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PSI

Wir schaffen Wissen – heute für morgen

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ActiLab activities at PSI

Certified System swissTS ISO 9001

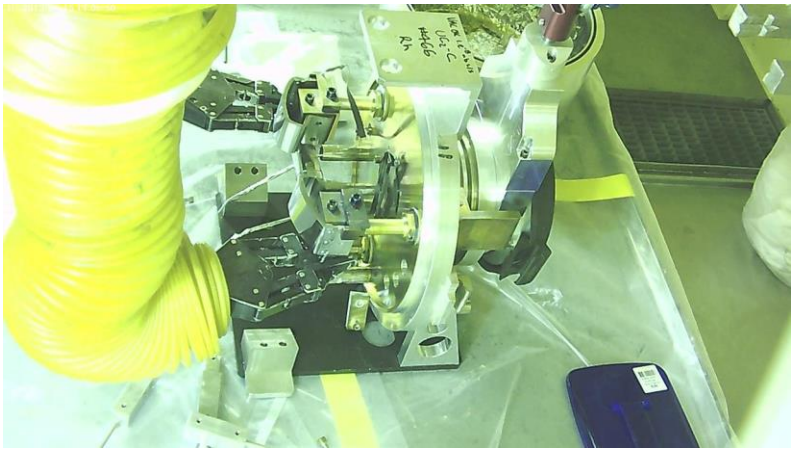
WP8 – JRA02 – 3 Characterization of irradiated targets in hot cell

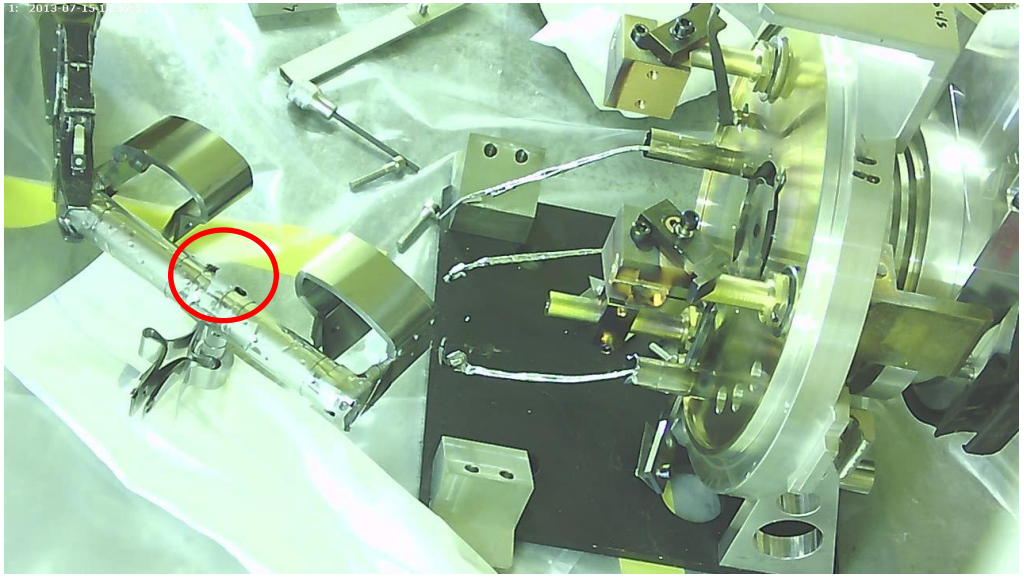
This Task 8.3 includes

- dismantling of the irradiated target from the containment in PSI hot cell
- extraction of sub-samples from the UC target for EPMA
- extraction of sub-samples from the UC target for microXAS analysis
- characterization of the different UC sub-samples by EPMA
- Summery

Dismantling of the target in July 2013

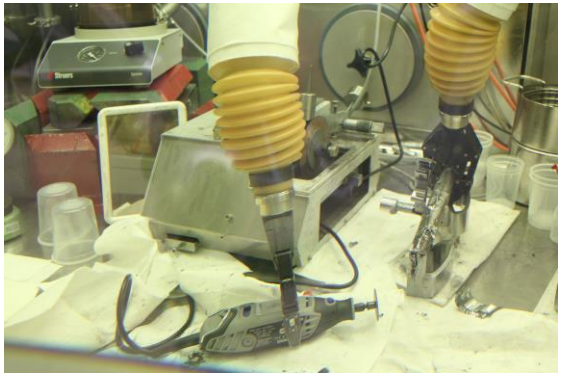
- Target dismantling in the Hot cell in July 2013 and seal the target
- The dismantling was performed under ambient atmosphere

