
奈米量測技術 (III)

XRD (X-ray Diffraction) (部分)

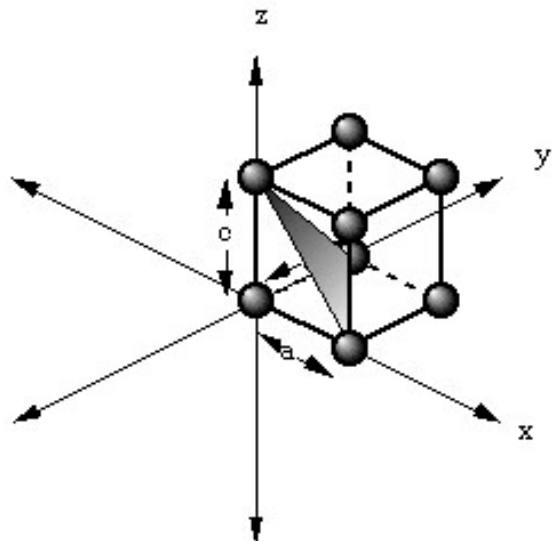
連興隆

X-ray Diffraction

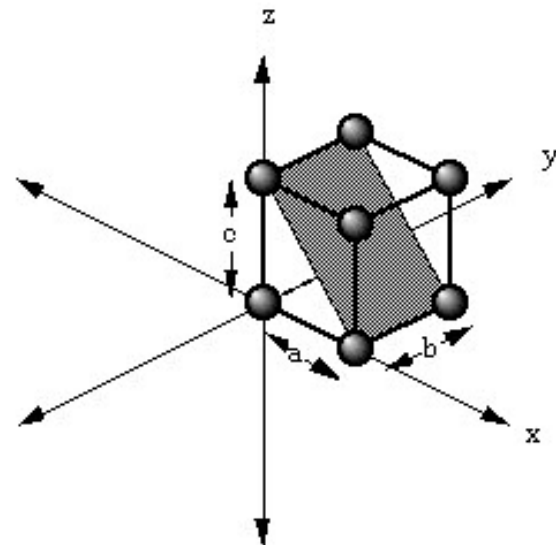
- Miller indices
 - 布拉格定律(Bragg's Law)
 - Scherrer's Formula
 - Data Analysis Examples
-

Miller Indices

- **Miller Indices** are a symbolic vector representation for the orientation of an atomic plane in a crystal lattice and are defined as the reciprocals of the fractional intercepts which the plane makes with the crystallographic axes.
 - (hkl) = parenthesis designate a **crystal face** or a **family of planes** throughout a crystal lattice.
-



	a	b	c
intercept length	1	1	1
reciprocal	$\frac{1}{1}$	$\frac{1}{1}$	$\frac{1}{1}$
cleared fraction	1	1	1
Miller indice	(111)		



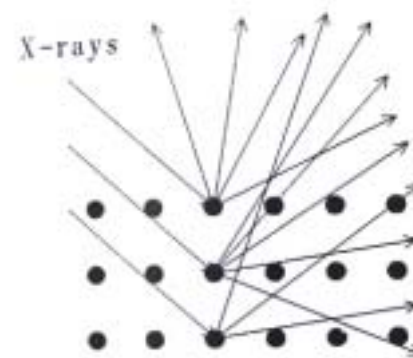
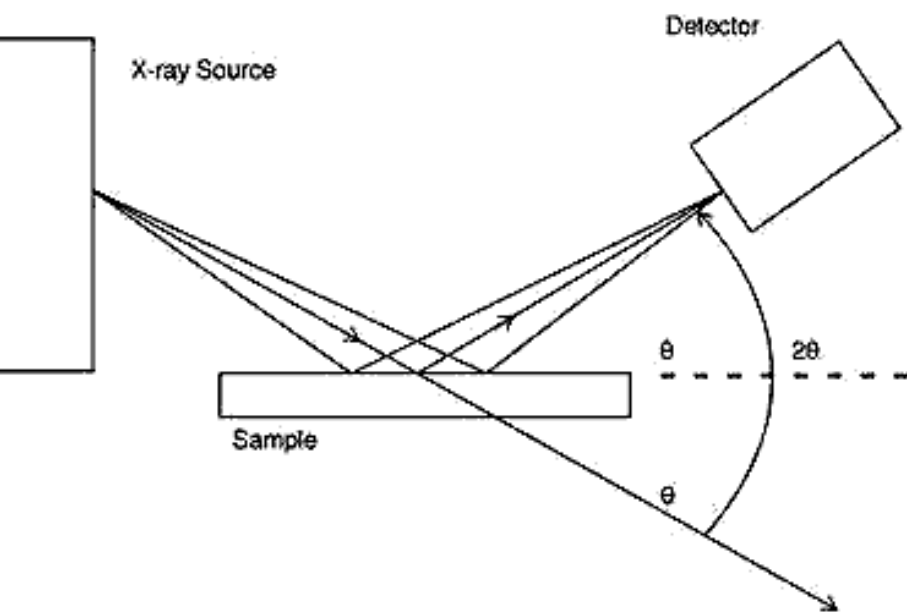
	a	b	c
intercept length	1	∞	1
reciprocal	$\frac{1}{1}$	$\frac{1}{\infty}$	$\frac{1}{1}$
cleared fraction	1	0	1
Miller indice	(101)		

