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HOT CELL IMPLEMENTATION AND SOME APPLICATIONS OF A SEM EQUIPPED WITH A BACKSCATTERED ELECTRON DETECTOR, A HEATING OBJECT-HOLDER AND WDS

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INTRODUCTION

This document presents the installation of a scanning electron microscope (SEM) in a shielded hot cell at the Irradiated Composants Study Laboratory (LECI) of the CEA-Saclay nuclear research center.
The SEM used is a JEOL instrument, type 5400. It has been equipped with a WDS (Wavelength Dispersive Spectrometry), a heating object-holder and back-scattered electron system.

The scanning electron microscope is designed for fractographic studies on fuels (UO_2 and MOX), cladding and other more specific expertise investigations.
Fractography on fuels provides a graphic image of the porosity and fission gas bubbles, occurring, as well as the microstructure, without requiring ion or chemical attack to expose these properties.
Fractography on mechanical tested samples provides mechanisms changes in fracture mode, whitch involves damages in mechanical properties.
Others expertise investigations reveal cladding stress corrosion cracking.

I] THE SECONDARY ELECTRON MICROSCOPE

Description of the instrument :

As with any scanning electron microscope, the electronic images can be formed with secondary electrons or backscattered electrons. Both types of electronic images are generated using the same detector.
To avoid decreasing the performance characteristics of the SEM, the standard microscope configuration has been kept as far as possible. However, different modifications have been realized for the nuclearisation of the microscope. The electronic column which is the physical part of the SEM is located to the left of the electronic control part. To nuclearise the microscope, the electronic control part has been separated from the physical part, placing the latter in a shielded cell. To limit the quantity of material introduced into the shielded cell, only the electronic column has been placed inside the cell (figure 2). The pump system is located under the cell work surface. Examination chamber and pump system are configured to the standard configuration.

Shielding of examination chamber :

Because of the highly radioactivity of studied specimens, a denal shielding (tungsten-based alloy with density of 18.5) has been installed in the chamber. This consists of a 20 mm thick plate placed around the electron detector window. It is the first γ radiation barrier between the specimen and the secondary electron detector.

Manipulation system drive :

To use the microscope in the shielded cell, it was necessary to be able to remotely drive the specimen stage, as well as opening and closing of the door. The drive system for the X, Y, Z, T and tilt axes was designed using stepper motors.

Shielded cell :

The cell is formed by 150 mm lead shielding for the walls, and 200 mm steel for the floor and roof, with a 600 mm x 400 mm port and a remote manipulator (Walischmiller type) with booting for remote manipulation.
The rear shielding, consisting of a double door, and a lexan panel, can be opened for periodic preventive maintenance operations on the microscope.

Radiation and contamination control :

Located inside the shielded cell are two probes used to measure the radioactivity and contamination of the specimens. The contamination probe is also used to verify the condition of the stage plate and the work surface of the glove box. The specimens can be β, γ emitters. The β, γ activities of the specimens will be limited to about 5.2 10^{10} Bq (1.4 Ci).

<u>Preparation of the specimens</u> :

In this part, we present different investigations we widely practice.
In order to keep the SEM shielded cell as clean as possible, the specimens are prepared in another cell.

