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Structure and Phase Stability in Extreme Environments Explored via In-situ TEM Experiments

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The ability to understand materials response to harsh and complex environments often found in real world applications has greatly improved due to recent developments in the field of in-situ transmission electron microscopy (TEM) [1]. These techniques permit direct observation of the dynamics of materials that can be incorporated into a range of atomistic to mesoscale models. The in-situ ion irradiation transmission electron microscope (I^3TEM), seen in Figure 1, is a unique tool that was developed to permit a range of in-situ experiments not possible elsewhere [2]. As such, a greater number of phase and structural stability of materials can be explored at the nanoscale.

As an exemplar, germanium antimony telluride (GST) is a phase-change material that undergoes a amorphous-to-crystalline phase transitions at specific temperatures, locking in optical and thermal properties making it useful for memory applications [3]. To qualify its use in extreme environments such as nuclear reactors or interstellar transport, the microstructure must be probed in real-time using in-situ microscopy with synergistic heating and irradiation [4]. Using the I^3TEM at SNL, we can couple the external stimuli with advanced nano-scale characterization to visualize the dynamics dictating microstructural evolution in GST in-situ during simulated real-world environment exposure. Combined thermal and irradiation extremes of amorphous and crystalline films of GST coupled with Automated Crystal Orientation Mapping offer insights into the crystallization and amorphization pathways under extreme conditions, see Figure 2.

In addition, this presentation will highlight recent developments in specialized holders and additions to the I^3TEM to enhance the coupled and extreme environments that can be reached and understood in greater depth. This includes the recent addition of a gas injection system (GIS) and differential pumping to the I^3TEM that permits two order of magnitude pressure change and thus permits a range of environmental TEM (ETEM) experiments during ion or laser irradiation [5].

