University of California Berkeley Nuclear Materials Laboratory

Berkeley ATR facility and instruments:

The University of California Berkeley offers the capability of radioactive sample preparation (polishing, cutting, grinding, mounting) as well as post-irradiation examination (SEM/FIB, nano-indentation, in-situ indentation) to the NSUF. The total allowable activity of the samples is determined by the specific isotopes, the sample geometry and volume. Small amounts of radioactive material can be handled and prepared upon contact with Prof. Peter Hosemann. The hard limit is currently 750mR/h on contact but can be expanded upon request depending on the task.

Rough-cutting tasks can be performed in a certified stainless steel glove box to avoid contamination of the environment. The glove box contains a minimet polishing setup, hot plate, microscale, and a slow speed cut off saw. A second 1-inch thick lead-shielded box is available upon request. Further polishing can be performed in the hoods certified for active work as shown in Figure 2.

Figure 1: Stainless steel glove box for radioactive sample preparation. The box contains an Allied slow speed cut off saw, a Buhler minimet polisher, and a hot plate. The glove box is located in a separate lockable part of the nuclear materials laboratory to ensure access only to trained users a). Further sample preparation on activated materials can be performed in the certified hoods as shown in b).

Polished and mounted radioactive and not radioactive samples can be transferred to a new state-of-the-art micromaterials nanoindenter. The instrument has two load ranges 0.1mN to 500mN and from 1mN to 20N. The loading is performed via a lever system and electromagnet. The instrument is also equipped with a high-power optical microscope and an automated lens changer to allow accurate positioning of indents on a sample. In addition, a scanning option exists and allows precise positioning at elevated temperatures. The current system is in an environmentally controlled box, which purges the system with noble gases to avoid oxidation upon heating. The low load option can heat the sample and the cBN tip up to 750°C. In high load mode sample and tip can be heated up to

500°C. After a sufficient waiting time at test temperature (to minimize drift) the measurements can be performed. An image of the instrument can be seen in Figure 2:

Figure 2: Nanoindenter available at UCB to NSUF users. The environmental box is visible allowing for inert environment a). Closer view of the horizontally mounted sample tip arrangement b) and microscopy image of the indenter and the sample (embedded TRISO fuel particles) c).

A quanta 3D FEG dual beam FIB is available that can handle activated samples. The system is equipped with GIS (Pt) EDS/EBSD (Oxford), S-TEM and Kleindick manipulators. In addition, a Hysitron PI85 with a load range up to 30mN is available and can be installed in order to perform in-situ mechanical testing.

Figure 3 shows images of the tools. The FIB can operate liquid nitrogen temperature and the mechanical tests can be performed at this condition using a hummingbird cry stage. The tool is part of the BNC user center at UC Berkeley but access for the NSUF has to be discussed with Peter Hosemann due to the high demand of the tool. Active samples are allowed.

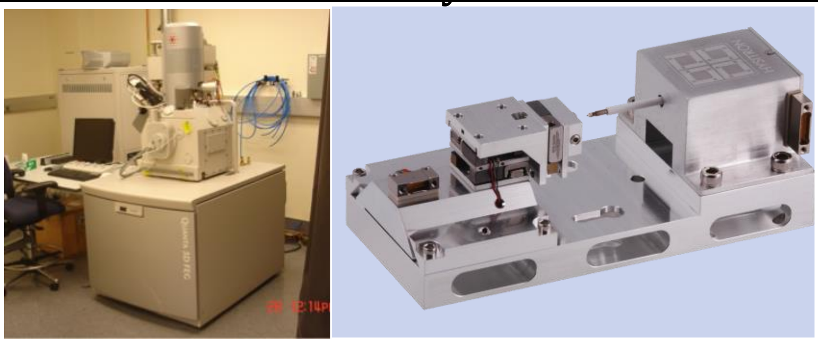

Figure 3: image of the Quanta 3d FEG tool as it is located at UC Berkeley a). Hysitron PI 85 in-situ nanoindenter available to fit this instrument b)

In 2015 a SEM (XL30) will be installed for initial SEM investigation. The tool will contain an Atomic Force microscope for in-situ AFM measurements in the SEM. This is especially interesting since the XL30 is a ESEM and in-situ studies can be performed. Also the above mentioned nanoindenter can fit in this tool. More details will be added as the tool comes online.

In 2015 the ORION Nanofab, a triple ion beam scanning microscope unifying He, Ne and Ga ions in one tool, becomes available. This tool is currently the only one of its kind at a user facility nationwide. While FIB precision is improved orders of magnitude he implantation can be performed in-situ. Further information will be provided as the tool becomes available in 2015.

