ESRF	**Experiment title:** Multimodal tomography for characterization of irradiated PWR fuel rod samples to observe the fuel-cladding interface	**Experiment number**: MA-4997
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Shifts: 12	**Local contact(s)**: Eleanor Lawrence Bright	

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Report:

In Light Water Reactors (LWR), extreme conditions (temperature, pressure and irradiation) lead to the oxidation of the Zr-based cladding by the UO_2 fuel, resulting in the formation of a zirconia layer at the Fuel-Cladding Interface (FCI). This zirconia is composed of a succession of phases and microstructures, as well as differences of the local chemistry due to the recoil implantation of fission products from the fuel periphery. For the first time, a XRD/XRF campaign with micro-samples from highly irradiated fuel rods performed by FIB preparation was performed at the ID11 beamline at ESRF.

In this goal, we have analyzed two irradiated samples processed by FIB-SEM and extracted from the FCI were processed (Fig. 1) to reduce the activities of the samples to avoid additional shielding such as their activities were under the detection limitation. XRD line scans (with rotation of +/-90°) have been operated in transmission mode, combined to XRF measurements using two beam sizes of 100 and 200 nm (Tab. 1) at 44 keV. A Dectris Eiger 4 2D imaging detector have been used for the XRD measurements, coupled with an energy dispersive detector placed at 90° to the incident beam measures the fluorescence signal (Fig. 2).

Table 1*. Overview of the combined XRD/XRF dataset acquired during ID11 campaign.*

Sample	**200-nm-beam**	**100-nm-beam**
E06	5 lines – 264 scans/line	3 lines – 543 scans/line
Q09	6 lines – 238 scans/line	2 lines – 484 scans/line

The correlation between XRD and XRF data allowed to have an accuracy localisation of each scan, and then, to identify the different materials (Fig. 3). Thus, the first refinement of the XRD patterns showed that the well-known cubic phase of UO_2 has here a bigger lattice than an unirradiated fuel, with an average lattice parameter of 5.475 Å. In addition, due to the very small beam size, the bottom of the peaks showed many smaller peaks which could highlight unidentified phases for the fuel and fission product precipitates.

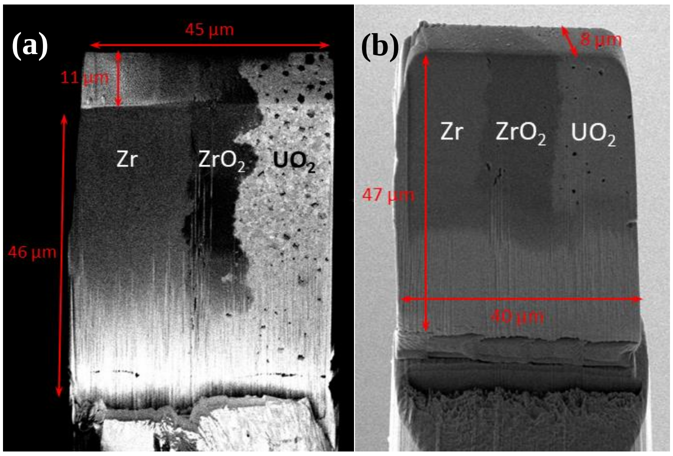

Figure 1. *SEM image of the FIB samples: (a) E06: Zircaloy-4/UO_2 – 61 GWd/tU (5 years in LWR); and (b) Q09: $M5_{FRAMATOME}$/UO_2 doped Cr – 50 GWd/tU (4 years in LWR).*

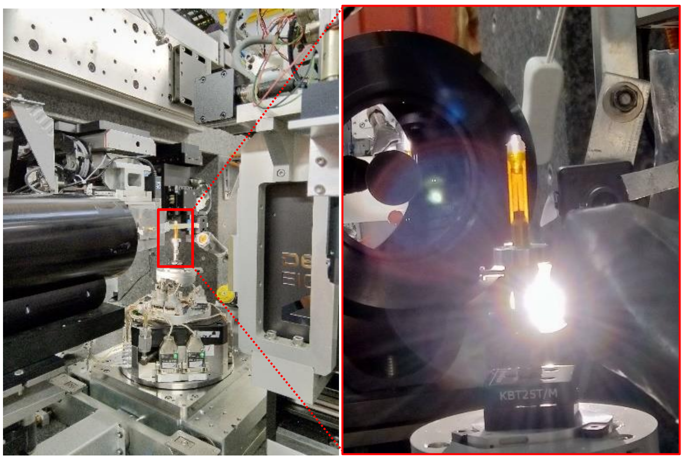

Figure 2. *Experimental set-up at the ID11 beamline of the sample holder in transmission mode and detail. The analysed sample is located at the top of the needle which is visible by the light through the kapton tubes forming the airtight double containment.*

