Solid State Physics
X-ray Scattering II: Transmission Laue Method

This experiment uses the Tel-X-Ometer (580) X-ray instrument located in Colton 14.
You MUST sign and return the X-ray information handout before using this instrument!

General instructions and information about the Tel-X-Ometer are found in the Teltron manual, entitled "The production, properties, and uses of X-rays", which is located near the instrument and is to remain in Colton 14. The present experiment is described on pg. 16 of the manual (D12 - Diffraction of X-rays; Laue). The goal is to produce an X-ray diffraction pattern on a piece of film from a small single crystal of LiF. The Laue method provides a nice demonstration of X-ray diffraction and analysis of a Laue diffraction pattern gives good insight into crystal structure. As a quantitative tool, the Laue method is used most often for accurate crystal orientation rather than for structure determination. Our goal here will be a quantitative interpretation of the LiF Laue pattern given that we know the alkali-halides have a face-centered cubic (fcc) structure. **As always, be sure to keep detailed records of your lab work in your lab notebook.**

For Operating the Tel-X-Ometer: See the instructions for "X-ray Scattering I" (which are posted in Colton 14) and see Sections 6.5-6.6 on pg. 2 of the manual. In this experiment we will be running the X-ray tube at 30 keV and with a tube current of around 80 μA (although the exact tube current value is not critical).

The X-Ray tube current must be monitored at all times with a digital multi-meter.

The X-Ray tube current should not be allowed to exceed 80 μA.

Experimental Procedure:

Follow the instructions for experiment D12 (pg 16). The LiF mini-crystals are inside the small plastic sleeves in the vial labeled TEL 582.007. These crystals are extremely fragile ... HANDLE WITH CARE ... and not with your fingers.

The crystal should be mounted (with tape) directly over the aperture of the 1mm circular beam collimator. Before mounting the crystal, be sure you know how the collimator/plastic slide holder gets assembled and attached to the X-ray tube. Practice this without the crystal in place!

The X-ray film is in a white box in the darkroom. There are two different filmpak types in the box. We're using the shorter one (filmpak 750/2). Load the film in the cassette labeled 562.013 (taking care not to break the plastic part of the cassette slide) and mount this in the slide holder after the latter has been attached to the X-ray tube. Note that at some point you will need to measure the crystal-film distance as accurately as possible for each of the following setups.

Place the film in slot #3 and take a two-hour (or longer) exposure. Since the timer on the Tel-X-Ometer only gives up to about 45 minutes, you will need to reset it more than once during the exposure. Time your exposure with a stopwatch rather than the Tel-X-Ometer timer (which is not that accurate). You should also check the tube current periodically as it will float around. Try to keep it below, but near 80 μA.

After the exposure, remove the filmpak cassette and then very carefully remove the collimator/slide holder from the X-ray tube. Replace the crystal in its vial. Please do not leave these crystals out when not in use as they will pick up moisture from the air.

You should also take a second photo with the film in slot #2. (This smaller crystal-film distance may allow you to capture some larger angle scattering peaks). Here the exposure time should be at least three hours.

Film development:

The film can be developed any time after the X-ray exposure. Read the filmpak instruction sheet before starting! I suggest loading both the developer and fixer syringes before starting the development. Injecting these solutions can be tricky. You may want to play with one of the "spent" film holders sitting near the developing solutions. I suggest using scissors to open one of the injection ports at least halfway down and then perhaps inserting a straightened paperclip into the channel just to make sure it's open. When you do inject the solutions, insert the tube to its hilt and hold the tube tightly to prevent solution from coming back up out of the port. Have a stopwatch ready for timing. Hold the filmpak over the red tray and inject slowly ... some solution will inevitably drip out ... it's not toxic, but you'll want to wash you hands! (Sink is in the dark room). Use the scissors and small plastic tweezers to remove your film. Follow the filmpak instructions to fix your film for permanent record. Although the film background may be dark you should be able to clearly identify a diffraction pattern. Once dry, you can make measurements on the film itself. Alternatively, you can take a digital photo of the film and do the measurements on the blown-up photograph.

