904
doi:10.1017/S143192761300651X

Microsc. Microanal. 19 (Suppl 2), 2013

Radioactive Sample Preparation using Focused Ion Beam

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In an effort to develop or adapt characterization techniques to radioactive materials, Idaho National Laboratory utilized focused ion beam (FIB) tool for radioactive specimen preparation. The analyzed materials ranged from neutron irradiated structural materials, to unirradiated and irradiated fuels (metal, oxide, intermetallic, carbide, and hydride). Following FIB-based preparation, nuclear fuels and materials can be analyzed using a number of techniques, including 3D electron backscatter diffraction (EBSD), nano/micro indentation, 3D atom probe tomography (APT), and transmission electron microscopy (TEM). The ultimate goal is to characterize nuclear materials at micro-, nano-, and sub-nano scales.

Tristructural-isotropic (TRISO) coated particle fuel will be used to explain the issues associated with radioactive specimen handling and advantages of using FIB for specimen preparation. TRISO fuel is used in high temperature gas-cooled reactors and will be used in very high temperature reactors in the near future. As it can be seen from Fig. 1, TRISO fuel consists of metal oxide or mixed oxide/carbide fuel kernel, which is coated with buffer carbon layer, inner pyrolitic carbon layer (IPyC), silicon carbide (SiC) layer and an additional outer PyC (OPyC) layer. The release of fission products from the fuel particle at elevated temperatures is retained by the SiC layer. Consequently, characterization of the interfaces between SiC, IPyC and OPyC is of great importance. Previous reports are based on scanning electron microscopy (SEM)-based characterization, sometimes coupled with EBSD analysis of the sample surface. Idaho National Laboratory is capable of preparing cross-sectional TEM specimens, and large blocks suitable for three dimensional microstructure analysis using EBSD technique. The combination of various instruments and methodologies allows extracting information on grain morphology, grain boundary orientation and elongation, as well as composition and structure.

In addition to traditional fuel that has been used for many years, new generation fuel will be discussed. The fuel is exposed to extreme conditions, which implies the possibility of fuel-cladding interaction, raises safety concerns and increases radioactive waste burden. Therefore, it should be thoroughly investigated prior to its implementation in power reactors. One of the examples of such fuels is provided in Fig. 2. Figure 2 shows the interaction zone formed between U-Pu-Zr metallic fuel and Fe clad upon thermal annealing at 750^{o}C. To determine the intermetallic phases formed between the fuel and cladding material at high temperatures and to investigate their microstructure properties, TEM lamella are lifted-out from various positions along the interaction zone. Conventional TEM sample preparation, such as jet-polishing, is not applicable to diffusion couples. Hence, capability to prepare site-specific samples from diffusion couples in FIB instrument opens new horizons to nuclear fuel characterization field.

The paper will discuss the issues, advantages, and limitations associated with characterization of radioactive nuclear materials. The advantages of using FIB for radioactive sample preparation are as follows: reduced personnel radiation exposure, improved quality of EDS analysis because of the lower radiation background noise, ability to prepare site-specific lift-outs for TEM analysis, and cross-sectioning of the fuel for SEM analysis of fission gas behaviour. In addition to this discussion, the safety relevant information will be summarized and good radiological practices will be outlined [1].

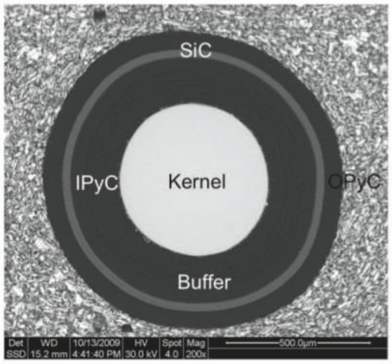

Figure 1. Scanning electron micrograph of the TRISO fuel cross-section: fuel kernel, buffer, inner pyrolitic carbon (IPyC), silicon carbide (SiC), and outer pyrolitic carbon (OPyC) layers are labeled for convenience. The scale bar denotes 500 µm.

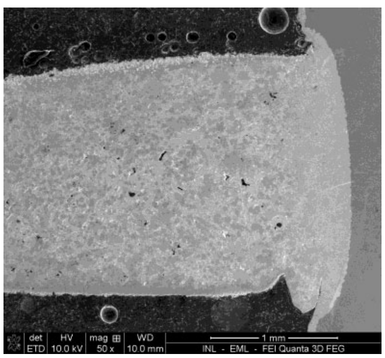

Figure 2. Scanning electron micrograph depicting interaction zone formed between U-Pu-Zr fuel and Fe clad. The scale bar denotes 1 mm.

Figure 3. The FIB stage survey, conducted upon loading and unloading of the specimen.

[1] This work is supported by the US Department of Energy through Fuel Cycle Research and Development (FCRD), Reduced Enrichment for Research and Test Reactors (RERTR), and Advanced Gas Reactor (AGR) programs at INL.

https://doi.org/10.1017/S143192761300651X Published online by Cambridge University Press