Development of the In-Situ Ion Irradiation SEM at Sandia National Laboratories

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Monitoring materials evolution in extreme environments using in-situ techniques allows for increased insight into the active mechanisms behind materials transformations and an enhanced understanding of the temporal dependencies of a given materials response. These types of experiments are especially useful for studying the combinatorial effects of super-imposed environmental stressors, such as those posed by nuclear reactor and radiation environments. The In-situ Ion Irradiation Transmission Electron Microscope (I³TEM) at Sandia National Laboratories (SNL) was developed to study such microstructural changes in thin, electron transparent specimens [1]. The capability to study the effects of radiation damage, high temperatures, mechanical stresses, and other environmental variables has been demonstrated in several publications [2-4]. However, the I³TEM does not allow for the study of mesoscale and surface transformation, and in-situ TEM experiments in general have been known to be subject to various thin-film effects. As such, a complementary microscopy capability enabling work at larger length scales has been developed in the In-situ Ion Irradiation Scanning Electron Microscope (I³SEM) facility.

The I³SEM facility mates a JEOL JSM-IT300HRLV FEG SEM with a 6 MV EN Tandem Van de Graaff-Pelletron accelerator. The SEM is equipped with the largest specimen chamber offered and is capable of 1.5 nm resolution and operating in low-vacuum modes with pressures up to 150 Pa. The Tandem is coupled with four different ion sources (SNICS, Alphatross, duoplasmatron proton source, and Hiconex 834 sputter source) allowing for a wide range of ion species to be produced and directed into the SEM chamber at energies ranging from 800 keV to 88 MeV. The two are mated via a custom adapter utilizing the WDS port of the stock JEOL SEM chamber. This port also allows for installation of a Kaufman & Robinson KDC 10 gas-fed ion source capable of high-current (>10 mA) implantation of He or other gaseous species with energies from less than 100 eV to 1.2 keV. The Tandem and the KDC 10 source can be operated simultaneously for dual-beam experiments. While the secondary radiation produced during ion irradiation tends to saturate the secondary electron detectors, the ion beam can be interrupted for electron imaging, allowing for pseudo in-situ observation of microstructural evolution.

These ion irradiation capabilities can be paired with various in-situ SEM testing stages, such as the Hysitron PI-85 picoindenter, the MTI-Fulham heating/straining tensile stage, and a custom-built piezo-fatigue stage [5,6], allowing for radiation creep, radiation fatigue, or simply high-temperature irradiation experiments to be performed. The SEM is also equipped with EDAX Velocity™ fast-scan EBSD camera and an EDAX Octane Elite silicon drift detector for EDS analysis, thus enabling studies on grain structure evolution or chemical segregation during pseudo in-situ irradiation. This suite of capabilities enables a plethora of mesoscale studies of materials behaviour in extreme environments.

Initial proof-of-concept experiments have been performed using 20 MeV Au⁺ ions into a boron-doped polymer. As demonstrated in Figure 2, the formation of ion-induced cracking on the surface of the
polymer was observed in-situ. Experimental access to the I$^3$SEM facility is available through both the Nuclear Science User Facilities (NSUF) and the Center for Integrated Nanotechnologies (CINT) user proposals [7].

References:

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Figure 1. Image of the I$^3$SEM facility at the Sandia National Laboratories Ion Beam Laboratory.

Figure 2. A boron-doped polymer imaged in the I$^3$SEM before irradiation and after 720 seconds of irradiation with 20 MeV Au$^+$ ions.