Physics 410/510

Experiment X-7

Diffractometer: Measurement of Atomic Vibrational Amplitudes and Atomic Electron Distributions

In this experiment, you will measure the Debye-Waller factor, i.e., the motion of atoms, in an aluminum lattice, and the atomic form factor, i.e., the charge distribution, in iron atoms. If you are not familiar with the elements of x-ray diffraction by crystals, it is recommended that you do the powder pattern (X-6) first. The attached chapters of the book by Warren may help in doing this experiment. Also, the attached papers are very useful. Read them!

1. Familiarize yourself with the diffractometer and the counting equipment. Read the notes attached to the x-ray power supply. Using CuK_α radiation obtain the integrated intensities of the lines from the pressed aluminum powder by measuring the areas (with planimeter) above background as recorded on the chart. Measure all reflections from (200) to (420).
   Using the appropriate function, construct a log plot vs. $(\sin\theta/\lambda)^2$ such that the slope will give the Debye-Waller factor for aluminum. From this determine the mean square amplitude of vibration. Compare with values in literature (International Tables for Crystallography, Vol. III).

2. Using a low-order aluminum reflection as a standard, measure the atomic scattering factor for the (110) and (200) reflections for the pressed iron powder. Use MoK_α radiation. Compare this with the values to be expected for the isolated iron ion with different numbers of 3d electrons.
   In you report discuss the various sources of error especially those other than involved with the actual taking of data. For example, extinction, surface effects, preferred orientation.

References:

B.E. Warren, "X-ray Diffraction," Addison-Wesley, Reading, 1969, Chapters 1-5, 11.
B.D. Cullity, "Elements of X-ray Diffraction," Addison-Wesley, Reading, 1956, in particular Chapters 4 and 7.
H.P. Klug and L.E. Alexander, "X-ray Diffraction Procedures," John Wiley, New York, 1954, Chapter 5.
R.W. James, "Optical Principles of the Diffraction of X-rays," Especially Part 3, Chap. 1 and 3.
C.S. Barrett and T.B. Massalski, "Structure of Metals," McGraw Hill, New York, 1966, 3rd ed.
W.P. Davey, "A Study of Crystal Structure," McGraw Hill, New York, 1934.

Papers:

R.J. Weiss, Revs, Modern Phys. 30, 59 (1958).
B.W. Batterman, Phys. Rev. 115, 81 (1959).
Batterman, Chipman and DeMarco, Phys. Rev. 122, 68 (1961).
B.W. Batterman, Transactions of the American Crystallographic Association 1, 1965.

August, 2, 1994.

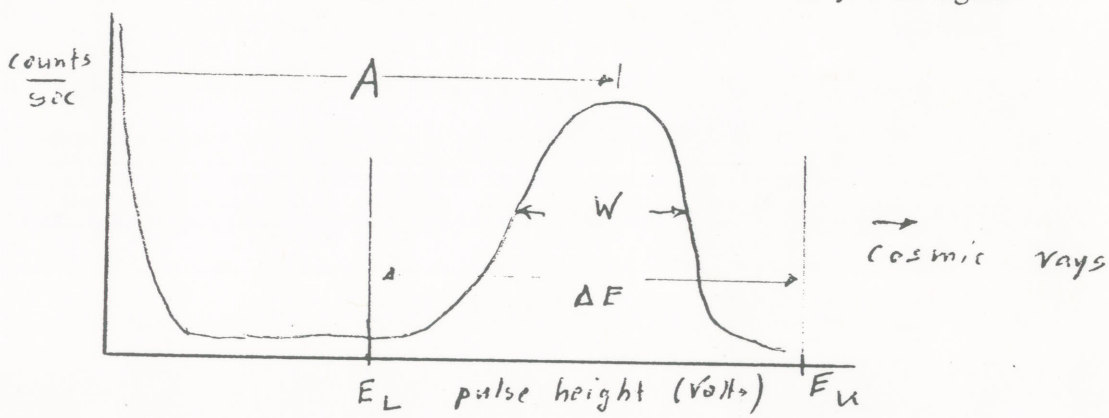
PHYSICS 410 - 510

Special Notes for X-7

It is a good idea to start out by taking a rapid scan over the angular range covering all the peaks of interest. When the areas for the separate peaks are to be measured, a slower scan in the neighborhood of the peak should suffice.

Adjusting the proportional counter high voltage and associated electronics:

The scaling electronics panel has associated with it a single channel pulse height analyzer, and this must be adjusted for proper detection. The detecting tube is a gas filled (Xenon) proportional counter, i.e. it produces short duration electronic pulses whose amplitude is proportional to the energy of the x-ray photon. For the purposes of this discussion, characteristic x-ray lines ($K\alpha$, $K\beta$, etc.) can be considered to be of infinitesimally sharp energy. Yet, the pulse produced by the counter has a certain energy spread. This is shown schematically in the figure.