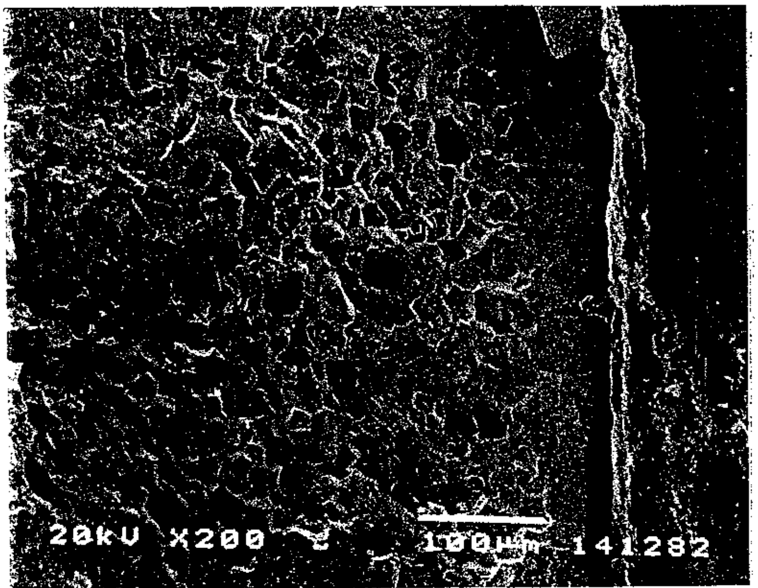
- Fuel specimens

A 15 mm cylinder of fuel is embedded in epoxy. A 3 mm-thick disk is cut out from this cylinder, in the longitudinal axis. This disk is then placed in the specimen support. A notch is made in the cladding to direct the fracture.
The fracture is made using a two-pillar crane fitted with a punch. This gives us a specimen holder containing the fractured fuel disk.
After fracturing, the specimen is systematically decontaminated in alcohol with ultrasonic cleaning. Following this, a conductive film (AuPd) is deposited by sputtering.

- Cladding specimen

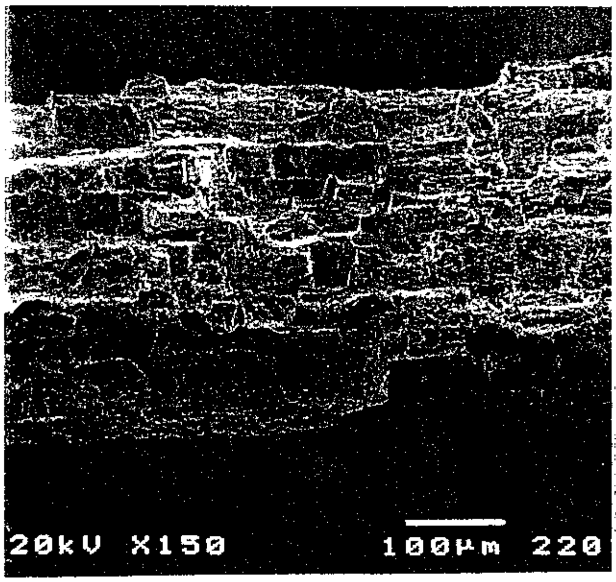
The cylinder is cut in its longitudinal axis and loaded until fracture.

<u>Exemples of investigations</u> :