Data Analysis:

The Laue diffraction pattern should be very symmetric with sets of diffraction spots arranged in rings about the center of the pattern. Measure the location, relative to the center, of all diffraction spots and use these distances to determine the scattering angles of each ring of spots (these angles are actually 2θ where θ is the angle between the X-ray beam and a crystal plane). To index our scattering spots we need to establish a relationship between scattering angle and Miller indices. In our scattering geometry, the X-ray beam is parallel to the (100) direction and thus the angle between the beam and the (*hkl*)-plane is given by

$$\cos(90° - \theta) = \hat{n}_{100} \bullet \hat{n}_{hkl} = \frac{h}{\sqrt{h^2 + k^2 + l^2}} \quad . \tag{1}$$

[See "X-Ray Sacttering I", Eqs. (2) and (3)]. Draw a picture of scattering from the (*hkl*)-plane which shows the direction vectors used in Eq. (1) and defines the angle θ and be sure you understand why the angle measured from the film is 2θ. For our scattering geometry, we can get a maximum of eight diffraction peaks in a single ring due to scattering from the following sets of lattice planes, which all make the same angle with the X-ray beam:

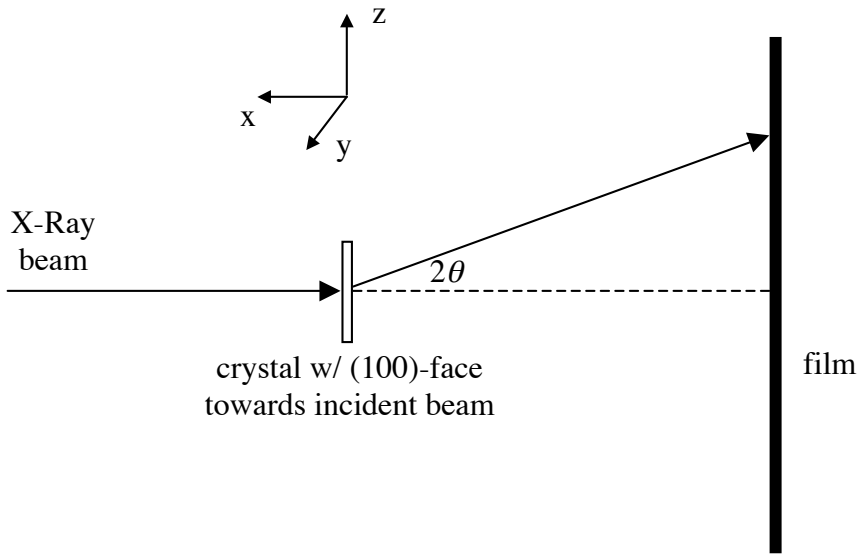
$$(hkl),\ (h\bar{k}l),\ (hk\bar{l}),\ (h\bar{k}\bar{l}),\ (hlk),\ (h\bar{l}k),\ (hl\bar{k}),\ (h\bar{l}\bar{k})$$

where the over-bars indicate a negative sign. Note that our beam does not see planes with negative *h* indices, since these face the "back" of the crystal, and that there is no scattering for *h*=0 planes (which are "edge-on" to the beam). Also note that for some *hkl* combinations (such as when *k*=*l*) we will have fewer than eight spots in a ring. Each ring of spots is due to a different X-ray wavelength that satisfies the "Bragg" condition

$$\lambda = 2d_{hkl}\sin\theta \qquad (2)$$

where, for a cubic crystal, $d_{hkl} = a/\sqrt{h^2 + k^2 + l^2}$ is the inter-plane distance.

The easiest way to index your diffraction spots is to make a table of allowed *hkl* combinations (for an fcc crystal these must be all even or all odd), starting with the combinations giving the largest d_{hkl} (which should give the strongest scattering). For each allowed (*hkl*) set determine the expected scattering angle θ from Eq. (1) and the needed X-ray wavelength λ from Eq. (2). We can exclude (*hkl*) planes that require $\theta > \theta_{max}$ or $\lambda < \lambda_{min}$ where θ_{max} is the maximum scattering angle allowed by your geometry and λ_{min} is the cutoff wavelength of the X-ray tube (which is set by the accelerating potential of the tube: $hc/\lambda_{min} = eV$). From your table you should be able to index each ring of diffraction spots.



Scattering geometry for
transmission Laue experiment.