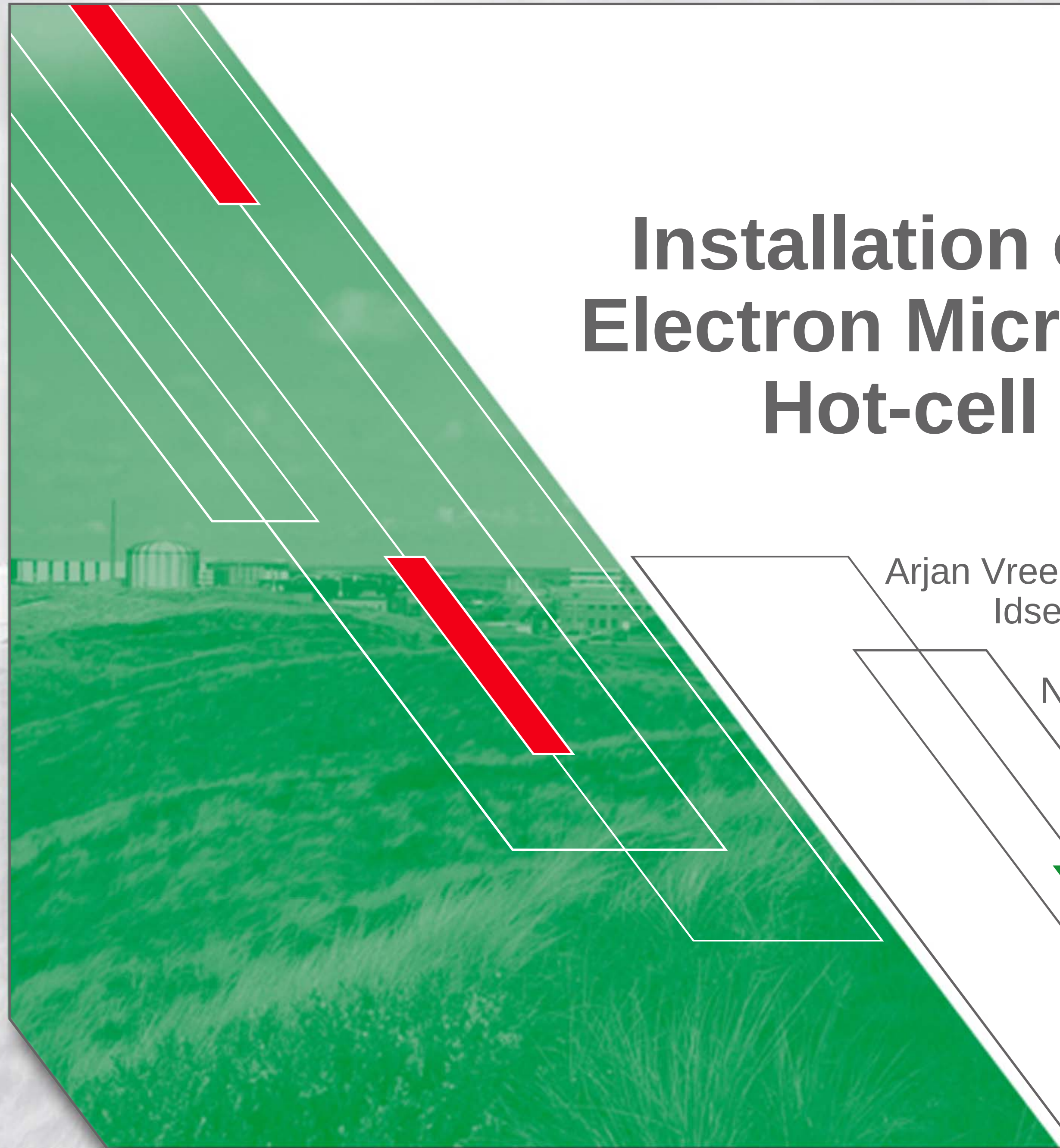

Installation of a Scanning Electron Microscope in the Hot-cell Laboratory of NRG Petten

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Outline

- Introduction
- Equipment
  - Specs
  - Modifications
- Building the facility
- Examples
  - Fuel
  - Composites
  - EBSD
- Conclusions

Materials Characterisation at NRG

- Mechanical Characterisations
  - Strength testing
  - Fracture Mechanics

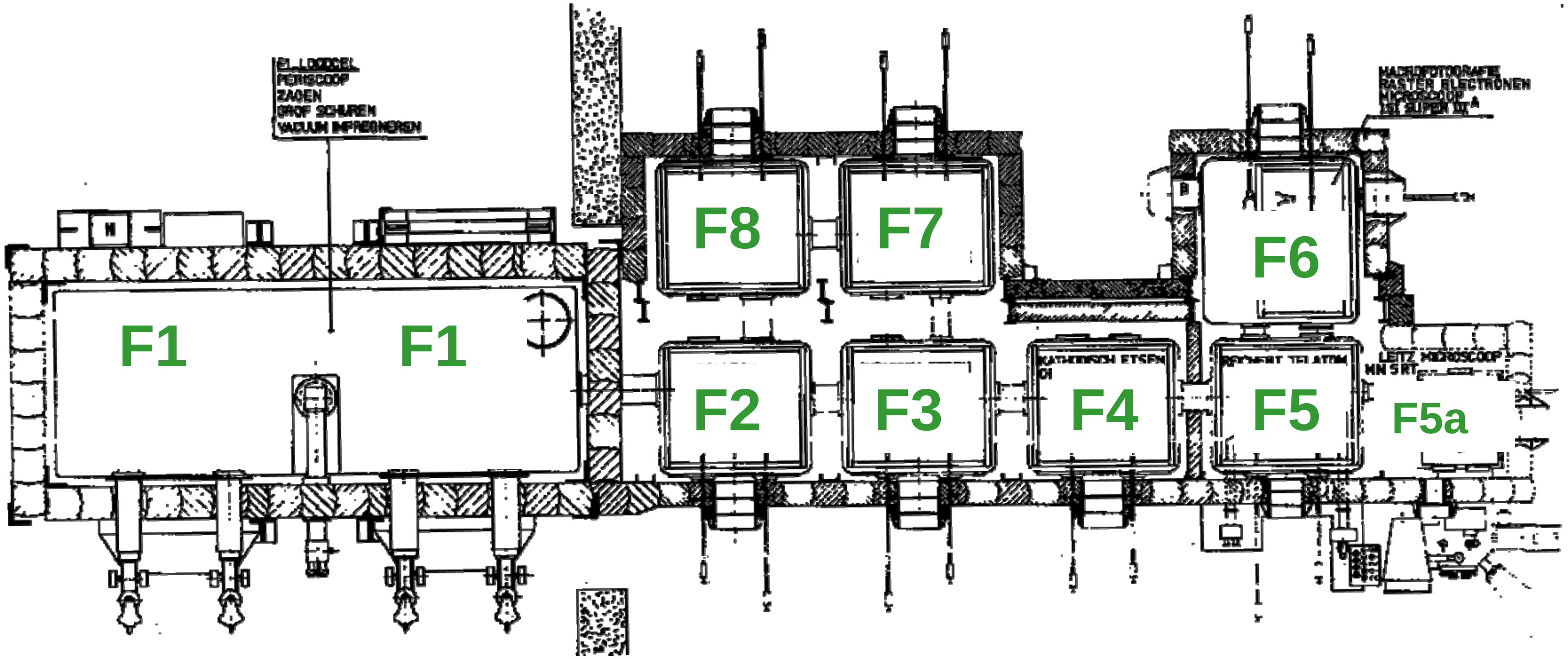
- Physical Characterisations
  - Dimensions, mass, density
  - Time of flight (Dynamic Young's Modulus)
  - Thermal Properties (Expansion, Conductivity)
  - X-ray Diffraction