XRD

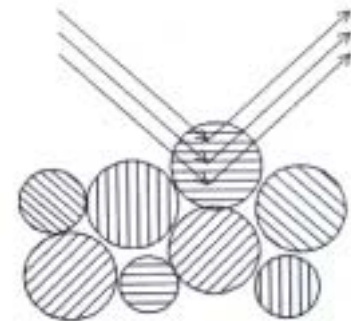
Energy source: X-Ray

Cu K 8.04 keV, $\lambda = 0.154$ nm

當X-ray透過具晶格結構之材料時，產生建設性干涉(constructive interference)



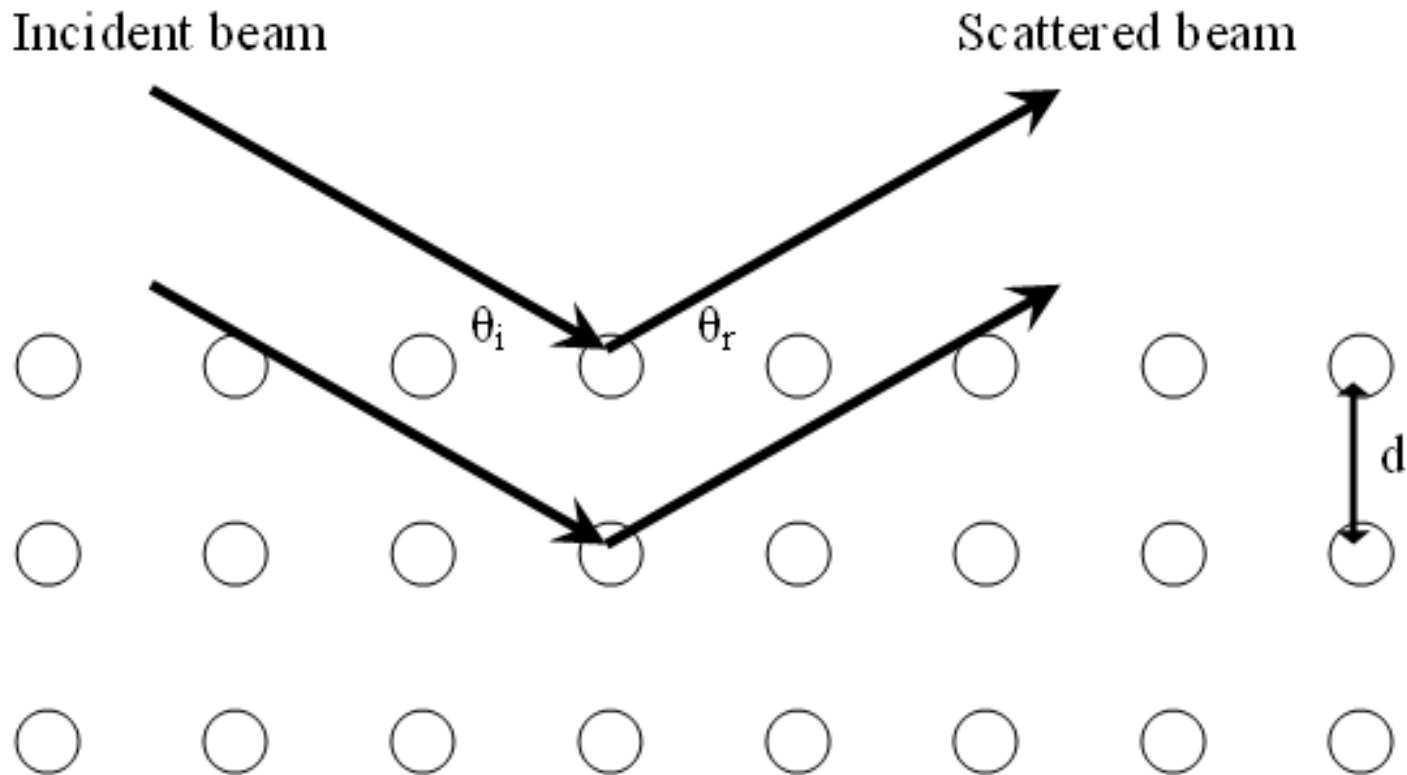
$$n \lambda = 2 d \sin \theta$$



Bragg's Law

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

- Constructive interference only occurs for certain θ 's correlating to a (hkl) plane, specifically when the path difference is equal to n wavelengths.



Summary of Bragg & Laue

- When a diffraction condition is met there can be a reflected X-ray

- Extra atoms in the basis can suppress reflections

- Three variables λ , θ , and d

- λ is known
- θ is measured in the experiment (2θ)
- d is calculated

- From the planes (hkl)

