Sandia Ion Beam Laboratory
NSUF Partner Facilities Description

Further information regarding Sandia's Ion Beam Lab:

http://www.sandia.gov/research/facilities/technology_deployment_centers/ion_beam_lab/

http://tours.sandia.gov/tours.html

In-situ Ion Irradiation Transmission Electron Microscope (I^3TEM):

The I^3TEM facility at the Sandia Ion Beam Laboratory is a dedicated in-situ microscopy station that specializes in characterization of material microstructural evolution in a variety of extreme environments relevant to nuclear applications [1-3]. The I^3TEM combines the characterization capabilities of a modern, 200 kV JEOL 2100 high-contrast TEM with the implantation/irradiation capabilities of the 10 kV Colutron and the 6 MV Tandem accelerators housed in the Sandia Ion Beam Laboratory (Figure 1). The Colutron allows for low-energy implantation of gaseous species (primarily H_2, D_2 and He, but also heavier diatomic and noble gases), while the Tandem can produce numerous species of sputtered, swift heavy ions using various cathodes (i.e. H, He, C, Si, Fe, Au, etc.). Their combined capabilities allow for dynamic observation of radiation damage events using a variety of ion species at energies up to 48 MeV, in addition to the ability to perform triple-beam ion irradiation experiments (deuterium, helium, and heavy ions simultaneously), providing the ability to fine-tune experimental irradiation conditions to most closely emulate synergistic effects of in-core neutron exposure response while providing the ability to deconvolute governing factors [4, 5]. A diagram illustrating the irradiating species and energies that have been used in the I^3TEM to date is shown in Figure 2.

This in-situ characterization capability is further supplemented by a number of specialized TEM stages. These include heating, cooling, electrical biasing, liquid mixing [6], and vapor phase stages [7], in addition to three straining stages (Gatan 654 Straining Stage, Gatan 672 Heating/Straining Stage, Hysitron PI95 Picoindenter Stage [8]) and two tomography stages (Gatan 925 double-tilt/rotate stage, Hummingbird 1000 Series high-tilt tomography stage [9]). This collection of sample holders allows for investigation of radiation damage effects and interactions in a number of extreme environments, including temperatures and coolant environments relevant to nuclear reactor operation, whereas the straining stages allow for direct correlation of radiation-induced microstructural changes to change in mechanical properties and deformation behavior.

Finally, the I^3TEM itself has the added means for photoluminescence, cathodoluminescence, and ion beam-induced luminescence experiments and detailed orientation imaging microscopy and precession electron diffraction capability via the Nanomegas and ASTAR systems [10], allowing for high resolution mapping of texture and grain boundary character in a specimen. Installation of dynamic TEM (DTEM) capability, allowing for imaging with nanosecond time resolution, is expected to be completed shortly. Combining this myriad of capabilities into one instrument has resulted in one of the most comprehensive and flexible facilities for in-situ characterization of material microstructure and performance in a variety of environments with a specific focus on radiation damage effects.

