Focused Ion Beam-Based Microstructural Characterization of Irradiated Materials at KAERI

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1. Introduction

Understanding irradiation-induced microstructural evolution is critical for predicting the performance and lifetime of reactor structural materials [1,2]. Transmission electron microscopy (TEM) remains one of the most powerful tools for direct observation of nanoscale irradiation defects such as voids, dislocation loops, and solute segregation at grain boundaries [3, 4]. However, the preparation of electron-transparent radioactive thin foils poses significant challenges in terms of safety, precision, and throughput.

To address this, Korea Atomic Energy Research Institute (KAERI) has established a FIB-SEM laboratory in Building 2, forming part of its radiation-controlled research infrastructure. The system provides an essential linkage between bulk irradiated samples and nanoscale characterization, thereby enabling the development of a national-level capability [5] comparable to those already in operation at INL, ORNL (USA), NNL (UK), and CEA (France).

2. Facility Description

The radiation-controlled laboratory in Building 2 at the KAERI is equipped with a state-of-the-art Focused Ion Beam-Scanning Electron Microscope (FIB-SEM) system. Manufactured by Thermo Fisher Scientific and integrated with the Oxford Instruments Aztec Ultim 65 Energy Dispersive spectroscopy (EDS) detector and Symmetry Electron BackScattered Diffraction (EBSD) detector, this analytical machine provides comprehensive capabilities for the microstructural characterization of irradiated materials.

The laboratory is systematically partitioned into three zones to facilitate efficient and safe specimen handling and analysis as shown in figure 1. The zone 1 is dedicated to the initial processing of irradiated samples, which includes cutting by a low-speed precision saw and surface preparation using an automatic polisher with colloidal silica slurry; electrolytic polishing can be employed when minimal surface damage is required. These preparation steps significantly reduce the overall radioactivity of the specimens, thus addressing radiological safety concerns for laboratory personnel.

The Zone 3 houses the FIB-SEM system, where microstructural analysis and advanced specimen fabrication is performed. Here, electron backscatter diffraction analysis is used to identify crystallographic orientations and high-angle grain boundaries of interest. The FIB enables precise microfabrication, including

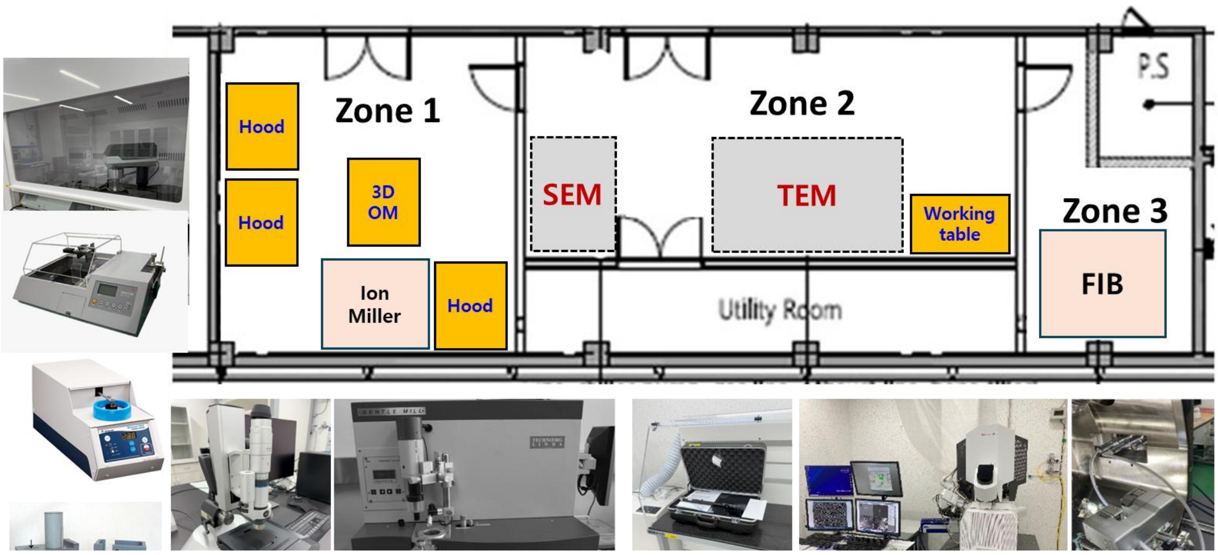

Figure 1 Laboratory Layout

trench milling and the preparation of TEM lamellae through site-specific deposition, lift-out, grid attachment, and final thinning. Low-energy ion milling is utilized to further reduce specimen thickness to below 100 nm, which is optimal for transmission electron microscopy investigations.