- Microscopy
  - Optical microscopy
  - Electron microscopy (Scanning and Transmission)

Microscopy at NRG

- Alpha tight hot-cells for sample preparation
- Optical microscope (Leitz) in alpha tight hot-cell
- SEM in alpha tight hot-cell
- TEM (Jeol JEM-1200EXII) in B-lab
- Optical microscope (Olympus) in glove box in B-lab

Microscopy in NRG Hot-cells



- F1: Posting, sawing and rough grinding of samples
- F2-F4: Grinding, polishing and etching of samples
- F5: sample handling
- F5a: Optical microscopy (Leitz)
- F6: Scanning Electron Microscoop (SEM)

New Scanning Electron Microscope

JEOL 6490 LV SEM

- High resolution (3.0 nm)
- Possibility of Low Vacuum mode for badly conducting surfaces
- Enhanced SE and BSE detection
- Five-axis motorized stage
- Equipped with EDS, WDS and EBSD detectors

Detectors from Oxford Instruments

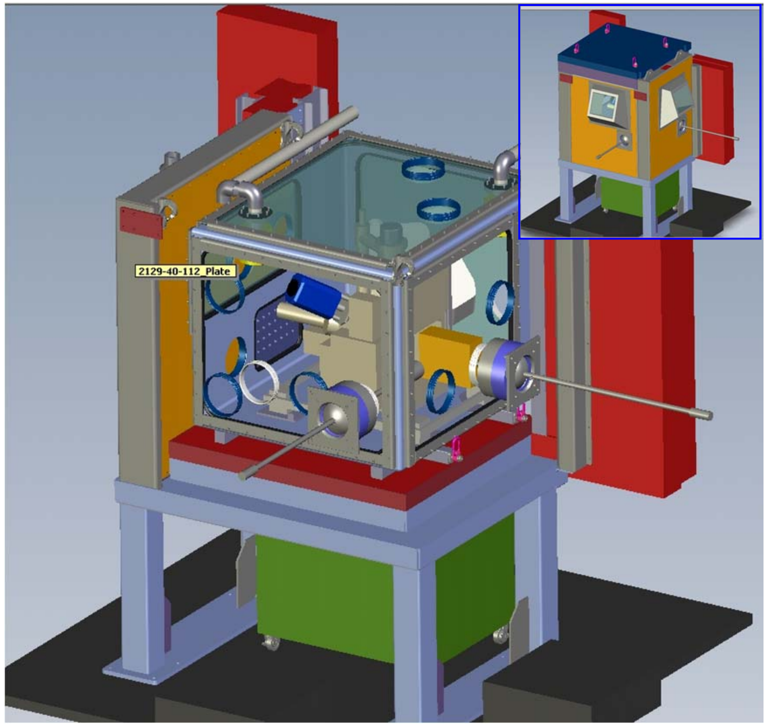
Energy-Dispersive Spectrometer (EDS)

Wavelength dispersive Spectrometer (WDS)

HKL Nordlyss Electron BackScatter Diffraction (EBSD)

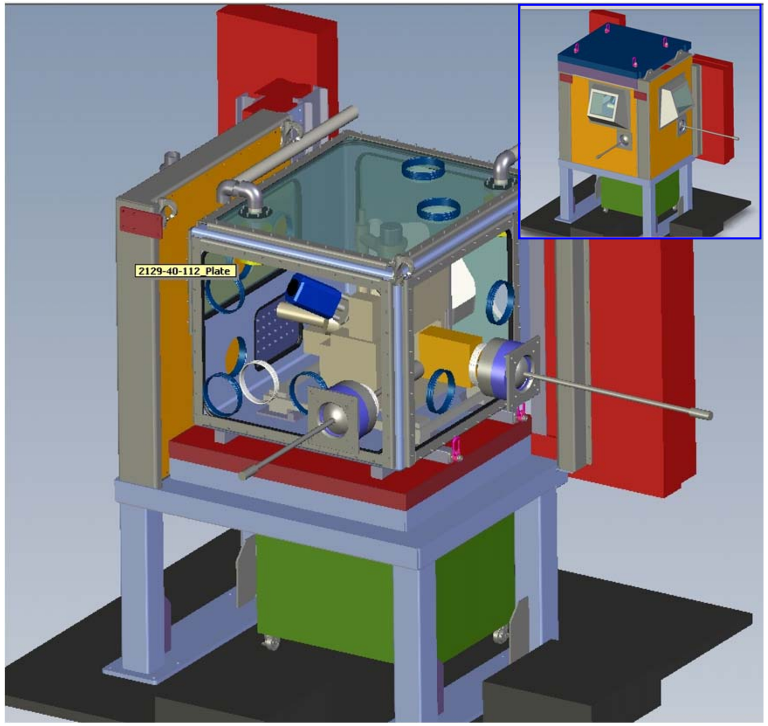
Modification to SEM (I)

- Alpha tight hot-cells: Leak tight box in lead cell
- Separate Electronics/radiation sensitive parts if possible
  - Extending cables



Modification to SEM (II)

- Electronics that could not be separated
  - Local shielding by Tungsten plates if possible
  - Limited space available at motor stage =>
    - Components tested in high gamma-field up to 25000 Gy



