

University of Toronto
ADVANCED PHYSICS LABORATORY

LAUE

Laue Back-Reflection of X-Rays

Revisions:

April, May 2016	David Bailey < dbailey@physics.utoronto.ca >
October 2013	P. Krieger (for use of medical X-ray film)
August 2006	Jason Harlow, with suggestions from student Jerod Wagman
September 1994	Derek Paul
June 1988	John Pitre

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INTRODUCTION

In 1912, Max von Laue used X-ray diffraction from crystals to prove that X-rays were very short wavelength electromagnetic radiation, for which he was awarded the Nobel Prize in Physics in 1914. In 1915, W. H. Bragg and W.L. Bragg were awarded the Nobel Prize in Physics for “the analysis of crystal structure by means of X-rays”. X-ray scattering is now one of the most powerful methods for understanding the structure of materials, from high temperature superconductors to proteins, and a key tool for Nobel winning discoveries ranging from the DNA double helix to quasicrystals.¹

This experiment uses the Laue method for identifying the type and orientation of single crystals. Another Advanced Lab experiment uses the Powder Method² to measure the crystal lattice spacings of a target consisting of many randomly oriented small crystals.

Production of X-Rays

When electrons of a few tens of keV are incident on the metal anode target of an X-ray tube, about 2% of their energy is converted into X-rays and the balance is converted into heat in the target. As shown in Figure 1, the radiation consists of a continuous spectrum spread over a wide band of wavelengths and a “characteristic” spectrum of superimposed lines. The former is the analogue of white light and is frequently called white radiation. The latter corresponds to monochromatic light, and is produced when the electron beam has sufficient energy to knock tightly bound electrons out of atoms, producing atomic spectral x-radiation that is characteristic of the metal emitting the rays. The continuous spectrum can be produced without characteristic radiation if the tube is operated at low voltage, but as soon as the voltage is increased beyond a critical value, the characteristic spectral lines appear in addition to the white radiation.

For this experiment we require white radiation so we use an X-ray tube with a tungsten target and accelerating voltages that are never greater than 50 kV, below the K excitations of tungsten. The maximum energy of a photon produced by an electron of charge e accelerated through a voltage V in volts is given by

$$hv_{\max} = \frac{hc}{\lambda_{\min}} = eV \quad (1)$$

where h is Planck's constant, c is the speed of light and v_{\max} is the highest frequency X-ray produced (which has the shortest wavelength λ_{\min}). Substitution of values for h , e and c gives

$$\lambda_{\min} = \frac{1239.8}{V} \quad (2)$$

where λ_{\min} is given in nm . We use voltages between 10 and 50 kV so that $\lambda_{\min} \leq 0.1 nm$, which is a wavelength short enough to determine crystal structures with unit cell dimensions typically a fraction of a nanometre. The material outlined here is discussed in Cullity Ch. 1.

¹ <http://www.iucr.org/people/nobel-prize>

² <http://www.physics.utoronto.ca/~phy326/pxr>.

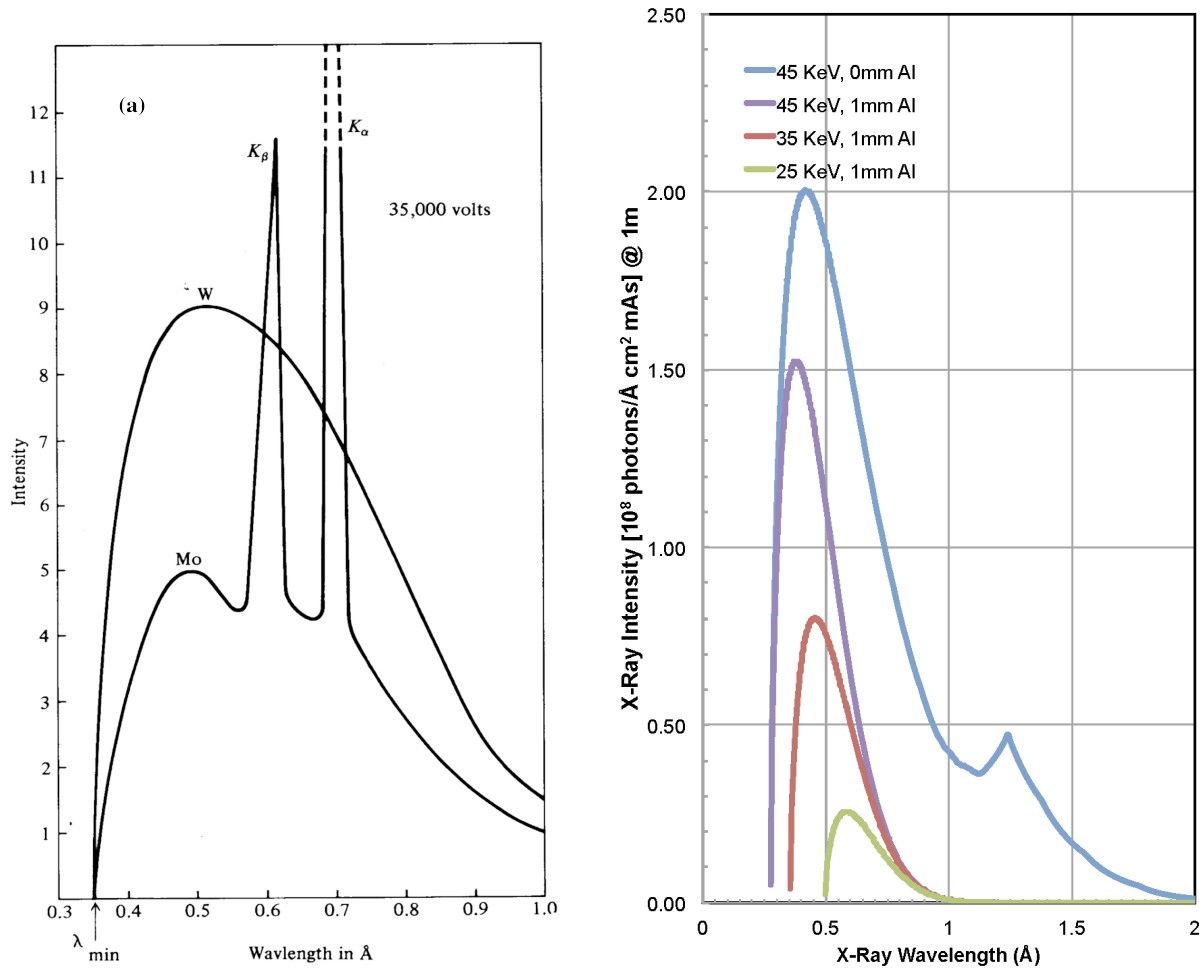


Figure 1: X-ray Spectra for (a) 35 KeV electrons striking tungsten or molybdenum Targets (from Semat, with arbitrary intensity scale), and for (b) 25, 35, and 45 KeV electrons striking tungsten with (and in one case without) an Al filter on the X-Ray beam.³ The short-wavelength limit for X-ray emission is set by the electron beam energy, but the position of the peak and shape of the long wavelength tail are determined by whatever material the x-rays have to traverse on the way to the crystal.