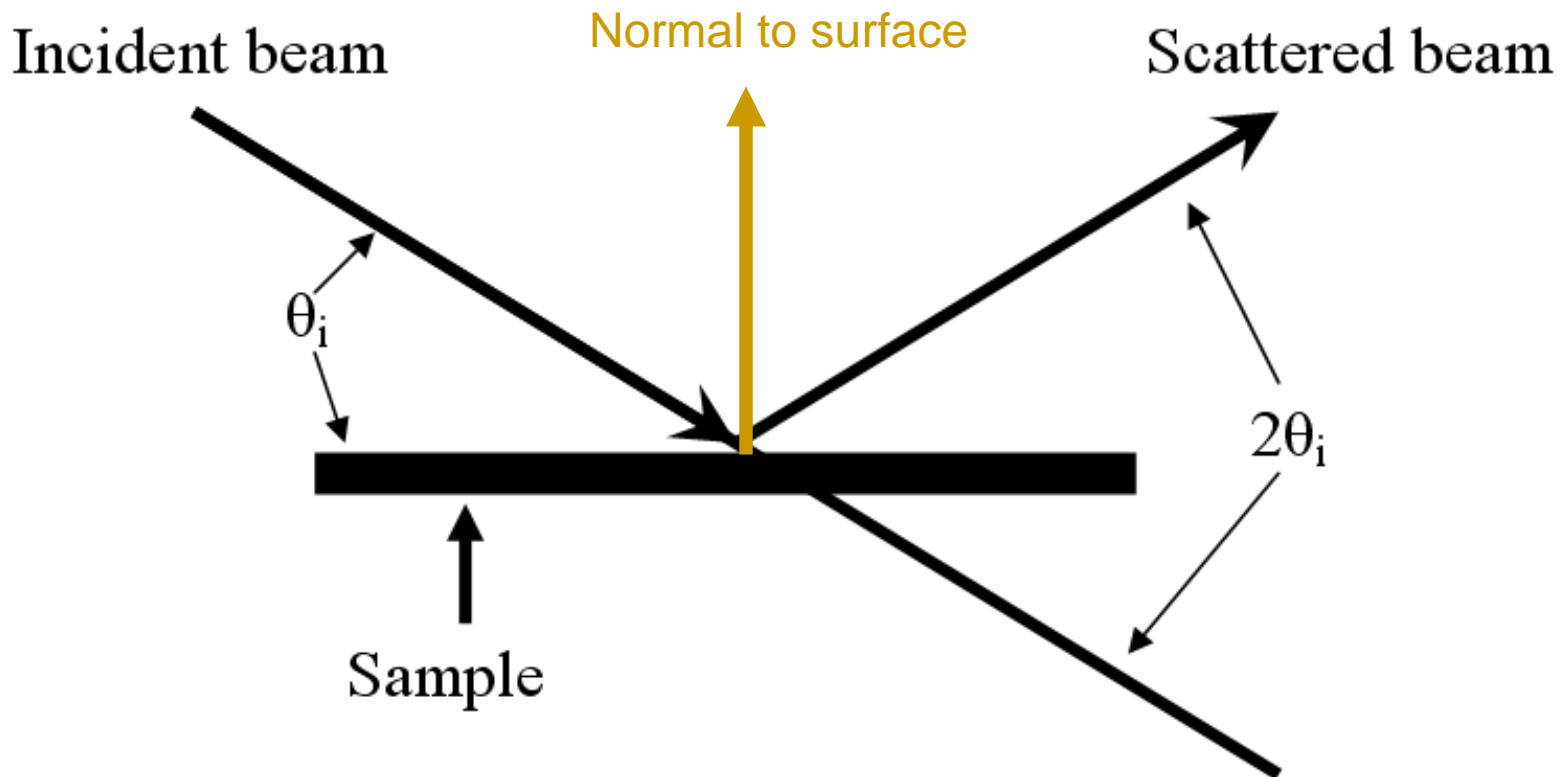
- ~~a is calculated~~

$$d = \frac{n\lambda}{2\sin\theta}$$

$$a = d\sqrt{h^2 + k^2 + l^2}$$

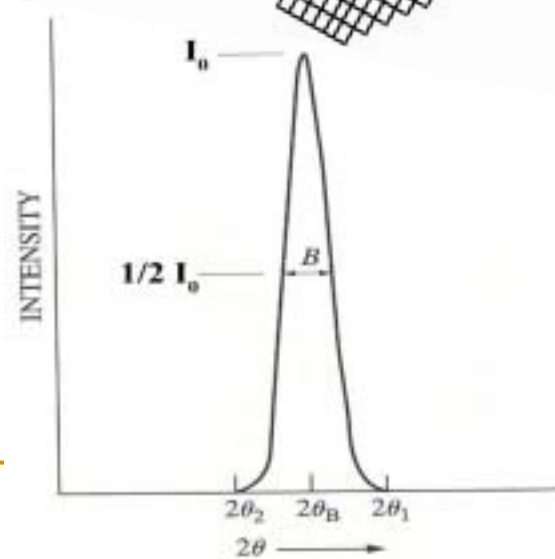
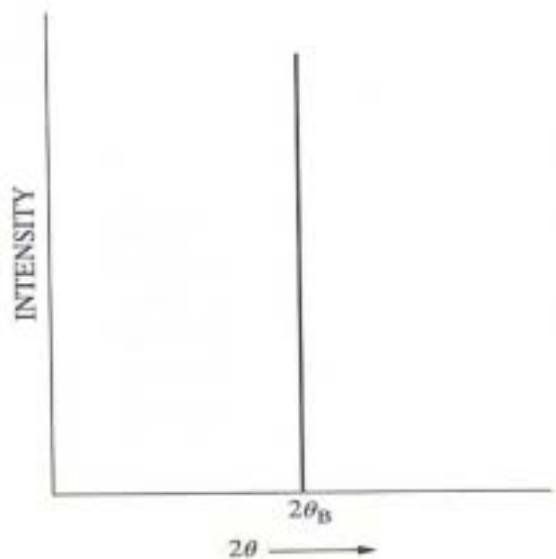
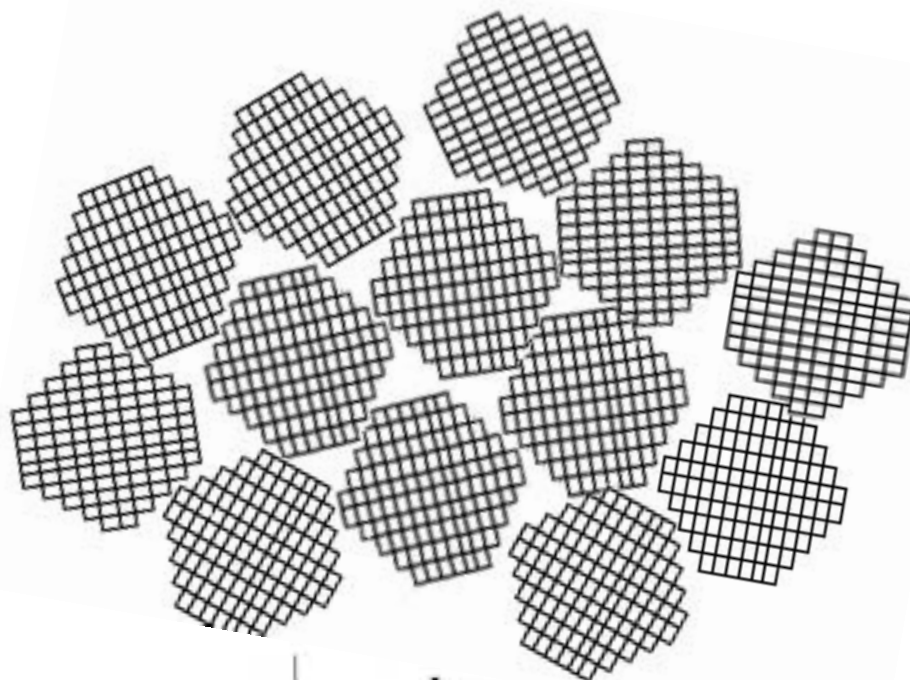
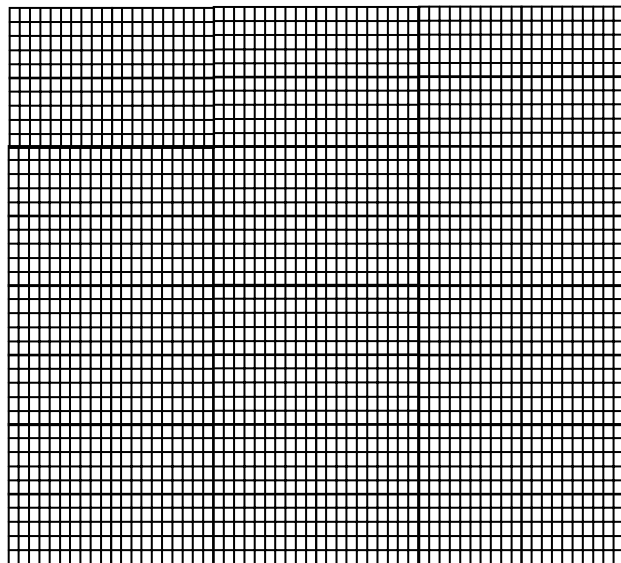
- 2 Scan

The - 2 scan maintains these angles with the sample, detector and X-ray source



Only planes of atoms that share this normal will be seen in the - 2 Scan

Smaller Crystals Produce Broader XRD Peaks



Scherrer's Formula

$$t = \frac{K * \lambda}{B * \cos \theta_B}$$

t = thickness of crystallite

K = constant dependent on crystallite shape (1-0.89)