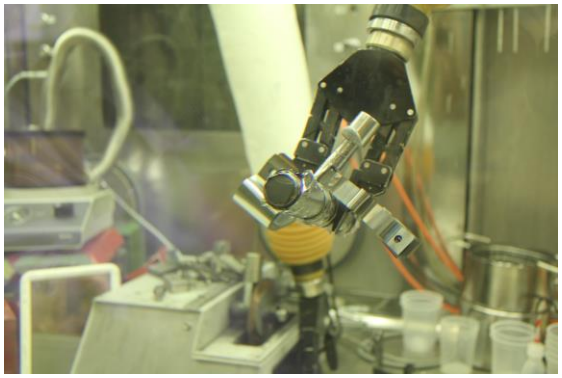




Target opening in October 2013

- Target opening in October 2013 under nitrogen atmosphere
- Sample extraction, separation and selection for microXAS and EPMA





Extracted samples for microXAS and EPMA analysis

- List of the extracted samples

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Sample cutting for microXAS beam line October 2013 and EPMA

Table 1: Samples fabricated out of the CERN target

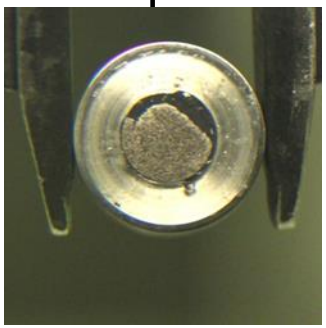
Sample name	Number of pellets	Responsible target area	comments
CERN_EPMA/Cer_B	1	Sample from the area of the graphite plug	EPMA examination, sort of ceramography
CERN_XAS_BC	0.5	Sample from the area of the graphite plug	microXAS sample take from the pellet center of the half pellet (FIB)
CERN_XAS_BR	0.5	Sample from the area of the graphite plug	microXAS sample take from the pellet rim area of the half pellet (FIB)
CERN_EPMA/Cer_M	1	Sample from the center of the target looking from graphite plug	EPMA examination, sort of ceramography
CERN_XAS_MC	0.5	Sample from the center of the target looking from graphite plug	microXAS sample take from the pellet center of the half pellet (FIB)
CERN_XAS_MR	0.5	Sample from the center of the target looking from graphite plug	microXAS sample take from the pellet rim area of the half pellet (FIB)
CERN_EPMA/Cer_E	1	Sample from the end of the target looking from graphite plug	EPMA examination, sort of ceramography
CERN_XAS_EC	0.5	Sample from the end of the target looking from graphite plug	microXAS sample take from the pellet center of the half pellet (FIB)
CERN_XAS_ER	0.5	Sample from the end of the target looking from graphite plug	microXAS sample take from the pellet rim area of the half pellet (FIB)
CERN_Test	1		Control of the xidation conditions

ab 12.5.

+ 1 unbeschichtete UC_2 (Nr. 4)

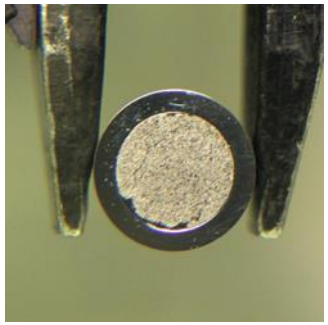
Characterization of the UC material by EPMA

- The EPMA analyses were performed on irradiated samples and on an unirradiated reference material
- It was scheduled to analyze elements of the bulk material, fission and spallation products and intermetallic precipitates
- Embedded, polished specimens were prepared under nitrogen atmosphere


Reference A


Specimen E


Specimen M


Specimen B

- Specimen E: location of the beam incoming
- Specimen M: location of the target center
- Specimen B: location of the outlet