The Zone 2 serves as a reserved area for the future installation of advanced microscopy equipment, such as a conventional LaB6 TEM and a W-SEM. At present, TEM specimens are loaded onto dedicated holders and transferred to separate analytical laboratories within KAERI for further characterization; after analysis, specimens are returned to the radiation-controlled area for storage or disposal.

3. Sample Preparation Workflow

The preparation of radioactive and irradiated specimens for microstructural analysis within the KAERI Building 2 laboratory follows a carefully controlled and systematic workflow designed to maintain radiological safety while achieving high-quality analytical samples.

Initially, irradiated materials stored in the underground irradiated sample repository are transferred to Zone 1 of the Building 2 laboratory. In this zone, radioactive specimens with activity levels up to 6000 MBq are securely mounted using epoxy resins, such as G1 epoxy or conductive sliver epoxy, within specially designed fixation jigs. The curing process typically requires approximately 4-24 hours to ensure complete hardening and immobilization of the specimens.

Once fixed, the mounted specimens undergo precision sectioning using a low-speed saw. The cutting procedure involves incremental removal of material, with each cut designed to yield micro-scale sections approximately 500 micrometers or less in thickness. The goal is to produce thin disk or square foils roughly 10 mm in diameter or width, containing the irradiated regions of interest, while simultaneously reducing the specimen radioactivity to approximately one-tenth of the original level for safer handling.

Subsequent mechanical polishing is performed in Zone 1 using an automated polisher using colloidal silica slurry to minimize surface damage. When required, electrolytic polishing may be employed to further reduce residual deformation and produce surfaces suitable for high-resolution analysis. The polishing process is critical to obtain electron microscopy specimens with sufficiently smooth and damage-free surfaces.

In Zone 3, specimens that have undergone preliminary preparation are transferred for detailed microstructural analysis and final specimen fabrication. The FIB-SEM system is employed to perform site-specific trench milling and to identify regions of interest based on EBSD or surface morphology data. After target areas are selected, a protective carbon or platinum layer is deposited to preserve the area during focused ion beam milling. Lift-out procedures are then conducted to extract lamellae from the bulk specimen. The extracted TEM lamellae are precisely thinned by ion milling to thicknesses below 100 nm, which is essential for electron transparency during TEM analysis. Following FIB thinning, low-energy ion milling is carried out within a separate workstation inside Zone 1 to further minimize surface damage and ensure optimal specimen quality.

Finally, the prepared TEM specimens are mounted on grids and placed into radiation-dedicated TEM holders in the Zone 2. These holders allow safe transport of the samples to TEM facilities located in other KAERI buildings, where primary high-resolution imaging and spectroscopic analyses including scanning TEM and electron energy-loss spectroscopy (EELS) thickness mapping are performed. Based on these analyses, further ion milling can be applied if necessary to achieve the desired specimen thickness.

All specimens post-analysis are either stored securely in the underground storage or disposed of following KAERI's radiation safety protocols. This integrated sample preparation workflow ensures that high-quality