Figure 4: ORION nanofab triple ion beam microscope. This tool will go online in 2015.

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Scientific capability:

The instrument allows one to measure hardness on multilength scales as well as perform microcompression testing using an in-house build goniometer and evaluate yield strength and work hardening rates on a localized level. Details about the instrument can be found at http://www.micromaterials.co.uk/the-nanotest/nanoindentation/

Examples of the type of tests that can be performed using the nanoindenter can be found in Figure 3. An array of nanoindents on a fiber matrix composite and the resulting hardness map are shown. Clear hardness differences between the fiber and matrix properties can be seen on this material. Figure 3b) presents hardness data gained on 1.1MeV ion beam irradiated steel. The ion beam induced hardening is clearly visible. In order to increase the depth resolution, the indents were performed in an angle. The scattering of the data results from multigrain measurements.

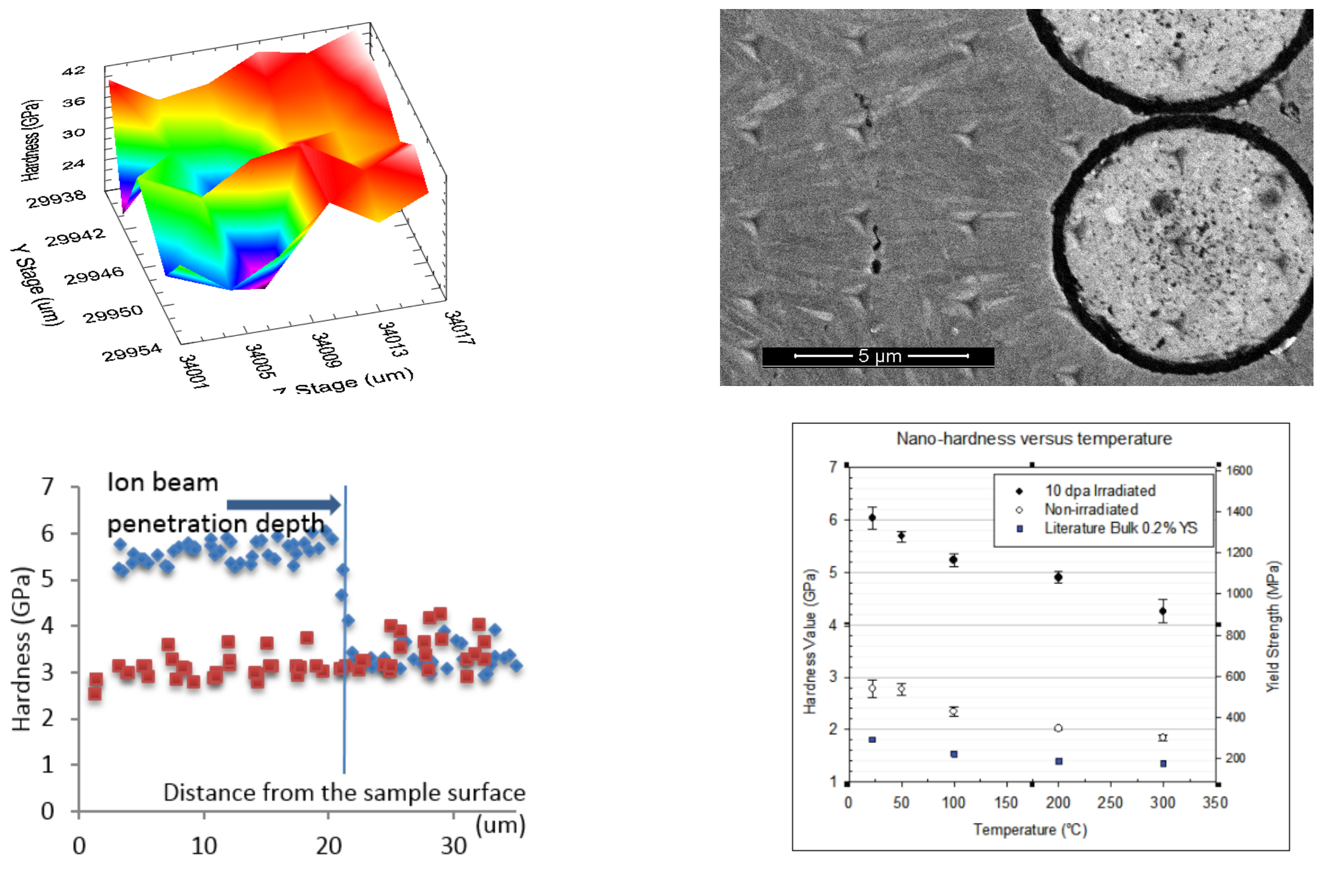
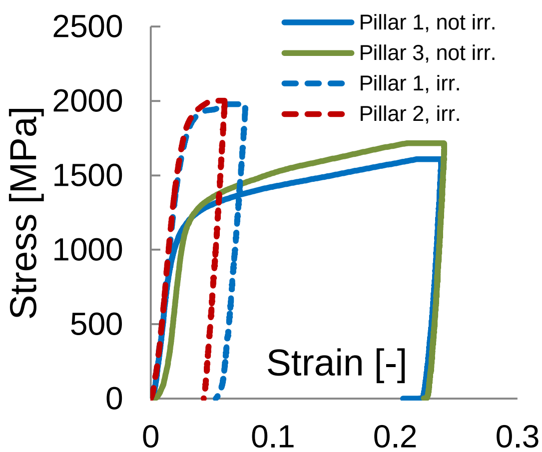