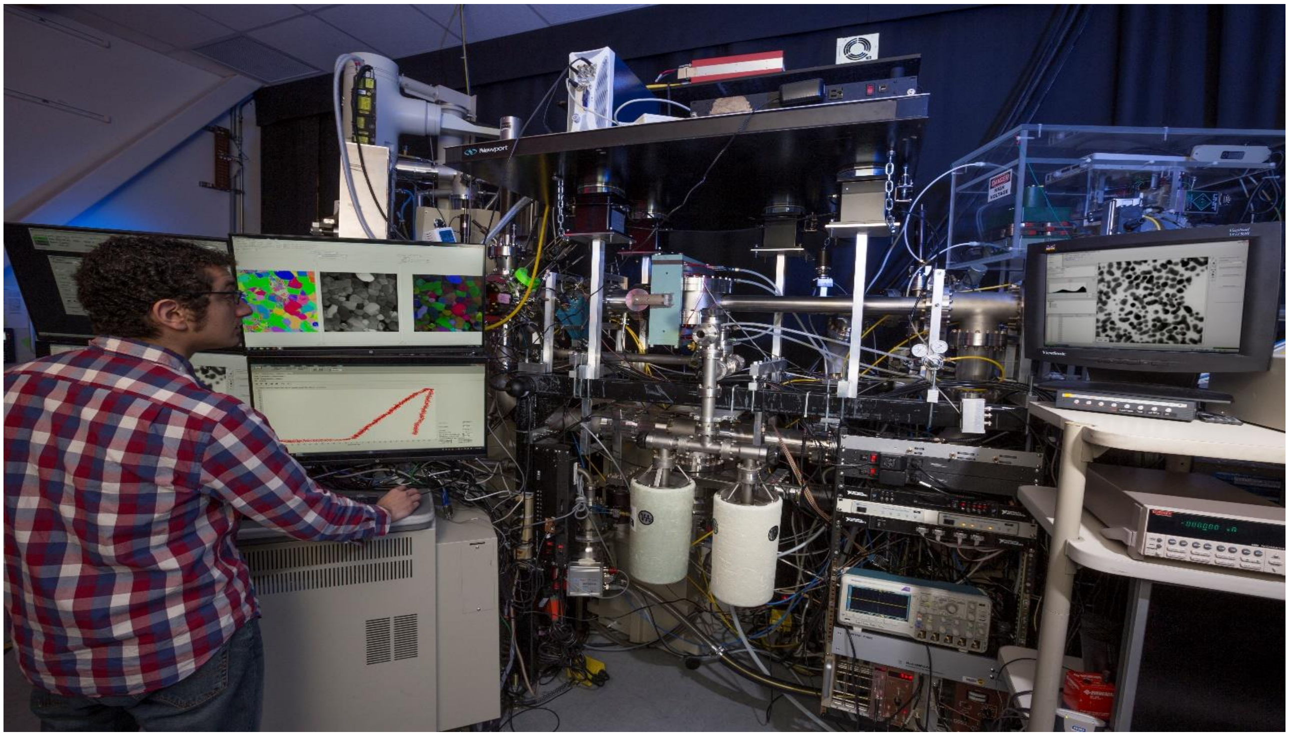

Figure 1. The in-situ ion irradiation transmission electron microscope (I^3TEM) that combines a highly modified JEOL 2100, a 6 MV tandem accelerator, a 1 MV tandem accelerator, a 10 kV Colutron, a 1064 nm pulsed laser, and a gas injection system (GIS) in order to explore a range of coupled extreme environments in real time.

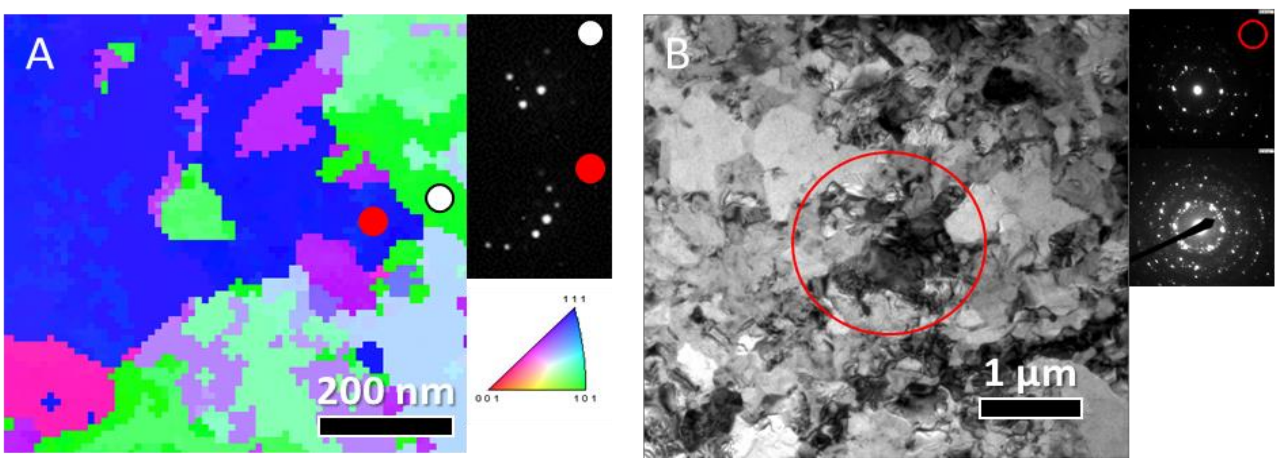

Figure 2. A) IPF-Z ACOM map of cubic GST crystallized following in-situ TEM heat treatment. Insets show diffraction patterns from indicated grains. B) BF-TEM micrograph of cubic GST crystallized and associated electron diffraction patterns indicate full crystallization following annealing and cooling demonstrating phase-change lock-in.

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[4] T Clark et al., Microscopy and Microanalysis **27**(S1) (2021), p. 1232-.
[5] This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. DOE's National Nuclear Security Administration under contract DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the U.S. DOE or the United States Government.

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