Shielded Focused Ion Beam Field Emission Scanning Electron Microscope (FIB-FE-SEM): Evaluation, technical modification and implementation

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Abstract

Scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDS) are important analyses techniques in the PSI Hot-laboratory to characterize nuclear materials. Since 1992 the PSI Hot-laboratory operates the SEM DSM962 on low active irradiated materials, but with the raising operation time the maintenance of the electronic spare parts becomes more and more difficult and therefore the acquirement of a new instrument was indispensable. On the other hand the PSI research division of nuclear materials has the request of materials characterization in the atomic scale, which is possible at synchrotron beam lines. Due to statutory specified limited activity values at these facilities the production of tiny specimens is essential.

To combine the requests the new shielded analytical tool should be equipped with a focused Ion beam (FIB) gun and a micromanipulator for in-situ sample preparation combined with an analytical system consisting of Energy Dispersive X-ray Spectrometry (EDS), Wavelength Dispersive X-ray Spectrometry (WDS) and Electron Back Scatter Diffraction (EBSD).

The instrument should allow to handle highly radioactive samples like irradiated fuel and cladding. To protect human health and life and the sensitive parts of the instrument modifications have to be performed on the conventional purchasable instrument and detectors.

The best offered instrument combined with the favorable conditions was a Zeiss Crossbeam 540 Zeiss is represented in Switzerland by the company Gloor Instruments AG.

The device in the final stage will be equipped with the following system:

- GEMINI II field emission electron optic
- Capella FIB column with liquid Ga ion source
- micro manipulator highly precise “Oxford Omniprobe Autoprobe 200.2”
- Everhart-Thornley-type chamber detector (SE)
- in-lens SE-detector
- a YAG-detector for Back-Scattered Electrons (BSE)
- in-lens EsB
- Energy Dispersive X-ray Spectrometry (EDS), with 3D analysis
- Wavelength Dispersive X-ray Spectrometry (WDS)
- Electron Back Scatter Diffraction (EBSD) with high resolution “DigiView 5” camera, with 3D analysis

Since there is a limitation of floor loading in the laboratory, the construction of the hot cell was challenging. The dimensions of the lead shielding had to be large enough for a small glove box and the instrument attached with all the detectors, but also small enough not to exceed the maximum floor loading allowed. In order to fulfill these requests the instrument is installed in

a cabin with a wall thickness of 10 cm lead, detectors are protected with shutters and the remote controlled loading system is realized with manipulators through an attached glove box, which allows safe handling of highly alpha/gamma radioactive and/or contaminated samples.
With this new installation it is planned to prepare tiny specimens for further analyses on various PSI-internal as well on PSI-external beam lines. The complex analysis system of the FIB/SEM itself allows qualitative and semi-quantitative elemental and crystallographic analyses of in-situ prepared surfaces and ion cut and polished open laid structures of any bulk material.

1. Introduction

Since more than 40 years SEM and EDS are important analyses techniques in the PSI Hot-laboratory to characterize nuclear material. In 1992 the 25 years old Cambridge Stereoscan S4-10 was replaced by the Zeiss DSM 962 [1]. This SEM is equipped with an Everhard-Thornley detector for secondary electrons, a Robinson-type backscatter detector and for the detection of X-rays a liquid Nitrogen cooled Pioneer Si(Li)-detector in combination with the Tracor-Noran Voyager analysis system, later upgraded with the Noran System SIX.
Since this instrument was not shielded, only analyses of inactive and low gamma active specimens were possible. Occasionally with a specially constructed specimen holder [2] analyses of α-contaminated material as well as fresh fuel can be performed. Now, with increased aging, the maintenance becomes more and more difficult, the availability of electronic spare parts decreases and therefore the acquirement of a new instrument was indispensable.
With the financial support of the SNF and the PSI directorate the tender procedure (WTO) started. Subsequent to the deadline for tenders, the best offered instrument combined with the favorable conditions was a ZEISS machine. Zeiss is represented in Switzerland by the company Gloor Instruments AG.
The planning and execution of the project of the NES FIB/SEM XB540 system is dependent on the organization and the collaboration of four different companies (Gloor Instrument AG as master, Zeiss France, EDAX and Défi Systémes). In January 2016 the order has been triggered. Subsequent of a short development period the first draft of the machine housing was presented to PSI, by the company Défi Systémes.

