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Coupling Extreme Environments in the SEM: Present and Future Developments

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A full understanding of material evolution during in-service applications requires visualization of material evolution in real-time. Micro- and nano-scale material features dictate material form and function, and thus dictate the properties and performance. The ability to image materials on this scale in real-time will offer the transient information lacking from typical pre- and post-mortem materials characterization [1, 2].

In addition, we must also probe materials in their simulated real-world extreme environments, including heat, cooling, irradiation, strain, and corrosion, and any number of combinations of those external stimuli providing a synergistic environment. The combined effects of these on the evolution of a material microstructure is not well defined with high time fidelity. To improve the understanding of material transients, we must rely on in-situ experiments in simulated environments. In-situ electron microscopy experiments in the transmission electron microscope (TEM) have routinely employed gas exposure, ion irradiation, liquid flow, heating, and cooling to investigate material evolution on the nanoscale [3]. However, limited systems exist for micro-scale, real-time analysis of composition, microstructure, and properties.

This presentation will highlight the development of an in-situ electron microscopy facility combining synergistic environmental stressors at Sandia National Laboratories for the micro-scale. The in-situ ion irradiation scanning electron microscope (I^3SEM) at SNL is a newly-operational, combined environment SEM for studying material composition and texture evolution under simulated extreme environments. The system combines a JEOL JSM-IT300HR/LV with multiple irradiation sources, heating, cooling, liquid cell, and mechanical testing stages. The I^3SEM is coupled to the 6 MV Tandem accelerator for MeV-level heavy ion implantation and a 1.2 kV KDC-10 high current ion source for low energy surface modification. Heating and cooling stages allow the ability to couple irradiation with thermal extremes, from -50 C to 800 ˚C. Rapid, localized heating is investigated through the integration of a JSDU High-Power 808 nm laser, connected through an optical viewport entering the chamber 30-degrees relative to the electron beam. A 4.0 W peak power output coupled with a 105 µm aperture allows for millimeter-sized, site-specific heating in continuous or pulsed mode for analysis of thermal transients. Liquid cell stages allow for in-situ studies of corrosion, and ion irradiation-enhanced corrosion, with future developments expanding beyond static liquids to integrating a flowing liquid cell. In-situ straining in compression, tension, and fatigue can be investigated with 3 mechanical testing stages and coupled with ion irradiation and heating for coupled analysis of material property evolution under synergistic stimuli. Finally, an EDAX Octane Super Elite EDS detector and Velocity Super EBSD camera allow for in-situ analysis of elemental composition and crystallographic changes during exposure to the synergistic extreme environments [4].

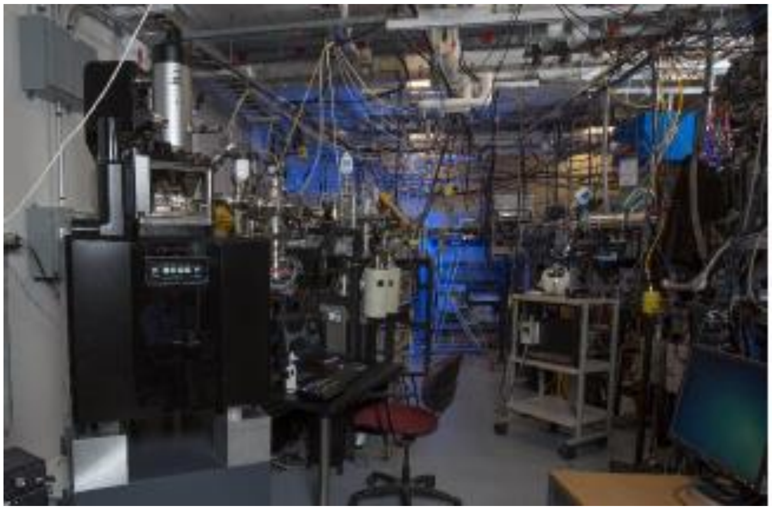

Figure 1. The I^{3}SEM at Sandia National Laboratories, showing the IT300 SEM, beamline for the 6MV Tandem, and 1.2 kV KDC-10 ion source.

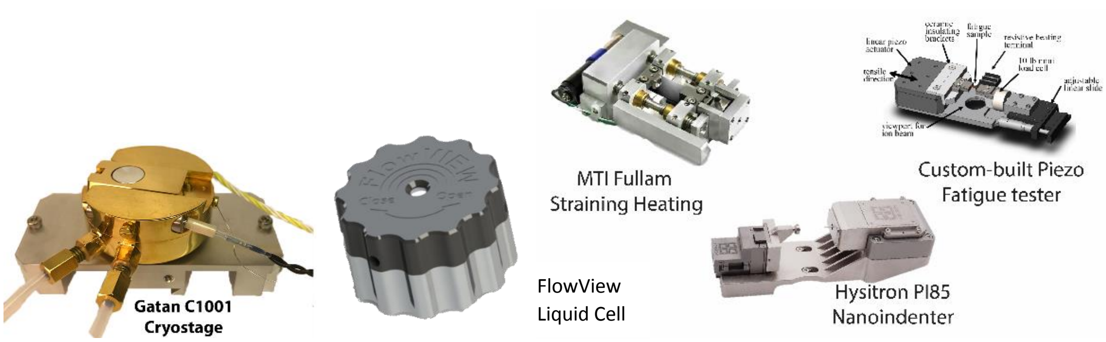

Figure 2. Suite of SEM stages for in-situ material evaluation under (from left to right) cooling, liquid immersion, heating, and straining.

References:

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[2] IM Robertson et al., Journal of Materials Research **26**(11) (2011), p. 1341.
[3] RJ Parrish et al., Microscopy Today **29**(1) (2021), p. 28.
[4] 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 multi-mission laboratory managed and operated by National Technology and 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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