Grain-Size Analysis in Ferroelectric Materials

NSF EFE 1757579

Alexandra Cabrera[1], Mentors: Prasanna Venkatesan Ravindran[2] and Sarah Lombardo[3],
Principal Investigator: Dr. Asif Islam Khan[2,3]
[1]Department of Physics and Astronomy, Georgia State University
[2]Department of Electrical and Computer Engineering, Georgia Institute of Technology
[3]Department of Material Science and Engineering, Georgia Institute of Technology

Abstract:
Recent discovery of ferroelectricity and antiferroelectricity in semiconductor-process compatible oxides, hafnia and zirconia has fueled a new wave of interests in FEFETs. In this project, we work on preparing zirconia and hafnia MOSCAP samples for Scanning Transmission Electron Microscope (STEM) imaging. In addition, we develop an application that processes the STEM images to create a statistical distribution of the grain sizes. We were able to produce more than ten samples and our application was able to denoise images, identify grains, and isolate them for further analysis.

Background:

Moore's Law states that computer power is expected to double every two years by optimizing the size of transistors, which has reached its limit by the 2000s [3].

Ferroelectric materials possess unique properties such as hysteresis, negative capacitance and semiconduction. Ferroelectric Field Effect Transistors (FEFETs) present an opportunity to build a variety of circuits that leverage these properties to address the diverse needs of modern computing [2].

In 2011 and 2012, Hafnia and Zirconia, the semiconductor-process compatible binary oxides, were discovered to have ferroelectric properties, thereby inspiring a fresh wave of research interests in FEFETs [1]. Both these technologically important materials are nanocrystalline, exhibiting a strong relationship between the underlying microstructure and the macroscopic electrical properties.

Sample Preparation Process:

To prepare the samples for STEM imaging, we are given Hafnia and Zirconia Metal-Oxide-Substrate Capacitors (MOSCAP) wafers that need to be cut down into 2mm-by-2mm squares. Next, the samples undergo a three-step process to be thinned as possible. This will allow the electron beam of the STEM to pass through the hafnia/zirconia layer and capture an image of its grain structure.

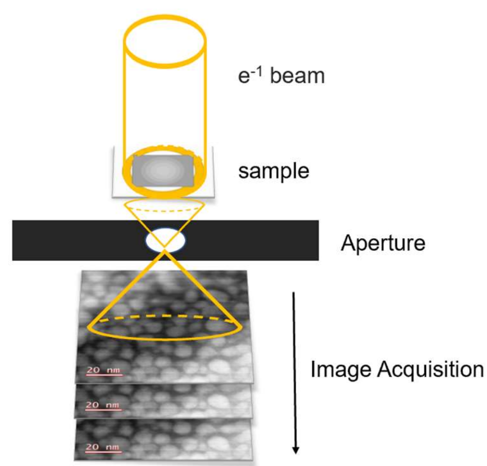

Figure 1. Illustration of how the STEM captures an image.

Step One: Manual Polish. After dicing, the silicon side of the 400µm-thick sample is faced down onto polishing paper and moved in a figure-8 motion until the sample is about 80µm thick. Four different polishing papers are used from roughest to finest grit: 22µm, 15µm, 10µm, and 5µm, with about 100-50µm polished off with each polishing paper.

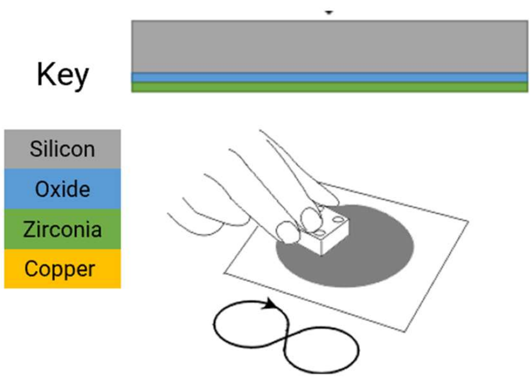

Figure 2. Displays key of MOSCAP sample and the setup for manual polishing.

Step Two: Dimple Polish. Then the sample is placed under a polishing wheel in a dimple machine. Using a 3µm and 1µm polishing paste, the sample is dimpled and checked periodically with the backlight of a microscope for a bright red-orange color indicating translucence of the sample.

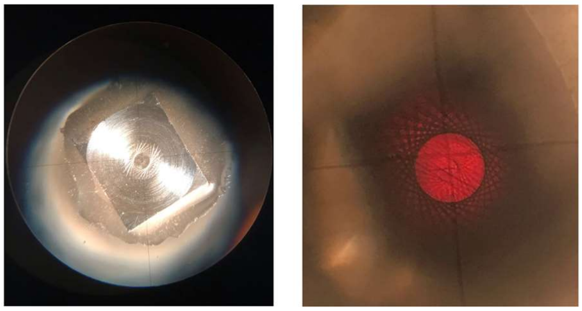

Figure 3. Left, top-light view of the sample under the microscope compared to the sample backlit in the right image.

