EECE5606 Laboratory Template

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Title: SEM and XRD **Lab week: 5**

1. **Lab Description (10 points)** ***(What is this lab about?)***

In this lab lesson, the students were introduced to the characterization techniques of SEM and XRD.

2. **Lab Objective (10 points)** ***(Why are you doing this lab?)***

This laboratory class was aimed at training the students to characterize a sample of MEMS resonators using the Carl Zeiss AG - SUPRA 25 system to perform scanning electron microscopy (SEM) and the Philips X'Pert MPD system to perform X-ray diffraction (XRD). The main objectives of this class were to teach the students the basic principles of SEM and XRD, the operation of the system tools, the correct choices of the software user interface settings, and how to interpret the resulting images or graphical plots.

3. **Methods (20 points)**

1. **Scanning electron microscopy (SEM):**

In this laboratory class, the instrument used for the SEM process was the Carl Zeiss AG - SUPRA 25 system. The SEM uses a focused beam of electrons to create high-resolution images of the surface of a sample. The electron beam is generated by heating a filament or cathode and is accelerated and focused into a narrow beam by a series of electromagnetic lenses. As the beam scans over the surface of the sample, it interacts with the electrons in the sample and generates a variety of signals, which are detected by detectors in the SEM. These signals are then processed to create an image of the sample, which can be displayed on a computer screen and analyzed. The resolution of SEM images can be extremely high, allowing for the visualization of features as small as a few nanometers.

For this class, the SEM was used to produce high-resolution images of a MEMS resonator sample. The sample was a small square piece cut from a lithium niobate ($LiNbO_3$) coated Si wafer with Al/$LiNbO_3$ devices, i.e., resonators with interdigitated (IDT) electrodes made with an Al layer on $LiNbO_3$. The sample must be coated with a conductive film so that charges do not accumulate from the impinging electrons. The sample is stuck to the sample holder with a conductive carbon tape.

The SEM system uses the software Smart SEM on the windows operating system. The various steps involved in the SEM process are as follows:

1. The load lock chamber is vented to 1 atm (760 Torr) and when the light on the load lock chamber turns green from red, it can be opened to place the sample holder (with the sample) and load it. After loading, the load lock chamber pressure is brought to vacuum and the sample is indirectly inserted into the main chamber by horizontally sliding the metal rod (that sticks out on the side of

the system) inside the chamber. Once the sample holder has been fastened in the main chamber, the rod slides out and the main chamber is closed.

2. Using the software, the power is set to 2 kV and electron gun is energized. The stage parameters, X, Y, Z, T, and R indicate the position and orientation of the sample. The Z value is set to 25 mm and "Set Focus" is clicked to automatically set the other parameters. The adjustment in parameters is done using the TV view as reference.
3. After the values are set, the electron gun is switched on. The initial SEM image is produced with distortion on the sides. Magnification is done by selecting a rectangle over a region and setting the contrast. The resolution can be adjusted by tuning the scan speed. The surface features of the resonators are observed.
4. After the scanned images are produced and measurements taken, the electron gun is set to standby mode. The sample is again adjusted using the TV view as reference and the main chamber is opened to remove the sample using the rod. After the sample has been evacuated from the main chamber into the load lock chamber, the former is closed, and the latter is vented to 760 Torr. Following this, the load lock is opened, and the sample is removed.

2. X-ray diffraction (XRD):

In this laboratory class, the instrument used for the XRD process was the Philips X'Pert MPD system. XRD is a technique used to determine the atomic and molecular structure of materials. It involves directing high-energy X-rays at a sample from the source and recording the scattered X-rays using a detector, which is the collector. The diffraction pattern generated by the scattering is analyzed using software to determine the atomic arrangement of the sample.

The XRD process makes use of a simple software on the windows operating system. The various steps involved in the SEM process are as follows:

1. The sample was placed in the XRD chamber and fastened. The sliding doors must be closed softly.

2. The software user interface is used to set the type of scan, Gonio or Omega. The Gonio scan involves rotating both source and collector at the same time, whereas the Omega scan involves rotating only the source, keeping the collector stationary.

3. After the type of scan is set, the values for various parameters such as "Range," "Step size," "Scan speed," and "2Theta". Then the scan is set to "Continuous" mode and started.