Diffraction of X-Rays by Crystals

The condition for constructive interference of the scattered X-rays is that the path difference for waves reflected from two consecutive surfaces must be an integral number of wavelengths. Bragg's law gives the relation between the X-ray wavelength λ and the interplanar distance d :⁴

$$2 d \cos \theta = n \lambda \quad (3)$$

where n is the order of the interference.

³ Calculated using SpekCalc (<http://spekcalc.weebly.com>). The calculation assumes the X-rays are emitted at an angle of 6° from the anode surface, and they pass through 20cm of air, 0.8mm of Be, and 1mm of C.

⁴ This is the “cos” version of Bragg’s Law with θ defined relative to the plane normal; be careful when reading the literature not to be confused if the “sin” version is used with θ defined relative to the plane surface.

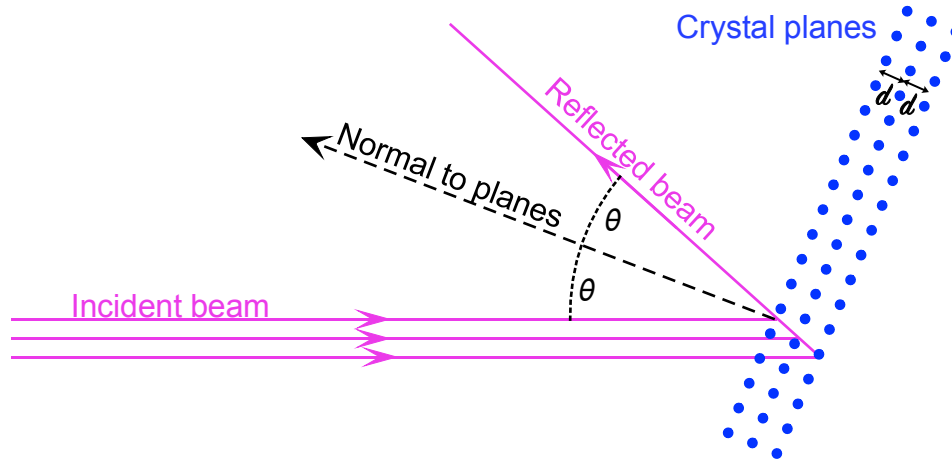


Figure 2: Diagram for Bragg Reflection.

Observing forward transmission diffraction requires thin crystals. This experiment uses backwards reflection so thick crystals can be studied.

Question: *Approximately how thin should a Tungsten crystal be for Laue forward transmission diffraction with 30KeV X-rays?⁵ To avoid degradation in reflection diffraction, surface imperfections must be small compared to this same thickness.*

The interplanar distance d depends upon the lattice constants and the Miller indices h, k, l of the crystal plane (see Wood, p.8). For example, in a cubic crystal,

$$d = \frac{a}{(h^2 + k^2 + l^2)^{1/2}} \quad (4)$$

and the expressions for other crystal systems are given in Wood p. 13.

Equations (3) and (4) are relevant in understanding which spots are visible and their intensity, since as shown in Figure 1 the X-ray intensity varies with λ . (Because the X-ray spectrum peaks at wavelengths smaller than typical values for a , higher order reflections often dominate the intensity of a Laue spot.) The smallest observable interplanar distance, corresponding to $\cos\theta=n=1$ in Equation (3), is the resolution limit $d_{\min}=\lambda_{\min}/2$. For a cubic crystal, this limits the observable planes to those with

$$h^2 + k^2 + l^2 < \left(\frac{2a}{\lambda_{\min}} \right)^2 \quad (5)$$

and sets a minimum scattering angle given by

$$\sin\theta_{\min} = \frac{\lambda_{\min}}{2a} \quad (6)$$

Equation (5) explains why the infinite number of possible hkl planes produce only finite number of spots on a Laue photograph.

⁵ e.g. See http://henke.lbl.gov/optical_constants/atten2.html or <http://physics.nist.gov/PhysRefData/XrayMassCoef/ElemTab/z74.html>.

Laue Back Reflection Method

In this method a beam of white X-rays is incident on a stationary single crystal. For each crystal plane in a particular orientation, d and θ are fixed; thus, for a given n , some λ will be available in the white radiation to satisfy Equation (3) producing a Laue spot in a direction making an angle 2θ with the incident X-ray beam as shown in Figure 2. Since d and θ are fixed, all orders of interference n , for a given crystal plane will be superimposed on the same spot. (Note: n is usually subsumed in h,k,l , i.e. $(hkl) = (222)$ is the (111) $n=2$ reflection.) The symmetry of the Laue pattern corresponds to the symmetry of the crystal, and directions of the crystal axes are determined by the symmetry axes of the Laue pattern. The information given here is expanded in Wood pp. 28-31 and Cullity Ch. 5.

