Laboratory #7 Guide: X-Ray Diffraction Studies of Polycrystalline Semiconductor Thin Films November 12, 2013

Introduction

Analysis of crystalline structure and the degree of crystallinity of semiconductor thin films by x-ray diffraction (XRD) provides key insights into material quality. Some materials exist in several different phases, with different crystal structures associated with each phase. Many materials show varying degrees of crystallinity, so that in some cases when one tries to make high quality, highly-crystalline material, they may instead end up with a film that contains both crystalline and non-crystalline (or amorphous) material blended together. All materials at one time or another incorporate strain, such that the average lattice constant of the material differs slightly from the equilibrium "standard" lattice constant. Differences between films of the same material can also arise when the density (per unit volume, or per unit area of the film) of crystalline grains varies – i.e., the average size of the crystallites (crystal grains) varies depending upon how the samples were grown and/or treated.

In each of these cases, XRD can measure samples' crystalline properties to provide essential information regarding the crystal formation process, the effect of chemical and thermal treatments, and the specific phases of material present within the film. Note that some samples, such as CdS, may grow in two different phases, or structures, within the same film. We will see if that's the case for our CdS sample.

In this lab, you will measure and analyze the XRD patterns for four thin film samples: (1) sputtered CdS, (2) spray-deposited copper indium diselenide (CISe), (3) as-deposited (aputtered) CdTe, and (4) $CdCl_2$–treated CdTe.

Goals of Lab #7

Your goals for Lab #7 involve developing a strong familiarity with XRD patterns for the materials we'll study, including a clear understanding of (a) the quantitative relationship between XRD peak locations and the Miller indices of the crystal lattice planes responsible for each peak, (b) how to use standard spectra to confirm identification of a material based on the XRD pattern, and (c) how to apply the Debye-Scherrer relation to calculate the apparent average crystal grain size based on the XRD line width.

Sample List

1. Sputtered CdS
2. Spray-deposited CIS
3. As-deposited (sputtered) CdTe
4. $CdCl_2$-treated CdTe

Experiment (Demonstrate understanding within your lab report)

With assistance from an experienced XRD instrument operator, measure the XRD signal intensity as a function of 2θ (where θ is the Bragg angle) for each of four samples. Take care to use measurement parameters that enable you to acquire high-quality data which clearly show as many peaks as can be resolved. Take notes on what parameters on the instrument affect the data quality, such as any bandwidth settings, angular step size, integration time, etc. What values for each of these parameters did you use to acquire your XRD spectra?

Laboratory Report

1. Plot the data as Intensity vs. 2θ for each of these samples. Annotate the graphs to denote the (*hkl*) values for each peak (see below) in the CdS and CdTe graphs.

2. Include two tables in your report, one for CdTe and one for CdS, listing the possible (*hkl*) Miller indices values, the value of the lattice spacing (d_{hkl}), and your calculated 2θ peak positions (in degrees). Your 2θ values should be calculated based on Bragg's Law and the values for the lattice constants provided in the lecture slides, and based on the use of Cu K-α X-rays (as opposed to K-β X-rays). Note that there are two possible crystal structures for CdS, zincblende (cubic) and hexagonal (wurtzite). Based on the XRD spectrum you measured for CdS, deduce whether the crystal structure of our CdS is zincblende or hexagonal (refer to the lecture slides for values of lattice constants *a* and *c* for the hexagonal structure).

3. Assign *hkl* values to all peaks in the CdTe *and* CdS spectra; describe/indicate which peaks you know with certainty and which have an uncertain origin (include your basis for each assignment). All peaks in your experimental CdTe and CdS XRD spectra should be assigned (and labeled, per Step 1 above) according the Miller indices. If you observe any peaks arising from Cu K-β X-rays, point this out.

4. Apply the Scherrer equation to the most prominent peak for each of the 4 samples to compute each sample's average crystal grain size (based on the FWHM peak width). Note that β is measured in radians.

5. Include a discussion in your text of various key aspects you note about XRD and our thin film samples -- such as the relationship between the lattice plane spacing (d_{hkl}) and the scattering angle (2θ), how the CdTe XRD pattern changes with $CdCl_2$ treatment, and how one can deduce the structure of our CdS based in part on the XRD measurement.