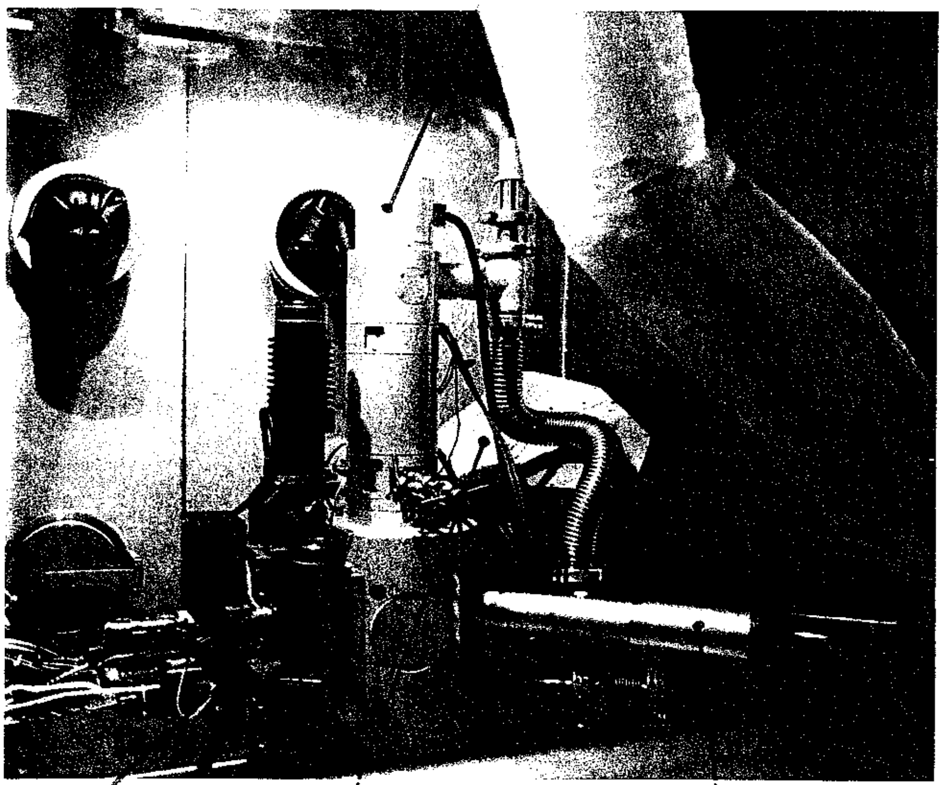
fuel | clad

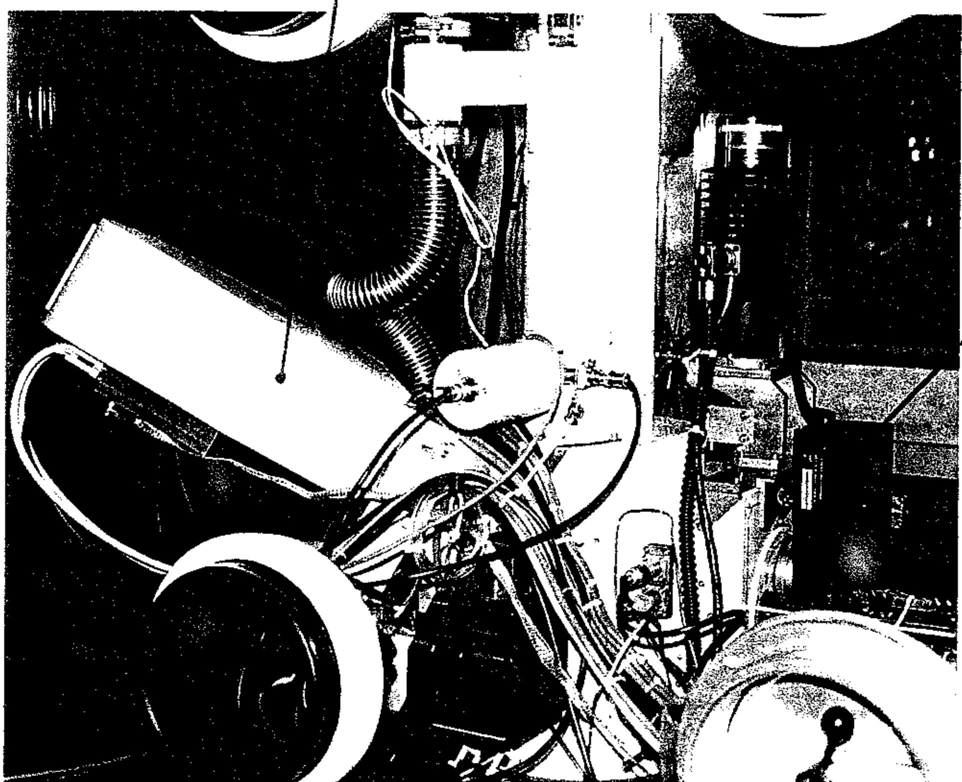
UO_2 with high burnup - Peripheral area
Dose rate on contact 1.8 Gy.h^{-1} (180 rad.h^{-1})



Longitudinal fracture on clad

SEM and its equipments





II] IMPLEMENTATION OF A RETRACTABLE BACKSCATTERED ELECTRON DETECTOR

In order to complete SEM analysis, the backscattered electron detector leads to atomic mass contrast, and so phase dispersion. Samples have to be well prepared (grinding and polishing).

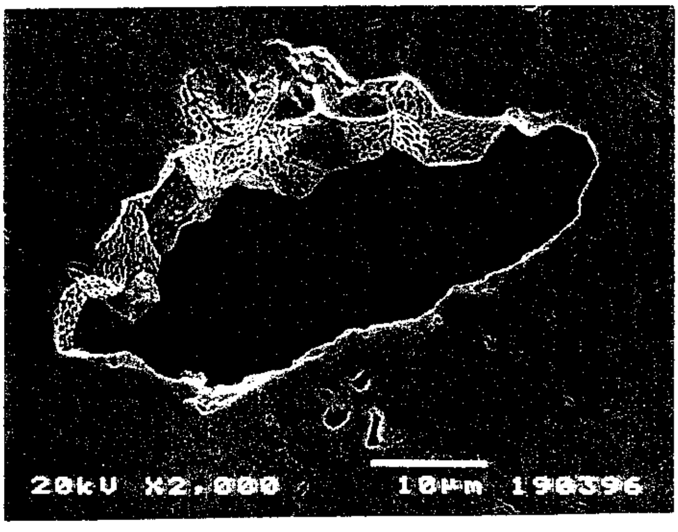
The ROBINSON detector (RBJ-5400) is a high gain retractable backscattered electron detector. It has been pre-aligned in manufacture to fit on the circular vacuum port on the right hand side rear of the specimen chamber.
It is correctly operated in the IN position and can be withdrawn out of the way when it is not used.