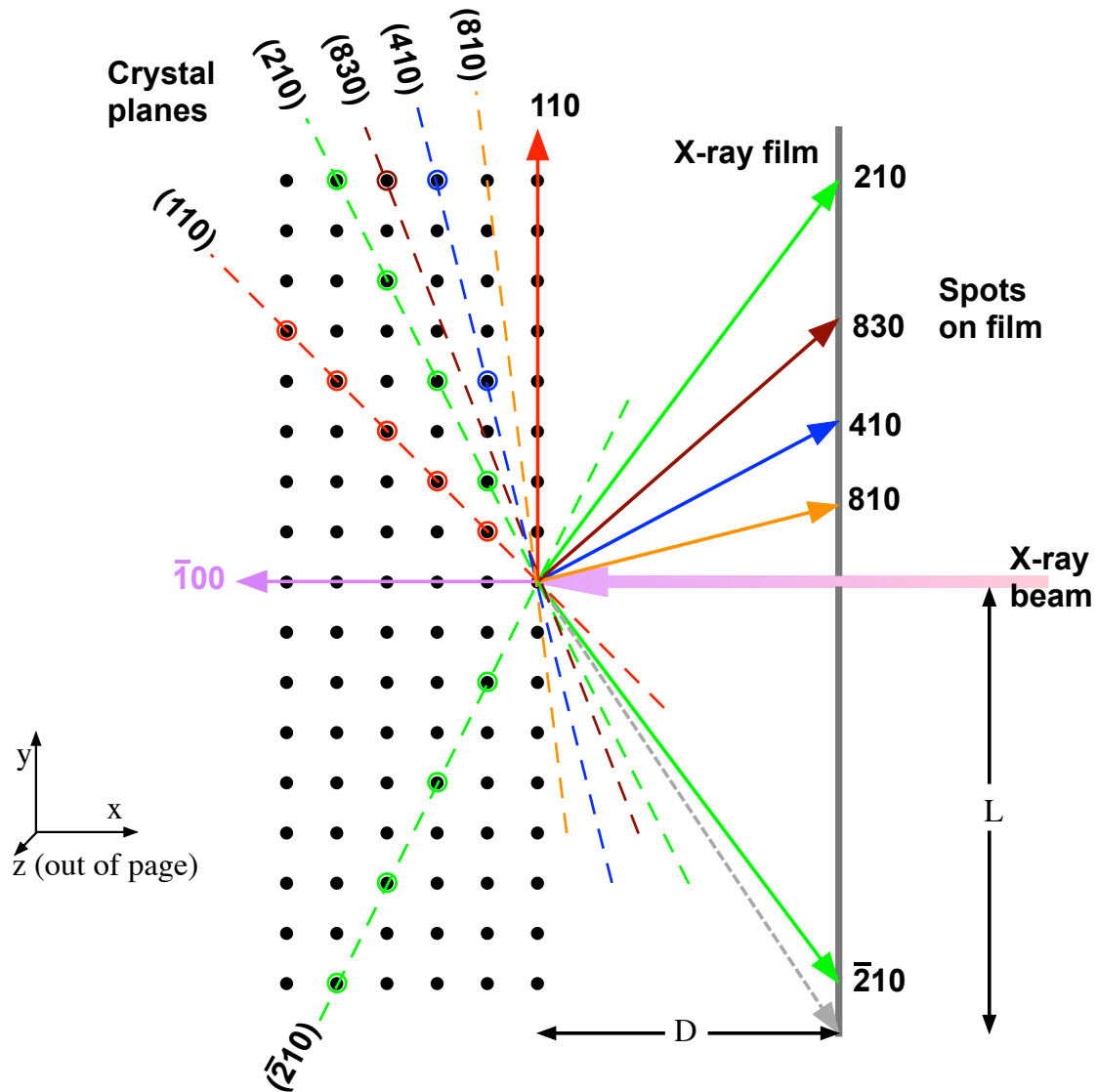


Figure 3: Plan view of simple cubic lattice (space group $Pm\bar{3}m$). A few planes and their reflections are shown. (A bar over a number implies a negative direction.)

When a crystal is oriented along an axis having a high degree of symmetry, it is often possible to identify the symmetry and all the spots with ease. Suppose, for example, you have a

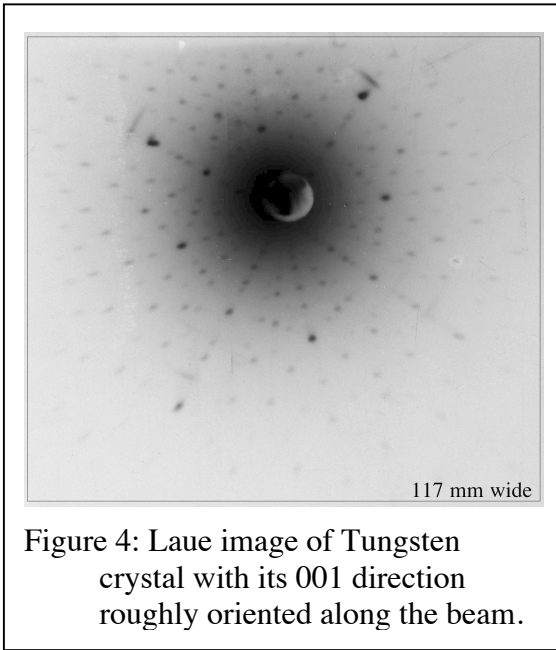


Figure 4: Laue image of Tungsten crystal with its 001 direction roughly oriented along the beam.

tungsten crystal aligned with a (100) plane perpendicular to the incident X-ray beam. The Laue spots will lie on a series of lines passing through the point where the X-ray beam crossed the photograph. These lines form a symmetric pattern which is predictable if you draw the two-dimensional lattice representing the front (100) face of the crystal. These lines and the symmetries are easily seen even if the crystal is slightly misaligned as seen in Figure 4. Each line is called a *zone* which consists of all the spots generated by reflections from planes that share a common axis, i.e. the $[uvw]$ zone consists of all planes (hkl) that satisfy

$$h \cdot u + k \cdot v + l \cdot w = 0 \tag{7}$$

In Figure 3, for example, all the diffractions shown belong to the $[001]$ zone since they all reflected from planes that are aligned with the z axis. Their spots form a straight line, as will the spots belonging to any zone whose axis is perpendicular to the X-ray beam axis. Reflections belonging to zones that are not perpendicular the beam form a cone, producing spots that lie on an hyperbola on the film as shown in Figure 5.

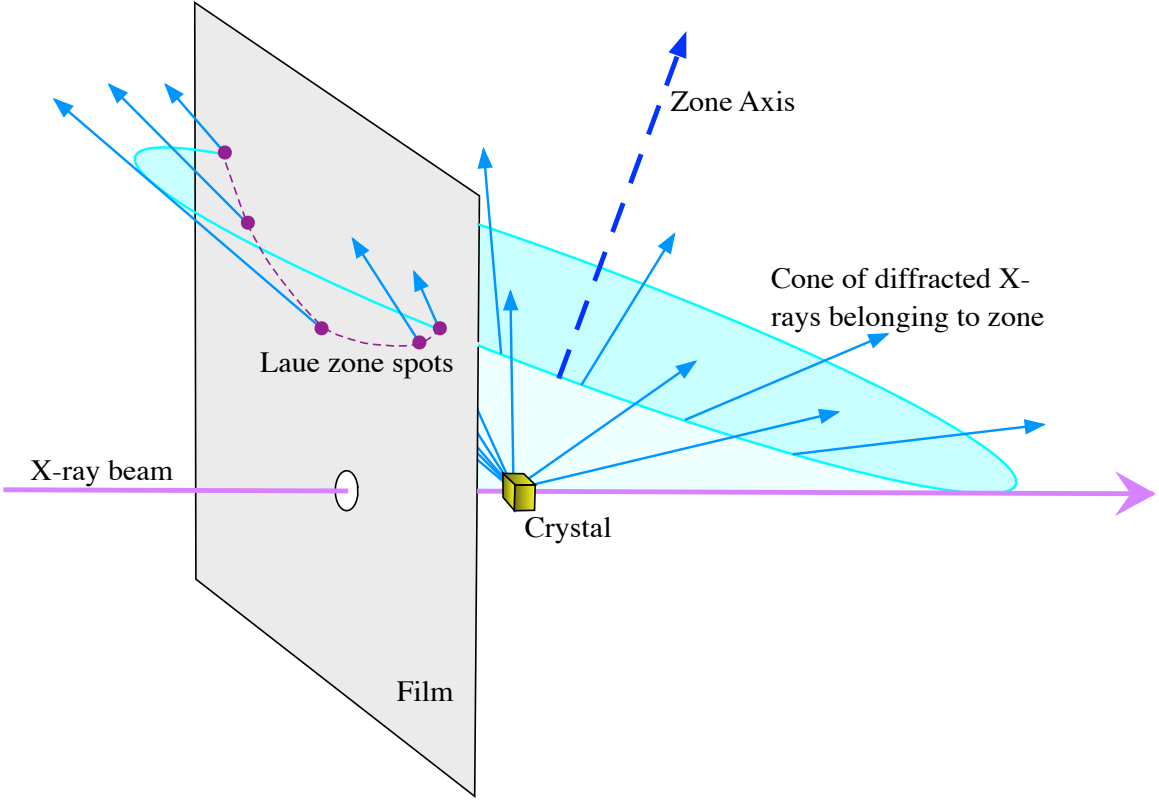


Figure 5: Cone of reflections corresponding to a zone axis.

