Physics 4780: Atomic and Nuclear Physics Laboratory

Experiment CM5: X-ray diffraction and crystal structures
Lab Location: MH 3010

References: Preston and Dietz, Expt. 10 pp. 180-197
Eisberg and Resnick, Quantum Physics, Sec. 9
Kittel, Intro to Solid State Physics, Chaps. 1-2 (7th ed)

Goals: *-to understand the generation of characteristic and continuum x-rays in x-ray tubes;*
-to understand Bragg diffraction from crystal powders;
-to understand the construction and operation of a Geiger-Müller detector ;
-to understand the x-ray absorption and use of x-ray filters.

Preston and Dietz have an excellent discussion of the Bragg diffraction from crystalline solids. Also read Kittel for the theory of X-ray diffraction (several of the key pages are reproduced in this section.) The oversimplified description of x-ray diffraction is to treat it as wave interference from alternate, smooth, continuous lattice planes. This gives the Bragg Law: $m\lambda = 2d \sin(\theta)$, where, unlike optics, θ is the angle of the beam measured from the lattice plane (NOT from the normal to the plane or surface). The spacing of the planes, d, depends on the fundamental unit cell length, a, and the Miller indices of the plane (h,k,l). [If not already familiar, you should review how the Miller indices are related to the intercepts with the axes established by the primitive translation vectors.] For a cubic system, the plane which cuts the x- and y- axes at 2 and the z-axis at ∞ has: (1/h=1/2,1/k=1/2,1/l=0) and thus the Miller indices are (1,1,0). For cubic systems, the separation between planes with Miller indices (h k l) is $d = a/(h^2+k^2+l^2)^{1/2}$.

➔ **Before you come to the lab class, compute the angles, θ, of important Bragg reflections of NaCl (a=0.563nm), LiF (a=0.4028nm) and GaAs (a=0.5653nm) for Cu K_α(λ=0.154nm) radiation. These should include (100), (200), (110), (300).**

Actually, of course, life is more complicated. The x-ray wavelength (0.154 nm for the case of Cu K_α) is about the same as the atomic spacing and atom size. Therefore one cannot really treat the planes as smooth and featureless. One must really include information on the structure of the atoms, most importantly the number of electrons (atomic form factor) and information about the "basis" or molecular units that are associated with the idealized lattice points (structure factor of the basis), and finally the structure factor of the particular lattice. A brief description of this is included in the pages from Kittel included here. Note, for example, that for the lattice of NaCl (fcc with a basis of two atoms -- Na and Cl), the (100) and (300) reflections are missing, as well as any plane for which h+k+l = odd integer.

BACKGROUND

The following pages are taken from Preston and Dietz pp. 191-2. They describe in a little more detail the production of x-rays (bremsstrahlung and characteristic radiation) and also the use of a Ni

foil to filter out the Cu K_α radiation. Also some typical diffractograms are shown for powdered Si, KCl, and KBr. Finally two tables of lattice constants are given for several common crystals having the zinc-blende and NaCl structures.