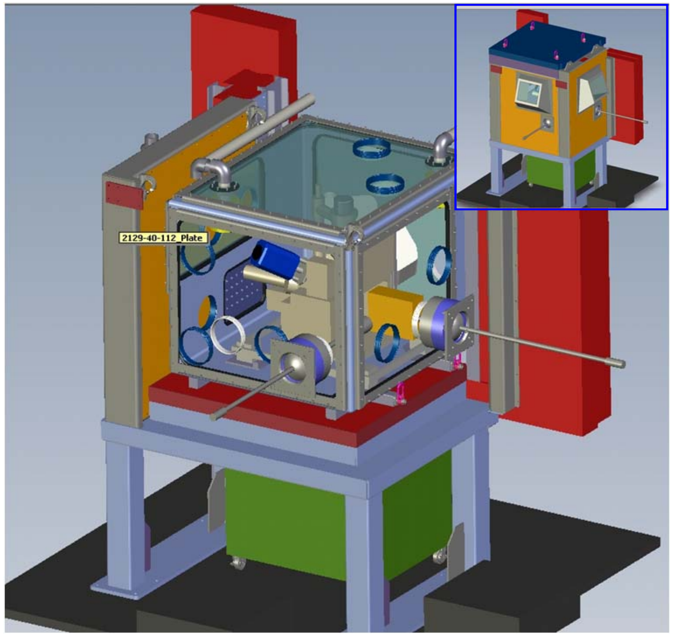
Modifications (III)

- Handling optimised for handling with tong manipulators

Tong 1: Getting sample+ sample holder

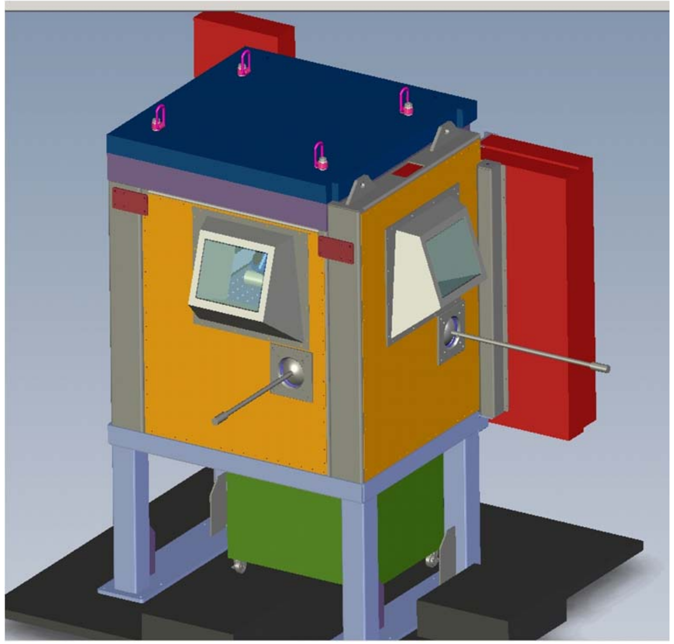
Tong 2: Opening door, placing the sample and closing the door:

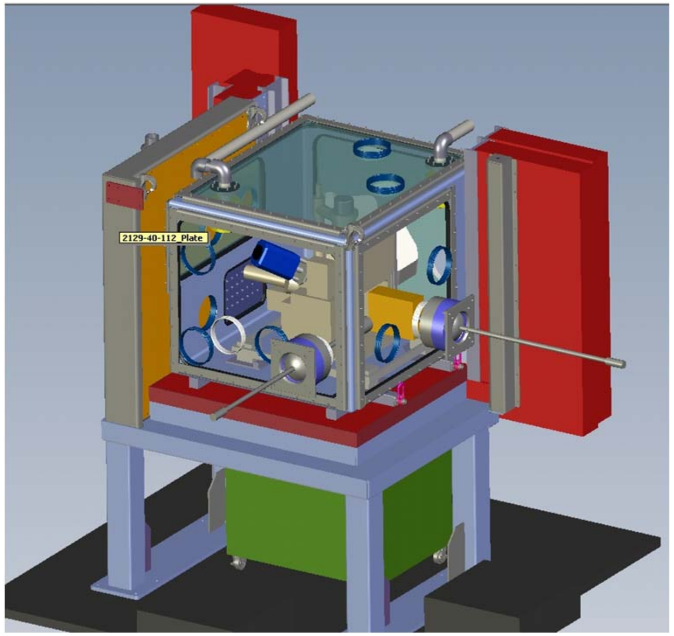
  - Door re-designed
  - Sample loading re-designed



Modifications (IV)

- Improve visibility by placing windows under an angle
- Accessibility for servicing, repairing and replacing parts





Building the new facility (PHASE I)

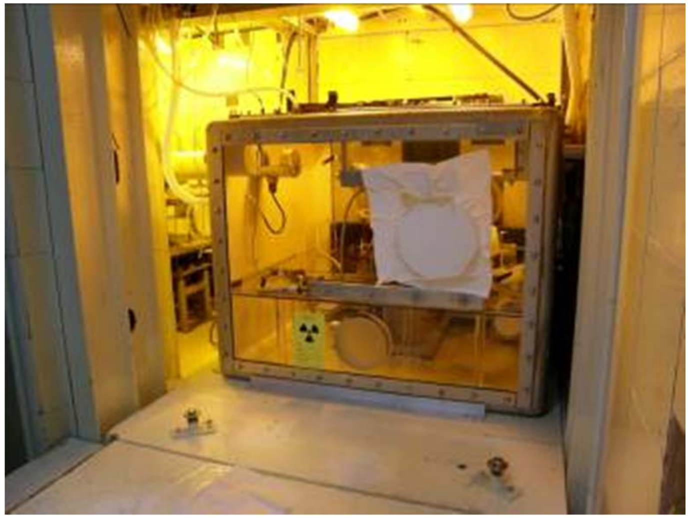
Removing old SEM and hot-cell

- Decontaminating box
- Decontaminating SEM
- Dismantling of hot-cell

Building the new facility (PHASE 2)

Modifying F5 cell

- Decontaminate box
- Remove old equipment
- Design posting lock between F5 and F6 (SEM cell)
- Modify lead window and tong positions



Design of new hot-cell

Dummy SEM (Maquette at real size) used to obtain the positions of openings and handling possibilities.

Building the new facility (PHASE 3)

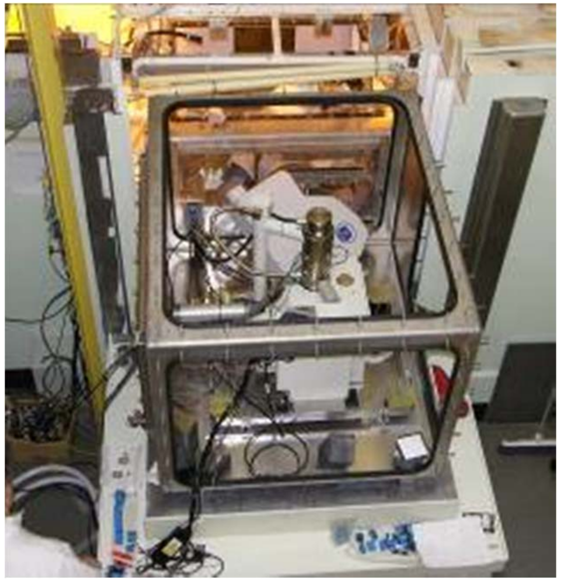
Building the new hot-cell

- Building hot-cell
- Acceptance test of hot-cell

Building the new facility (PHASE 3)