Both these traight and curved zones are visible in Figure 4.

It is easy in crystallography, however, to make a mistake. If one thinks one is looking at the (100) orientation of a cubic crystal, but important zones are missing from a Laue image, or if the symmetries are wrong,⁶ or if the spot do not match the positions calculated by Equation (12), then the hypothesis of (100) orientation must also be in error, or maybe the structure isn't cubic.

Crystal structures

Crystallography is a large complicated subject. In classical crystallography, there are 7 crystal systems⁷ (cubic, tetragonal, orthorhombic, rhombohedral, hexagonal, monoclinic, and triclinic), 14 Bravais lattices (e.g. cubic systems have 3: simple, body-centred, face-centred), and 230 symmetry groups⁸ (e.g. there are 10 body-centred cubic symmetry groups). Most commonly a symmetry group is referred to by its short name or number. For example, most pure elements (including Tungsten) with body-centred cubic (bcc) crystal structures have symmetry group $Im\bar{3}m$ (#229) and a simple 2 atom unit cell. Manganese also has a bcc structure, but with symmetry group $I43m$ (#217) and a 58 atom unit cell! CaF_2 is a face-centred cubic (fcc) $Fm\bar{3}m$ (#225) crystal with a 4 Ca + 8 F unit cell. Multi-element crystals, especially biological macromolecules, can have very complicated structures well beyond the scope of this experiment.

Diffraction intensities

The size, intensity, and shape of a Laue spot depends on many factors, including the spectral intensity and angular dispersion of the X-ray beam, the perfection of the crystal, the thermal motion of the atoms in the crystal planes, the spectral absorption of beam and diffracted X-rays in the crystal, the angle of the spot,.... Only a few of these factors are discussed here; for more detail, see Cullity Ch. 4 and Preuss Ch. 2.3.

X-rays primarily scatter from electrons (because they are so much lighter than nuclei), so we start with the intensity for Thompson scattering of unpolarized electromagnetic radiation from a free electron:

$$I_e(\theta, \lambda) = I_0(\lambda) \frac{e^4}{m^2 c^4 R^2} \left(\frac{1 + \cos^2 2\theta}{2} \right) \quad (8)$$

where $I_0(\lambda)$ is the X-ray beam intensity (e.g. as shown in Figure 1), e and m are the electron charge and mass, and R is the distance from the electron at which the intensity is measured.

Electrons in a crystal are not free, however, so the scattering by a unit cell in a crystal is parameterized by the structure factor :

$$I_{\text{unit cell}}(\theta, \lambda) = I_e(\theta, \lambda) \times |F_{hkl}|^2 \quad (9)$$

where the structure factor F_{hkl} depends on the atomic scattering form factor f_n and location (x_n, y_n, z_n) of the atoms within the unit cell.

$$F_{hkl} = \sum_{\text{atoms in unit cell}} f_n e^{2\pi i(hx_n + ky_n + lz_n)} \quad (10)$$

⁶ E.g. See Preuss p. 102-124 for the symmetries expected for Tungsten crystals. Note that Preuss shows stereographic projections, not image patterns.

⁷ A reasonable quick reference is https://en.wikipedia.org/wiki/Crystal_system

⁸ https://en.wikipedia.org/wiki/List_of_space_groups

Depending on the structure of the unit cell, F_{hkl} may be zero. For example, a simple body-centred cubic (bcc, space group Im3m, #229) structure has two atoms in each unit cell, one at (0,0,0) and the other at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, so if both atoms are identical with $f_n=f$, then

$$\begin{aligned} F_{hkl} &= f \left[e^{2\pi i(h \cdot 0 + k \cdot 0 + l \cdot 0)} + e^{2\pi i(h/2 + k/2 + l/2)} \right] \\ &= f \left[1 + e^{i\pi(h+k+l)} \right] \\ &= \begin{cases} 0 & \text{if } h+k+l \text{ odd} \\ 2f & \text{if } h+k+l \text{ even} \end{cases} \end{aligned} \quad (11)$$

Similar reflection condition rules can be found for other crystal structures.

Question: What hkl values are allowed for face-centred cubic (fcc) crystals? See Cullity Ch. 4.6 or Preuss Ch. 3.⁹

Remember, however, that the intensity of a Laue spot is the sum of all the diffractions with the same scattering angle θ , so a nominally forbidden odd hkl Laue spot will still be observed for a bcc crystal, albeit with weaker intensity. For example, if $a=3.5\text{\AA}$ and $\lambda_{\min}=0.35\text{\AA}$ then Equation (5) requires $h^2 + k^2 + l^2 < 400$, so the observed nominal (120) spot for a bcc crystal will be the sum of (240) + (480) + (6 12 0) + (8 16 0) reflections. This is also why lower index spots tend to be brighter than high index spots. In this case, the nominal (110) spot will be the sum of 14 reflections – (110)+(220)+...+(14 14 0), while the (970) spot will have only one reflection, since the next higher reflection (18 14 0) violates the requirement that $h^2 + k^2 + l^2 < 400$.

Intensities can be reduced if a crystal is too perfect. Real crystals have a mosaic structure composed of tiny ($\sim 1000\text{\AA}$) domains that are slightly rotated by a very small angle with respect to each other. (The angle can be as large as 1° , but is usually much, much smaller.) This is actually a good thing, since “extinction” can reduce the intensity of the diffraction in a perfect single crystal. This is where X-rays diffracted backwards and forwards at the same angle from many exactly parallel planes end up destructively interfering. The mosaic structure of a real crystal changes the angle of diffraction slightly for different domains, reducing the magnitude of this destructive interference. (See Cullity Ch. 3-7 and 14-11.)