2. Requirements

The Lab of Nuclear Materials (LNM) had the wish to procure a Focused Ion Beam Milling (FIB), a device, which enables the production of tiny specimens precisely cut out of highly radioactive samples to be below the activity limits for samples at the beam lines.
The unshielded SEM is still in operation, but due to the described limitations (maintenance, low sample activity etc.) it has to be replaced as soon as practicable.
Consequently, the procurement of a device which combines the SEM and the cutting technique was seen as an advantage. Therefore, the NES division decided to procure a FIB.
Thus the device has to enable the production and pre-characterization of high quality tiny specimens below the activity limit of beam lines and conform to the requirements of the AHL PIE questions.

3. Instrument & Equipment

The electron source of the SEM unit is a GEMINI II field emission gun [Fig. 1]. The lateral resolution is 2 nm at 1 kV and 1 nm at 30 kV. The ion source is a Zeiss Capella ion gun with a huge probe current range from 1 pA up to 100 nA with a maximum resolution of 3 nm.

Secondary electrons (SE) are detected with an Everhart-Thornley-type chamber detector and with the in-lens SE-detector. For the detection of Back-Scattered Electrons (BSE) a YAG-detector and an in-lens EsB (Energy selective Backscatter) will be available.

The EDAX-Pegasus analysis system contains the “Octane SUPER” silicon drift detector for EDS, the high resolution “DigiView 5” camera for crystallographic analyses with EBSD and the TEXS WDS spectrometer.

To lift out and handle the prepared specimen in the vacuum chamber after ion cutting and polishing a highly precise “Oxford Omniprobe Autoprobe 200.2” micro manipulator will be available.

Fig. 1: Zeiss XB 540, Glove box

4. Shielding and Modification

To protect human health and sensitive parts of the instrument several kinds of shielding have been partly completely new developed. There is an alpha glovebox with tele-manipulator [Fig.4] and loading equipment [Fig.2]. A hot-cell around the alpha-box and the FIB/SEM-instrument with all the detectors was constructed. To protect the detectors from gamma irradiation internal shielding was constructed.


Fig. 2: Hot Cell with transfer systems

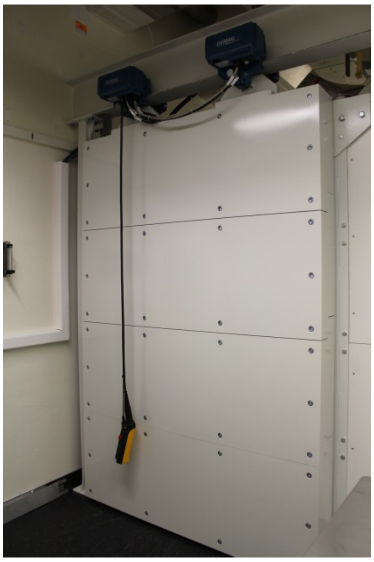
Fig. 3: Hot Cell back door closed

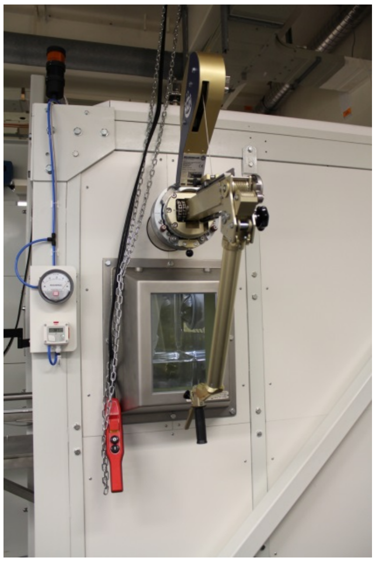
Fig. 4: lead glass window and tele-manipulator

The alpha box is built of stainless steel and is fitted with two acrylic glass windows. Through a “La Calhène” lock (LCL) [Fig. 5-7] inactive and low active material and through a double cover lock (DCL) [Fig. 8-10] high active and highly contaminated samples may be transferred in and out of the containment. With a specially developed magnetic driven loading system, specimens can be load and unload to/from the chamber of the FIB/SEM. A tele-manipulator [Fig.4] supports the loading/unloading process. During absence of high active samples five gloves allow manipulations inside of the alpha- box e.g. for service if necessary.