4. The graphical plot (Counts vs 2Theta) is produced with various peaks at different angles, each denoting a particular material. The angles at which the count peaks are produced can be used to calculate the atomic arrangement of the material.

5. Both the Gonio and Omega scans are done for each sample and the peaks produced in both must occur at the same angle (2Theta = 2 * Omega).

6. Once the graphical plots are produced, the FWHM value can be read by opening the "Peak Parameters" window.

7. After this, the XRD is brought to stand-by mode (current value of 20 mA) and the samples are removed. The glass doors must be carefully shut.

4. Outcomes and Measurements (15 points)

The outcome of this laboratory class was that the students were introduced to the SEM and XRD characterization techniques.

1. Scanning electron microscopy (SEM):

The SEM images showed many micro-scale devices on the sample. The sample cantilevers (one coated with an Al metal layer and another without) were probed. The nonuniform coarse coating of Al metal on the LiNbO3 cantilever. This is because liftoff was used for this sample. Etching would have produced a smoother and cleaner layer of Al on LiNbO3. The width or pitch of the cantilever can be estimated to be approximately 7 µm.

2. X-ray diffraction (XRD):

The results of XRD show that for sample 1, the Gonio scan has its peak at ° and Omega scan at °. Similarly, for sample 2, the Gonio scan has its peak at ° (not shown) and Omega scan at °. We can see that in both cases, the peak 2Theta value is approximately or exactly twice of the peak Omega value.

5. Comments (5 points)

The following were observed in this laboratory class:

- The SEM can be used to perform e-beam lithography for power equal to or greater than 30 kV.
- The smallest feature size for SEM is 100 nm.
- The XRD peaks are different for same materials of different doping concentrations, stress, and abnormally oriented grains (AOG).

6. Analysis Questions (30 points)

(a) You observed two different structures under the SEM. How does a scanning electron microscope work, on a general basis?

The operation of a scanning electron microscope (SEM) relies on the interaction between electrons and the surface of the specimen. Here's a broad overview:

Electron Source: An electron gun produces a tightly focused beam of electrons.

Electron Lenses: Electromagnetic lenses are utilized to focus and manage the electron beam.

Specimen: The specimen is positioned within a vacuum chamber to prevent electron scattering.

Electron-Beam Interaction: Upon striking the specimen, the electron beam undergoes various interactions, including elastic scattering, inelastic scattering, and the emission of secondary electrons.

Detectors: Various detectors capture signals generated by these interactions, encompassing secondary electrons, backscattered electrons, and characteristic X-rays.

Image Formation: These signals are processed to generate intricate images depicting the surface characteristics of the specimen.

(b) Can you image all the materials? Are there any limitations/challenges?

Although the scanning electron microscope (SEM) is a formidable imaging instrument, it does come with its set of constraints:

Conductivity: SEM is most effective when dealing with materials that are conductive or semiconductive. Non-conductive specimens have a tendency to accumulate charge, which can adversely impact the quality of the images produced.

Sample Size: The specimen must be accommodated within the vacuum chamber, thereby imposing limitations on the size of objects that can be effectively imaged.
Sample Preparation: Prior to imaging, specimens typically necessitate coating with a conductive layer to enhance image quality, adding an extra step to the sample preparation process.
Resolution: While SEM is renowned for its high resolution, it may not reach the atomic scale resolution achievable by certain other microscopy techniques.

(c) Quickly describe the charging phenomenon. How can it be avoided?
When charging occurs, it results from the accumulation of electrons on the insulating surface of a specimen, causing distortion or drift in the image. To mitigate or prevent charging, several methods can be employed:
Conductive Coating: A solution involves applying a thin layer of conductive material, such as a coating of gold or palladium, to the specimen. This assists in dissipating any accumulated charge.
Grounding: Grounding either the specimen itself or utilizing conductive sample holders aids in discharging any accumulated electrons, thereby reducing the likelihood of charging.
Low Beam Voltage: Employing a lower electron beam voltage can effectively minimize charging effects, as it reduces the energy imparted to the specimen surface.
Environmental SEM (ESEM): The utilization of Environmental SEM (ESEM) offers an alternative approach by enabling imaging of specimens within a gas environment. This environment helps alleviate charging issues associated with insulating samples.

7. Attendance (10 points)
Leave this answer blank – to be used by TAs.