A new look on irradiated fuels at the CEA Cadarache

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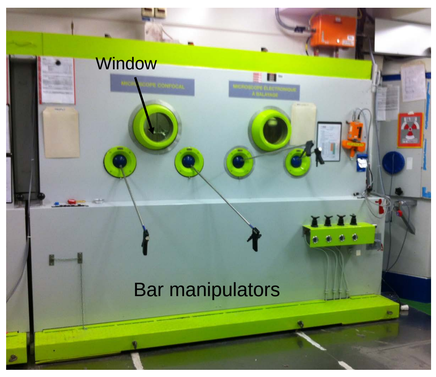
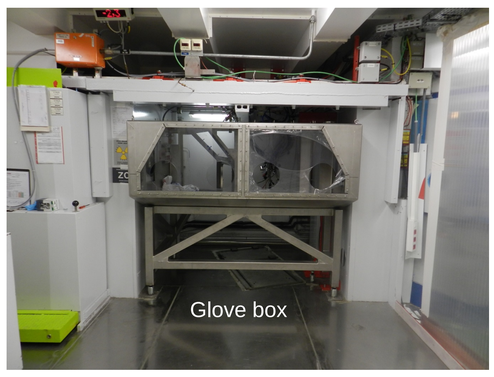
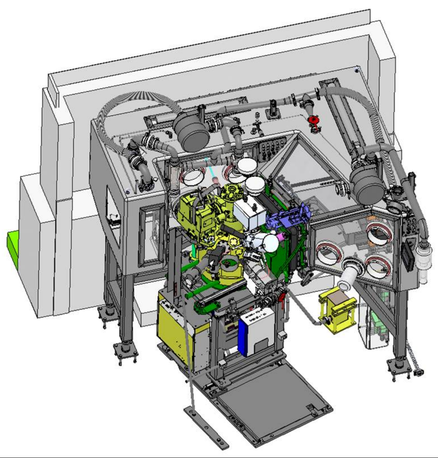
Hot cell facility LECA-STAR – FIB SEM

Shielded room



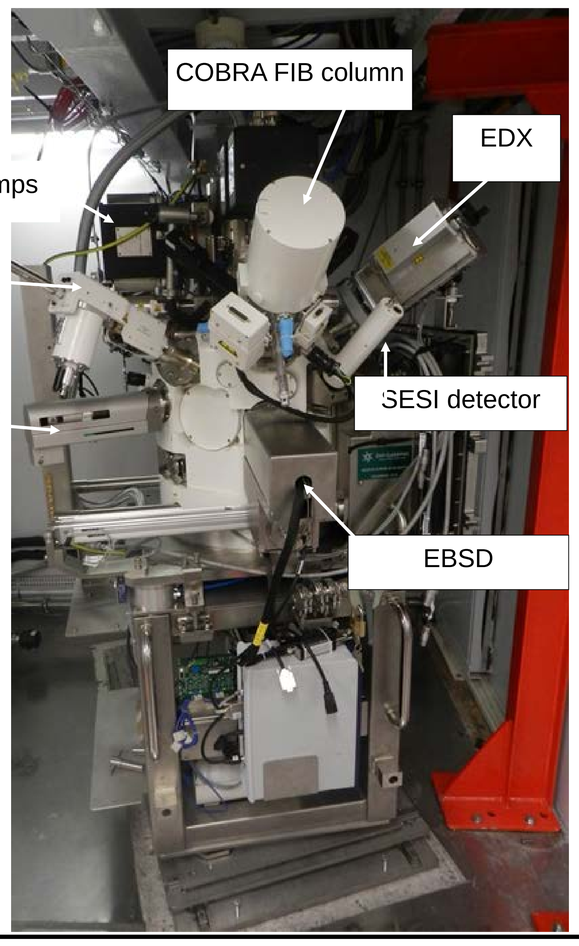


Glove Box and SEM FIB

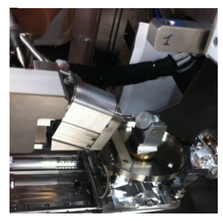


A **new FIB SEM** is operationnal for **research on nuclear fuels** at the CEA Cadarache. The microscope is installed in a shielded room. It is connected to a glove box, containing the contamination of the samples. The loading of the samples on the FIB SEM stage is done using bar manipulators.