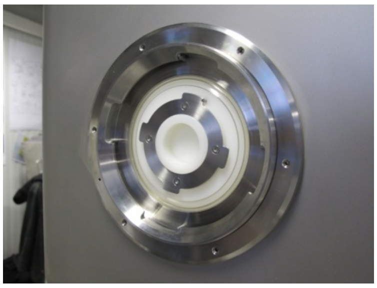
Fig. 5: LCL access


Fig. 6: LCL connection


Fig. 7: LCL container

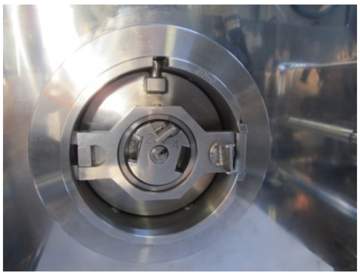
Fig. 8: DCL closed

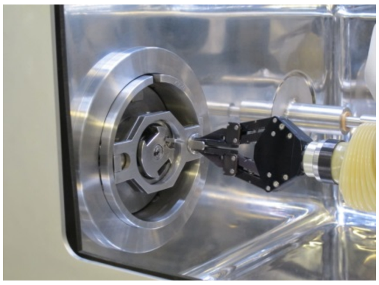
Fig. 9: DCL manipulation

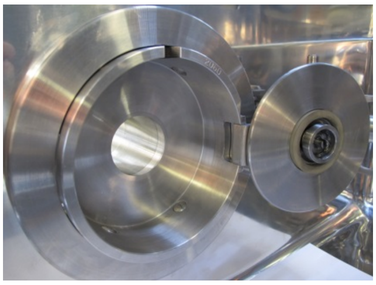
Fig. 10: DCL open

The hot-cell [Fig. 2-4] encloses the alpha box and the Instrument (specimen chamber, interlock system, detectors, vacuum system). The inner dimensions are 2.17 m x 1.37 m x 2.39 m (l/w/h). The walls are built from 10 cm lead. The roof and the wall facing the laboratory concrete wall were built of 5 cm lead to reduce the weight. Using the tele-manipulator the 70 cm lead glass allows the view into the alpha box. . On the back side of the hot-cell an electrically moveable steel door allows the access to the enclosed instrument parts for maintenance. The total weight of the hot-cell including door [Fig.3], lead window and steel construction is some 25.8 metric tons and since the maximum floor load of the laboratory is 5 t/m^2 the maximum allowed weight was almost achieved.
EDS- and EBSD-detector are sensitive to the irradiation with gamma rays and will lose performance with increasing energy deposed. Therefor those detectors are retractable and at the outer position they are protected with special and individual constructed shutters [Fig. 11-16].


Fig. 11: EBSD retracted

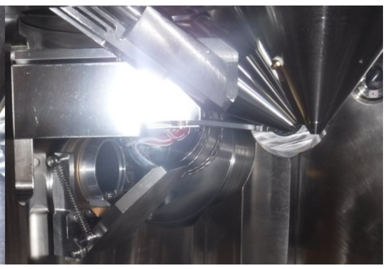
Fig. 12: EBSD halfway

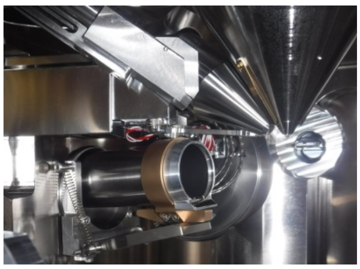
Fig.13: EBSD inserted

Fig. 14: EDS retracted

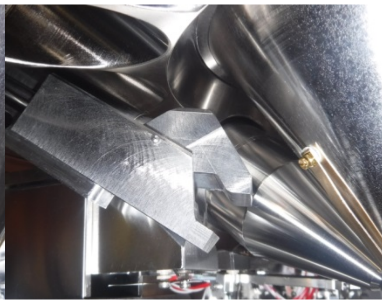

Fig. 15: EDS halfway

Fig. 16: EDS inserted

5. Planned field of application

The installed equipment will allow the preparation of tiny specimens from highly radioactive and contaminated materials as well as the characterization of these materials with state of the art analysis tools.
It is planned to prepare specimens from fuel (fresh and irradiated), cladding material etc. for e.g. Transmission Electron Microscopy (TEM) and various other facilities on PSI campus outside the hot-laboratory with statutory specified limited activity values, e.g. Synchrotron Light Source (SLS) beam lines etc.
With the integrated analysis systems in situ characterizations of fresh prepared surfaces in nanometer scale will be performed, e.g. crystallographic structures, strain and orientation, size and composition of secondary phase particles etc.

Acknowledgment

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References

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