Implementation of a scanning electron microscope (SEM) in hot cell

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• The LEMA lab (Actinide based Materials Study Laboratory) is located within the ATALANTE facility.
  • The SAFETY referential of the laboratory is:
    – $\alpha$ activity of the laboratory: $\leq 4\ 000\ Ci$
    – $\beta\gamma$ activity of samples: $\leq 20\ mCi$
• The laboratory was equipped with a nuclear scanning electron microscope dating from the eighties, model JEOL T 330A,
  Totally implemented in hot cell except electrical (remote) control
  – Obsolete technology, inadequate performance
  – Recurrent breakdowns for several years
    • Impacts on the quality of results and the planning process.
  – Its replacement was necessary.
The New SEM: Zeiss SUPPRA 55/55VP

• **Performance increase:**
  – A field emission super-high resolution SEM
    • Magnification range expanded up to 900,000 x in conventional operation
    • Imaging: increased surface detail thanks to low acceleration voltage
    • Best resolution: 1 nm at 15kV to 2.5 nm at 30kV
    • Best image quality
  – **Analytical Satellites**
    • In energy dispersive (EDS: Energy-Dispersive X-Ray Spectroscopy)
    • In dispersion wavelength (WDX: Wavelength dispersive X-Ray Spectroscopy)
      – Resolution gain
      – Semi-quantitative analysis with possible discrimination of U, Np, Pu, Am, Cm
Main Challenges of the SEM Nuclearization

- to make easier maintenance and repairing
  - Nuclearize only the sample chamber of the microscope
  - Be able to deconnect the microscope from the glove-box
  - Redesign biological protection
- to increase the image quality
  - Amortize the vibrations due to the ventilation of the facility and the glove box
Innovative Solution n° 1: Development of specific bellows*

- Use of elastomeric bellows to connect the chamber to the SEM glove-box

Advantages of bellows:

- Preserve the glove-box tightness
- Absorb vibrations and fluctuations of the pressure in the glove-box
- Life time estimated to several years (still to be confirmed)

* CEA patent n° 11 60216
Innovative Solution n° 2: Sealing plate*

- Sealing plate placed in the glove box, in front of the opening of the glove box allowing access to the microscope slide
  - To absorb excessive vibrations and to protect the microscope from the pressure fluctuations in the glove-box.

* CEA patent n° 11 60216
Innovative Solution n°3: Deconnection System*

– Deconnect the microscope from the glove-box while maintaining the glove-box confinement
  • The decoupling system consists in two plates (or doors) connected by a mechanical device that can dissociate one door without opening the second one.
  • The tightness and therefore the containment is guaranteed by the presence of an inflatable seal on the glove-box side and a seal on the microscope side.

Implementation of the decoupling system in the glove-box

1: Side with inflatable seal  
2: Side with seal

Seal between the two plates preventing contamination dispersion.

* CEA patent n° 11 60216
Kinematics of the disconnection operations of the SEM from the glove box

1— Position analysis: SEM is connected to the glove box

2— Installation of a rail to extract SEM

3— SEM extraction for maintenance
Technical solution n°4: Silent-block

- Absorb the vibrations to optimise the SEM performance
  - Silent-block placed between the metallic glove box support and the glove-box.

a: Glove box fixed on the frame  
b: Silent block fixation
• In 2009:
  – Old SEM dismantling
  – Biological protection modification
  – Design of biological protection to the new seism standards
Dismantling Operations

Old SEM JEOL T 330A
In 2010:

- Implementation of the new glove-box
- Implementation of the microscope and electrical connection
- Connection to the ventilation
- Leak test of the glove-box
**Authorization to introduce active sample:**

28/07/10

**First sample analysis:**

sept 2010
Some views of the new SEM installation in operation

SEM/Glove-box disconnected
As a conclusion… some observations (1/2)

MARIOS dense pellet \((U_{0.85}Am_{0.15})O_2\)

SEM image of a pore \((x2500)\)

SEM image of a pore \((x7500)\)

SEM image of grains \((x10000)\)

SEM image of grain boundaries \((x35000)\)
As a conclusion... some observations (2/2)

(UPu)O$_2$ powder after coprecipitation and calcination