Installation of SEM

- Firstly the SEM was tested outside hot-cell
- SEM installed in hot-cell
- Testing of SEM in hot-cell
- Acceptance test of SEM and hot-cell



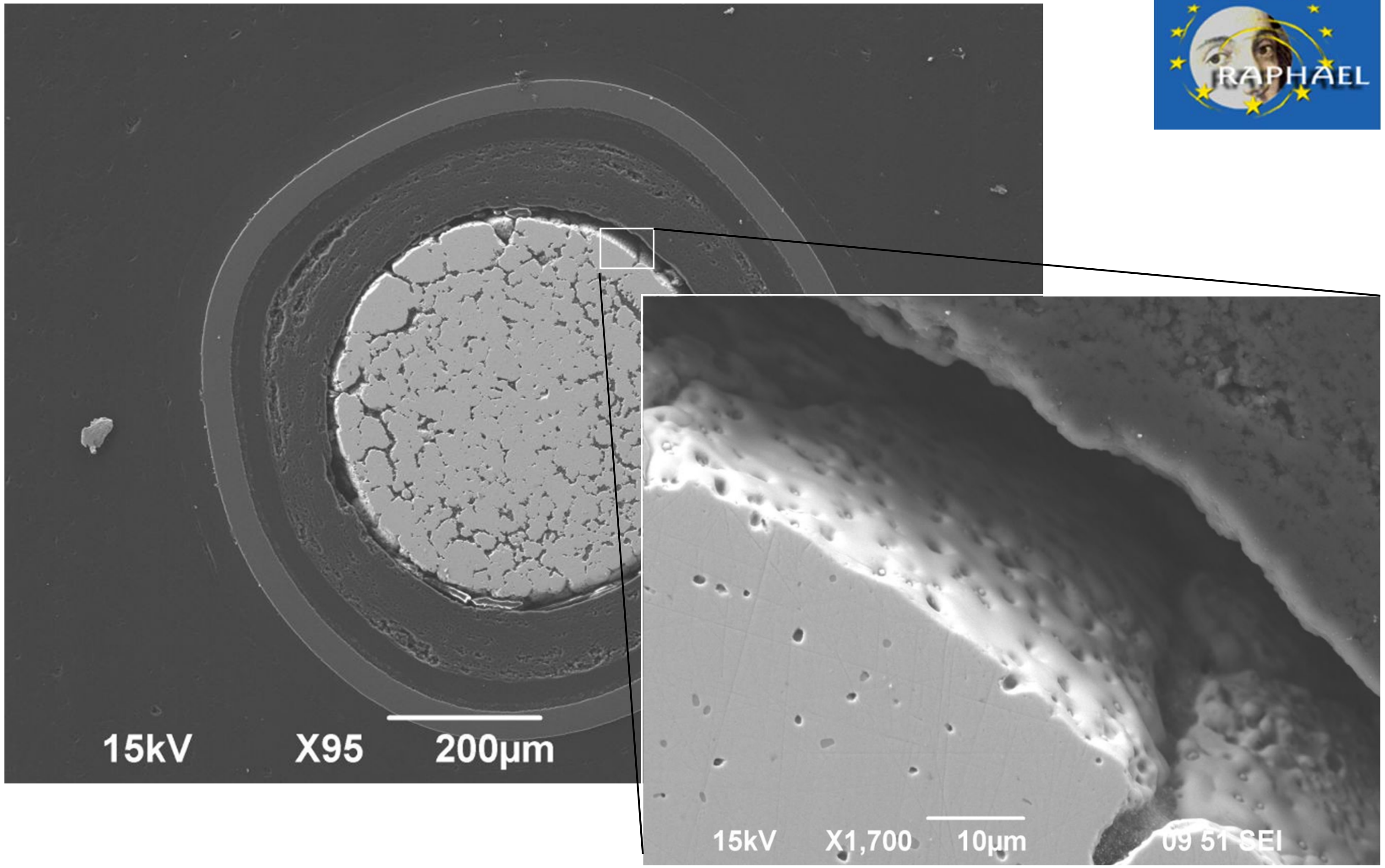
August 2010: SEM ready for use

(Lead removed to show SEM, no sample loaded!)

- Hot-cell connected to HCL system (off gas, alarms)

