X-RAY DIFFRACTION & DIFFERENTIAL SCANNING CALORIMETRY

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1. Introduction

The aim of the experiments is to use X-rays to characterise crystals and to measure using differential scanning calorimetry, the evolution of changes in an initially glassy polymer.

You will be using a desktop X-ray diffraction machine as illustrated in Figure 1. The equipment is designed so that the high voltage (20 or 30 kV) can only be applied when the shielding cover is closed, and another safety device is correctly activated. The cover is secured such that it cannot be opened by lifting the lid; *do not open the lid*. You will not have to lift the cover in any of your experiments. There is an additional screening plate at the front of the kit with a radiation warning label. The machine is well-shielded so that the emitted radiation is not detectable above the background radiation.

The intensity of X-rays at a particular 2θ is measured using a Geiger counter (detector); you can rotate it on the side where the deterctor is located, using the orange device that is located outside of the shield, taking care not to get the wire connecting the detector to the counter, fouled with the shield.

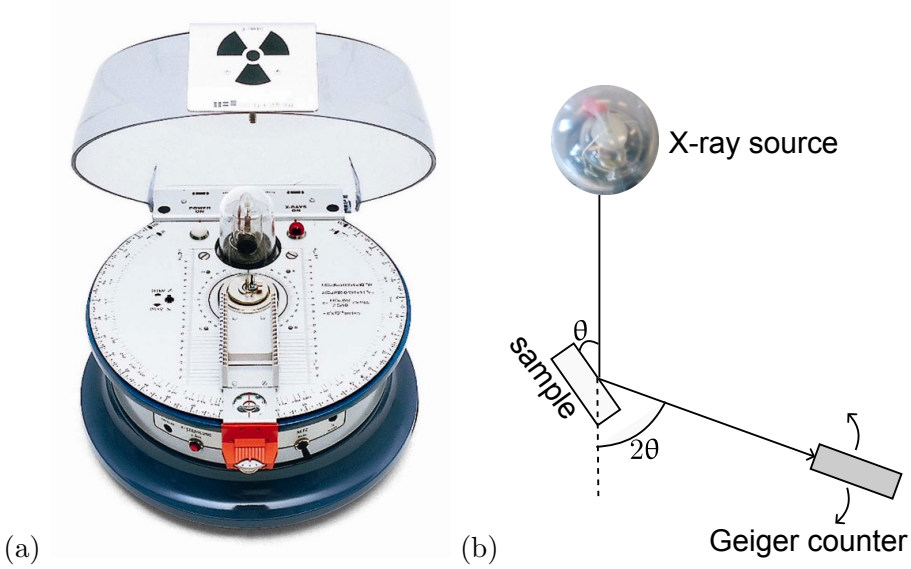
2. X-ray experiment, LiF

The X-ray tube emits both Cu-K_α ($\lambda = 0.154\,\text{nm}$) and Cu-$K_\beta$ radiations, together with some white X-rays. However, we have placed collimators and a filter before the detector so you can assume that only Cu-K_α data reach the detector.

For a fixed value of θ, you will see that the count rate varies, given that the process of X-ray generation has some randomness. You may use an average of the range indicated by the needle on the meter.

The Demonstrator will have set up a single crystal of LiF, cut so that its edges correspond to $\langle 100 \rangle$ directions.

When the X-rays are switched on, scan about the $2\theta = 44.4^\circ$ and $2\theta = 98.2^\circ$, rotating the detector through just 1° steps to measure the count rate. Plot the count rate measured on the meter against the value of the 2θ angle. Given that these peaks correspond to the $\{200\}$ and $\{400\}$ planes, and that LiF has a cubic lattice, **estimate** values of the lattice parameter of LiF using the peak intensity recorded in each of the peaks plotted. **Are these**



(a) (b)

FIGURE 1. (a) General view of the equipment, with the shield open, in which case the X-ray source is not operational. (b) Schematic illustration of the source, sample and detector. The detector moves through 2θ as the sample turns through θ.

values identical and if not, why not? Comment on the accuracy of the measured lattice parameter. The measurements and plotting should take about 30 min.

3. X-RAY EXPERIMENT, BARIUM TITANATE

Ask the Demonstrator to replace the LiF sample with a compacted powder of barium titanate, which has a slightly tetragonal lattice. The strongest X-ray diffraction peak using Cu-K_α radiation occurs at about $2\theta = 32°$. The intensity you will observe is not as large as was seen with the LiF. Plot the peak, which according to your lecture notes (Figure 1.6) corresponds to closely spaced {101} and {110} planes. **Comment** on whether you have resolved these two planes on your plotted peak, and if not, **comment** on the shape of the observed peak. The measurements, plotting and commentary should not take more than 30 min.

4. DIFFERENTIAL SCANNING CALORIMETRY

We will be using a polylactic acid (PLA) polymeric sample, to measure the DSC curve as a function of heating at $10\,\mathrm{K\,min^{-1}}$ over the temperature range 30-200 °C. The polymer is initially in a glass state – you will be able to observe certain changes as the sample is heated. A schematic diagram of these two stages is shown in Figure 2.2 of your notes, although it will not be possible to melt the sample.

Identify the location of events such as the glass transition or crystallisation, giving an explanation of the shapes of the curve in the vicinity of the transitions. **Comment** on what to expect if the experiment is done at a greater than $10\,\mathrm{K\,min^{-1}}$ heating rate over the same temperature range?

The differential scanning calorimeter works by measuring a small temperature difference that develops between the sample pan and a reference pan. **Comment** on what reference you used in your experiment.

Comment on how you could use your data to determine the enthalpy of crystallisation of the polymer, in units of $\mathrm{J\,g^{-1}}$?

5. Report

You should write your individual report and hand it in before leaving the laboratory at the end of your two-hour slot. Your report does not need to include a description of the equipment used, but it should cover the following points:

(1) Have you included enough information to enable someone to reproduce your work. If, for example, I am measuring the strength of a material, then I must at the same time indicate the strain rate used to measure the strength which depends on the rate.

(2) Your results should be included.

(3) You have been asked to **comment**, **estimate**, **identify** etc., items in the text above – you should address all of the items marked in bold font.

(4) It always is important to be concise and legible.