doi:10.1017/S1431927617002082
Microsc. Microanal. 23 (Suppl 1), 2017

Sample Preparation for Nano-mechanical Testing on Radioactive Materials

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Nuclear Materials research focusing on the degradation of materials utilized in nuclear environments has been struggling with the fact that the materials are radioactive after reactor exposure and therefore are difficult to handle. Large scale mechanical testing requires shielding and elaborate laboratory equipment to ensure the workers' safety. In addition, reactor irradiations take extensive amounts of time to reach appreciable doses on materials, making materials investigations a time and cost intensive effort. Ion beam irradiations can accelerate the accumulation of dose in materials, compared to reactors achieving the same dose in a matter of days that takes years in a reactor irradiation. However, ion beam irradiations do have the challenge of small penetration depths into the materials.

Both the enhancement of safety by reducing the volume of reactor irradiated materials and ion beam irradiations call for small volume investigations. While in the first instance one is interested in reducing the exposure of a scientist to radiation by reducing the volume of the material, in the second case one is interested in obtaining mechanical data from ion beam irradiated materials.

Regardless of the reason there is motivation for reducing the material's mechanical testing evaluation to smaller length scales. Of course it is also a key interest to investigate the material's microstructure as a result of the irradiation exposure and correlate the mechanical properties measured to the microstructural changes. Therefore, performing mechanical testing inside a microscope can help address some of these issues.

With the desire to create nano- and micro-mechanical test samples comes the challenge of sample preparation. Today, Focused Ion Beam (FIB) techniques are widely utilized to create such samples. In this work we will outline several procedures of creating a variety of nano- and micro-mechanical sample geometries as well as utilizing the modern He ion beam microscopes to induce He beam implantation.

Reactor irradiated samples have the challenge of a) being radioactive requiring radioactive working areas and dedicated Focused Ion Beam instruments and b) being bulk samples, making targeted preparation of specific regions of interest difficult, while ion beam irradiated samples have limited volume available to examine. Depending on the property of interest, various sample geometries are needed. While compression samples do allow the measure of yield strength and sudden slip deformation they do not allow a measure of strain to failure. Microbend samples allow more insight into the plasticity aspects, but the data analysis is difficult to conduct and may require modeling. Micro-tensile testing by contrast allows an assessment of plasticity while also reducing the need for modeling due to the uniaxial loading condition. Several approaches of manufacturing any of the aforementioned sample geometries exist using FIB techniques, and examples of the final geometries are shown in Figure 1. Depending on whether the samples are radioactive or specific targeted sample preparation is needed (e.g. grain boundary examination, crack tip examination, etc.) various techniques can be utilized with advanced FIB work.

In addition, this work features the manufacturing of TEM foils and micro pillars for subsequent *in situ* nano-compression testing where each pillar is irradiated with a different dose. Modern helium ion beam microscopes also allow implanting of helium in specific regions of interest in a sample, allowing for an array of multiple pillars to be created within the same grain, with each pillar implanted to a different dose.

We present different FIB based approaches to manufacture micro-mechanical and nano-mechanical samples for *ex situ* or *in situ* irradiation and subsequent mechanical testing. The aim is to demonstrate what is possible today in the processing of irradiated materials using FIB techniques [1].

References:

[1] The Authors want to thank the National Center for electron microscopy at Lawrence Berkeley Laboratory as well as the DOE-NEUP program for support. Work at the Molecular Foundry was supported by the Office of Science, Office of Basic Energy Sciences, of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231. Additional Instrument access was provided by the BNC.

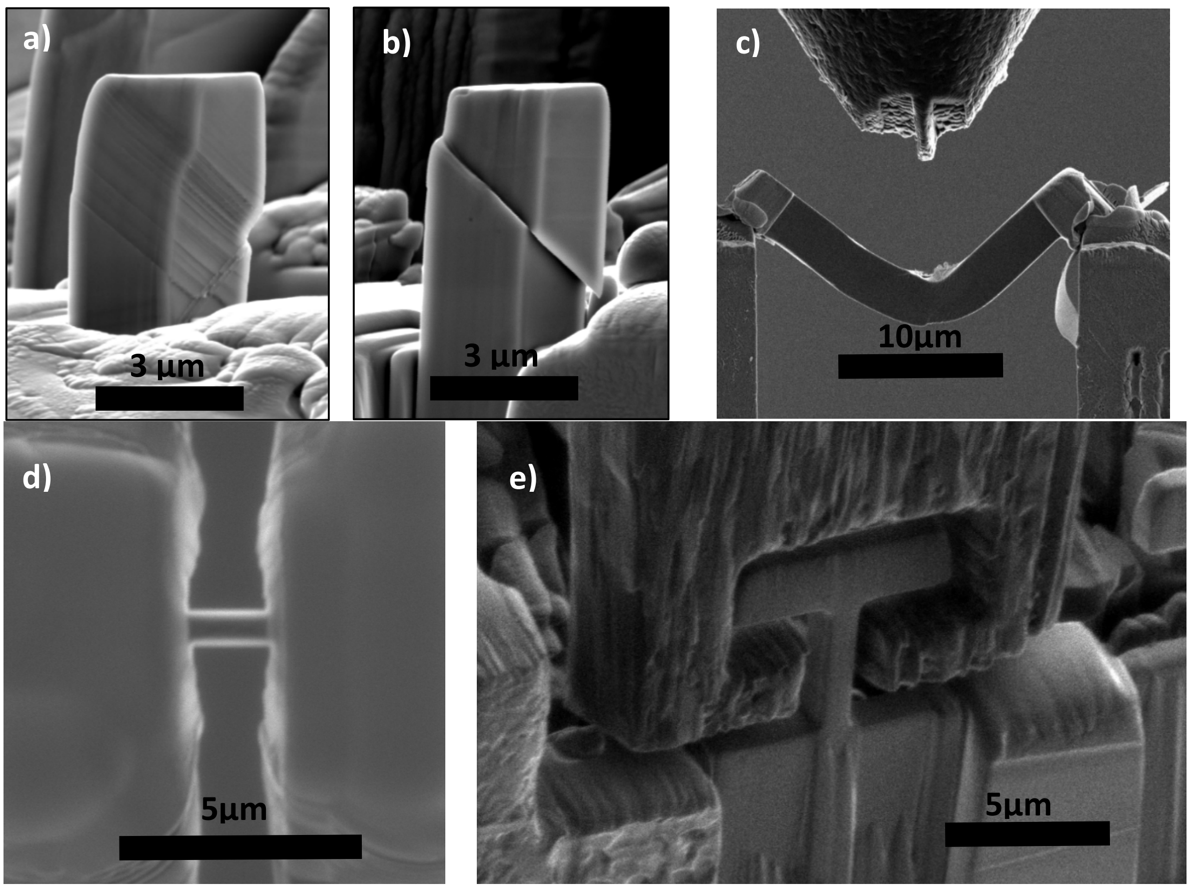

Figure 1. Micro-compression sample post testing on 304SS a) non-irradiated and b) irradiated Micro-compression sample post-testing c) Three-point bend testing on neutron irradiated material d). Tensile testing on irradiated material using a Push-to-pull device allowing targeted sample preparation e). Micro-tensile testing on irradiated material.

https://doi.org/10.1017/S1431927617002082 Published online by Cambridge University Press