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Correlative Micro-CT and FIB-SEM Tomography for Refined Macro-scale Pore Volume Measurements in TPBAR $LiAlO_2$ Pellets

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Tritium producing burnable absorber rods (TPBAR) are used for production of the hydrogen isotope tritium (^3H). These rods are formed by concentric cylindrical layers; the main tritium producing part being the $LiAlO_2$ pellet layer in which ^6Li, under neutron irradiation, is converted into ^3H and ^4He ions, often creating small (<1 um) pores in the process. Porosity in these materials, whether from irradiation or from the manufacturing, can significantly affect the performance of a material, so it is important to characterize the prevalence, morphology, and networking of these pores. Micro- X-ray Computed Tomography (µCT) has been an important technique for non-destructive analysis of the physical 3D structure and is capable of analyzing samples on the macro-scale (> 1 mm); however, the resolution is often not fine enough for resolving features such as pores on the scale of 1 um or smaller. Additionally, actual resolution in µCT varies widely with samples size. Correlative µCT and Focused Ion Beam Scanning Electron Microscope (FIB-SEM) tomography have in the past few years been found useful for locating features well below the sample surface for extraction by FIB or for chemical or crystal orientation analysis. The two techniques have also been combined to analyze volumes for a finer understanding of the microstructure observed by µCT.

Previous work shows nano-CT (nCT) is capable of successfully mapping microstructural features down to ~100 nm in this material.[1] However, the scale of those volumes was < 10 um in any one dimension, a scale on which the microstructure in these materials varies wildly and cannot tell us about the porosity on the macro-scale. Here we apply a correlative µCT and FIB-SEM tomography to measure pore volume in TPBAR pellets using features below those resolvable by µCT but well within the bounds of SEM resolution to obtain a refined and corrected µCT pore volume that can be scaled to the macro-scale. Unirradiated and neutron irradiated samples are examined to determine if this technique can even account for increased porosity arising from irradiation.

Standard unirradiated TPBAR pellet tubes are obtained and fiducials are milled onto the surface using a Thermofisher G4 Hydra PFIB with the Xe+ source. The full tubes are analyzed by a Thermofisher Heliscan µCT for maximum volume analysis, though this requires a lower resolution. The samples are then cut so the portion with a fiducial is roughly 1 x 1 x 12 mm^2. This reduced sample volume is run again through the µCT to obtain higher resolution data. All the µCT data are then reconstructed and analyzed in Thermofisher's 3D imaging and analysis program Avizo. Due to time constraints, FIB-SEM tomography with the Hydra PFIB is volume limited, so regions of particular interest are selected from the higher resolution µCT data. The FIB-SEM tomography data for each region are aligned and reconstructed in Avizo. Porosity volumes from each method are compared to determine if a consistent deviation from the more precise FIB tomography measurement could be obtained. Figure 1 shows the general workflow of the study.

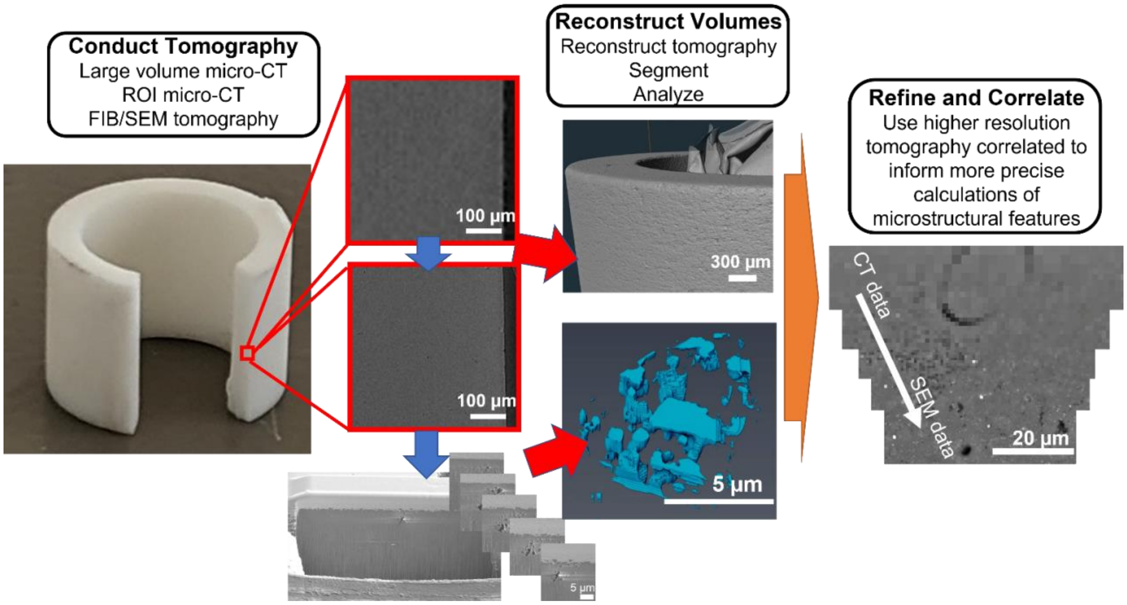

Figure 1. A schematic overview of the work. TPBAR pellets are analyzed by µCT as full tubes at a lower resolution. Regions of interest were cut from this tube for µCT at higher resolution and portions of that are analyzed by FIB-SEM tomography. Volumes are reconstructed, segmented, and analyzed for pore volume. Higher resolution tomography is used to inform the larger scale, lower resolution tomography.

References:

[1] BE Matthews et al., Technical report PNNL-30558 (2020).
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