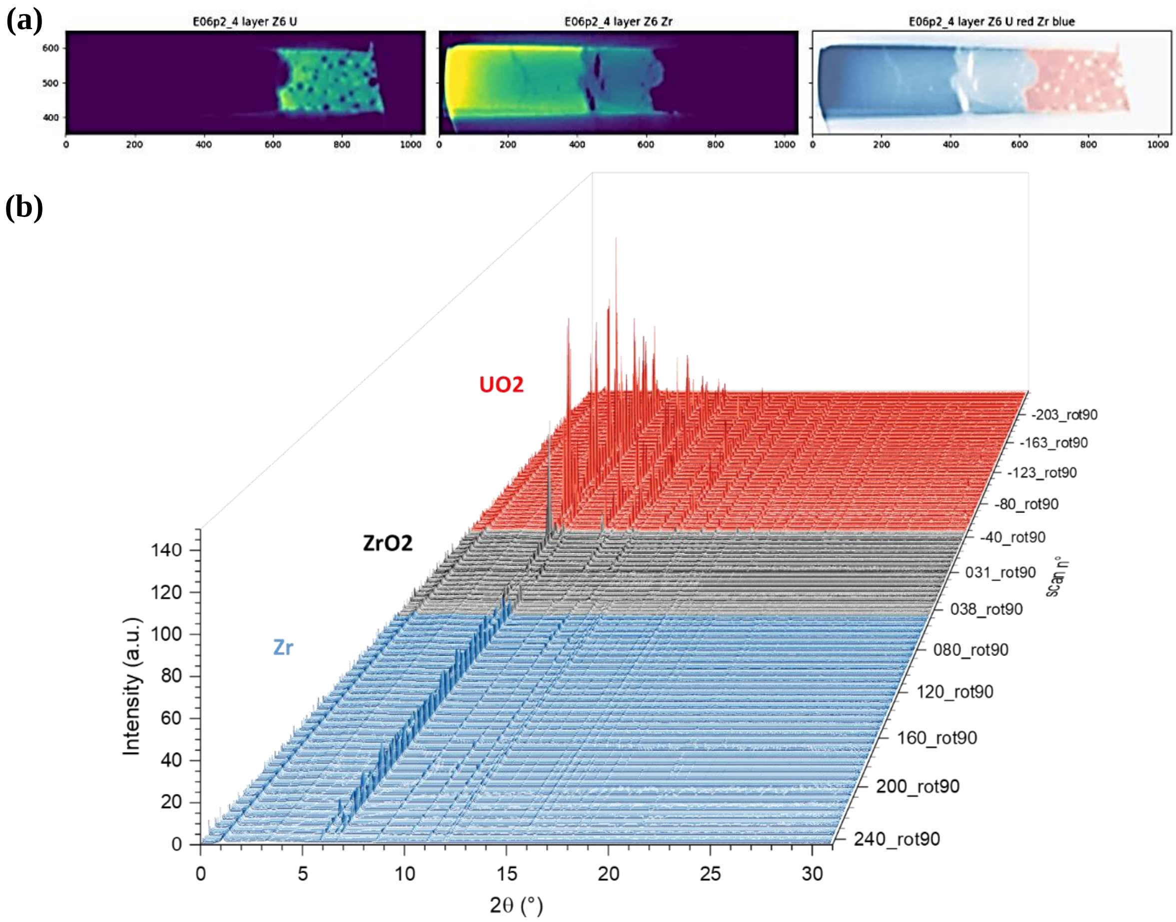

Figure 3. *Example of dataset acquired with the 100-nm-beam: (a) XRF maps for U and Zr elements of sample E06 and (b) correlated XRD patterns. The colours correspond to the three materials within the sample.*

In summary, this experiment was dedicated to transmission X-ray diffraction and fluorescence measurements on two nuclear fuel samples with different burnup prepared from a fuel irradiated in LWR. The specific area of interest of the fuel samples for this experiment was the intermediate layer of zirconia at the Fuel-Cladding Interface (about 8-10 µm thickness). The main goal was to use the multimodal tomography characterization: the high sensitivity and the spatial resolution allowed observing fuel and zirconia phases structures, and their components. Thus, the results of this experiment, which has been a first experiment on this kind of samples, improve the knowledge of the interface between fuel and cladding and FCI evolution with irradiation. The analysis is still currently in progress and will be publish subsequently.