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Recent Development in *In situ* Ion Irradiation Transmission Electron Microscopy

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In order to understand the structural response of materials at the nanoscale to various extreme environments, an effort has been made since nearly the inception of transmission electron microscopy (TEM) to simulate these environments within the microscope. To simulate radiation environments in the TEM, microscopists have capitalized on the original serendipitous observation of dislocation loop formation from *in situ* oxygen ion irradiation from a contaminated tungsten filament made by Pashley and Presland in 1961 [1]. In the first 50 years of transmission electron microscopes with *in situ* ion irradiation capabilities, a total of 31 facilities around the world have been publicly demonstrated with 11 known to be operating in 2011 [2]. Recent advancements occurring in both TEM and ion beam line technology are subsequently incorporated into the new *in situ* TEM ion irradiation facilities providing ever increasing ion beam control, TEM resolution, analytical capability, and *in situ* capabilities.

The newest *in situ* ion irradiation TEM (I^3TEM) facility to be brought into operation is at Sandia National Laboratories' new Ion Beam Lab that contains five accelerators and custom end stations for *in situ* optical, electron, and ion microscopy studies. The I^3TEM is a combination of a JEOL 2100 LaB_6 TEM with a 6 MV Tandem Accelerator and a 10 kV Colutron that provides adequate ion beam characterization and control, pumping capability, as well as vibration and electrical isolation of the three systems (Figure 1A). Recent real time videos of experiments will be presented in which the combination of heavy ion irradiation and light ion implantation were applied to a well-characterized severe plastically deformed tungsten sample [3]. This facility also permits enhanced three-dimensional data collection with a high-tilt pole piece, automated software, and a high-tilt TEM stage (Figure 1B). This presentation will highlight some of the initial work done to create four-dimensional reconstructions of both gold nanoparticles and tungstate nanoscintillators, as a function of ion dose and ion species [4]. Other advanced capabilities that achieved with the I^3TEM incorporate the combination of *in situ* ion irradiation and quantitative small scale mechanical property testing (Figure 1C). With this technique, Mo-alloy nanofibers [5] were irradiated *in situ* with 3 MeV Cu to a dose of approximately 1×10^7 and then strained to failure using a Hysitron "push-to-pull" (PTP) device. Initial results from these studies will be discussed and demonstrate the significant potential for the combination of *in situ* ion irradiation and quantitative small scale mechanical testing (Figure 2). Finally, a discussion on the continued development of the I^3TEM facility at Sandia National Laboratories and the possible future directions for transmission electron microscopes with *in situ* ion irradiation capabilities will be presented [6].

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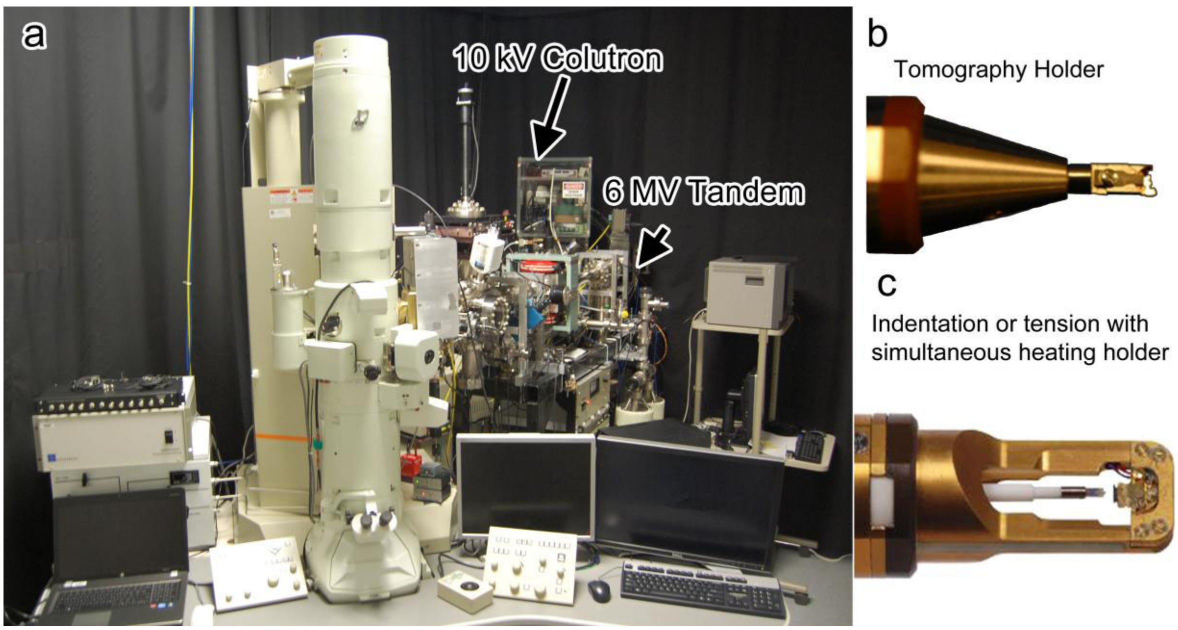