Some computations for expected positions of spots

Very simple formulas exist for backscattering from a (hkl) plane in a cubic structure. If the (100) crystal axis is aligned with the X-ray beam that defines the x axis perpendicular to the yz plane of the film, then the y, z positions of the hkl spots will be

$$(y, z)_{hkl} = (k, l) \frac{2h}{h^2 - (k^2 + l^2)} D \quad (12)$$

The scattering angles $(2\theta, \phi)$ for a Laue spot are shown in Figure 6. The angles of the planes that produce this spot are (θ, ϕ) :

⁹ http://www.cryst.ehu.es/cryst/get_hkl.html can provide the reflection conditions for any space group, but goes beyond what you will likely need for this experiment.

$$(\theta, \phi) = \left(\tan^{-1} \frac{\sqrt{y^2 + z^2}}{D}, \tan^{-1} \frac{z}{y} \right) \quad (13)$$

The angular distance between two spots is

$$\Delta = \cos^{-1} (\cos \theta_1 \cos \theta_2 + \sin \theta_1 \sin \theta_2 \cos (\phi_1 - \phi_2)) \quad (14)$$

The angular distance between two planes is

$$\Delta = \cos^{-1} \left(\frac{(h_1 h_2 + k_1 k_2 + l_1 l_2)}{\sqrt{(h_1^2 + k_1^2 + l_1^2)(h_2^2 + k_2^2 + l_2^2)}} \right) \quad (15)$$

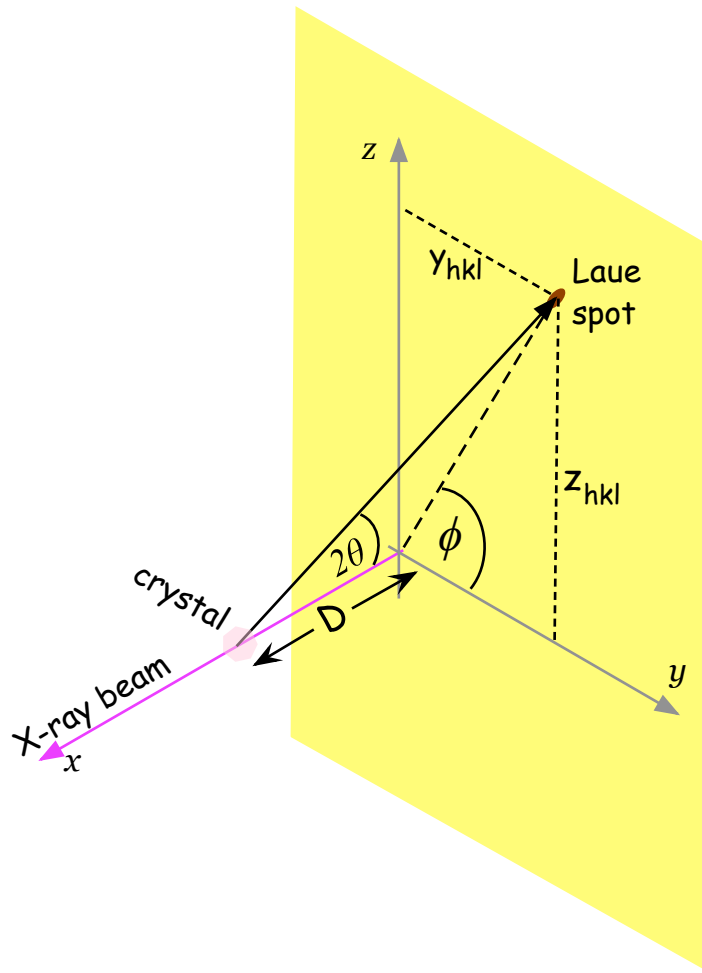


Figure 6: Laue scattering angles.

Safety Reminders

- Before using the X-ray machine, you must read the “SAFETY OPERATING PROCEDURES FOR ANALYTICAL X-RAY EQUIPMENT” posted on the X-ray machine (also available at <http://www.ehs.utoronto.ca/Assets/ehs+Digital+Assets/ehs3/rad/XRay/SOP+model.pdf>).
- An X-ray dosimeter must be worn at all times when using the X-ray machine.
- The X-ray shielding must be in place and its door closed before turning on the X-ray machine. Under no circumstances are the safety interlocks or other safety systems to be tampered or interfered with. If you have problems, contact your Demo, Professor, or Technologist.
- Make sure the water cooling outlet hose is inserted into its drain, before turning on the X-ray machine cooling water, to avoid flooding the lab.
- Before developing any film, read the MSDS sheets for the chemicals used.
- Properly dispose of any used chemicals, following the instructions of the lab technologists.

NOTE: This is not a complete list of all hazards; we cannot warn against every possible dangerous stupidity, e.g. opening plugged-in electrical equipment, juggling cryostats, Experimenters must constantly use common sense to assess and avoid risks, e.g. if you spill liquid on the floor it will become slippery, sharp edges may cut you, If you are unsure whether something is safe, ask the supervising professor, the lab technologist, or the lab coordinator. If an accident or incident happens, you must let us know. More safety information is available at <http://www.ehs.utoronto.ca/resources.htm>.

EXPERIMENT

The experiment's objectives are:

1. Make a Laue photograph of the (001) oriented tungsten crystal and answer the following questions: What is the crystal structure of tungsten? Are the spots what you expect? Are there any spots you cannot explain, or any spots that should be visible but are missing?
2. Make a Laue photograph of the (110) oriented tungsten crystal. Based on the information from this photograph, rotate the crystal so that it should be in the (111) orientation and make a new Laue photograph. How close to the (111) orientation is it?
3. After discussing the possibilities with the professor supervising this experiment, try at least one of the following exercises:
 - A. (a) Make Laue photograph of CaF_2 crystal. What is the crystal structure of CaF_2 ? What is orientation of the crystal's cleavage face?
(b) Confirm that you have correctly identified the orientation of the CaF_2 crystal by rotating it to another direction of high symmetry and making a new Laue photograph that demonstrates the expected pattern of spots.
 - B. (a) Use the Laue method to identify the crystal structure of one or more of the unknown crystals that are provided, or any crystals that you may bring to the lab. Be sure to show at least one Laue photo where you have aligned the crystal to a direction of high symmetry.

- (b) Estimate the lattice constant using Equation (5), the X-ray beam energy,¹⁰ and the highest $h^2 + k^2 + l^2$ values observed.
- C. Predict what you will see if you take a Laue photograph for a couple of the following: a sintered powder tungsten slug, a bit of metal, some magnetized iron, a rock, a piece of glass, Pick samples that you think will give qualitatively different images. Make the photographs. Do they match your prediction? If not, try to explain what you observe.
- D. Compare Laue photographs of crystals made with varied parameters, e.g. different film-crystal distances, X-ray tube voltages, X-ray beam filters, surface quality, Try to explain what you observe. For example, can you understand how the intensities of the spots change?
- E. If you have other ideas, discuss them with the supervising professor.

Radiation Monitoring and Protection

Anyone operating the X-ray machines must be wearing a radiation monitor, available from the technologist in Room 250.

Before turning on the unit, to ensure that no one is inadvertently exposed to X-rays, be certain that the cap covering the X-ray port is down or the camera is in place with the lead shutter closed, and that the large wood/lead shield box is in place with the door closed. **Note:** There is an interlock switch which renders the X-ray power supply inoperative unless the shield box is in position. The leaded glass in the window is so that the X-ray beam can be observed visually if alignment is needed.