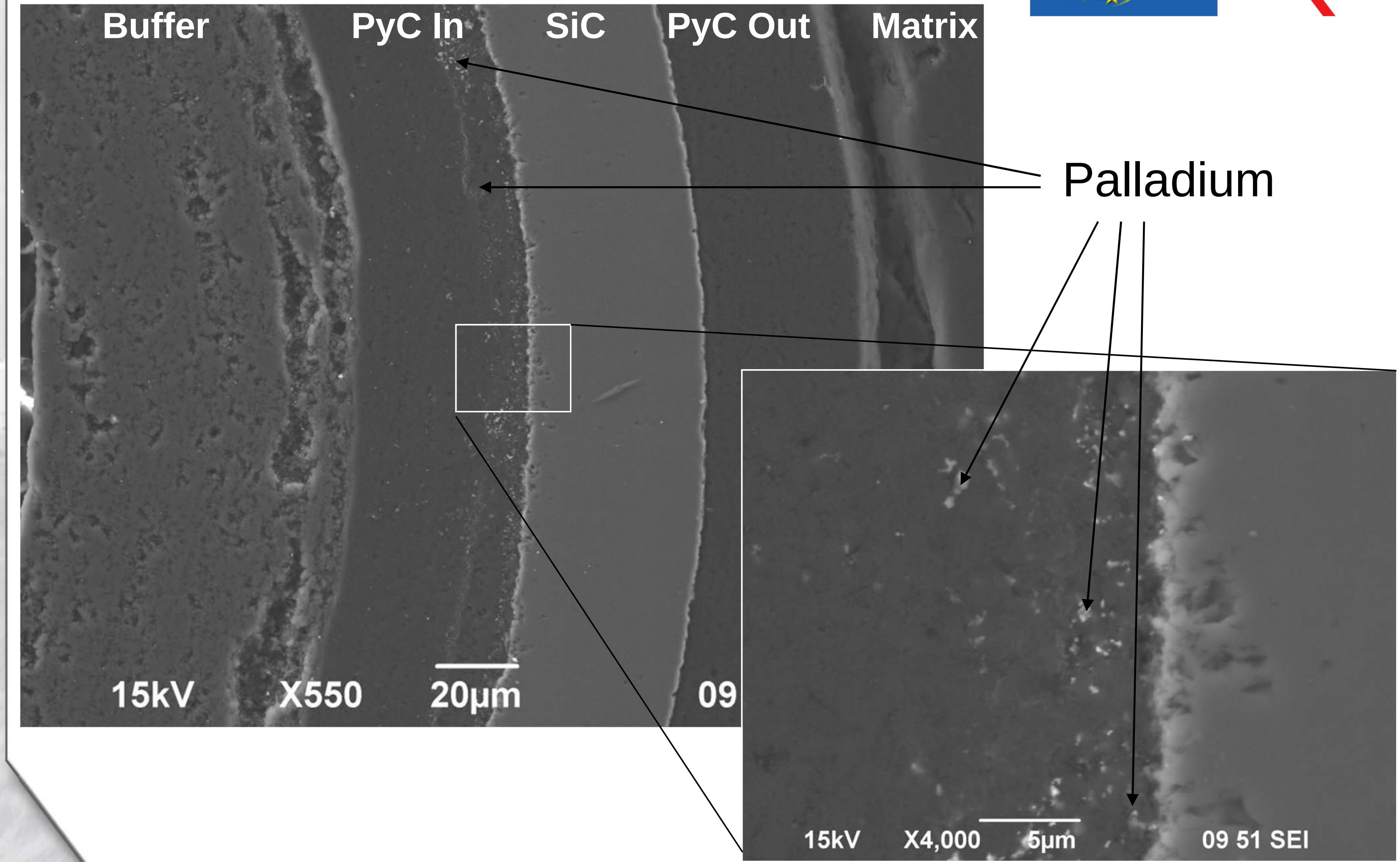
Example 1: HTR FUEL

- HFR-EU1bis irradiation has been designed, built and irradiated under JRC-IE coordination in the HTR-F and Raphael EU framework programs
- 5 AVR HTR fuel pebbles have been irradiated in the HFR Petten, in 249 Effective Full Power Days (10 HFR cycles).
- Requirement to keep central pebble temperature at 1250°C (very high temperature performance test)
- Fuel pebble: graphite matrix containing TRISO particles (coated UO_2 kernels)

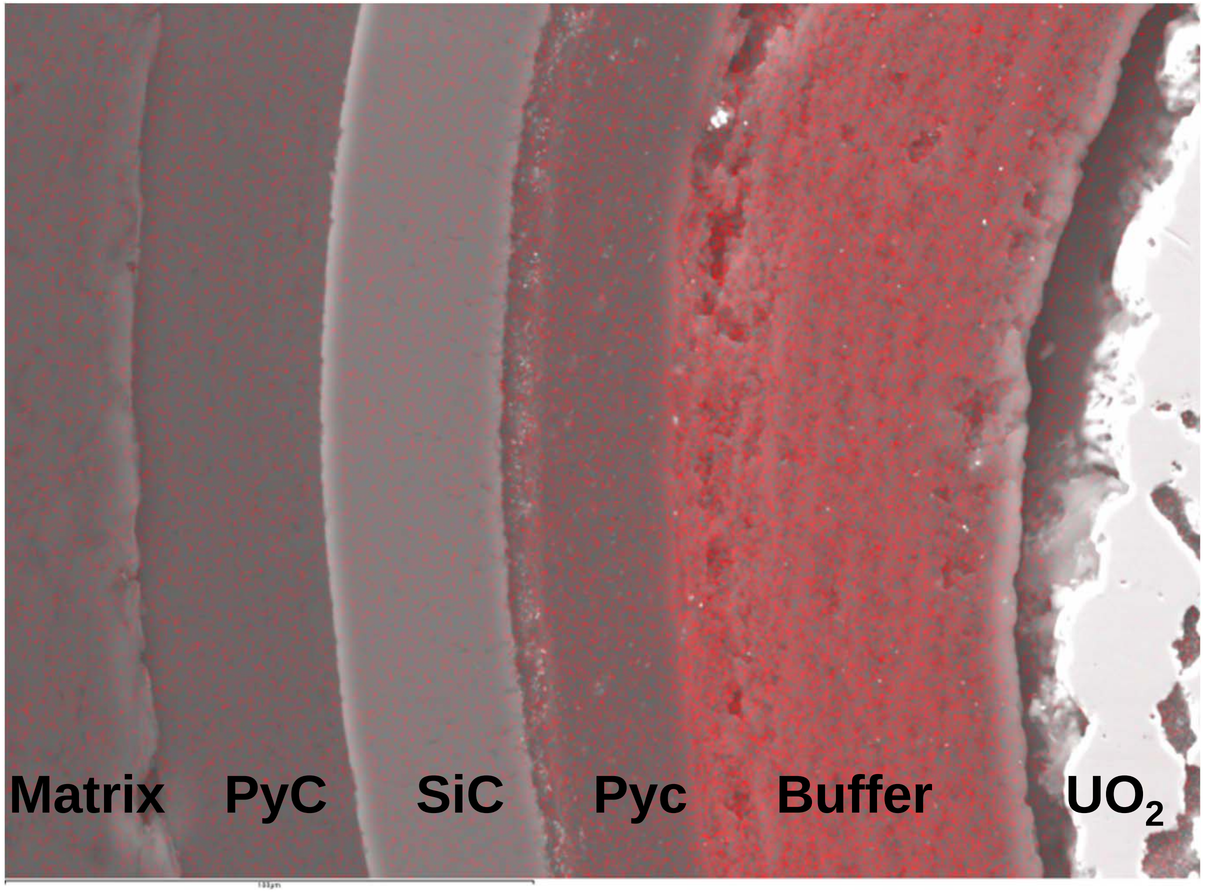


- SEM-imaging of one HTR TRISO fuel particle after irradiation
- UO_2 kernel, buffer, inner pyrocarbon, SiC and outer pyrocarbon layers visible
- Images show fission gas bubble formation at grain boundaries, metallic fission product precipitates, and a peculiar kernel surface, indicating fission gas release.

HFR-EU1bis Coatings



Cs mapping by WDS



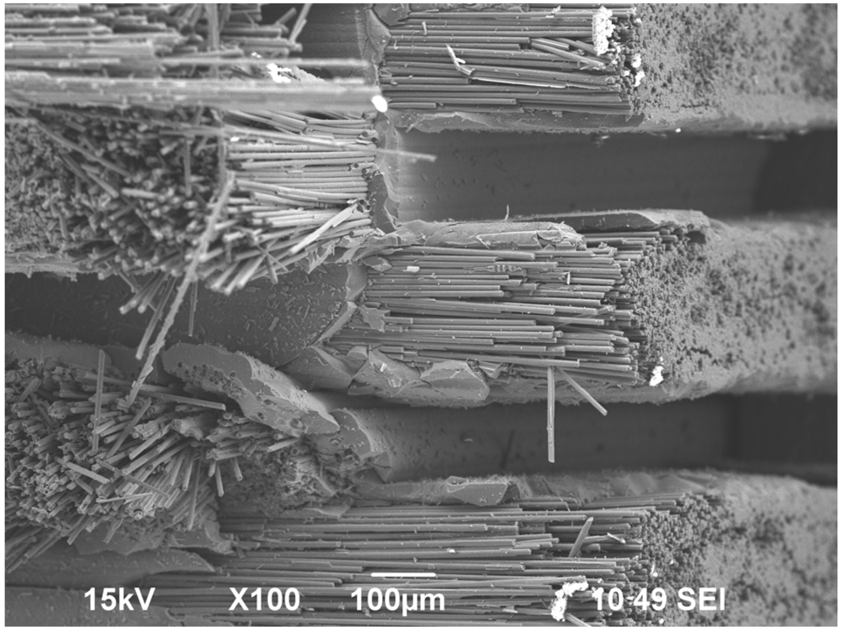
WDS mapping powerful tool to investigate and visualise element distribution

