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FIB-SEM for Nano-CT of Tritiated $LiAlO_2$ Pellet Nanopores

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Tritium producing burnable absorber rods (TPBAR) are used for production of the hydrogen isotope tritium (H^3) which is used for various national security applications. These rods are formed by concentric cylindrical layers, as shown in Figure1a; the main functional parts being an outer steel cladding for support, a zircaloy getter, and the $LiAlO_2$ pellet layer from which the tritium is converted. For production of tritium, these rods are placed in reactors and irradiated with neutrons which, in the pellet, convert the Li^6 to He^4 and H^3. This irradiation causes large changes in the microstructure. While as-fabricated pellets are typically porous, particularly around grain boundaries, after irradiation, pellets become porous near the center of the grains (Figure1b and 1c), with pores on the scale of ~1um or smaller. Since differences in pore formation can significantly affect the performance of a bulk material, it is imperative to characterize the development of the pores that form during irradiation[1]. To capture the morphology and networking of these pores and grain boundaries, a 3D mapping technique with resolution on the nano-scale is needed.

X-ray Computed Tomography (CT) has been an important technique for non-destructive analysis of the physical 3D structure and layout of samples ranging from soft organic tissues to geological specimens. Recent advances have allowed nano-scale features to be resolved even in general lab-based instruments, down to 50 nm resolution for the Zeiss Xradia, a feat which previously had only been available at synchrotrons [2]. This powerful advancement allows us to probe features that are key to understanding defects in material performance, such the porous nature developed in $LiAlO_2$ pellets under irradiation.

To achieve the desired resolution, the sample must be quite small in order to achieve enough intensity penetrating through the entire sample, typically smaller than 65 um in diameter. For high-quality data, it is best to have as uniform a sample shape as possible to mitigate signal attenuation due to varying thickness. Therefore, a cylindrical shape is often used. To attain the minute size and specific shape required from site-specific regions, a Helios 660 dual beam Ga FIB-SEM is employed.

The use of the FIB-SEM for creation of CT samples has been well documented [3]; however, many of the methods require removal of large amounts of material, which may be undesirable when preparing a radioactive sample and attempting to minimize the amount of contamination produced. Considering this, the method often used for preparation of atom probe tomography (APT) needles was used, creating a wedge-shape sample which is mounted on the tip of a needle, as seen in Figure 2. In this case, both W needles and steel sewing pin needles were used. The needle tips were polished flat, then the samples were mounted on the tips and coated with both Pt and Au. The samples were then probed at Washington State University (WSU) at their Institute of Materials Research in the Zeiss Xradia Ultra 810. Resulting 3D reconstructions were analyzed in Thermofisher’s data analytic software Avizo [4].

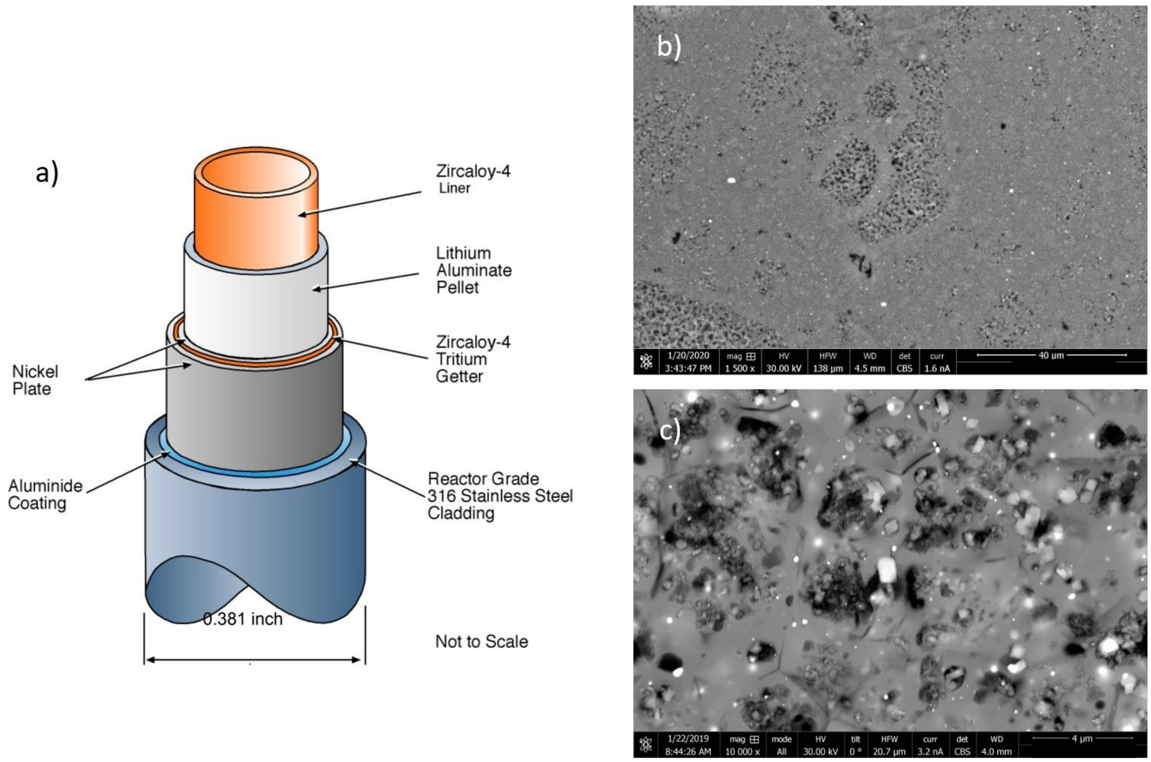

Figure 1. a) Schematic of the TPBAR core. b) SEM images of the surface of an irradiated LiAlO2 pellet.

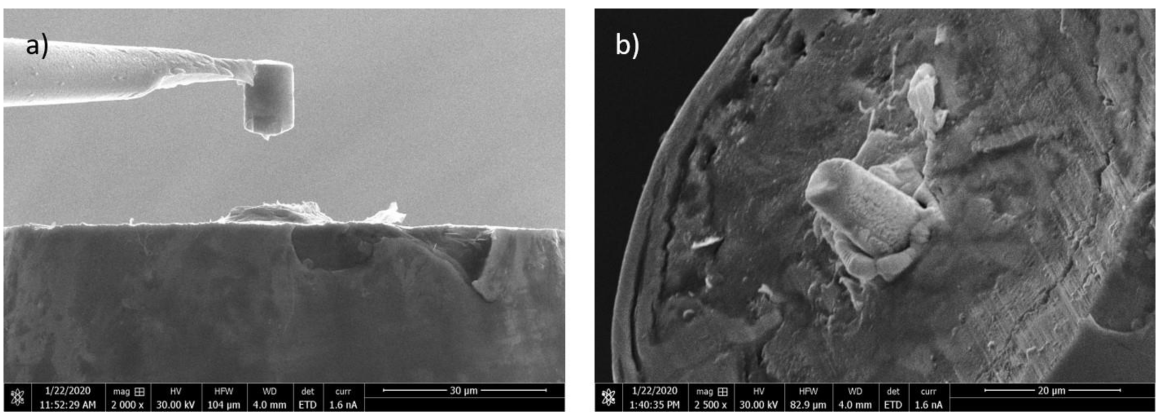

Figure 2. a) The sample being mounted on the polished needle tip. b) The needle polished to a cylindrical shape.

References
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[4] We would like to thank Marc Weber at Washington State University for his expertise and use of the Zeiss Xradia Ultra 810.