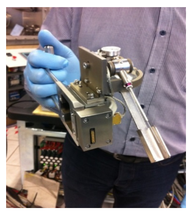
Detectors and accessories



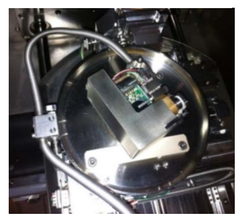
Nano-indenter



Micromanipulator

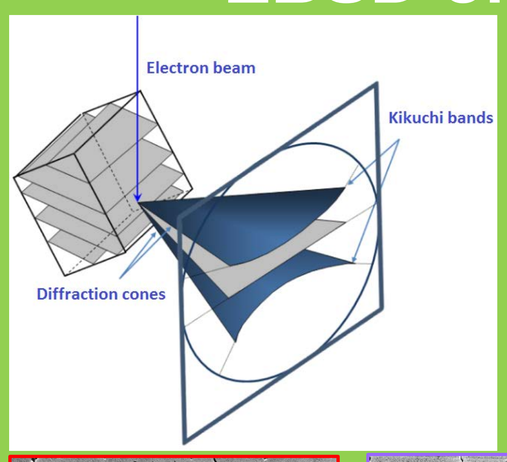


STEM

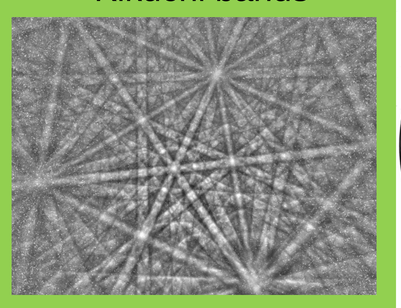


The FIB SEM is a Carl Zeiss Auriga 40 equiped with an Orsay Physics COBRA FIB column.
The nano-indenter and the micro-manipulator are mounted on the door of the SEM chamber and the STEM is installed on the stage. All these accessories have to be positionned and connected manually via the gloves, prior to the loading of the radioactive samples.
The EDX and the STEM detector must only be used on micro-samples with very low irradiation levels to avoid damage to the sensitive parts of the detectors.

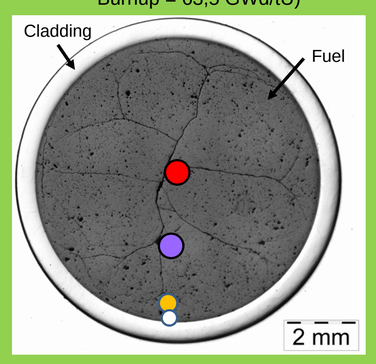
EBSD on high burnup UO_2

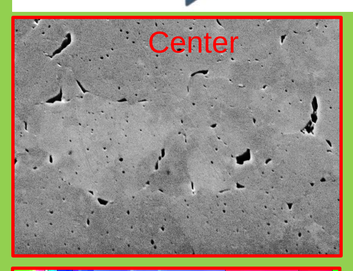


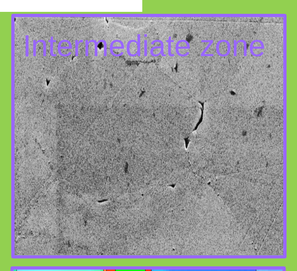
Kikuchi bands

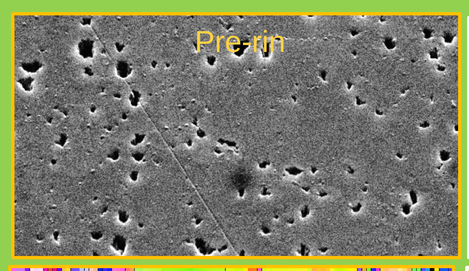


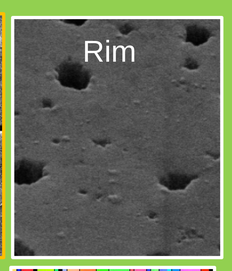
Macrography of the sample
Burnup = 63,5 GWd/tU)