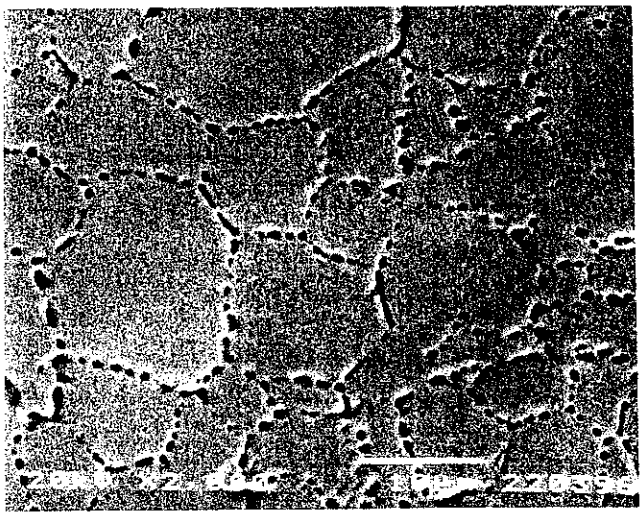
A classic diode backscattered electron detector cannot be used because of the badly resistance of the diodes to radiation. We have then installed a wide angle scintillator photomultiplier backscattered electron detector, with capability for retraction. The collection angle depends upon working distance, but typically 5 steradian, 80% of available hemispherical detection. The spatial resolution is better than 6 nm.

In the IN position, the detector is directly situated in the electron beam and above the specimen. We are then working in contrast phase.
Placed in retracted position, the detector leads to topography contrast.

Examples of different investigations :

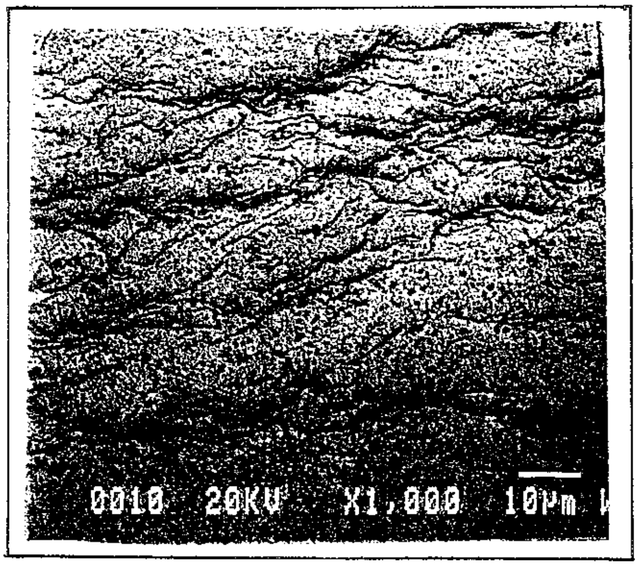
1) Porosity analysis in fuel materials
This analysis involves contrasted images, without shadow effect.





Porosity in UO_2 (secondary electrons) **Porosity in UO_2 (backscattered electrons)**

2) Displaying of hydrides in zircalloy cladding, which appeared during irradiation.