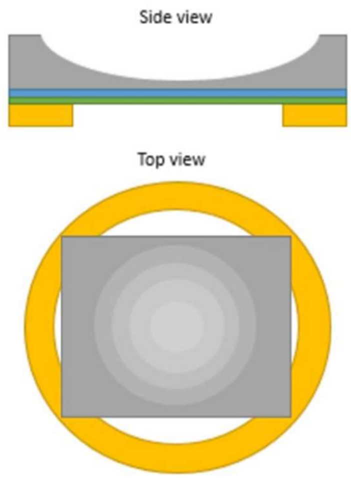

Figure 4. Side-view and top-view illustration of the sample after it is dimpled and transferred onto a copper grid.

Step Three: Ion Milling. Finally, the sample is transferred onto an ion milling machine, where ions are shot at a 5° angle to the center of the sample as it rotates. It is then finely polished until a small hole appears. The area around the hole is at its thinnest and the process is complete.

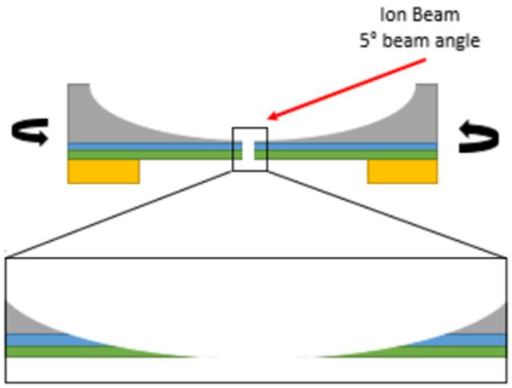

Figure 5. Side-view illustration with a close-up on the thin area around the formed hole.

I have made more than ten hafnia and zirconia samples and two were imaged underneath the STEM.

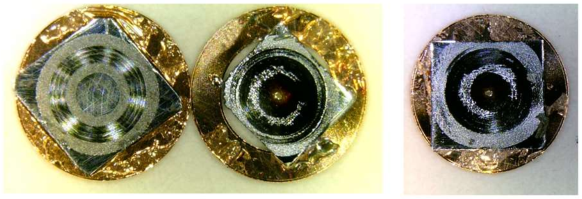

Figure 6. Left, one hafnia sample before ion milling and one finished hafnia sample. Right, one finished zirconia sample.

Grain-Size Analysis:

Ordinarily, after capturing the STEM images, we will measure individual grains manually to then analyze the microstructure of the ferroelectric. However, this process is not the most efficient method as we compile larger sets of data.

Instead, we want to create an application using MATLAB software to automate the process. So far, we have been able to denoise images, identify grains, and isolate them.

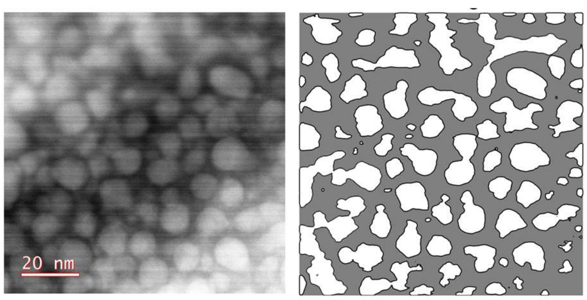

Figure 7. Left, top-down planview STEM image of Zirconia MIMCAP. Right, MATLAB generated image of edited left.

Our next steps are to improve image processing and use a best-fit shape, such as an ellipse or circle, to measure the size and area of the grains.

Future Work:

Overall, this project focused on sample preparation and grain-size analysis for top-down planview of STEM images. In the future, we would like to get a statistical distribution of the grain sizes and understand their relationship to device performance.

References:

[1] Boske, T.S, Muller, J., Brauhaus, D., Schroder, U., & Bottger, U. (2011). *Applied Physics Letters,* 99, 102903.

[2] Khan, A.T., Keshavarzi, A., & Datta, S. (2020). *Nature Electronics,* 3, 588-597.

[3] Waldrop, M.M. (2016). The chips are down for Moore’s law. *Nature*, 530, 7589.

Acknowledgements:

This work was supported in part by the Semiconductor Research Corporation (SRC) — Global Research Collaboration program and in part by the National Science Foundation (Grant No. 1718671). This work was performed at the Georgia Tech Institute for Electronics and Nanotechnology, a member of the National Nanotechnology Coordinated Infrastructure (NNCI), which is supported by the National Science Foundation (ECCS-1542174).