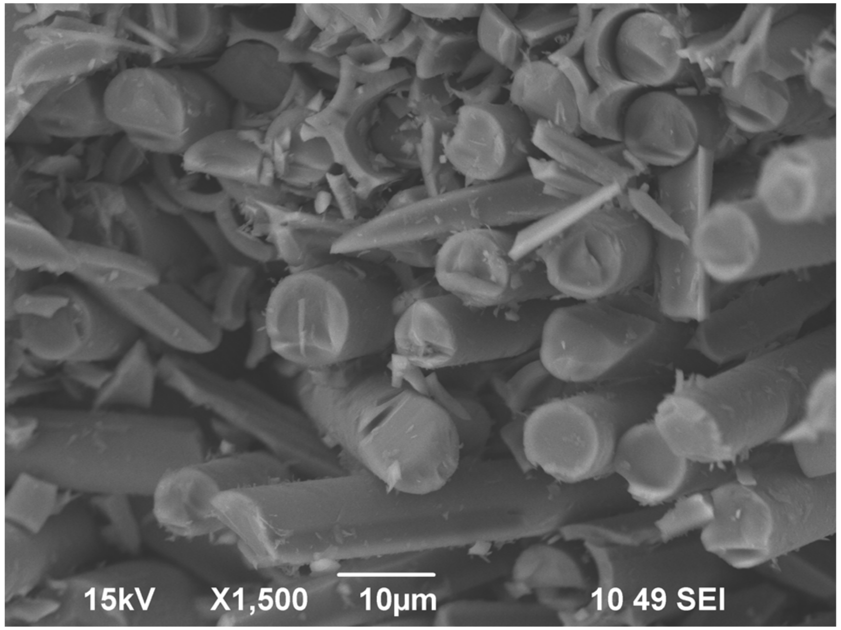
EXTREMAT

EXTREMAT-IP (6th framework programme EU):
Investigate new materials in extreme environments

- Two irradiation experiments performed in HFR Petten
- Among other materials fibre reinforced composites studied (SiC/SiC, SiC/C, C/C)
- Applications in fusion and advanced fission reactors
- Samples tested on mechanical and physical properties after irradiation
- Microscopy performed on fracture surfaces after 4 point bending tests

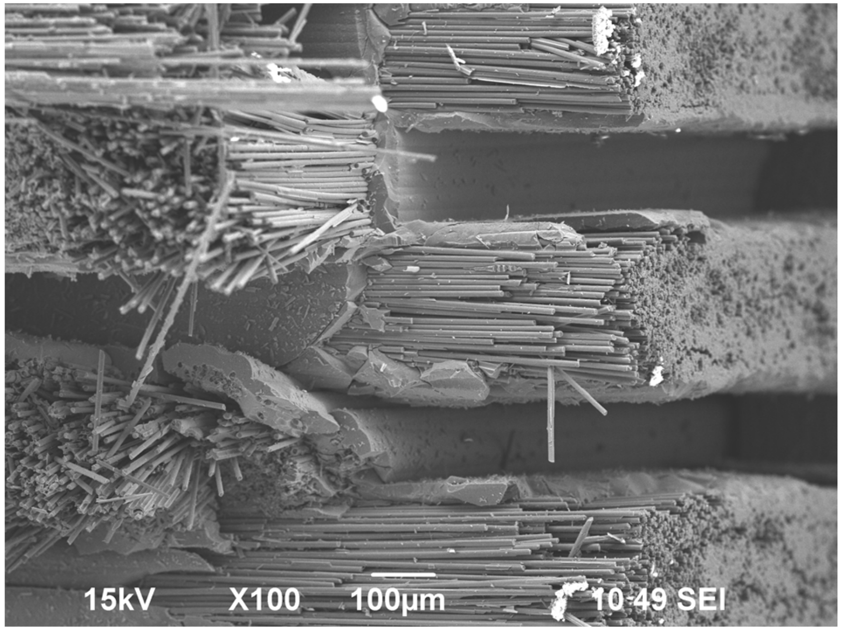
EXTREMAT Fracture surfaces

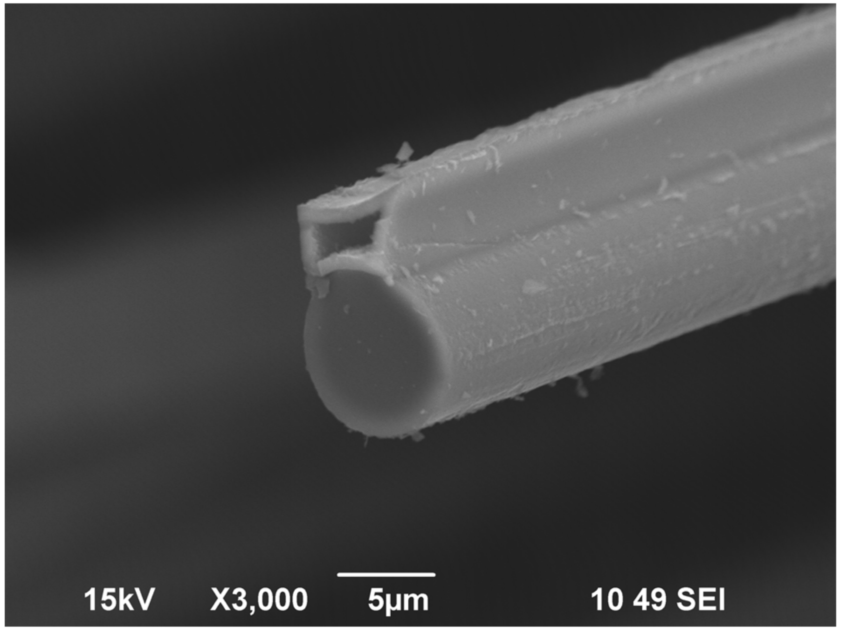




Fracture surface of SiC/SiC 3D woven to 4.5 dpa (steel) between 800 and 900ºC.