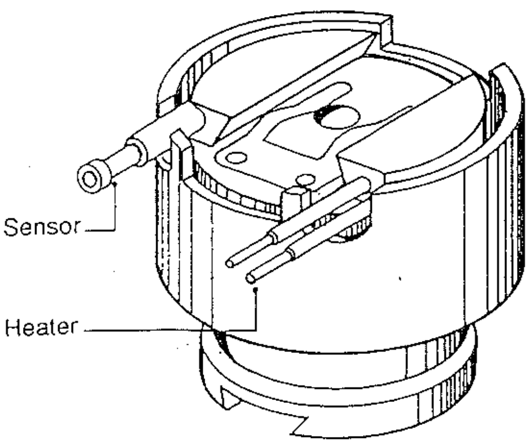


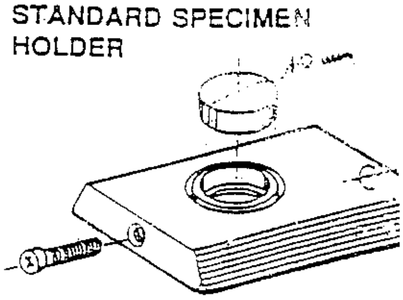
Hydrides in zircaloy cladding (20°C)

III] <u>HEATING OBJECT-HOLDER</u>

To provide the microstructure evolution with temperature, we have developped a hot stage which provides sample heating to 773 K (500 °C).

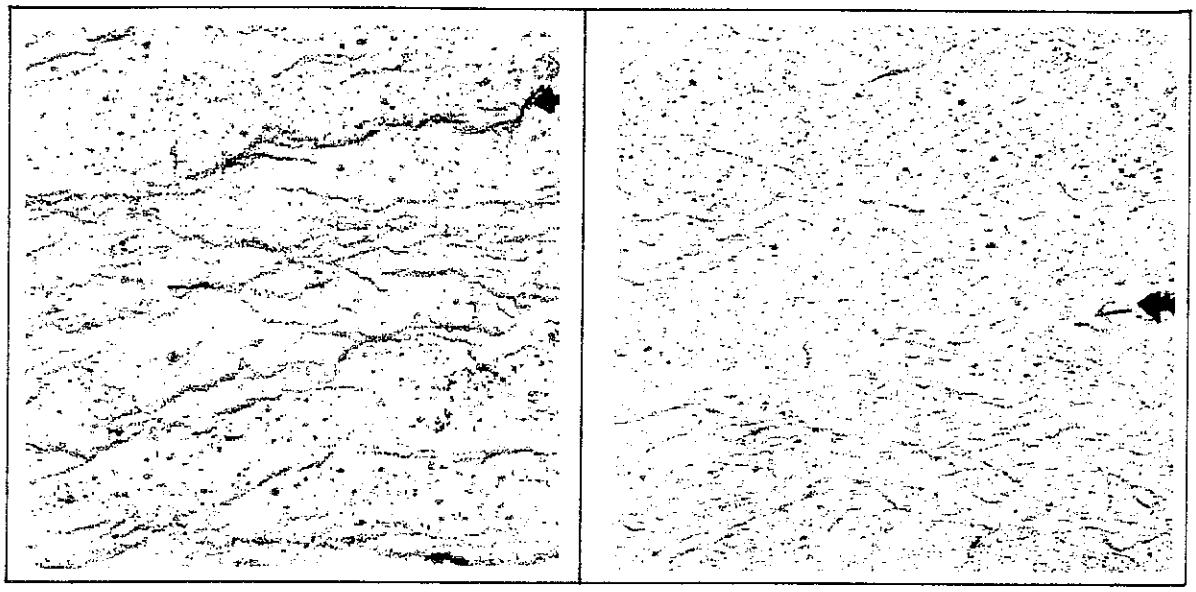
The system consists of :
- the sample hot stage with radiation shield, temperature sensor, heater and dovetail,
- flange with thermometry and heater leadthroughs,
- temperature controller





The hot stage is made of copper and is thermally isolated from the SEM goniometer and an earth wire ensures that specimen changing does not occur.
A platinum resistance thermometer and a heater element are mounted in the stage for temperature measurement and control.

Application :
purpose of displaying the kinetics of hydrides in the zircalloy cladding under temperature conditions near to those occurring in an operating reactor.



