical quality. To replace 5-490. Plus 6 reflections to 0.9089. Pattern at 25 C. Pattern reviewed by J. Holzer and G. McCarthy, North Dakota State University, Fargo, North Dakota, USA, \ITJCPDS Grant-in-Aid Report\RG (1990). Agrees well with experimental and calculated patterns.				

39 reflections in pattern.

2 θ	Int.	h k l	2 θ	Int.	h k l	2 θ	Int.	h k l	2 θ	Int.	h k l
20.8501	22	1 0 0	55.3283	2	1 0 3	77.6619	1	2 2 0	94.6651	1	1 0 5
26.6519	100	1 0 1	57.2380	1	2 1 0	79.8778	2	2 1 3	95.1187	1	4 0 1
36.5421	8	1 1 0	59.9485	9	2 1 1	80.0461	1	2 2 1	96.2267	1	2 1 4
39.4559	8	1 0 2	64.0009	1	1 1 3	81.1475	3	1 1 4	98.7375	1	2 2 3
40.2836	4	1 1 1	65.7605	1	3 0 0	81.4721	3	3 1 0	102.1991	1	4 0 2
42.4650	6	2 0 0	67.7497	6	2 1 2	83.8208	1	3 1 1	102.5594	1	3 1 3
45.8092	4	2 0 1	68.1305	7	2 0 3	84.9713	1	2 0 4	103.8839	1	3 0 4
50.1407	14	1 1 2	68.3227	8	3 0 1	87.4640	1	3 0 3	104.1993	1	3 2 0
50.6111	1	0 0 3	73.4620	2	1 0 4	90.8583	2	3 1 2	106.1453	1	2 0 5
54.8689	4	2 0 2	75.6706	2	3 0 2	92.8224	1	4 0 0			