λ = x-ray wavelength

B = FWHM (full width at half max) or integral breadth

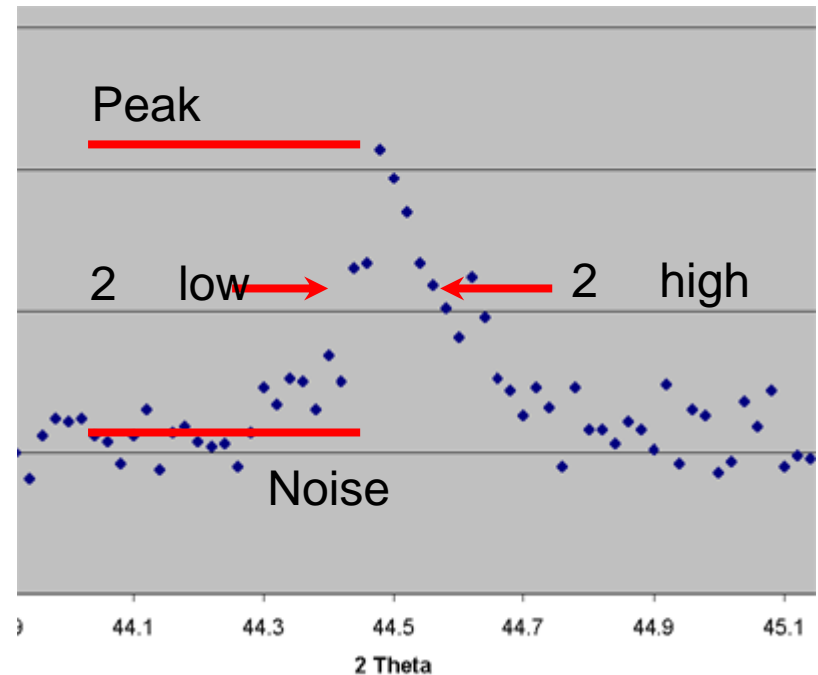
θ_B = Bragg Angle

Scherrer's Formula

What is B ?

$$B = (2 \theta_{\text{High}}) - (2 \theta_{\text{Low}})$$

B is the difference in angles at half max



When to Use Scherrer's Formula

- Crystallite size $< 1000 \text{ \AA}$
- Peak broadening by other factors
 - Causes of broadening
 - Size
 - Strain
 - Instrument
 - If breadth consistent for each peak then assured broadening due to crystallite size
- K depends on definition of t and B
- Within 20%-30% accuracy at best

Scherrer's Formula References

Corman, D. Scherrer's Formula: Using XRD to Determine Average Diameter of Nanocrystals.