Hydrides disappearance at 350 °C

IV] X-RAY MICROANALYSIS

Two different spectrometers lead to X-ray microanalysis :

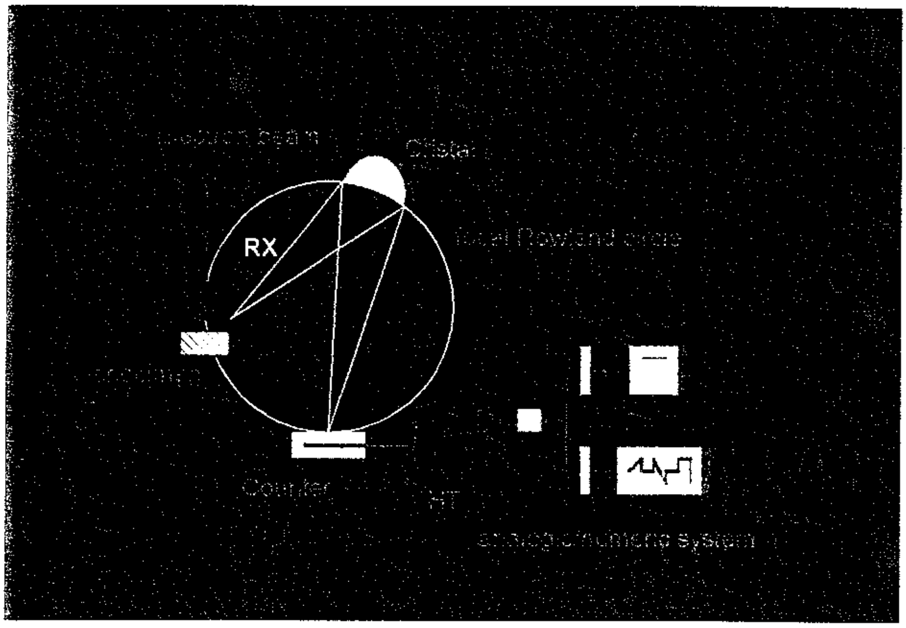
- The Wavelength Dispersive Spectrometer (WDS)
- The Energy Dispersive Spectrometer (EDS)

X-ray microanalysis by spectrometry leads to the identification of elements and the intensity of X-rays of specific energy emitted by a specimen submitted to an electron beam.

The choice of WDS is based on different arguments :

- the diode system detection of the EDS is too sensitive to radiation,
- WDS provides superior resolution and peak-to-background ratio, yielding order of magnitude improvement in sensitivity,
- WDS provides better spectral resolution which leads to the detection of trace elements (< 0.5 weight per cent),

- WDS gives better quantitative analysis quality (better counting rate for each ray).



Wave length Dispersive Spectrometry

The diffractor is positioned at a precise angle relative to the emerging X-ray beam to reflect only X-rays of specific energy into a counter.

cristal	**OV60**	**TAP**	**PET**	**PET**	**LIF**	**LIF**
Raie K	N7 F9	Na11 Si14	P15 Cl17	Ar18 Tl22	V23 Fe26	Co27 Zn30
Raie L	Tl22 Fe26	Ga31 Sr38	Zr40 Ru44	Rh45 Sb51	I53 Ho67	Er68 O76
Raie M		Sm62 Re75	Pt78 Bi83	Th90 U92		

Cristal diffractors for different elements

A multilayer structure of Mo/B_4C can be used for Z = 4 to 7, quartz is used for heavy elements.

Qualitative analysis : element identification is made loading Bragg's angle for which the Bragg law ($2d\sin\theta = n\lambda$) is satisfied.

Quantitative analysis : mass concentration of element A : $C_A = IA / I(A)$
IA : caracteristic intensity of A element, measured on the specimen
I(A) : caracteristic intensity of A element, measured on a calibration specimen

Adaptation to the SEM :
The spectrometer can be mounted horizontally or inclined. We have chosen the inclined montage. Inclined mounted, the detector leads to qualitative analysis of samples presenting an irregular (rough) surface. The vertical mounting alloys only perfectly plane specimens.

CONCLUSION

The nuclearized SEM gives very satisfactory results, the performance of the microscope has not been altered by nuclearization. Futhermore, during observations, we have become aware of the limitations of electronic imaging alone ; the equipements which have been installed are now opérationnels.
The backscattered electron detector gives information in contrast phase. The detector is protected from radiations in retracted position. The WDS, for the purpose of identifying and quantifying the different elements, is in validation instance. The heating plate gives temperature in the same order of magnitude than those occurring in a reactor.