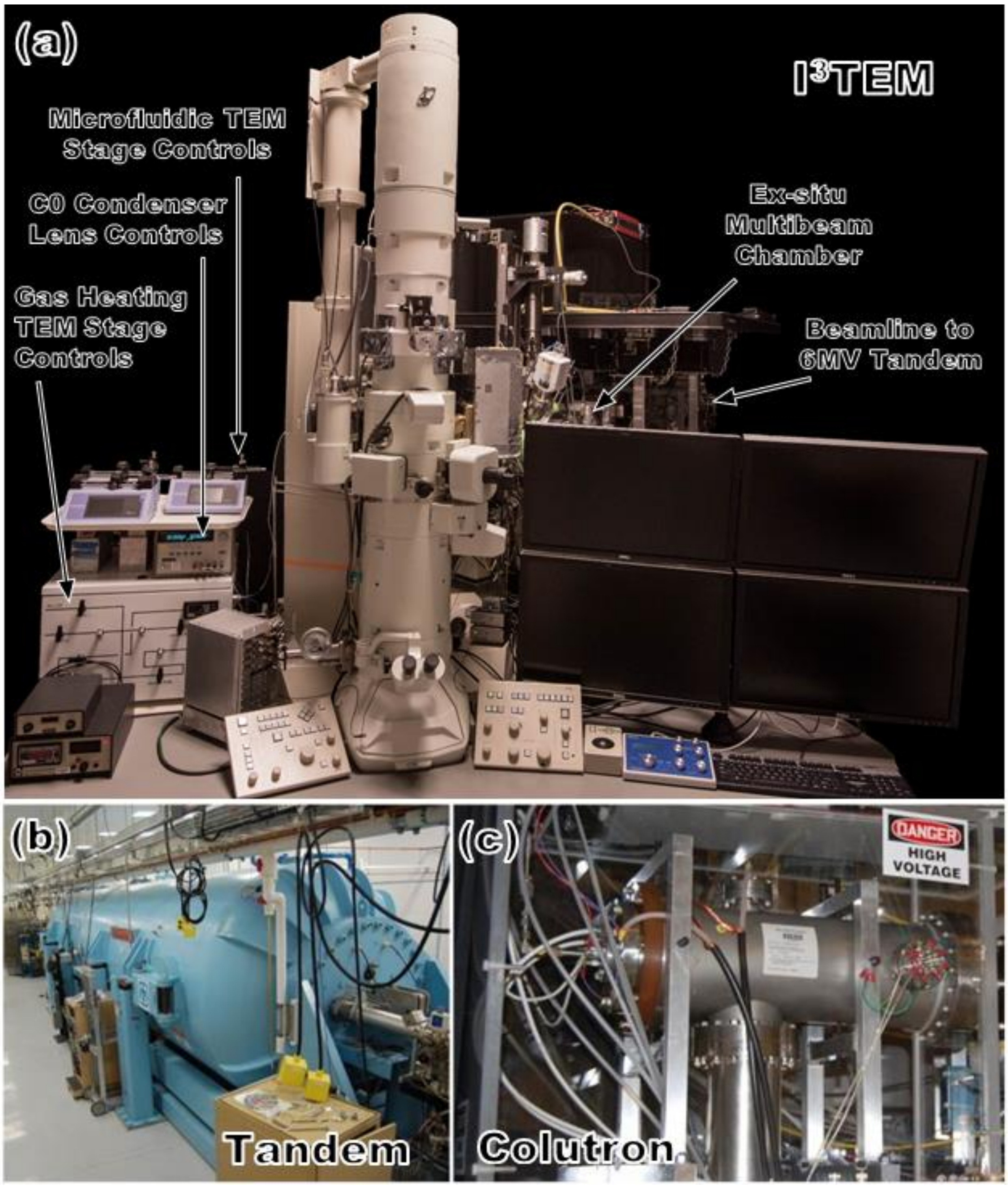

Figure 1: (a) I^{3}TEM and associated controllers and instrumentation. (b) 6 MV Tandem accelerator. (c) 10 kV Colutron accelerator.

In-situ Ion Irradiation Scanning Electron Microscope (I^{3}SEM)

The JEOL JSM-IT300HRLV 30 kV FEG SEM housed at the Sandia Ion Beam Laboratory is meant to complement the I^{3}TEM facility by providing similar analytical capabilities for larger-scale and bulk specimens and has been designed to be the world's best in-situ SEM for overlapping extreme environments. However, development of this facility is still a work-in-progress and as such, the final configuration is still fluid.

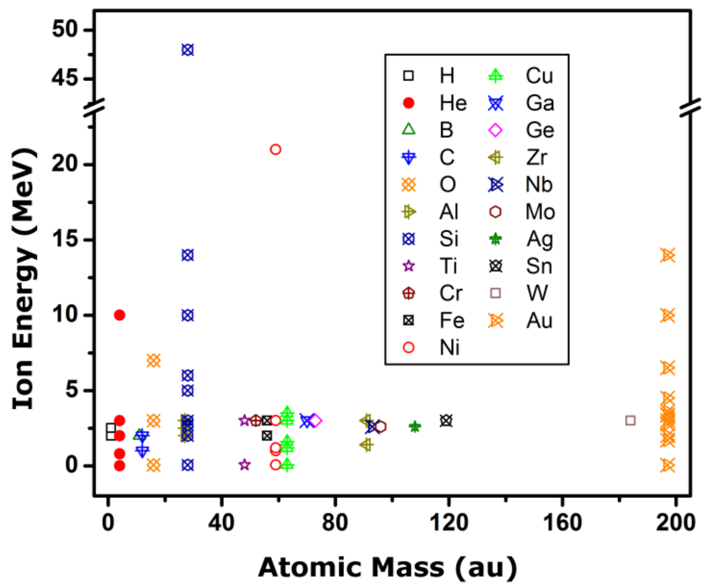

Figure 2: Diagram illustrating ion species and energies introduced into the I^{3}TEM to date

First and foremost, the final instrument will be directly connected to the 6 MV Tandem beamline, allowing for similar heavy ion irradiation capability to the I^{3}TEM (Figure 2). Low-energy gas implantation will be accomplished with a separate low-energy ion source mounted directly to the I^{3}SEM chamber. Together, high dpa and gas implantation levels can be achieved on the surface of bulk specimens through micrometer thick samples, allowing for in-situ studies of blistering in ceramic fuel specimens in addition to small-scale mechanical testing. Stages for in-situ mechanical testing experiments include the Hysitron PI-85 picoindenter, the MTI/Fullham heating/straining stage, and a custom piezo fatigue stage [11-13].