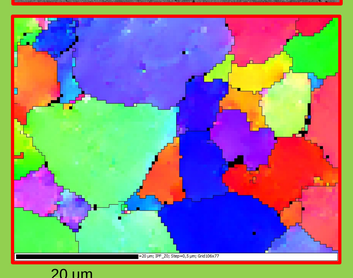




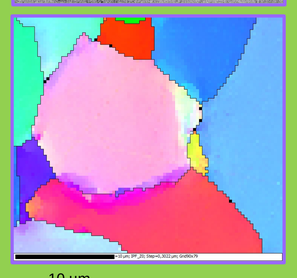




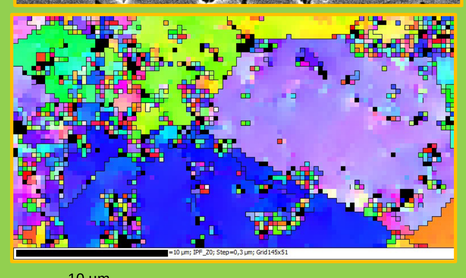




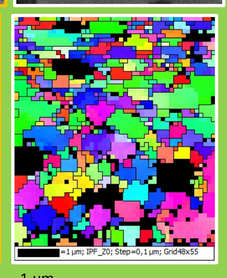
20 µm



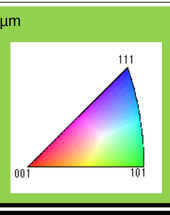
10 µm



10 µm

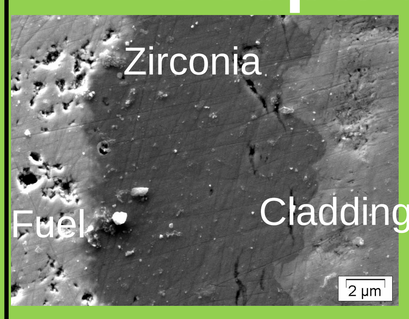


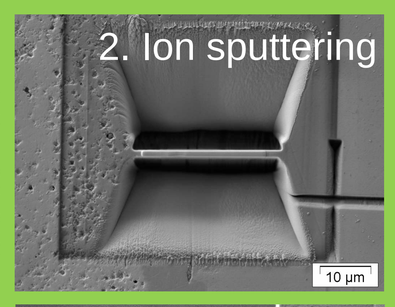
1 µm

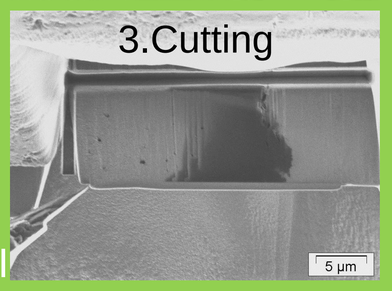


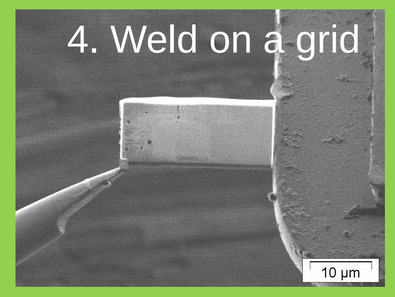
An irradiated UO_2 fuel was analysed by EBSD to observe the grains. Different radial positions in the fuel were studied : here, the center, the intermediate zone, the pre-rim and rim. The grain boundaries have been drawn for a misorientation superior to 10°. In the rim, the initial grains are subdivised in smaller grains.