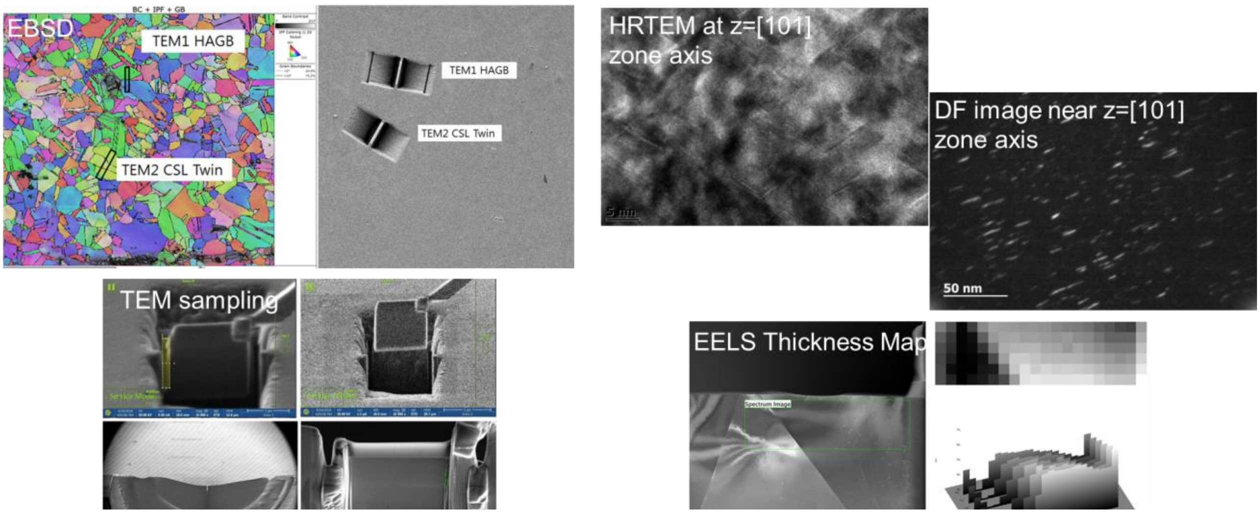

Figure 2 TEM Sampling and Analysis Capabilities for Characterization of Irradiated Materials

microstructural and nanoscale investigations of irradiated nuclear materials can be conducted while maintaining rigorous radiological safety standards.

4. Key achievements

The FIB-SEM facility at KAERI has successfully demonstrated advanced specimen preparation and microstructural characterization capabilities for irradiated materials. The key achievements now include the fabrication of electron-transparent TEM lamellae from ion-irradiated stainless-steel alloys and Zr alloys. Using optimized milling parameters, uniform lamella thicknesses below 100 nm were consistently accomplished, enabling high-resolution TEM and scanning TEM (STEM) analyses.

Microstructural observations revealed characteristic irradiation-induced features such as segregation of chromium, nickel, and silicon at high-angle grain boundaries as identified by EBSD and STEM-EDS mapping. Moreover, nanoscale irradiation defects, including voids, bubbles, and dislocation loops, were visualized, confirming effective irradiation damage simulation and specimen preparation protocols.

Several technical challenges were encountered and addressed during specimen preparation. Thickness control was refined through iterative STEM-EELS thickness mapping, ensuring samples met the stringent electron transparency requirements. Radiological safety concerns, including exposure reduction, were mitigated by a stepwise sample cutting process that lowered radioactivity by approximately an order of magnitude before detailed processing.

5. Future upgrades

To further enhance the analytical capabilities and research throughput, several key facility upgrades are planned at KAERI Building 2. First, the installation of a Lab6 transmission electron microscope (TEM) within the radiation-controlled Zone 2 is envisaged. This on-site TEM will eliminate the current requirement to transport radioactive specimens between buildings, substantially reducing handling risks and increasing operational efficiency.

Second, a high-temperature nanoindentation module will be integrated within the FIB-SEM platform. This upgrade will enable in-situ mechanical property measurements of irradiated specimens at reactor-relevant temperatures, providing invaluable data to understand irradiation hardening and deformation mechanisms under realistic service conditions. Such thermal-mechanical testing capabilities are expected to significantly contribute to the design and qualification of structural materials for advanced reactor technologies.

These upgrades will position the KAERI building 2 laboratory as a cutting-edge center for nuclear materials research, supporting both fundamental science and applied material development efforts. The enhanced infrastructure will promote international collaborations and foster the generation of highly innovative research outcomes to meet growing demands in the nuclear energy sector.

Overall, the integration of the FIB-SEM preparation workflow and advanced electron microscopy analyses has enabled comprehensive studies of irradiation effects at micro- to nano-scale resolutions on representative nuclear reactor structural materials. These capabilities enhance the understanding of irradiation damage mechanisms critical for the qualification and development of materials for current and next-generation reactors, including small modular reactors (SMRs).

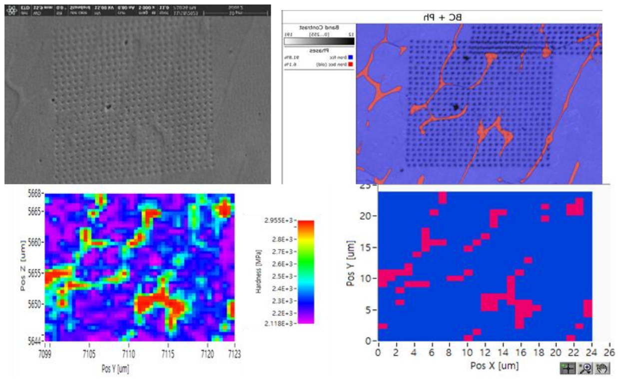

Figure 3 Combined Nanoindentation and EBSD Analysis in the FIB-SEM Facility

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