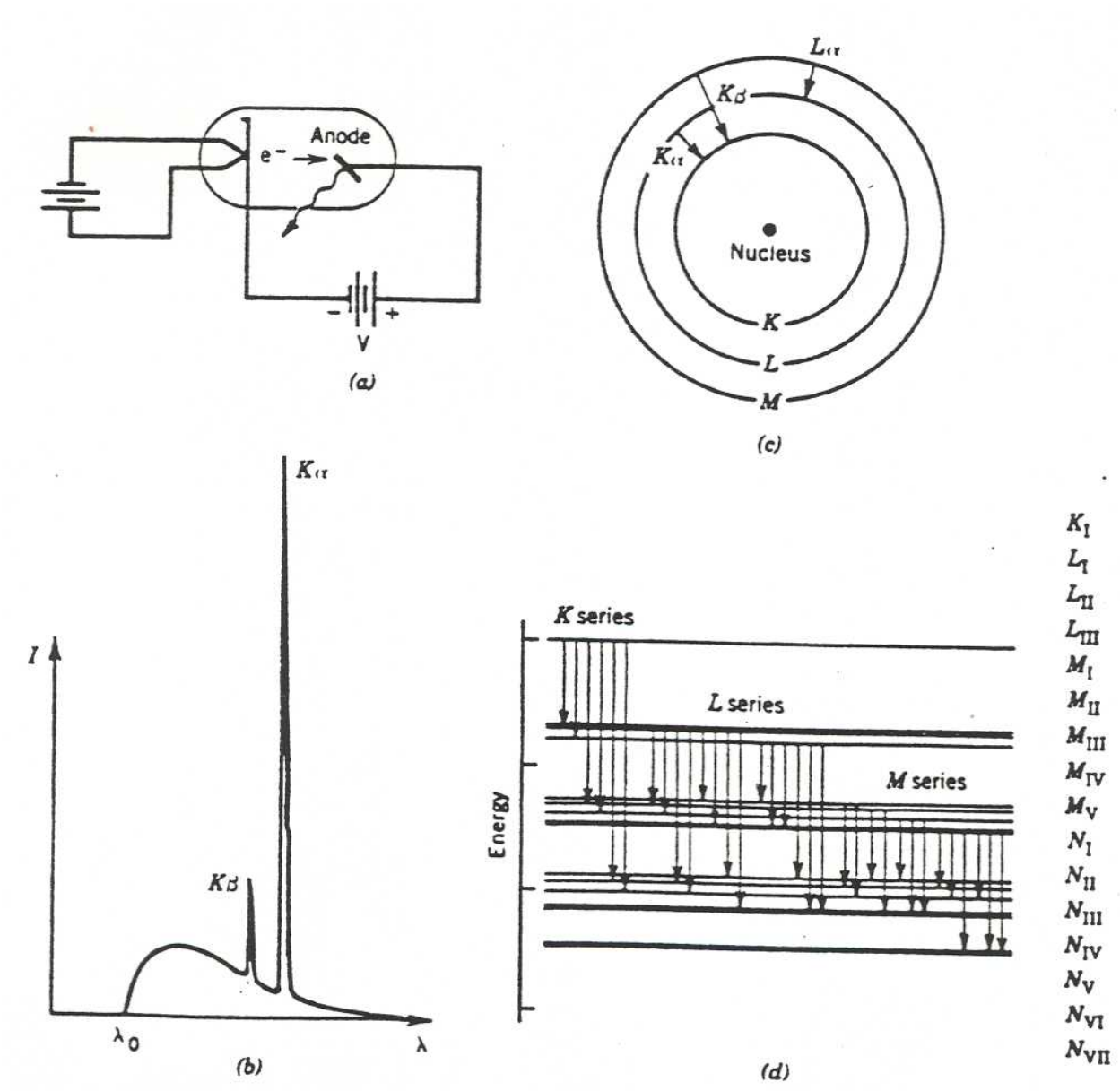

Fig. CM5-1: (a) production of x-rays; (b) typical spectral intensity distribution of the x-ray tube; (c) atomic transitions associated with the production of characteristic radiation; (d) energy-level diagram and transitions for various allowed x-ray transitions. [From Preston and Dietz, p. 191]