Preparation of a thin foil

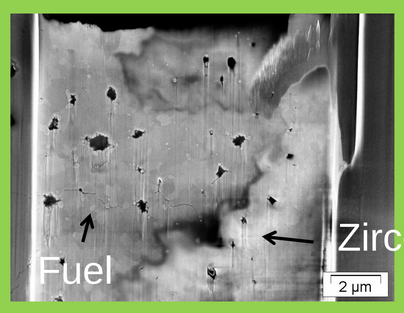




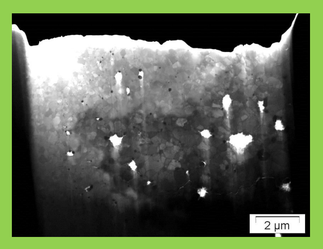


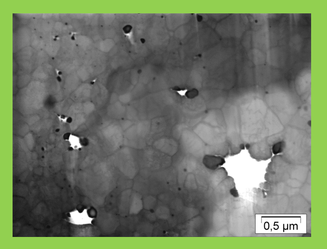


5. Finition of the thin foil



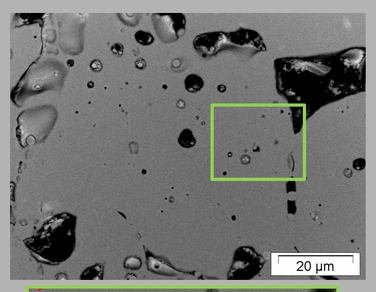
STEM : Control of the preparation

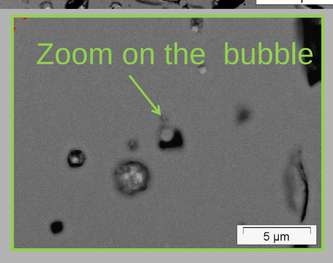


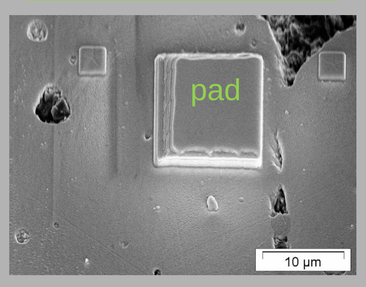


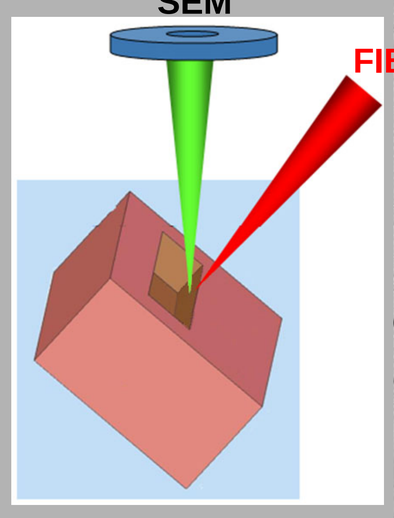
A thin foil was made by FIB in a zirconia layer created by oxidation between the fuel and the cladding. A pad of platinium (1) was deposited to the area of interest. Then two holes (2) were milled on either side of the layer to provide space for extraction with the micromanipulator. The micron thick specimen was welded at the end of the micromanipulator; cut (3), extracted, welded on a grid (4) and released from the manipulator. The sample was gradually thinned to a thickness of approximatively 100 nm using a low intensity and low voltage beam to reduce most of the defects created by the gallium (5). A control of the preparation was realised with a STEM detector.