Figure 5: Indentation hardness mapping on SiC/SiC composite materials a) and b). Indentation hardness mapping on ion beam irradiated materials c. Hardness as a function of temperature on ion beam and not ion beam irradiated materials d).

For pillar fabrication, UCB also houses a quanta dual beam FIB equipped with cryostage, Kleindiek micromanipulators and a Pt GIS deposition system. While the FIB is currently not part of the user facility it is available to outside users upon request for our standard user rate. The FIB system is certified for work with radioactive materials and is currently in the process of being added to the NSUF.

Microcompression testing examples are shown in Figure 4. Data on several (irradiated and unirradiated) microcompression pillars are shown. A significant change in yield strength due to radiation damage can be found. The subsequent SEM image shows the slightly deformed pillar that developed a crack during testing. Recently, bend bar testing for brittle materials is of interest because fracture toughness data became accessible. Again, the UCB-NSUF instrument allows good tip positioning due to a high power optical microscope and accurate stage transfer.



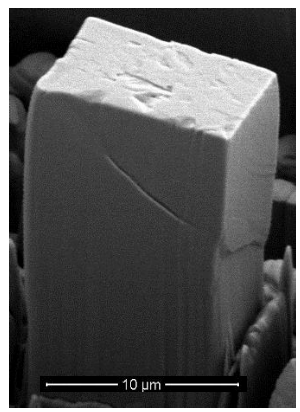
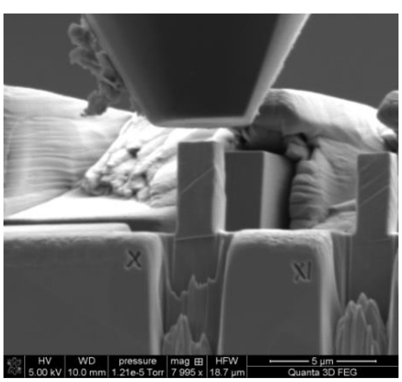
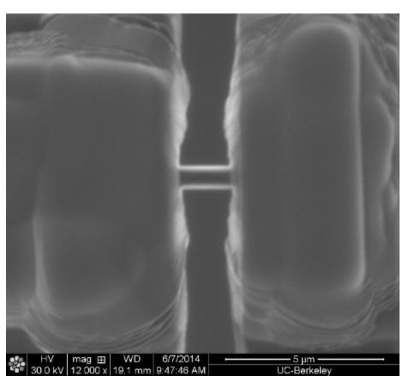


Figure 6: Microcompression test data on irradiated and unirradiated steels performed with the ex-situ micromaterials indenter at UCB.





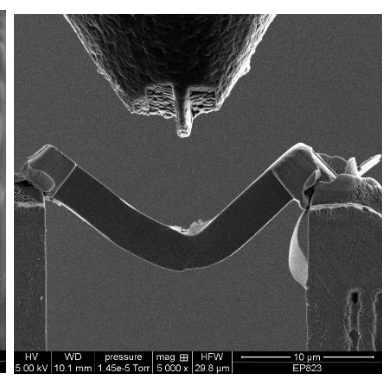


Figure 7: microcompression a), microtensile b) and micro 3-point bending c) geometry fabricated on irradiated materials using the Quanta dual beam FIB and tested using the Hysitron PI85 indenter.

User arrangement:

Before submitting a user proposal, it is recommended that the user contacts the UCB contact (Peter Hosemann) to discuss the planned experiment and to clarify details such as sample geometry, dose rate, etc. If the proposal is awarded, the details of the visit have to be discussed with the hosting organization. Several days to weeks can be scheduled at the UCB facilities. FIB time can be made available upon request. Please contact peterh@berkeley.edu for more information.