Figure 3: JEOL JSM-IT300HRLV being installed in the I^{3}SEM facility

Finally, the 12 ports featured by this large chamber SEM allow for the installation of several analytical instruments and detectors to supplement the I^{3}SEM's capabilities. EBSD detectors will be installed initially allowing for orientation and chemical mapping, respectively.

Sandia Ion Beam Laboratory Neutron Capabilities

The Sandia Ion Beam Laboratory also has the ability to generate neutrons through either D-D or D-T fusion reactions. This is performed by using a High-Voltage Engineering 350 kV Cockcroft-Walton accelerator to direct energetic deuterium ions into a deuterium- or tritium-based source [14, 15]. This results in near-isotropic emission of monoenergetic neutrons with energies of 2.45 or 14.1 MeV for the D-D and D-T reactions, respectively. This technique can generate a chosen flux of neutrons up to approximately 10^6 n/cm^2·s and allows for pulses on timescales of approximately 100 ns. This out-of-core neutron source allows for a variety of experimental capabilities, including fast/prompt neutron activation analysis, fast neutron radiography, subcritical fission, fast neutron damage, neutron time-of-flight, and neutron microscopy experiments.

Relevant publications:

1. Hattar, K., D.C. Bufford, and D.L. Buller, *Concurrent in situ ion irradiation transmission electron microscope.* Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 2014. **338**: p. 56-65.
2. Bufford, D. and K. Hattar, *The Design and Implementation of a Single, Double, and Triple Concurrent Beam In Situ Ion Irradiation TEM Facility.* Microscopy and Microanalysis, 2014. **20**(S3): p. 1562-1563.
3. Chisholm, C., K. Hattar, and A.M. Minor, *In situ TEM concurrent and successive Au self-ion irradiation and He implantation.* Materials Transactions, 2014. **55**(3): p. 418-422.
4. Sun, C., et al., *Microstructure, chemistry and mechanical properties of Ni-based superalloy Rene N4 under irradiation at room temperature.* Acta Materialia, 2015. **95**: p. 357-365.
5. Barr, C.M., et al., *Grain boundary character dependence of radiation-induced segregation in a model Ni–Cr alloy.* Journal of Materials Research, 2015. **30**(09): p. 1290-1299.
6. Chee, S.W., et al., *Studying localized corrosion using liquid cell transmission electron microscopy.* Chem Commun (Camb), 2015. **51**(1): p. 168-71.
7. Hattar, K., S. Rajasekhara, and B.G. Clark, *In situ TEM ion irradiation and atmospheric heating of cladding materials.* MRS Proceedings, 2012. **1383**.
8. Bufford, D.C., et al., *High Cycle Fatigue in the Transmission Electron Microscope.* Nano Lett, 2016. **16**(8): p. 4946-53.
9. Hoppe, S.M., et al. *Application of in-situ ion irradiation TEM and 4D tomography to advanced scintillator materials*. in *Penetrating Radiation Systems and Applications XIII*. 2012. San Diego, CA, USA.
10. Bufford, D.C., et al., *Unraveling irradiation induced grain growth with in situ transmission electron microscopy and coordinated modeling.* Applied Physics Letters, 2015. **107**(19): p. 191901.
11. Battaile, C.C., et al., *Quantifying uncertainty from material inhomogeneity*. 2009, Sandia National Laboratories.
12. Carroll, J.D., et al., *The effect of grain size on local deformation near a void-like stress concentration.* International Journal of Plasticity, 2012. **39**: p. 46-60.
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14. Styron, J.D., et al., *Predicting the sensitivity of the beryllium/scintillator layer neutron detector using Monte Carlo and experimental response functions.* Rev Sci Instrum, 2014. **85**(11): p. 11E617.
15. Ruiz, C.L., et al., *Progress in obtaining an absolute calibration of a total deuterium-tritium neutron yield diagnostic based on copper activation.* Rev Sci Instrum, 2012. **83**(10): p. 10D913.