This curve represents the distribution of pulse heights obtained when a monochromatic beam is being counted. The high counting rate at low voltages is due to electronic noise in the system. The peak of mean pulse height A is due to the x-ray beam. The value of A in volts depends on many variables. For a given counter tube, it depends on applied tube voltage, gain in the pre-amp and amplifier, and of course, x-ray photon energy. The width W at half maximum is a property of the counter and the photon energy. (Since the initial photon energy is extremely sharp, why should there be a non-zero W?) A measure of resolution of the tube is the W/A ratio and for a proportional counter and an 8 kV x-ray (1.54Å) it has a value of about 0.3. The W/A ratio usually gets smaller as the initial photon energy increases.

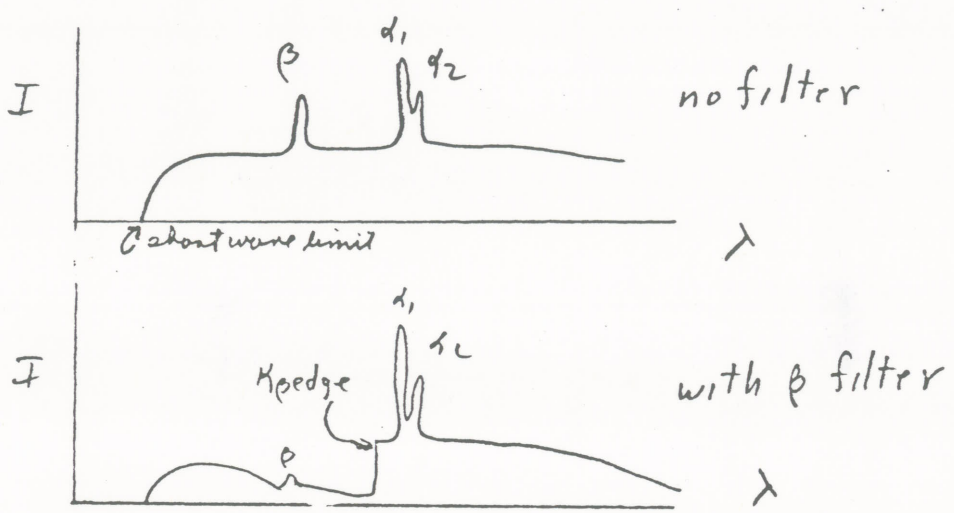
The single channel analyzer allows an electronic window to be inserted into the counting system so that only a particular range of pulse amplitudes are accepted. The upper and lower levels are separately adjustable. Let's call E_L the lower window level E_u the upper level, and ΔE the window width. We would like to adjust these so that only the pulses associated with our wanted radiation are to be detected. A simple procedure to do this is as follows: Set the counter tube voltage somewhere around 1500 V. Remove the upper window (integral mode-simple single level discriminator) and lower the base line E_L to be just above the system noise. Put a powder sample in the diffractometer and adjust for a strong K_α peak. Observing the count rate meter, raise E_L slowly until the count rate starts to drop off rapidly, then back off to a somewhat lower level. Put in the upper window at its wide open position. Then lower ΔE until the count rate again starts to drop rapidly and raise it above this point. The procedure should now provide a window which allows the order of 90% of the K_α pulse distribution to be counted. Shut off x-rays and measure the count rate. It should be smaller than 0.5 counts per second. If it's too high, it probably means that the signal pulses are close to noise. Increase tube voltage and/or amplifier gain for optimum results.

For part (1) the main experimental problem is to measure the different peaks so that the proportionality between them is maintained. One danger is that the beam intensity changes with time. One hour of warm up is usually sufficient to stabilize the x-ray generator. However, it would be a good idea to re-measure a given peak from time to time to act as a check and also as correction if the primary intensity changes. Also, do not assume that the rate-meter is linear if you change scales. Either check this, or take all the relative intensities on the same range scale.

