FIB, SEM in hotcell
(Focus Ion Beam and Scanning Electron Microscopy)

Smail Chalal
Application Engineer
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We make it visible.
FIB, SEM in hotcell

Agenda

1. Project Quick overview
2. Development description
3. Detectors protection against radiation
4. Sample fall recovery
5. Conclusion
# Project quick overview

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FIB, SEM in hotcell

projects for SEM

This project was studied for CEA Valduc to install an SEM Supra 40 in a glove box, and has been carried out to handle contamination issues from Pu samples.

At CEA Marcoule, the challenge was to insert a Supra55WDS in an existing hotcell together with a sealed box. Rails are used to allow extraction of SEM outside the hotcell for maintenance purposes.

- Column and chamber
- Gloves port
- Empty plinth
- EHT unit
- SEM
- Sealed box
- Plinth for cable support
- Rails
FIB, SEM in hotcell

projects for SEM

The next project concerning an SEM was challenging for two reasons, to place a new generation SEM inside an existing Hotcell and separate it in two parts.
Two projects are actually ongoing concerning Crossbeam adaptation in radioactive Environment for CEA Saclay and Cadarache. For these projects, the sample activity can reach 52 GBq and a dose of 1 Gy/h to 4 Gy/h. On both configuration, the Crossbeam’s are connected to a glove box inside a hotcell.
## Hotcell Project for SEM and FIB

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Hotcell Project for SEM and FIB

Project description

The following detailed description will show all the feature carried out to handle Operators safety, electronics devices protection and contamination confinement.

- Outer shield
- Glove box
- Tele manipulators
- Crossbeam (SEM/FIB)
- Chicane for cables out
Hotcell Project for SEM and FIB

Project description
Hotcell Project for SEM and FIB

Project description

Original pendulum damping system kept

Pneumatic actuator
Lead bricks

Chamber door pneumatic actuator
Bellow
Double door slave part
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Detector protection against radiation

The light guide has been bended to avoid PMT and preamplifier damage from Gamma rays
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Detector protection against radiation

In our case, the activity is around 1 to 12 Ci. The average dose rate measured on sample is 1Gy/h.

D1 = Dose rate without shielding on EDS
D1 = 1Gy/h x $\left(\frac{5}{15}\right)^2 = 111$ mGy/h

E = Screen thickness, 0.8 cm (densimet 19.3 g.cm$^3$)
D2 = D1 x $e^{-\frac{5}{15} \times 0.8}$
D2 = D1 x $e^{-0.1093 \times 17.2 \times 0.8}$
D2 = 111 mGy/h x $e^{-0.1093 \times 17.2 \times 0.8}$
D2 = 111 mGy/h x $e^{-1.5039}$

D2 = 24 mGy/h

Dose rate without shielding on EBSD
D1 = 1Gy/h x $\left(\frac{5}{25}\right)^2 = 40$ mGy/h

E = Screen thickness, 0.8 cm
D2 = D1 x $e^{-\frac{5}{25} \times 0.8}$
D2 = D1 x $e^{-0.1093 \times 17.2 \times 0.8}$
D2 = 40 mGy/h x $e^{-0.1093 \times 17.2 \times 0.8}$
D2 = 40 mGy/h x $e^{-1.5039}$

D2 = 8 mGy/h
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Detector protection against radiation

EDS detector has a sensitive part and to avoid crystal and transistor damage, a Movable screen has been mounted in front To protect it against radiation when in Park position.
Hotcell Project for SEM and FIB

Detector protection against radiation

On EBSD detector, the sensitive part is the CCD camera. And in a same way than EDS a movable screen is fitted in front of it. Like EDS this screen is made of high density Matter (Tungsten alloy)
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Nano-indenter and micromanipulator
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New Stem design

5 segments:
- Bright field
  - Dark field
  - ADF
  - HAADF
Hotcell Project for SEM and FIB

Electronics devices protection against radiation

In our installation, we have chosen to move electronic plinth outside hotcell to avoid Radiation damages. Of course, in some cases this solution was not possible due to cable length limitation, like EHT unit and Ebsd control unit.
Hotcell Project for SEM and FIB

Electronics devices protection against radiation

As we do not have technical feedback on electronic component sensitivity against radiation, we have limited the dose rate to the controlled zone (or green zone) limitation.

\[ D_1 = \text{Dose rate without screen on EHT unit and EBSD control unit} \]
\[ D_1 = 1\text{Gy/h} \times \left(\frac{5}{66}\right)^2 = 5.74\text{ mGy/h} \]

\[ E = \text{Average thickness below SEM chamber, 9.7 cm} \]
\[ D_2 = D_1 \times e^{-\frac{\mu \times \rho \times E}{\frac{\rho}{\mu}}} \]
\[ D_2 = D_1 \times e^{-0.07 \times 8.02 \times 9.7} \]
\[ D_2 = 5.74\text{ mGy/h} \times e^{-5.445} \]
\[ D_2 = 24.8\text{ µGy/h} \]
# Hotcell Project for SEM and FIB

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Hotcell Project for SEM and FIB

Sample fall recovery

First barrier plate

Second barrier plate

Lengthened rods
Hotcell Project for SEM and FIB

Collection of sputtered materials

We have carried out a long study to define optimized collection screen of sputtered material during etching FIB process. Unfortunately, I am not allowed to show results and solutions because a patent deposit is ongoing.

*Mean size of holes etched for TEM lamella prep:*
  *TEM: 20μm x 25μm x 10μm.*
  *Volume given: 5000μm3*
  *Holes on each side of the lamella: 2 x 10000μm3*
  *Volume in mm3: 10000μm3 x 10-9 = 0.00001mm3*

*Summary: To generate a volume of 1mm3, at least, carried out 100000 TEM lamellas.*
Conclusion
Conclusion:
Study and manufacturing have been carried out together with a partner called Défisystèmes installed at Nîmes, south of France. For information, a FIB project like CEA Saclay and CEA Cadarache took 15 months from the order to the delivery. All the necessary technical information are given by the factory as 3D drawings, Cable description, wiring…
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