Operating Procedure for the Norelco X-ray Machine

Turning the unit on

The X-ray unit with the green front has a tube with a tungsten target and is to be used for this experiment.

- Begin with the unit turned completely off:
- Push the lever on the electrical service box on the wall to ON.
- Open fully the tap (counter-clockwise) on the wall. This connects the cooling water. No water will flow yet.
- Push the overload reset switch upward to ON if it is down.



Figure 7: Laue X-Ray Apparatus

¹⁰ You may want to confirm the beam energy by checking what were the highest $h^2 + k^2 + l^2$ spots observed with the known tungsten crystals.

- e) Set the timer to some nominal figure (e.g. two hours) to cover the duration of the testing.
- f) Turn the key-operated line switch clockwise to ON.
- g) Push the line START button. The fluorescent panel light will come on and you will hear the cooling water flowing.
- h) Turn the KILOVOLTS and MILLIAMPERES control knobs to their full counter-clockwise position
- i) Push the X-RAY ON button. The rectifier pilot light will light, indicating that the filament circuit of each high voltage rectifier tube is receiving power. After a few seconds warm-up delay, the X-RAY light will come on, indicating that the X-ray tube high voltage circuit is receiving power.

Using the X-ray unit.

Do not exceed the maximum load rating for the tube with a tungsten target which is 50 *kV* and 20 *ma*. Lowering the voltage (*kV*) does not permit increasing the current (*ma*) above the maximum rated limit, even though the product $kV \times ma$ does not exceed the maximum power limit.

With a sample in position and all shields properly in place, apply power to the tube as follows:

- a) Turn the KILOVOLTS control knob slowly, in increments of 5 *kV*, to the desired value as indicated on the KILOVOLTS meter.
- b) Turn the MILLIAMPERES control knob slowly, in increments of 5 *mA*, to the desired value as indicated on the MILLIAMPERES meter.

Turning the unit off

- a) Turn the MILLIAMPERES control knob fully counter-clockwise to a minimum.
- b) Turn the KILOVOLTS control knob fully counter-clockwise to a minimum.
- c) Push the X-RAY OFF button.
- d) Push the LINE OFF button.
- e) Turn the key operated line switch clockwise to OFF.
- f) TURN OFF THE COOLING WATER TAP ON THE WALL (CLOCKWISE).
- g) Push the lever on the grey electrical service box on the wall to OFF.

Measurements

Make Laue Photographs of Oriented (001) and (110) Tungsten Crystals

Do not touch the faces of the tungsten crystals. They have been etched clean.

1. Uncover the X-ray port and set the filter holder to OPEN (no filter in the beam) as shown in Figure 8a.
2. Load the film into the camera as instructed by the lab technologist. This must be done in the darkroom in MP227. The film has photographic emulsion on only one side, and this side must be held firmly against the white Calcium Tungstate image intensifier in the camera to avoid blurring the spots. This image intensifying screen converts X-rays that pass through the film into fluorescent light that contributes to the intensity of the spot. (See Cullity p.142.)

Note the position of the notched corner of the film, otherwise you may reverse positive and negative horizontal spot positions when analysing the image, causing confusion.

Question: *What will be the approximate diameter of the X-ray beam hitting the crystal?*
(Hint: Before loading the film, look down the collimator tube with the collimator shutter open.)

3. Place the camera in the position as shown in Figure 8b and Figure 9. The camera collimator tube should fit snugly against the tube port as shown. If the collimator and the port do not appear to be properly aligned, contact the Lab Technologist to realign the track. **Do not change the alignment of the track yourself**; it is tricky, and if misaligned you will get poor results. The collimator shutter is shown in Figure 8b in the open position that allows X-rays to pass through the collimator; if the shutter is closed, your photos will be blank.

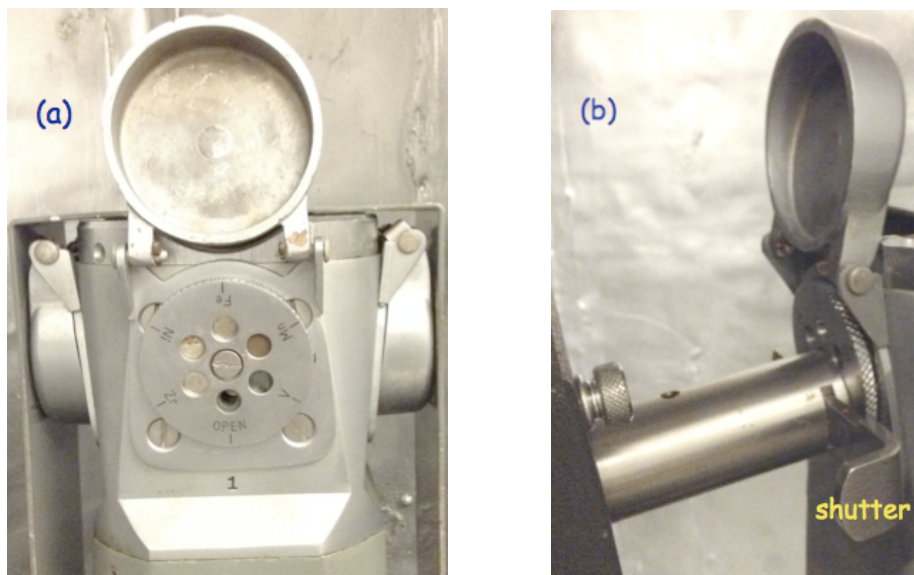


Figure 8: (a) Uncovered X-ray port with no (OPEN) filter. Rotating the wheel allows various metal filters to be placed in the X-ray beam, if desired. (b) Radiation cap against filter, with shutter open.

4. Without touching the faces of the crystal, mount the (001) crystal on the top of the pin at the centre of the goniometer so that the (001) face will be perpendicular to the X-ray beam. Use a small dab of Duco cement and be very careful not to get Duco cement on any part of the goniometer except the pin.
5. Mount the crystal position jig at some arbitrary point on the camera track with the point facing **away** from the camera and secure it by hand, turning the locking bolts.
6. When the Duco cement has dried, mount the goniometer on the camera track with the crystal face to be photographed facing the point of the crystal position jig. Adjust the goniometer so that the bases of the goniometer and jig are in contact and the point of the jig is at the centre of the crystal face and is as close to it as possible without touching it, as shown in Figure 10. Be careful not to scratch the crystal.



Figure 9: Crystal mounted on goniometer in front of Camera on the X-ray Machine.