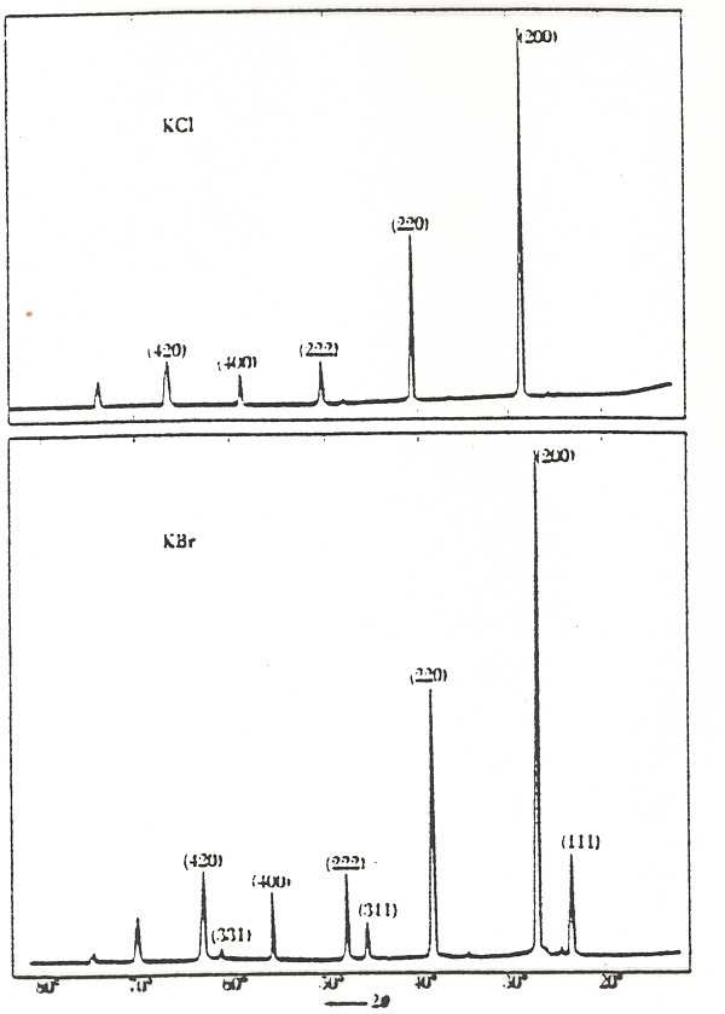

Fig. CM5-2: X-ray diffractograms of KCl and KBr. [From Kittel, p. 46]

Table A3-1. Some lattice constants. [mostly from Kittel pp. 19, 21.]

Crystal	a(Å)	Crystal	a(Å)	
GaAs	5.653	CuF	4.26	zinc blende or diamond structure
Si	5.431	ZnS	5.41	
CdTe	6.481	ZnSe	5.65	
CdS	5.82	SiC	4.35	
C (diamond)	5.545			
ZnS	a-3.81, c=6.23	ZnSe	a=3.98, c=6.53	hexagonal zinc sulfide structure
CdS	a=4.13, c=6.75	CdSe	a=4.30, c=7.02	
ZnO	a=3.25, c=5.12	SiC	a=3.25, c=5.21	
MgO	4.20	PbS	5.92	NaCl structure (fcc)
NaCl	5.63	KCl	6.29	
KBr	6.59	AgBr	5.77	
MnO	4.43	LiF	4.028	

Procedure:

A. Setup:

1. Follow the instructions in Appendix XVIII regarding the operation of the model 580M Tel-x-ometer x-ray system. Note that the basic system marketed by Tel-Atomic has been augmented by a stepper-motor system to automate the angular movement. In addition, the output of the Geiger tube and amplifier is input into the PCLabs digital input card for data acquisition.

2. X-ray emission from the tube inside the lead glass dome emerges as a beam of about 5 mm diameter from the "basic port." This beam diverges to about 15 mm diameter at the crystal mounting post. For these experiments we will want the beam collimated with the slit collimator to form a vertical "ribbon." To do this, use the 1 mm slit collimator at the basic port of the small glass dome around the x-ray tube. The slit should be oriented vertically.

3. Additional collimation is needed in front of the Geiger Muller tube detector. Start with the 3 mm slit mounted in a rectangular slide. Mount the collimator in one of the slots of the rotatable arm ("experimental station, E.S."). *Always use a spring clip to be sure that this second slit presses firmly against the numbered side of the carriage arm.* This collimator should be mounted in front of the G-M tube but leaving at least one slot for later mounting of the Ni foil filter.

4. Mount the NaCl crystal in the chamfered post in the center with the reflecting face against the post. This is the precise center of the rotation. Note that the system is constructed with a θ -- 2θ gearing mechanism so that as the crystal rotates by θ, the detector arm rotates by 2θ. (If you stop to think about it, you should conclude that with θ-2θ motion, you will only observe reflections from planes which are parallel to the face of the chamfered mounting post.)

B. Measurements:

1. With the 3 mm slit in front of the detector and no Ni filter, obtain a θ-2θ scan for the NaCl single crystal. Start your scan at a small value of 2θ, perhaps 15^o and scan to a large value of 2θ, perhaps 120^o. For your first scan you should use a relatively large step size. Later you can use a smaller step size for more precise measurements when you verify which region is the most interesting. Note carefully the starting position using the vernier read-out and note carefully the ending position, again with the vernier. When you plot up the data you can use these values to correct the step size to yield the correct total 2θ rotation angle.

   You should clearly observe the appropriate diffraction peaks for NaCl with a peak due to the K_α wavelength and a slightly shifted peak due to the K_β wavelength for every reflection. Since NaCl has the face centered cubic structure with a basis of two [Na at (0,0,0) and Cl at (1/2,1/2,1/2)], you should be able to calculate the allowed peaks for this system from the structure factor.

2. Repeat the scan of #1 above with the Ni filter in place. You should find that the K_β peaks are significantly reduced. Be sure you can explain this effect of selective absorption!

3. Observe the effect of using a narrower collimating slit in front of the G-M tube. Replace the 3 mm slit with the 1 mm slit. Repeat part 2 above, now using the 1 mm slit.