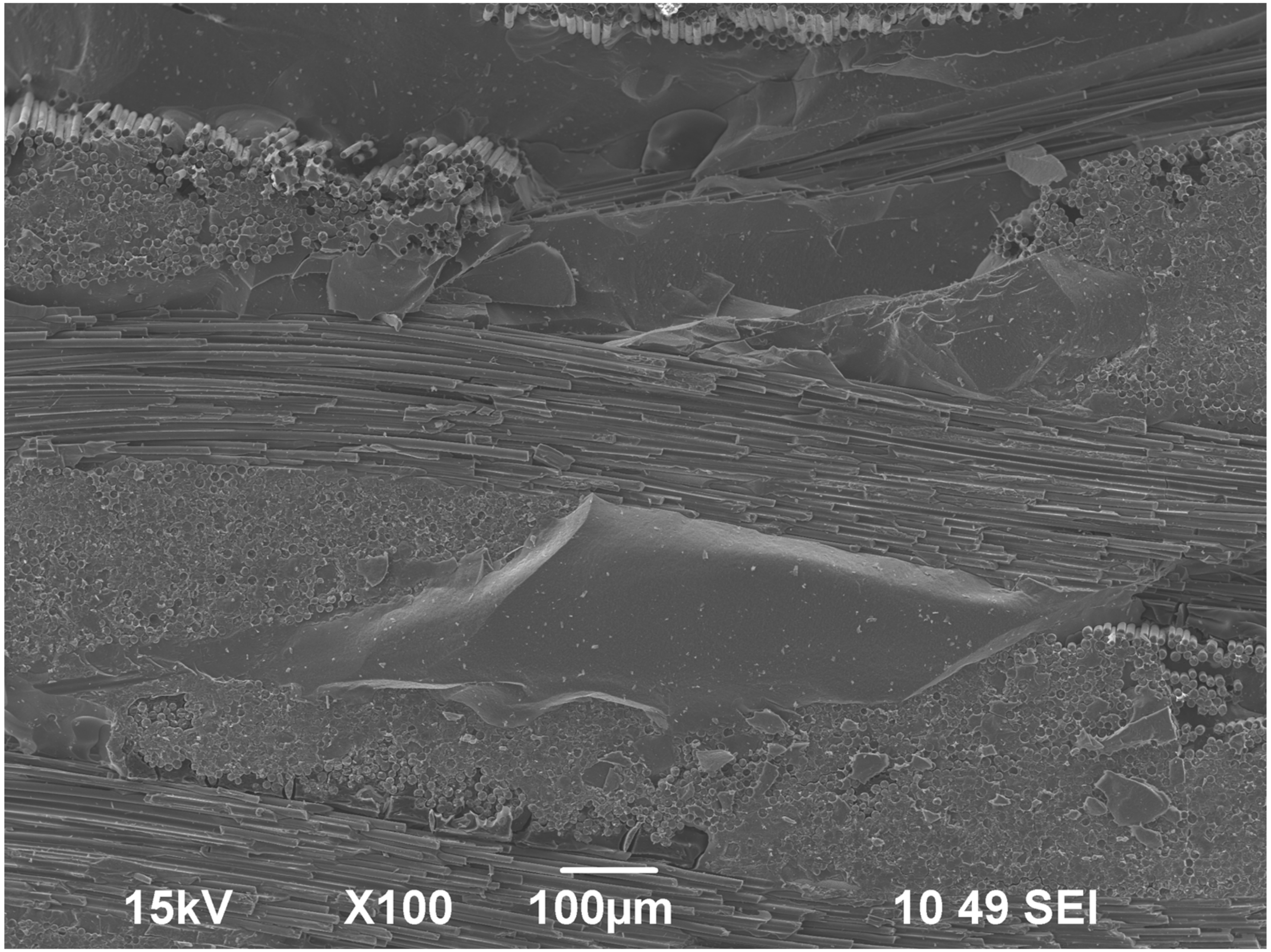
EXTREMAT Fracture surfaces





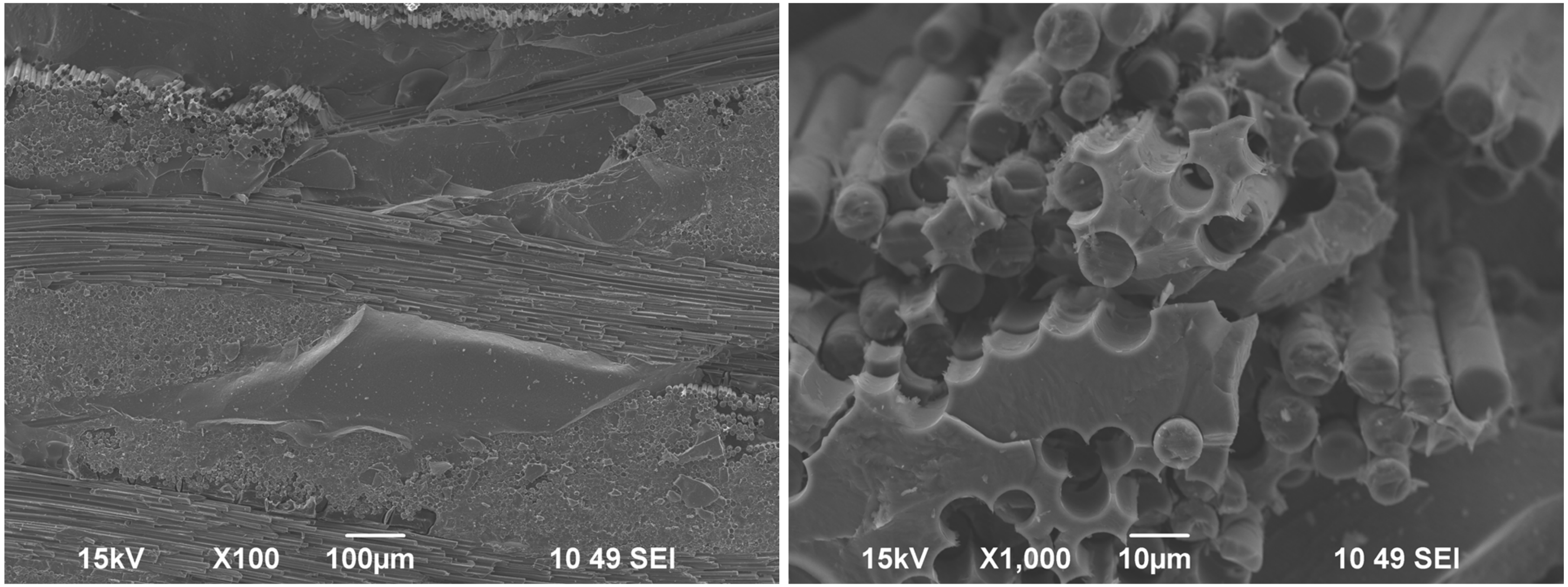
Fracture surface of SiC/SiC 3D woven 1 to 4.5 dpa (steel) between 800 and 900ºC.

