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TEM Identification of Phases in Metallic Pu-based Fuels

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Metallic fuels are considered for application in advanced fast reactors because of their high burn-up, high fissile and fertile density capability, and high thermal conductivity with significant safety benefits [1]. Metallic fuels have several potential advantages, such as simple fabrication, robust performance, benign response to reactor transients, and relatively easy recycling using compact molten salt electrochemical processing. The uranium-plutonium-zirconium (U-Pu-Zr) alloys are considered to be one of the most promising metallic fuels. The addition of Zr in U-Pu matrix was sought to increase the melting temperature of U-Pu alloys and to enhance compatibility between the fuel and stainless-steel cladding by suppressing the interdiffusion of fuel and cladding constituents during steady-state reactor operations.

Phases formed in Pu-based systems have been previously experimentally investigated using thermal analysis techniques, X-ray diffraction (XRD), and wavelength and energy dispersive spectroscopy (EDX/WDX) in scanning electron microscopes (SEM) [2-4]. The issue associated with both XRD and SEM analyses is that both techniques are surface-based. In addition, spatial resolution, magnification-dependent sensitivity of EDX/SEM, and overlap between U and Pu peaks in EDX results in questionable quantitative chemical analysis of Pu-based phases. To avoid such uncertainties, detailed structural and chemical composition analysis of phases and microstructure of Pu-Zr and U-Pu-Zr fuels has been conducted in the transmission electron microscope (TEM). The high spatial resolution of TEM allowed characterization of individual matrix phases. Selected-area electron diffraction (SAED) using transmission electron microscopy provided important space-group information and lattice parameters of the phases formed in ternary U-Pu-Zr system.

Surface of the specimen was initially examined using EDX/WDX techniques in a JEOL JSM 7000F field emission SEM operated at accelerating voltage of 20 kV. SEM was used to examine the formed microstructure and determine the location of the phases of interest. To reduce charging in the SEM, a layer of Au coating was applied to the sample surface prior to the insertion into the instruments. Cross-sectional specimens for transmission electron microscopy (TEM) were prepared in a FEI QUANTA 3D field emission gun (FEG) dual beam focused ion beam/scanning electron microscope (FIB/SEM) using a lift-out approach. Specimens for transmission electron microscopy were characterized in a FEI Tecnai TF30-FEG STwin TEM operated at 300 kV.

Figure 1 shows backscattering electron micrograph acquired from the U-Pu-Zr fuel. Several distinct phases were observed in the fuel. Initial SEM examination indicated formation of three phases in the fuel – Zr enriched globular inclusions, U and Zr enriched phase, and U, C, and O enriched matrix. SEM examination was followed by detailed TEM analysis. Implementation of a FIB instrument allowed for the preparation of cross-sectional TEM specimens from these transuranic samples. Multiple lamella containing features corresponding to different phases have been prepared to ensure a thorough and accurate identification of the phases formed in the alloy. Approximate lift-out locations in one area are identified with white rectangles in Fig. 1. An overview of the microstructure in one of the lift-outs is provided in Fig. 2.

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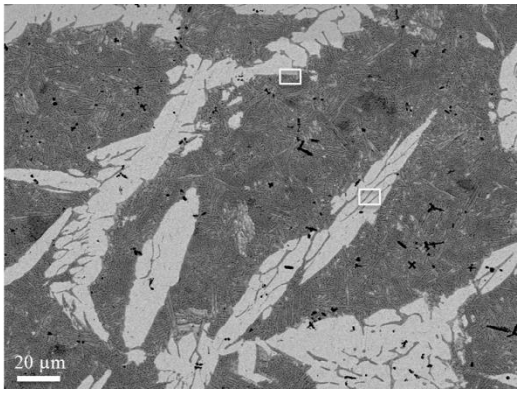

Figure 1. Backscattering electron (BSE) micrographs showing the microstructure of the U-Pu-Zr fuel. Two white rectangles identify positions of TEM lift-outs.

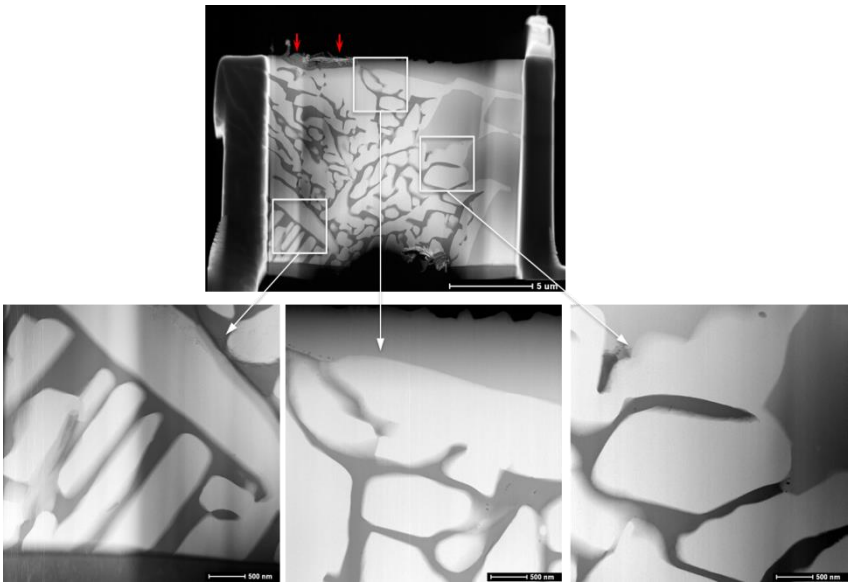

Figure 2. STEM micrographs of the cross-sectional specimen lifted-out from U-Pu-Zr fuel.

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