4 Obtain a good diffraction spectrum for the KCl crystal. In this case it is important to recognize that the K^+ and Cl^- ions have the same number of electrons so that the atomic form factors are nearly the same. This may change the allowed reflections.

5. Repeat for LiF, or GaAs, or Si.

6. For a challenge, if time permits, try the experiment with either a powder sample or a thin CdS or CdTe film on glass.

On your synopsis for this experiment:

1. Provide plots of the diffractograms for NaCl:
   - -with 3 mm slit
   - -with 1 mm slit
   - -with Ni filter and either 3 mm or 1 mm slit.

2. Provide a diffractogram for one other crystal (in addition to NaCl).

3. From your diffractograms with the two different slits, determine whether the resolution is linear in the slit width.

4. Annotate your diffractograms in #1 and #2 above. Calculate the predicted peak positions for K_α radiation for NaCl and your second crystal.

5. Explain the different effects of the Ni filter on Cu K_α and K_β radiation.

6. Be prepared to describe the operation of the Geiger Müller tube as a detector of x-rays.

7. Calculate the structure factor for NaCl and show why the (100) reflection is missing and the (200) reflection is present. What do you predict for the (300), (400), or the (111)?

Table 4 Crystal structures of the elements

The data given are at room temperature for the most common form, or at the stated temperature in deg K. For further descriptions of the elements see Wyckoff, Vol. 1, Chap. 2, and Table A-6 of Barrett and Massalski. Structures labeled complex are described there. The notation *ABAC* refers to the sequence of close-packed planes.

H^1 4K hcp 3.75 6.12																	He^4 2K hcp 3.57 5.83
Li 78K bcc 3.491	Be hcp 2.27 3.59											B rhomb.	C diamond 3.567	N 20K cubic 5.66 (N_2)	O complex (O_2)	F	Ne fcc 4.46
Na 5K bcc 4.225	Mg hcp 3.21 5.21	← Crystal structure. →										Al fcc 4.05	Si diamond 5.430	P complex	S complex	Cl complex (Cl_2)	Ar 4K fcc 5.31
		← a lattice parameter, in A. →															
		← c lattice parameter, in A. →															
K 5K bcc 5.225	Ca fcc 5.58	Sc hcp 3.31 5.27	Ti hcp 2.95 4.68	V bcc 3.03	Cr bcc 2.88	Mn cubic complex	Fe bcc 2.87	Co hcp 2.51 4.07	Ni fcc 3.52	Cu fcc 3.61	Zn hcp 2.66 4.95	Ga complex	Ge diamond 5.658	As rhomb.	Se hex. chains	Br complex (Br_2)	Kr 4K fcc 5.64
Rb 5K bcc 5.585	Sr fcc 6.08	Y hcp 3.65 5.73	Zr hcp 3.23 5.15	Nb bcc 3.30	Mo bcc 3.15	Tc hcp 2.74 4.40	Ru hcp 2.71 4.28	Rh fcc 3.80	Pd fcc 3.89	Ag fcc 4.09	Cd hcp 2.98 5.62	In tetr. 3.25 4.95	Sn (α) diamond 6.49	Sb rhomb.	Te hex. chains	I complex (I_2)	Xe 4K fcc 6.13
Cs 5K bcc 6.045	Ba bcc 5.02	La hex. 3.77 *ABAC*	Hf hcp 3.19 5.05	Ta bcc 3.30	W bcc 3.16	Re hcp 2.76 4.46	Os hcp 2.74 4.32	Ir fcc 3.84	Pt fcc 3.92	Au fcc 4.08	Hg rhomb.	Tl hcp 3.46 5.52	Pb fcc 4.95	Bi rhomb.	Po sc 3.34	At —	Rn —
Fr —	Ra —	Ac fcc 5.31															

Ce fcc 5.16	Pr hex. 3.67 *ABAC*	Nd hex. 3.66	Pm —	Sm complex	Eu bcc 4.58	Gd hcp 3.63 5.78	Tb hcp 3.60 5.70	Dy hcp 3.59 5.65	Ho hcp 3.58 5.62	Er hcp 3.56 5.59	Tm hcp 3.54 5.56	Yb fcc 5.48	Lu hcp 3.50 5.55
Th fcc 5.08	Pa tetr. 3.92 3.24	U complex	Np complex	Pu complex	Am hex. 3.64 *ABAC*	Cm —	Bk —	Cf —	Es —	Fm —	Md —	No —	Lw —