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Focused Ion Beam Serial Sectioning and 3D-Reconstruction of Nuclear Fuel

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The combined use of focused ion beam (FIB) milling and scanning electron microscopy (SEM) via serial sectioning is a technique used to characterize the microstructure of a material in three dimensions [1, 2]. It has gained popularity over the last decade due to improved ion beam capabilities, and matured characterization capabilities and software to study microstructure, chemical state, etc. Three-dimensional data provides information that might be missed or unavailable using classic 2D imaging techniques. When studying radiation enhanced effects such as fission gas bubble density and microstructural evolution at fuel-cladding interfaces, radiation limitations reduce the number of techniques readily available for post-irradiation examination, while some tomography-related techniques have resolution limits that are unable to capture sub-micron features adequately. Additionally, using a dualbeam SEM/FIB for 3D reconstruction provides an opportunity to capture energy dispersive electron spectroscopy (EDS) data or electron backscatter diffraction (EBSD) data on targeted regions of interest between each serial sectioning step. Here, the application of this technique to different fuel systems will be discussed, including its benefits and limitations. These systems include tristructural isotropic (TRISO) coated particle fuel and high burnup light water reactor fuel.

FIB/SEM 3D-reconstruction was used to study TRISO fuel from the Advanced Gas Reactor (AGR) program [3]. TRISO fuel is an encapsulated fuel form which includes a fuel kernel surrounded by a layer of porous carbon, enclosed in a layer of pyrolytic carbon (IPyC), silicon carbide (SiC), and a final outer pyrolytic carbon layer [4]. It is the fuel of choice for high temperature gas reactors and its design is being considered for other reactor concepts such as as an accident tolerant fuel form for fully-ceramic microencapsulated (FCM) fuel. This study focused on the IPyC/SiC interface. The IPyC/SiC interface is of interest for many reasons: for example, it acts like a sink for fission products during irradiation and certain fission products can degrade the SiC at this interface, among others. Reconstruction of this region of the fuel kernel, particularly when combined with EDS and EBSD at regions of interest, can therefore provide insight on the fission product, pore, and microstructural relationships at this interface before and after irradiation. It allows for a larger area to be targeted as compared to TEM lamella, to provide better statistics for fission product migration in the material and microstructural changes as a function of irradiation. Here, serial sectioning and analysis on as-fabricated uranium-oxide/uranium-carbide (UCO) two phase kernel particles at the IPyC/SiC interface was conducted using a Thermo/FEI Versa 3D DualBeam FIB/SEM and Amira-Aviso reconstruction software for 3D visualization and analysis. Data will also be taken on samples that have been irradiated in the Advanced Test Reactor at Idaho National Laboratory under the AGR program. Figure 1 shows preliminary results of the IPyC/SiC interface from the as-fabricated fuel. Porosity is observed at the interface where the two layers are stitched together. As this was an unirradiated fuel, no fission products are observable at the interface. It is expected that this information will provide valuable insight on interfacial interactions of this fuel form under irradiation.

FIB/SEM 3D reconstruction was also used to study pore evolution and grain restructuring along a radially cut piece of irradiated fuel from a fuel pin that had been inserted in the H.B. Robinson Unit 2 pressurized water reactor (PWR) plant. A recent paper by Gerczak et. al. [5] discussed the restructuring process in this UO_2 fuel based on the radial microstructural variation (grain boundary distribution and pore/gas bubble behavior). An apparent relationship between gas bubble density and restructuring was observed where small bubbles (nanometer scale) are located intragranularly in unrestructured regions, while no bubbles are found in restructured areas. These small bubbles discharge into larger bubbles during restructuring in a process involving the redistribution of intragranular bubbles. Serial sectioning was conducted on six regions across the fuel slice, from the center of the fuel out to the fuel/cladding interface, with particular focus on the high-burnup restructured region. Results were complimentary to those found in [5,6].

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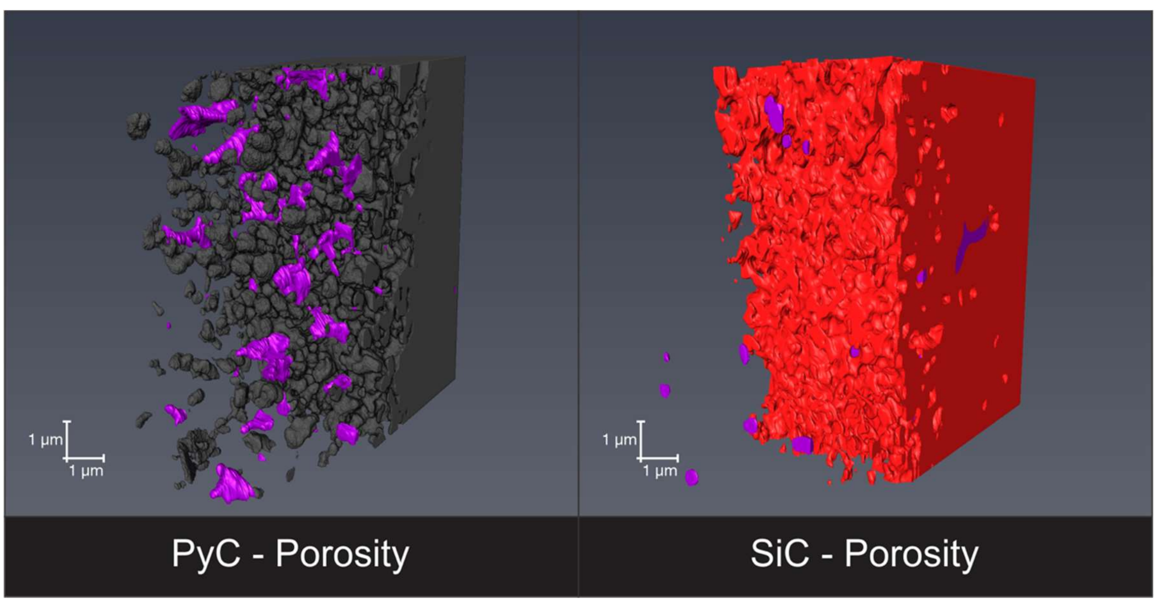

Figure 1. A 3D reconstruction of the IPyC/SiC interface. The PyC is on the left, and the SiC on the right. The purple highlights the major contributions to porosity at the interface.

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