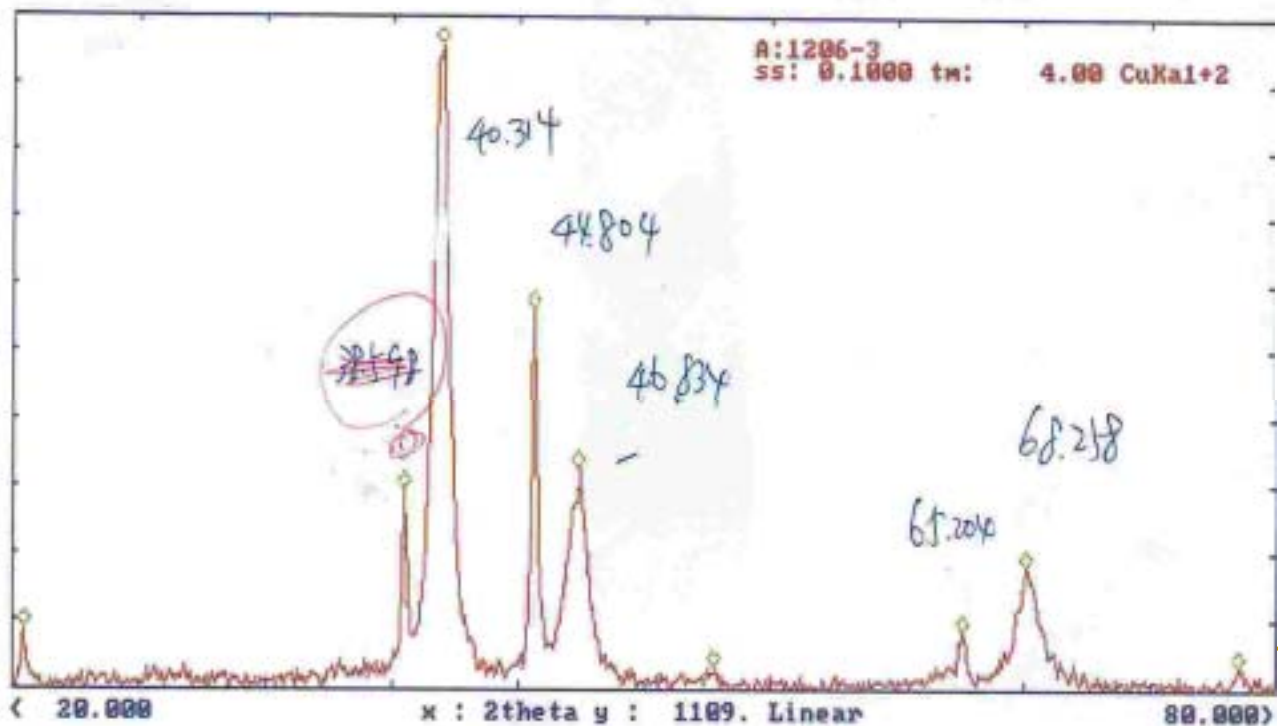
Data Analysis

- Plot the data (2θ vs. Counts)
 - Determine the Bragg Angles for the peaks
 - Identification of samples
 - International Centre for Diffraction Data (ICDD)- Joint Committee on Powder Diffraction Standards (JCPDS). (資料庫, 很貴)
 - X Ray Spacing (<http://webmineral.com/X-Ray.shtml>) 免費, 但查文獻
 - Calculate d and a for each peak
 - Apply Scherrer's Formula to the peaks
-

Sample: A:1206-3
Data file: B:1206-3.RAW

8-Dec-2005 16:03:52

Seq	2theta	d	rel. I	Seq	2theta	d	rel. I
1	20.442	4.3419	8.99	3	40.314	2.2358	100.00
2	38.598	2.3312	30.77	4	44.804	2.0216	58.64
3	40.314	2.2358	100.00	5	46.834	1.9386	34.01
4	44.804	2.0216	58.64	2	38.598	2.3312	30.77
5	46.834	1.9386	34.01	8	68.258	1.3732	18.61
6	53.300	1.7177	3.52 ?	1	20.442	4.3419	8.99
7	65.204	1.4299	8.83	7	65.204	1.4299	8.83
8	68.258	1.3732	18.61	6	53.300	1.7177	3.52 ?
9	78.341	1.2198	3.30	9	78.341	1.2198	3.30



<http://webmineral.com/X-Ray.shtml>

X Ray Spacing

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Minerals Arranged by X-Ray Powder Diffraction

Powder X-ray Diffraction (XRD) is one of the primary techniques used by mineralogists and solid state chemists to examine the physico-chemical make-up of unknown solids. This data is represented in