EXTREMAT Fracture surfaces



Fracture surface of SiC/SiC bonded irradiated to 2.4 dpa (steel) at 600ºC

EXTREMAT Fracture surfaces



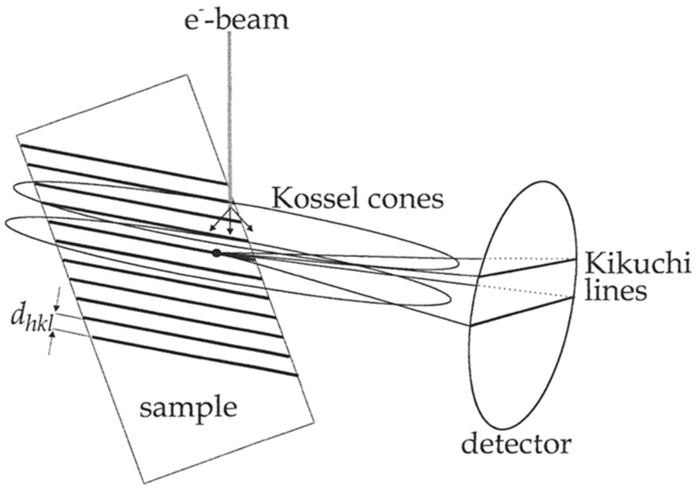
Fracture surface of SiC/SiC bonded, irradiated to 2.4 dpa (steel) at 600ºC

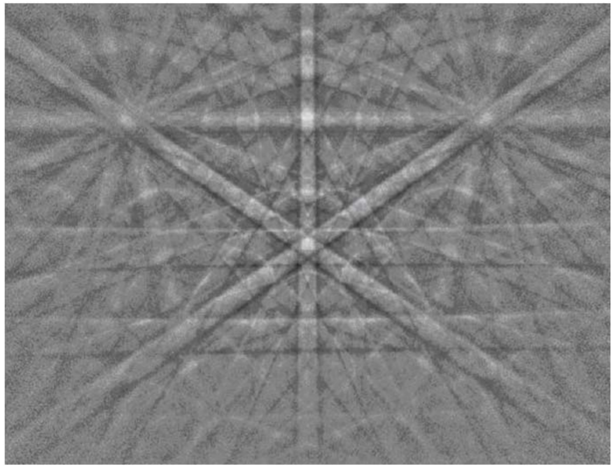
EBSD: Project started to develop technique on irradiated material

Electron Back-Scatter Diffraction

Obtains crystallographic information and can be used for:

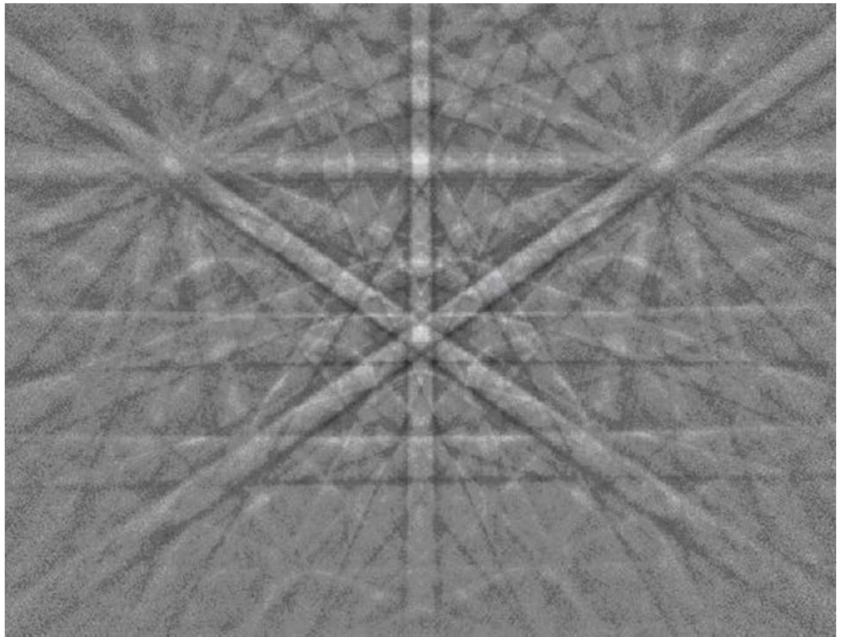
- Phase identification
- Texture
- Grain boundary identification
- Orientation relationships between phases

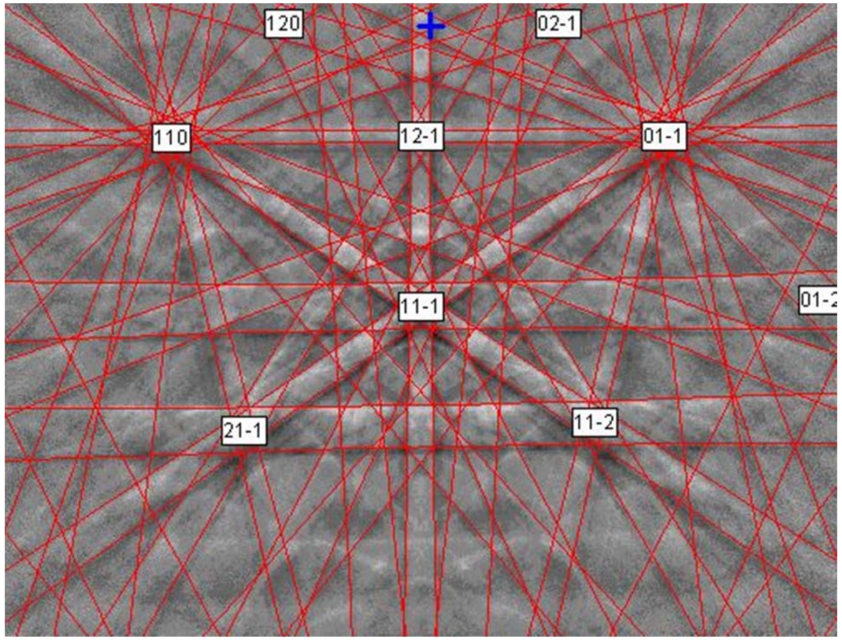




EBSD development project

First test on unirradiated Si:





Next step: irradiated material

Conclusions

- A JEOL JSM-6490 SEM, equipped with Oxford EDS/WDS/EBSD detectors, was successfully installed in a new hot-cell in the NRG hot-cell Laboratory
- Modifications where implemented to allow analysing radioactive samples in the SEM
- First radioactive sample loaded in August 2010
- EDS and WDS detectors successfully used to analyse nuclear fuels and structural materials
- Possibilities of EBSD on radioactive samples currently investigated
- SEM powerful instrument to collect valuable information from samples from irradiation experiments