Exploring the Structural Dynamics of Thin Films using X-ray Diffraction  
Kendall Vanderhoof '20, and Frank Peiris, Ph.D.  
Kenyon College Summer Science 2017

Abstract
Multiple thin films were scanned with an x-ray diffractometer and their diffraction peaks were analyzed to study their crystal phases and to use those crystal phases to discover more information about the zirconia (ZrO₂) and HgCdSe samples. The ZrO₂ samples were spin coated on silicon substrates and treated with oleic acid (OA) or trioctylphosphine oxide (TOPO) and annealed at various temperatures to influence their crystal phases. The TOPO samples were amorphous for longer, but transitioned from tetragonal to monoclinic before the OA samples. The second set of samples had different alloy concentrations of HgSe and CdSe, and were grown by molecular beam epitaxy (MBE) on GaSb and ZnTe/Si substrates. The diffraction peaks of HgCdSe were used to verify the alloy concentrations predicted by the MBE growth technique. In addition, these studies confirm that the samples are of high quality, deduced from the widths of the diffraction peaks in the x-ray spectra.

Background

ZrO₂
- ZrO₂ is spin coated on a silicon substrate
- HgCdSe is grown via molecular beam epitaxy (MBE) which produces more even samples than spin coating

HgCdSe
- HgCdSe is grown via molecular beam epitaxy (MBE) which produces more even samples than spin coating
- Each sample has a goal percentage of HgSe and CdSe. Through calibrations, MBE determines the alloy concentrations; however, after samples are grown, the exact percentage needs to be calculated
- This can be done by using Vegard’s Law; exploiting the linear dependence between the two lattice parameters of the compounds (see Fig. 3 & 4)
- HgCdSe is grown via molecular beam epitaxy (MBE) which produces more even samples than spin coating
- The crystal phase of the film
- Through X-ray Diffraction, we can determine the crystal phase of the sample

Approach

X-Ray Diffraction
- X-ray diffraction can find:
  - The lattice parameter (related to the distance between atoms)
  - The quality of the crystal
  - The crystal phase
  - The crystal structure

HgCdSe Thin Films

<table>
<thead>
<tr>
<th>Sample</th>
<th>%Hg</th>
<th>%Cd</th>
<th>L₁₁₁</th>
</tr>
</thead>
<tbody>
<tr>
<td>S20037</td>
<td>0.194</td>
<td>0.607418</td>
<td>0.423</td>
</tr>
<tr>
<td>S20074</td>
<td>0.360</td>
<td>0.607082</td>
<td>0.478</td>
</tr>
<tr>
<td>S20076</td>
<td>0.263</td>
<td>0.607211</td>
<td>0.457</td>
</tr>
<tr>
<td>S20021</td>
<td>0.245</td>
<td>0.607205</td>
<td>0.448</td>
</tr>
<tr>
<td>S20011</td>
<td>0.208</td>
<td>0.607376</td>
<td>0.430</td>
</tr>
<tr>
<td>S20022</td>
<td>0.209</td>
<td>0.607373</td>
<td>0.364</td>
</tr>
<tr>
<td>S20015</td>
<td>0.179</td>
<td>0.607463</td>
<td>0.349</td>
</tr>
<tr>
<td>S20027</td>
<td>0.249</td>
<td>0.607253</td>
<td>0.384</td>
</tr>
</tbody>
</table>

Figure 12. The given x-values for each sample is the percentage of CdSe in each sample. Using the lattice constants of each sample’s substrate, the percent difference in the lattice parameter of each sample is calculated.

- Finding the percentage a sample is different to it’s substrate is important, because when their lattice constants are similar, their diffraction peaks show up on top of each other, making the sample hard to analyze.

- As shown in figures 7 & 8, samples below 700 degrees treated with TOPO, and below 600 degrees treated with oleic acid are observed to be amorphous.
- The tetragonal peaks of the TOPO samples were found to be the most different to its substrate, but its peaks are still on top of its substrate peaks.
- The full width at half minimum of the intensity peaks is inversely related to the height of the intensity peaks; the peaks are becoming more defined as the annealing temperature increases, indicating that the zirconia is transforming its crystal phases inside the film.

- The holographic properties of ZrO₂ can be found in figures 10 & 11, which shows the peaks are found to be on top of the substrate peaks, which doesn’t allow one to calculate the actual percentages of HgSe and CdSe
- The goal of MBE is to grow samples on substrates that have lattice constants similar to them, to aid in growth.

X-Ray Spectra of ZrO₂

• TOPO
  
  Figure 7. The TOPO samples are stacked from highest annealing temperature to lowest annealing temperature from top to bottom. Below 600 degrees, the absence of diffraction peaks indicate the samples are amorphous. The high intensity peak at 33 degrees is the substrate.

• Oleic Acid
  
  Figure 8. The OA samples are stacked from highest annealing temperature to lowest annealing temperature from top to bottom. Below 500 degrees, the absence of diffraction peaks indicates that the samples are amorphous. The high intensity peak at 33 degrees is the substrate.

Phase Transformations of ZrO₂

- TOPO
  
  Figure 10. The tetragonal peaks of the TOPO samples were graphed, and their full widths at half maximum (FWHM) and peak heights (in red) were graphed above. The decrease in peak width and increase in peak height prove that the 900 peak is most tetragonal. Below 700, the samples were amorphous and are not included.

- Oleic Acid
  
  Figure 11. The tetragonal peaks of the OA samples were graphed, in the same way as the above TOPO samples, but the OA samples transitioned from amorphous to tetragonal before the TOPO samples did, so the 600 degree sample is included.

References

Acknowledgments
This work was funded by the Kenyon College Summer Science Scholars program. I would also like to thank Professor Frank Peiris for his assistance throughout the project.