a collection of single-phase X-ray powder diffraction patterns for the three most intense D values in the form of tables of interplanar spacings (D), relative intensities (I/I₀), hkl plane, mineral name and chemical formul.

The XRD technique takes a sample of the material and places a powdered sample in a holder, then the sample is illuminated with x-rays of a fixed wave-length and the intensity of the reflected radiation is recorded using a goniometer. This data is then analyzed for the reflection angle to calculate the inter-atomic spacing (D value in Angstrom units - 10⁻⁸ cm). The intensity(I) is measured to discriminate(using I ratios) the various D spacings and the results are compared to this table to identify possible

X Ray Spacing

D1(I _o)(hkl)	D2(I/lo)(hkl)	D3(I/lo)(hkl)	Mineral Name	Chemical Formula
0.7768(1)	0.7877(1)	3(1)	<u>Bowieite</u>	(Rh,Ir,Pt)1.77S3
0.779(1)	2.18(1)	0.8237(0.9)	<u>Sudburyite</u>	(Pd,Ni)Sb
0.7874(1)	0.8623(0.8)	0.8847(0.7)	<u>Rhodium</u>	(Rh,Pt)
0.8666(1)	2.122(1)(110)	0.8021(0.7)(321)	<u>Skaergaardite</u>	! CuPd
1.019(1)	1.081(0.75)	1.887(0.63)	<u>Nowackiite</u>	Cu6Zn3As4S12
1.019(1)	1.757(1)	1.91(0.8)	<u>Kalininite</u>	ZnCr2S4
2.246(1)	1.945(0.42)	1.376(0.25)	<u>Palladium</u>	Pd,Pt

回家作業(II)

1. 決定是何物質?
2. 計算粒徑大小