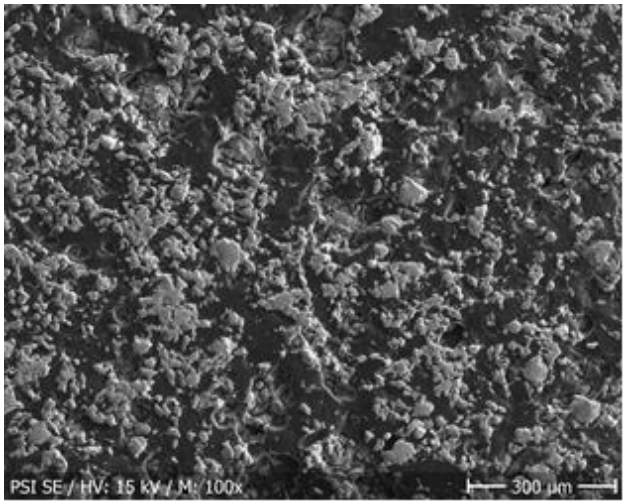
Characterization of the UC material by EPMA

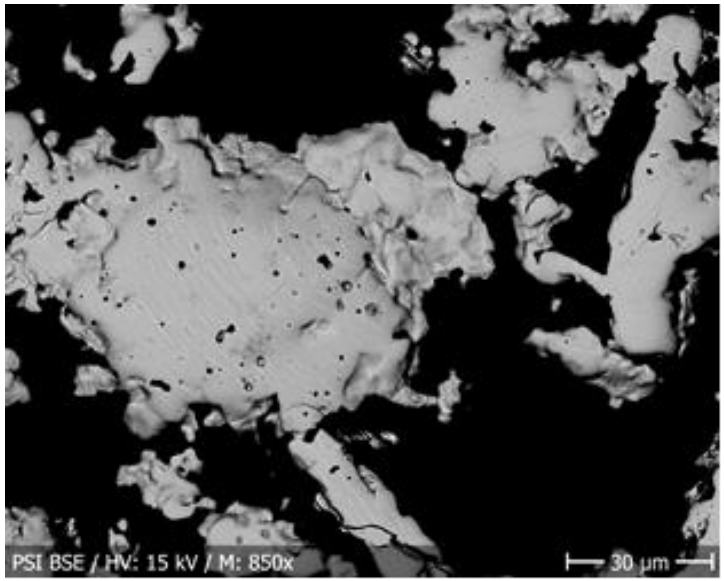
Analysis conditions

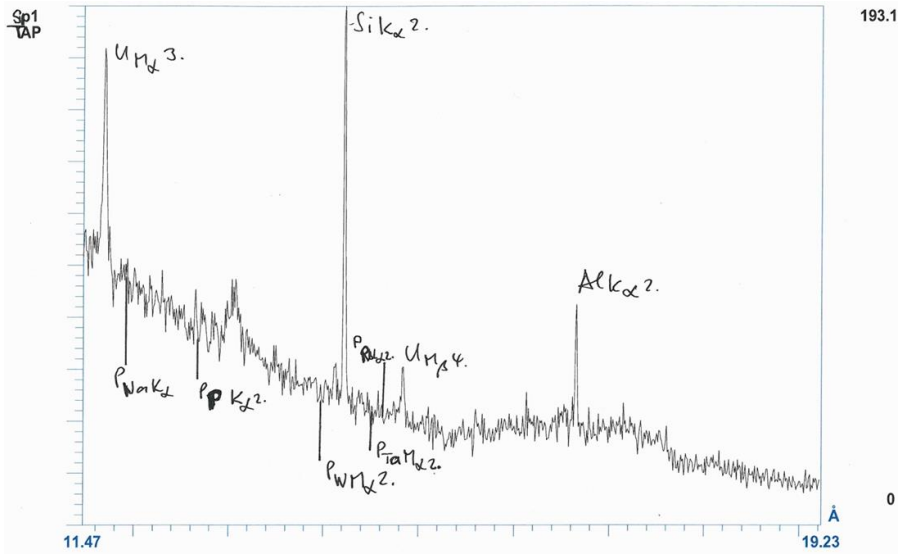
High voltage electron beam	20 kV
Beam current	200-210 nA ± 0.5 nA
Beam diameter	≈ 0.2 µm
Lateral resolution for X-rays	≈ 1 µm
Beam	1 µm beam ϕ for each point of linescan, 2 µm beam ϕ for spectrum scan and focused beam for mapping.
Elements	U, Cl with PET diffracting crystals of spectrometer 3 (SP3) and 2 (SP2). O and C with SP4/LD1 and SP1/LD2.
Measuring time for peak or point	2.5 s for linescans. 3.5 s for spectrum acquisition.
X-ray mappings	18 x 18 μm^2 up to 50 x 50 μm^2 with beam scanning (256 x 256 pixels). Acquisition time: typically 110 min. per element (100 ms/pix.).
Step size in spectra	0.11 mm which corresponds to sin Θ= 0.00039 (see below)