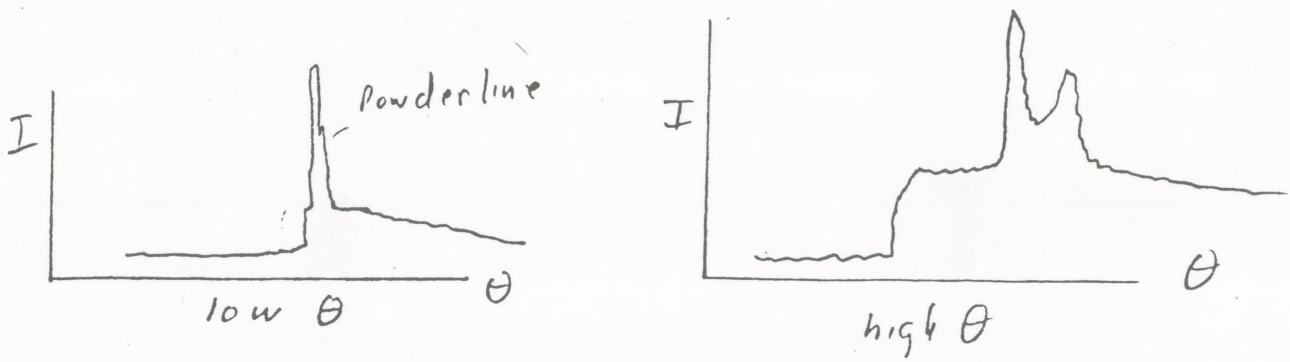
The problem comes up as to the choice of goniometer speed, slits, time constants on rate meter, use of filters etc. The rate meter is an analogue instrument whereas the counter is digital. The rate meter produces a voltage (which the chart records) which is proportional to the rate at which pulses are produced by the detector. Since the pulses are produced randomly (even for a fixed average counting rate) the instantaneous rate will fluctuate - hence, the wiggle of the pen. The optimum time constant will depend on the rate at which the peak is transversed. It is generally better to have too short a time constant. This way you are sure the pen is following the intensity, in fact it is over following. Draw a smooth line through the fluctuations and use this to define the peak area.

Try measuring a given peak with different slits. Since in this experiment you are primarily interested in this experiment you are primarily interested in area, resolution is not too important. Does the relative integrated intensity of the reflections depend on the slit size, or is this an intrinsic property of the specimen?

β-filters can pose somewhat of a headache and are worth discussing in some detail. The β filter, as the name implies, is used primarily to get rid of the $K\beta$ line from the tube spectrum. The figures below show the effect of the filter.



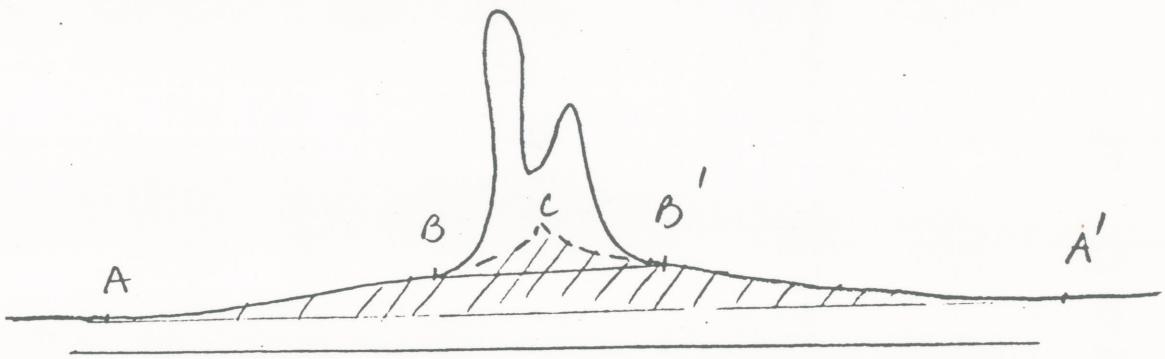
The $K\beta$ line of the target is effectively removed, but in the vicinity of the α lines there is a sharp discontinuity due to the K edge of the filter. Now, any powder line is really a wavelength analysis of the tube spectrum and hence will show the edge. For low angle reflections the edge will be close to the α doublet, whereas for higher angle lines it will be progressively further away. This is shown schematically below:



The difficulty comes in when one has to determine the background line under the peak. For the high angle line, the peak is well separated from the edge and one would draw the background line at the level of the white spectrum under the α doublet. At low angles, the edge is close to the peak and perhaps not too well defined so that the low angle background level is taken at the short wavelength side of the filter K edge. Thus, in the latter case, a piece of the white spectrum is counted in with the peak, while in the former case it was not included. The way to

avoid the problem is not to use a filter. This may cause the occasional problem of a β peak from some higher order line getting in the way. Correct for this by simply drawing a background line under the β peak.

If you look carefully at the tails of the higher order peaks they will extend a rather large distance from the peak center. It had always been assumed that these tails were part of the integrated intensity and the background was taken at the point A-A in the sketch below.



It has been shown* that most of the tail region is due to thermally diffuse scattered x-rays (TDS). The approximate shape of this scattering is ABCB'A' in the figure. The correction due to thermal scattering taking the background at AA' is the hatched area in the figure and depending on the order of the reflection and the elastic softness of the crystal can range from a few percent to upwards of 20 to 30%. In the present case the corrections would be relatively small.

The net result of all this merely says don't take the background A-A' but at BB' closer into the peak but still on the gradually sloping tails. This applies only to the highest order reflections. For most peaks in this experiment pick the points AA where the background becomes horizontal.

B. Batterman

*N. Nilsson, Arkiv Fysik, 12, 247 (1957).

D. R. Chipman and A. Paskin, J. Appl. Phys., 30, 1998 (1959).