Figure 1. A) Sandia National Laboratories' *in situ* ion irradiation TEM. B) Hummingbird high-tilt stage for 4D reconstructions. C) Hysitron PI-95 stage for quantitative small-scale mechanical testing.

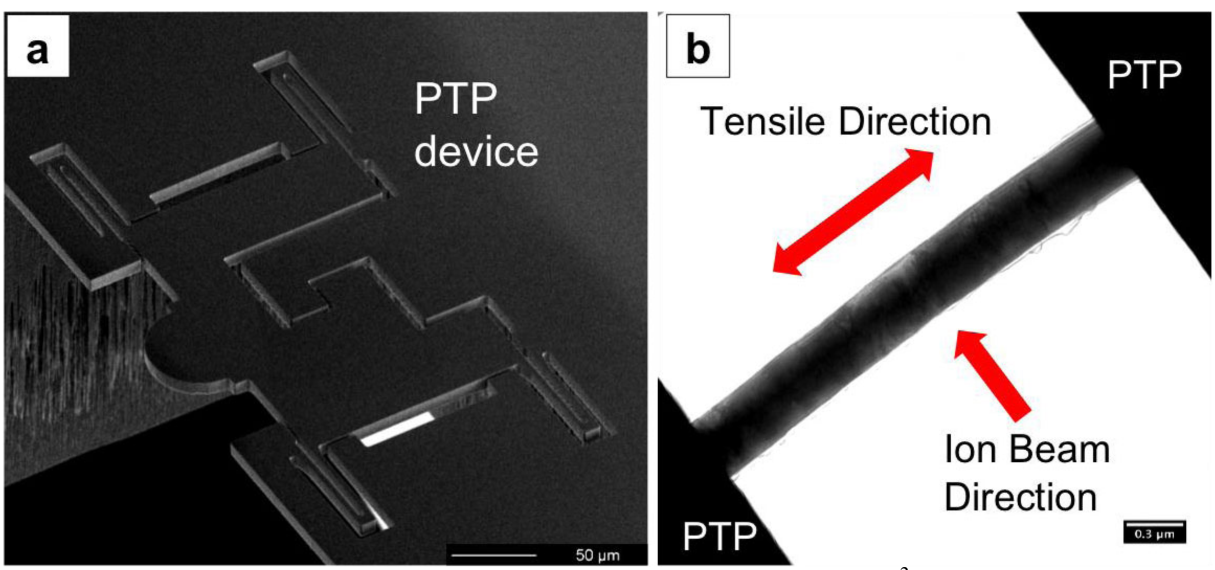

Figure 2. A) SEM micrograph of the Push-to-Pull device operated in the I^3TEM B) TEM image of Mo-alloy tensile sample in the experimental setup.

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