7. Slide the goniometer back from the jig, remove the jig and then slide the goniometer forward until its base is in contact with the base of the camera track adapter. The crystal face is now 3.0 ± 0.1 cm from the photographic film plane and the X-ray beam will fall on the centre of the crystal face. (See Figure 9)
8. Turn on the X-ray unit as outlined in the section above called **Turning the unit on**. For a setting of 40 kV and ~ 18 ma a typical exposure time (when medical X-ray film is used) is about 30-40 minutes.
9. After the correct exposure time, turn down the current and then the voltage and press the X-RAY OFF button. Then reach in to close the shutter. It is not necessary to turn the machine entirely off as outlined in the section above called **Turning the unit off** if you are going to take more pictures.
10. Develop the picture in the MP227 darkroom as instructed by the Lab Technologist. (The film in use as of April 2016 requires 2 minutes in developer solution, then 2 minutes in fixer, followed by careful rinsing in water and drying in a cool oven at $\sim 60^\circ\text{C}$.)

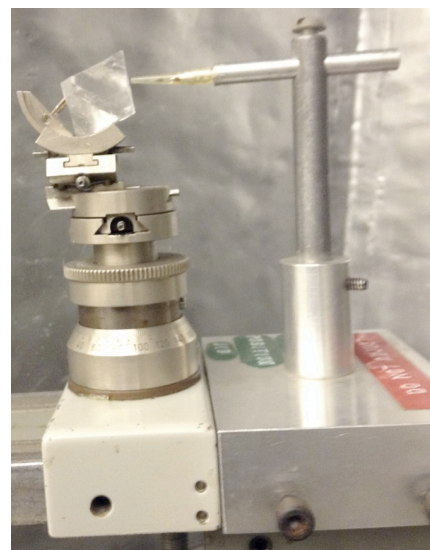


Figure 10: Large crystal on goniometer correctly adjusted with the crystal position jig.

- The centre of symmetry should be close enough to the centre of the film so that the axis of symmetry of the crystal can be determined. Write the axis of symmetry of the crystal at the bottom of the film.
- Remove the (001) crystal, then mount the (110) crystal and proceeding as before, photograph and identify the prominent planes. Do not dismount the (110) crystal when finished, since you will be rotating it in the next step.
- Verify the result of (12) by turning the crystal through 35° so that the beam is parallel to (111) and take a photograph. (It is easy to mix up coordinate systems and angles, so think twice before making the rotation.) Identify prominent spots and write down the symmetry at the bottom of the photograph. The stereogram p. 59 and photograph p. 67 in Wood may be useful. Are there any extra spots or expected spots missing? See Wood p. 71-74 for examples of possible problems.

Laue Photograph of a CaF_2 Crystal

- Remove the tungsten crystal and mount the CaF_2 crystal, shown in Figure 11, with the sanded bottom on the top of the pin and the cleavage face perpendicular to the X-ray beam.

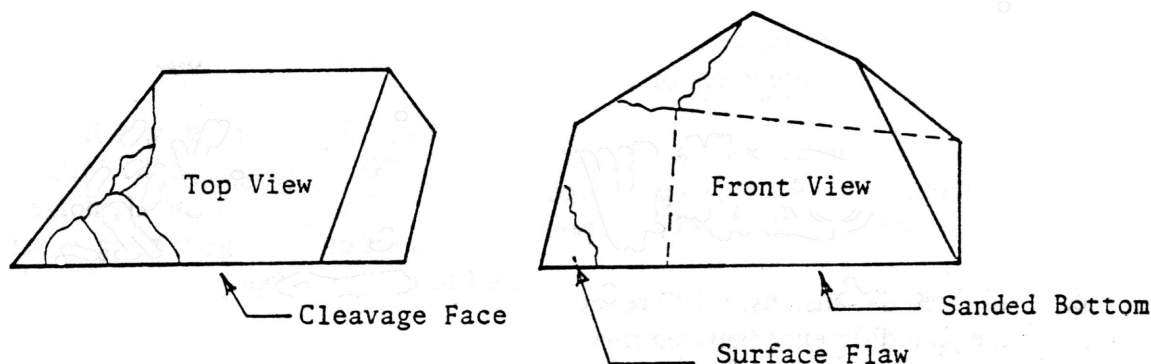


Figure 11: Top and Front Views of the CaF_2 Crystal.

- Take a Laue photograph and determine the direction of the cleavage face. For 40 kV and ~ 18 ma, exposure times may be several hours.
- Confirm (2) by finding two other directions of high symmetry. Turn the crystal so that one of these two faces is perpendicular to the X-ray beam. Take a Laue photograph. If the symmetry of the picture is what you would have predicted then this confirms your findings in (2).

Laue Photograph of an Unknown Single Crystal

- Remove the previous sample and mount the unknown crystal on the goniometer with the flattest, smoothest crystal surface facing perpendicular to the X-ray beam.
- Take a Laue photograph. Less dense samples will typically need longer exposures, but the only way to find out whether an exposure is long enough is to try. If you have several different samples, it may be worth making short 30-40 minute exposures of each to see which is likely to worth a longer exposure.
- Try to identify the crystal structure and orientation. It is easy to make a mistake, so unless the photograph shows an obvious major symmetry (e.g. 100 or 110) pattern, it is necessary to confirm the orientation by taking another Laue photograph with the crystal rotated such that

one of its major symmetry planes is perpendicular to the X-ray beam. If the expected pattern is not observed, the original structure or orientation hypothesis is incorrect.

Laue Photograph of an Unknown Sample

1. Remove the previous sample and mount the unknown on the goniometer, with the flattest, smoothest surface facing perpendicular to the X-ray beam.
2. Take a Laue photograph. What you see will depend on how crystalline the sample is. Try to explain what you see, and extract any possible information about the sample.

Measuring and Scanning Laue Images

To measure the location of spots, it is easy and accurate to simply tape the Laue film to a piece of graph paper on a light table and read off the spot positions. The positions can also be read from within QLaue, but the image first needs to be scanned in.

Spots near the beam spot are often harder to see on scans than on the original film; it is best to use a transmission scanner such as the Epson 4990 (or newer) photo scanner in MP226. When using the Epson 4990, be sure to remove the reflective document mat which is used from reflective scanning. Reasonable choices for 4990 settings are: Select Professional Mode, Original Document Type = Film, Film Type = Positive Film, Destination Image Type = 16-bit Greyscale, Resolution = 300dpi, Save as highest quality JPEG; QLaue cannot handle pdf files.

Analysis discussion

The primary goal of Laue analysis is to identify the type and orientation of single crystals. In addition, for this experiment you want to understand as well as possible the images you make. If they don't match an ideal pattern, can you understand why? It could be imperfections in your method or the sample. Wood, Cullity, or Preuss are all excellent resources for figuring things out.

Traditional Laue analysis used graphical methods to measure and analyse the spots on images, but computer software is now normally used. Commercial X-ray diffraction equipment will typically come with proprietary software, and there are also open source packages such as LaueTools¹¹, which are very powerful but not always easy to install.