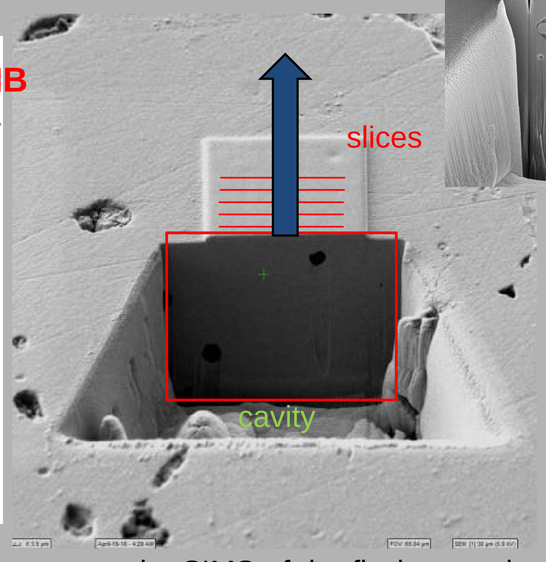
3D –Volume of a fission gas bubble

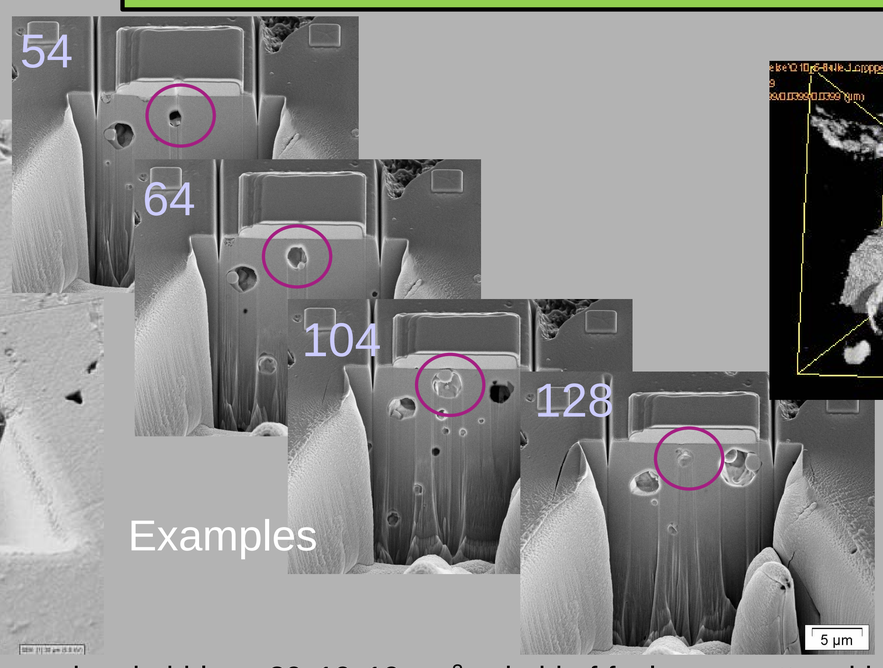






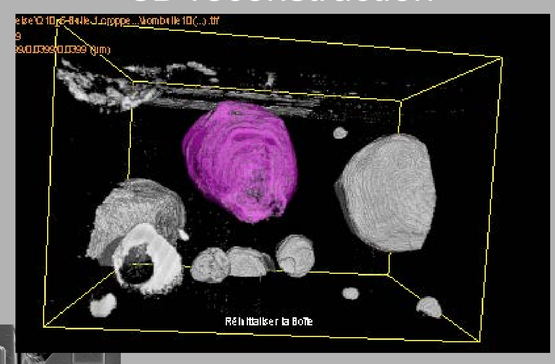


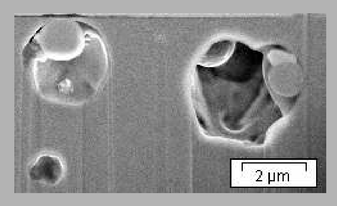




Examples

3D reconstruction





Zoom on the bubbles

In connection with the measurement by SIMS of the fission gaz in a bubble, a 20x10x10 µm³ cuboid of fuel was sputtered by ion beam to measure the exact volume of this same bubble. A 1 µm thick pad of platinium was deposited to protect the surface of the cube. A large cavity was made in front of the cube to provide a clear SEM view. The cube was sliced with the ion beam with 30kV-600 pA conditions, acquiring a SEM image at each step of 40 nm with a voltage of 10 kV. A stack of slices were then registrated and the bubbles were reconstructed in 3D using ORS software. The 3D shape of the bubble is ellipsoidal and the volume of the bubble of interest measures 14,1 µm³.