Solid State Physics: Experiment #4
X-ray Diffraction - Debye-Scherrer Method

The goals for this experiment are two-fold. First you will learn how to prepare a powder sample appropriate for X-ray analysis in our scattering device. Second, you will use the Debye-Scherrer method to determine the lattice structure and lattice constants for a powdered sample of LiF and a polycrystalline sample of pure niobium. We will be using Cu $K_{\square}$ radiation and we will record the scattering patterns on X-ray film. **As always, be sure to keep detailed records of your lab work in your lab notebook.** The various parts of the present experiment are described in Sections D27 and D28 of the Tel-X-ometer manual located in Colton 14.

For Operating the Tel-X-Ometer: See the instructions for experiment #2 (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.

The X-Ray tube current should 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:

Part 1. LiF powder analysis [See sections D27.1-D27.18 in the manual (pp. 24-35).]

Sample Preparation: Most of the sample preparation actually involves preparing a very small tube to hold the powder sample. This sample tube will be made from acetate cement as follows (see D27.1). Insert a piece of thin "sample tube" wire all the way into the tube of cement. Rotate the wire a few times and then slowly remove (you may get better results by doing this twice). Now let the wire dry for at least an hour hanging from a clip (perhaps over the radiator). When dry, place one end of the wire in a vice (now mounted in room 14), grasp the other end firmly with a pair of pliers and slowly pull on the wire. When you pull hard enough, the wire will start to give and then break. Remove the wire from the vice and clip off the smashed end that was in the vice with a pair of wire cutters. If there are any rough spots or burrs on either end of the wire try to smooth these out with your fingers or some emory cloth. Use the wire cutter to clip off a 15 mm length of the wire. You should find that the acetate cement now forms a sheath which can be slid along the wire. Slide the cement sheath to expose about a 10 mm tube ... this is your sample tube. (If this tube is messed up, try for another from your original piece of wire; you can make three of these tubes from one piece of wire).

To load powder into the tube (see D27.2) you first need to transfer a small amount of powdered LiF onto a small watchglass (or a spoon will do). Lightly thrust the tube into the powder. Let the tube make contact with the spoon to drive powder up into the tube. Continue this "tamping" process until you fill the 10 mm long sample tube. To seal the sample tube, place a drop of acetate cement on a piece of paper and dip the open tube end in the cement. Let this "cap" dry and then lightly press the free wire into the tube to compact your sample. Use wire

cutters to clip off all but about 5 mm of the wire. This sample can now be mounted in the powder camera.

Camera setup and X-ray exposure: (see D27.3-11) Mount your sample tube in the chuck of the camera sample post. The wire part should be all the way in the chuck so only the LiF sample is exposed to the X-rays. Take the camera, mounted sample, scissors, and X-ray filmpak (with long skinny piece of film) into darkroom. You can use the red light in the dark room but still let your eyes adjust for a few minutes. Cut the end off the filmpak and slowly pull out the film. Insert the film strip into the camera with the two film ends on each side of the internal collimator. The film should be all the way down in the camera and should be held against the wall by two posts in the camera base. Securely mount the camera cover and insert the sample mount. The camera is now ready for mounting in the Tel-X-ometer. The crystal mounting post should have been removed and the moveable arm should be in the 90° position. The 1 mm slit primary beam collimator should be installed on the X-ray tube in the vertical position. The camera should just set in place on the collimator. **Take a 3 hr exposure** (X-ray tube potential = 30 kV, tube current ≈ 80μA).

For developing the film, we will use two rectangular plastic boxes as developing trays. Put about 5 ml of developer in the shallow tray and about 5 ml of fixer in the deep tray. (You don't need a lot of these solutions). In the darkroom (red light use is okay) ... remove the film from the camera and place it in the developer. Slosh the developer around/move the film around in the developer for at least 1.5 minutes (use big timer in darkroom). Transfer to the fixer and again slosh the fixer around for at least 4 minutes before coming out of the dark room. You can continue to fix for 10 more minutes in the light, then let the film rinse under running water for 20 minutes. Once dry, you can make measurements on the film using a divider and micrometer. You may want to use the overhead projector in Colton 2 as a light box.

Data Analysis: Note that the lines on your film come in pairs. Rather than trying to measure line positions from the center (zero angle position), measure the distances between each pair of lines and convert these to scattering angles (note that your measurements give you 4□). Only consider lines due to the $K_{\square}$ radiation, i.e., the ones that continue up into the top of the film where a nickel absorber is placed in the camera. Analyze this set of scattering angles to determine both the lattice structure and the lattice constant (with uncertainty) for LiF.

Part 2. Polycrystalline niobium analysis

Insert a piece of niobium wire into the camera sample holder chuck. Then just follow the above instructions for camera setup and X-ray exposure. **Take at least a 3.5 hour exposure** for this one. Data analysis is the same as for the LiF.

The Write-up:

... should include a brief discussion of the Debye-Scherrer method, some figures of the setup, quantitatively accurate drawing of the patterns on your two pieces of film, and your data analysis.

Write-up due date: Monday, Mar. 31.