

***In Situ* Transmission Kikuchi Diffraction Observation of Thin-Film GST Crystal Phase and Grain Evolution**

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Transmission Kikuchi diffraction (TKD) is known for its ability to map crystal orientation and phase with a high spatial resolution [1, 2]. Requiring only a scanning electron microscope (SEM) equipped with an electron back-scattered diffraction (EBSD) detector, TKD inherits SEM's advantages such as low cost and high accessibility. Unlike traditional EBSD, TKD requires electron transparent samples. The beam interaction volume is thus much smaller, which allows TKD to take full advantage of SEM's nanometer resolution. Accordingly, *in situ* TKD is a promising technique for examining the time-evolution of thin film nanostructure [3]. By collecting TKD patterns on an electrically contacted, rectangular germanium-antimony-tellurium (GST) strip, we observe the GST's crystallization and grain growth *in situ*.

Using magnetron sputtering and electron-beam lithography, we deposit a 50 nm thick, $2.5\ \mu\text{m} \times 0.4\ \mu\text{m}$ amorphous GST strip bridging a TiN electrode to a TiN heater (Fig. 1a). A stage (Fig. 1b) custom-designed to avoid shadowing the camera (Oxford Symmetry EBSD) electrically connects the sample to a source-measure unit outside the SEM (a Helios G4 PFIB UXe SEMW). We crystallize the GST by powering the TiN heater to 100 μW and 200 μW , acquiring a TKD map with nanometer spatial resolution after each bias. The TKD maps are analyzed using Oxford Instruments' AZtecHKL and AZtecICE software to generate band contrast (BC) maps, phase maps, and inverse pole figure (IPF) color maps (Fig. 2).

After powering the TiN heater at 100 μW , the GST sample crystallizes. The BC and phase maps identify a few face-centered cubic (fcc) grains in the sample, which is predominantly hexagonal. The major hexagonal grains have an average size of $0.037\ \mu\text{m}^2$, and the IPF color map reveals that the grains prefer to orient such that their (0001) crystal planes lie parallel to the surface of the sample. After powering the TiN heater at 200 μW , the sample fully transitions into the hexagonal phase with a 37% increase in average grain size. The IPF color map reveals an even stronger (0001) orientation preference. We thus demonstrate *in situ* TKD's capability to study crystal dynamics such as grain growth and orientation in real time with high spatial resolution [4].

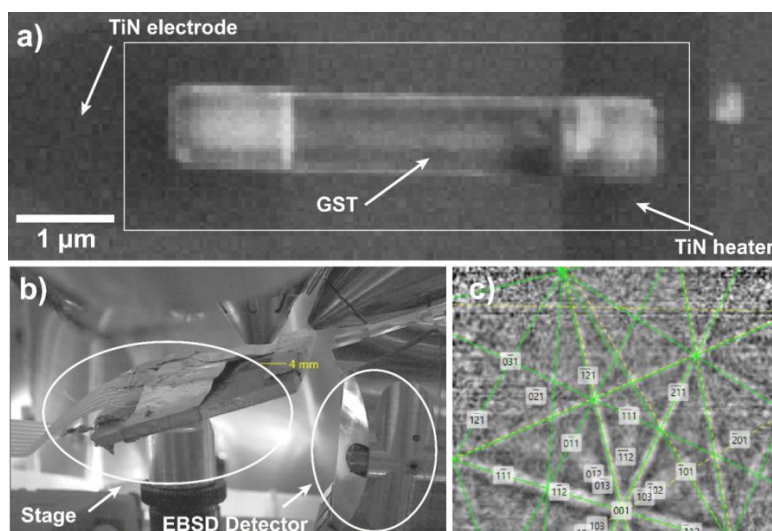


Figure 1. a) An SEM image of a GST strip making electrical contact to a TiN heater and a TiN electrode on a Si_3N_4 electron transparent window. The white box indicates where the TKD maps are taken. b) An optical image showing the SEM stage and EBSD detector inside the chamber. c) An indexed TKD pattern.

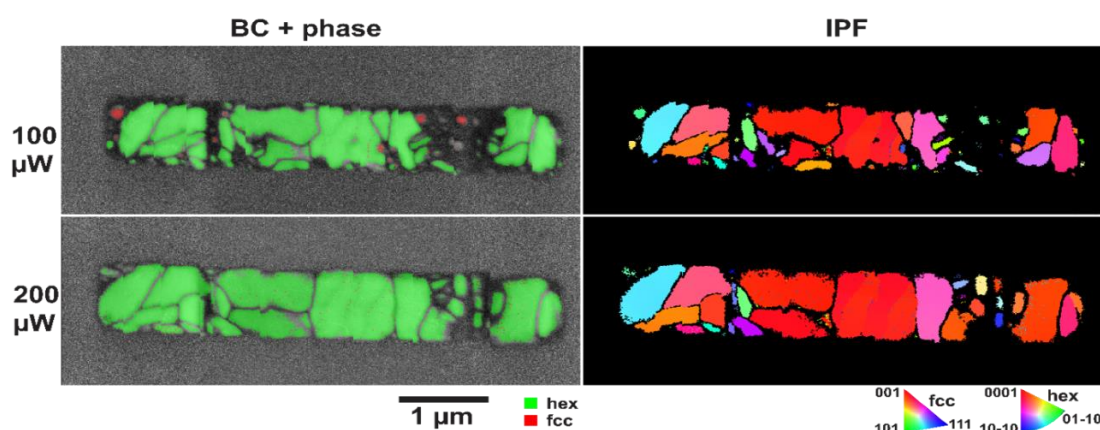


Figure 2. BC combined with phase maps (left) and IPF color maps (right). The phase maps show grain growth as heater power increases, and the IPF maps show a preferred orientation.

References:

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- [2] V Tong et al., *Journal of microscopy* **267** 3 (2017), p. 318-329.
- [3] Gwénaëlle Proust et al., *Journal of Visualized Experiments* **122** (2017) 55506.
- [4] The data were acquired at the Core Center of Excellence in Nano Imaging (CNI), University of Southern California. This work was supported by National Science Foundation (NSF) Science and Technology Center (STC) award DMR-1548924 (STROBE) and by the Semiconductor Research Corporation (SRC) and the BioPACIFIC Materials Innovation Platform of the National Science Foundation under Award No. DMR-1933487.