Characterization of the UC material by EPMA

- **Reference A**
- Very high porosity
- Twinning is observed
- Interferences of uranium side lines with different degree on main characteristic X-ray lines of possible fission and spallation products are identified
- A Si, Al and possible Cl contamination was found

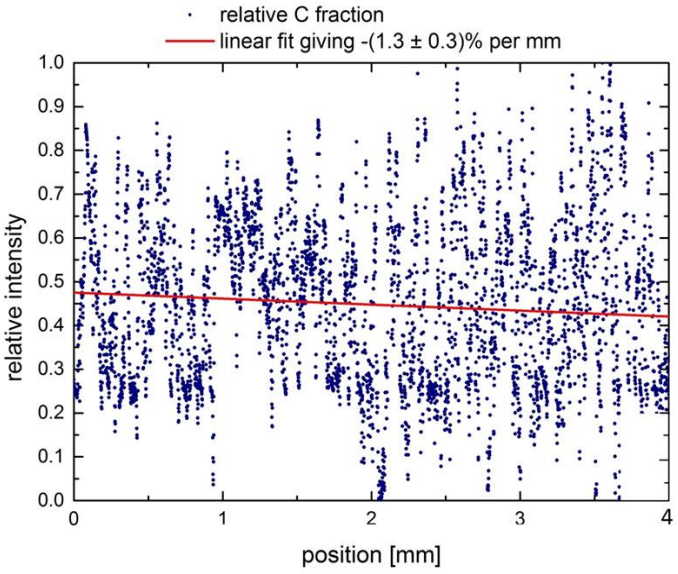


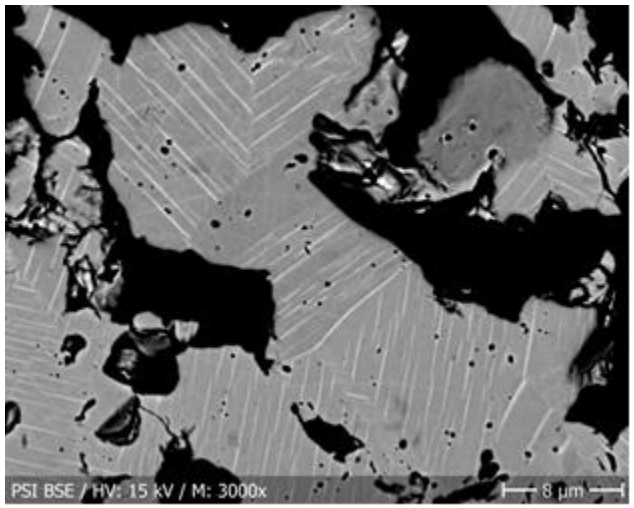




Characterization of the UC material by EPMA

- **Specimen E**
- The spectra show no evidence of significant amount of spallation products
- Because of the detection limit the trace elements in the scale of tenth or hundreds of ppm could not be detected.
- A decrease in the carbon content from the border to the center could be determined as for the reference
- No change in the high porosity
- Strong twinning in the grains is generally present
- Graphite phases can be met not only at the border but also in the center
- Surface oxidation has occurred and seems to be high in the pores





Characterization of the UC material by EPMA

- **Specimen M**
- No differences as to spectra and SE-Images are seen at a glance in comparison to specimen E
- Twinning is again present
- Cl was not present on the mappings as seen on the reference specimen

- **Specimen B**
- No differences as to spectra and SE-Images are seen at a glance in comparison to specimen E
- Twinning is again present
- Cl was not present on the mappings as seen on the reference specimen

Summery

- All EPMA SE, BSE and element mappings are reported in the document TM-43-14-07
- Some oxidation of the surface especially of pores during preparation could not be avoided.
- No significant changes through irradiation could be observed and detected
- Traces of fission or spallation products could not be clearly verified, because of the EPMA detection limit
- Crystallographic and stoichiometric information could be available under use of other techniques like XRD
- Content of spallation products could be determined by using ICP-MS and SIMS