For this experiment we recommend QLaue, Laue_Spot_Identify.py, and Laue_Spot_Patterns.py, which are easy to install and run on either PCs or Macs. QLaue allows you to display expected Laue Spot patterns for different crystal structures, and see if they match a scanned Laue image. QLaue has some ability to identify spots, but a more robust way to do this for cubic lattices is with the Python scripts Laue_Spot_Identify.py. For displaying spot patterns, Laue_Spot_Patterns is an alternative to QLaue for the cubic crystals you are studying in this experiment; it is a straightforward python script, so its intensity calculation and other aspects can easily be modified or improved. QLaue, Laue_Spot_Identify, and Laue_Spot_Patterns are all available from this experiment's web page (<http://www.physics.utoronto.ca/~phy326/laue>), along with some notes and examples.

Notes: (1) When comparing different programs, you may have to mentally rotate or otherwise convert coordinate systems to match spots, e.g. a 132 spot in one program, but be 123 in another. These differences are not important, but don't be confused by them. (2) Do not assume all the software is error free and will accurately predict what you see. There are no known

¹¹ <https://sourceforge.net/p/lauetools/lauetoolswiki/Home/>

serious mistakes, but if something doesn't make sense, feel free to check inside Laue_Spot_Identify or Laue_Spot_Patterns.

Example analysis

When analysing a Laue image, the usefulness of a spot in figuring out the crystal orientation or structure depends more on how many visible zones it belongs to, not how bright it is. This is because low index spots belong to more zones than high index spots. For example, in Figure 12, spots A, D, E, F and G all stand out, but they are not all equally important. F and G belong to only one obvious zone (the line of spots highlighted by a dashed yellow line), while A is at the centre of at least 6 converging lines of spots. Spots B and C are very faint, but careful examination of the image shows that each belongs to 3 visible zones, and they probably belong to more zones that are not visible because they fall outside the useful area of the image. Spots D and E appear to each belong to at least 2 visible zones

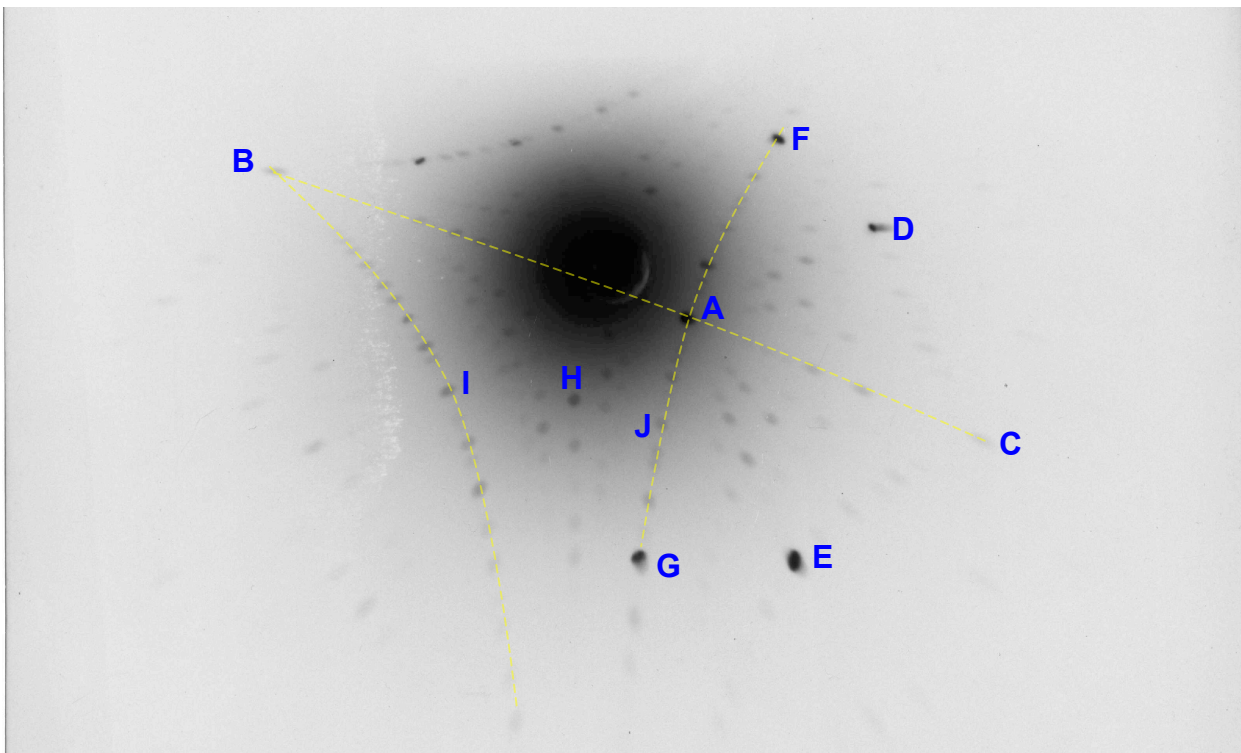


Figure 12: A sample CaF_2 Laue image (from Example_CaF2.xtl¹²).

If one analyzes spots A-G with Laue_Spot_Identifier.py, several almost equally good possible spot identifications are returned. Adding H and I, both belonging to 2 or more zones leads to a unique set of spot identifications. It turns out that dropping F and G still produces the same unique solution, since such “one zone” spots are less powerful at identifying the orientation, even if they are intense. The unique solutions shows that B, A, and C are the 100, 101, and 102 spots belonging to the [010] zone, D and E are the 213 and $2\bar{1}3$ spots, F and G are 535 and $5\bar{3}5$ on the $[10\bar{1}]$ line of spots, and H and I are $3\bar{1}2$ and $3\bar{1}1$ spots.

¹² Available at <http://www.physics.utoronto.ca/~phy326/laue>.

We seem to understand the spot positions, but it is not obvious why the $E(2\bar{1}3)$ and $G(5\bar{3}5)$ spots are so intense compared to other spots. Looking at this crystal orientation in QLaue or Laue_Spot_Patterns.py, or at the $\text{CaF}_2(110)$ pattern in Preuss (p. 386)¹³, one would expect $E(2\bar{1}3)$ and $H(3\bar{1}2)$ to be the same intensity, and the $J(3\bar{1}2)$ spot to be brighter than $G(5\bar{3}5)$. This is an example of the interesting questions that may come up as you analyse your images and investigate your crystals. If one wanted to investigate this spot brightness issue, one might rotate the crystal onto the 110 axis and take another image to see how the spot intensities change, e.g. if the $2\bar{1}3$ and $3\bar{1}2$ have the same intensity when they are symmetrically located relative to the beam.

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¹³ Note: The central spot in the Preuss pattern is labeled 220 not 110 because of the rules for allowed hkl values for fcc crystals; see Preuss Ch. 3 or Cullity Ch. 4.6. Laue_Spot_Patterns also has the same feature, but not Laue_Spot_Identify since the latter does not know the space-group of the crystal, so it cannot know whether a hkl value is forbidden.