

Quiz 3 4/13/01 XRD

The diffractometer in lab has a scintillation detector and a graphite monochromator attached to the end of an arm that scans diffraction angle. The detector is about 5 inches from the sample. *The incident beam passes through two slits near the tube, a small slit then a larger slit and the detector also has two slits first a large and then a small.* The sample is in reflection geometry (i.e. you can consider it a mirror for the purpose of optics) and rotates at an angle θ while the detector rotates at an angle 2θ .

- a) **-What** is a scintillation detector.
-Why does it require a high voltage supply?
- b) **-Explain** how a crystal monochromator works including the equation that govern its use.
- c) If the arm of the diffractometer were longer, the diffraction angle could be resolved to a higher degree of accuracy.
-Give 2 functions that explain the disadvantage of a longer arm.
- d) **-Sketch** the slit arrangement of the diffractometer (*described above*) and explain why the distance from the tube to the sample must equal the distance from the sample to the detector for matched slits by sketching the limits of the possible paths of the beam through the slits (a ray diagram).
-What is the smallest spot that can be resolved on the detector from this diagram?
-How does this explain the chosen distance of about 6 inches from sample to detector?
- e) **-Explain** the meaning of the following 4 expressions:
[100]
{100}
<100>
(100)
-For two planes (planes of a zone) how is the line of intersection (the zone axis) calculated?

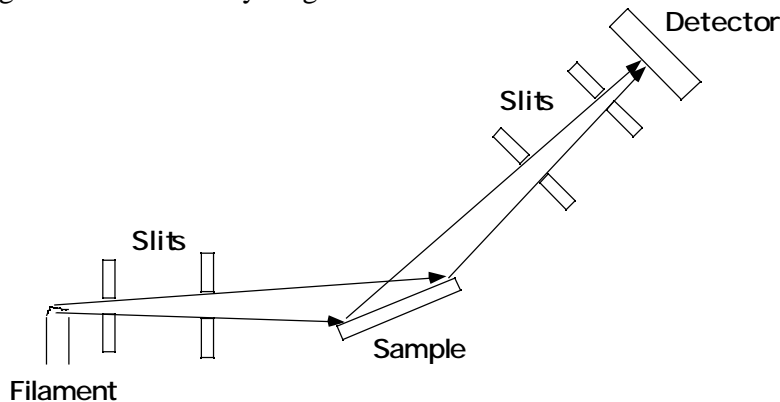
Answers: Quiz 3 4/13/01 XRD

a) Figure 6-21 on page 207 shows the scintillation detector. x-rays are incident on a scintillating film of NaI (doped with Thallium Tl^+). The film is behind an aluminum sheet that prevents light from entering the detector. The scintillator gives off violet light when exposed to x-rays. Abutting the scintillator is a PMT that is composed of a cesium antimony crystal which is a photocathode. The light from the scintillator causes the photocathode to emit electrons in a vacuum chamber away from the x-ray source. The electrons fall down a potential gradient towards a slalom of dynode plates of increasing voltage drop (about 200 V per plate with about 10 dynode plates). The impact of the high potential electron with the plate leads to ejection of many electrons (4 to 5 per incident electron). The signal (current) is amplified by about $1e7$ in the dynode cascade. A high voltage supply is needed to give the potential drop across the dynodes.

b) The crystal monochromator is governed by Bragg's law, $d = 2 / \sin \theta$. When a polychromatic beam is incident on a perfect crystal the reflections of various wavelengths from a given crystallographic plane are spatially resolved in angle. That is if d is fixed then θ can be resolved in λ . By the use of slits to select a fixed incident angle and fixed exiting angle, a single wavelength of very narrow breadth in wavelength can be selected. The breadth in wavelength is decided by the goodness of the collimation.

c) The two functions are $I = k/r^2$ (one by r -squared decay) and Beer's law, $I = I_0 \exp(-\mu r)$, where μ is the absorption coefficient of air. Also, if a longer path were chosen and the sample remained of the same size then the slits would have to be much narrower as shown in question d. This would also lead to a dramatic loss in intensity.

d) Figure 6.1 page 187 shows this ray diagram.



The smallest spot that can be imaged is the filament size as viewed from the sample. This limits the useful angle resolution at the detector so determines the distance of the arm.

e) $[100]$ is the direction 100 in the unit cell which is along the "a" crystallographic axis.
 $\{100\}$ is the family of planes of the type (100) , so (100) , (010) , (001) for a cubic lattice.
 $\langle 100 \rangle$ is the family of directions of the type $[100]$ so $[100]$, $[010]$, $[001]$ for a cubic lattice.
 (100) are the Miller indices for the plane (100) . This is the plane made by the b-c axis.

If the two planes are (h_1, k_1, l_1) and (h_2, k_2, l_2) , then the direction of the line is given by $[u, v, w]$, where uvw are given by,

$$u = k_1 l_2 - k_2 l_1$$

$$v = h_1 l_2 - h_2 l_1$$

$$w = h_1 k_2 - h_2 k_1$$