The Development of the Shielded Secondary Electron Microscope at KAERI

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1. Introduction

KAERI developed a shielded secondary electron microscope, which was remodeled from the Philips XL-30 model in 1998. The SEM was installed inside a glovebox, which has the shielding wall of 17 cm carbon steel and the confinement wall of hardened glass. Also, the glovebox was connected to the deep under pressure (DUP) line of a HVAC system. The shielded SEM has been applied to fractography of a spent fuel pellet, crud analysis of a PWR cladding, surface inspection of the contact area between grid spring and fuel rod, hydride morphology analysis of a cladding and so on.
Currently, we have developed the quantitative analysis techniques by the WDS-SEM. As a part of the development, we remodeled a sputter coater for hotcell and made a quantitative analysis program for a WDS-SEM including the function of inspecting the beam stability. We have plan to analyze fission products like Xenon and Neodymium inside a spent fuel and dopants like Manganese and Chromium of doped UO2 by the techniques.

2. Shielded SEM

The XL-30 SEM has maximum accelerated voltage of 30 kV with the electron gun of LaB6 and W hairpin. The SEM equipped with secondary electron detector (SED), back scattered electron detector (BSED), energy dispersive spectroscope (EDS) and wavelength dispersive spectroscope (WDS). In order to install inside a shielded glovebox, the SEM of Philips XL-30 model was modified as follows;

- Glass light conductor of SED instead of acrylate conductor
- Additional scan filter set for XL-FEG (Field Emission Gun)
- Chamber modification due to the confinement area of the glovbox
- Signal and electrical line extension
- Extension of chamber door open length

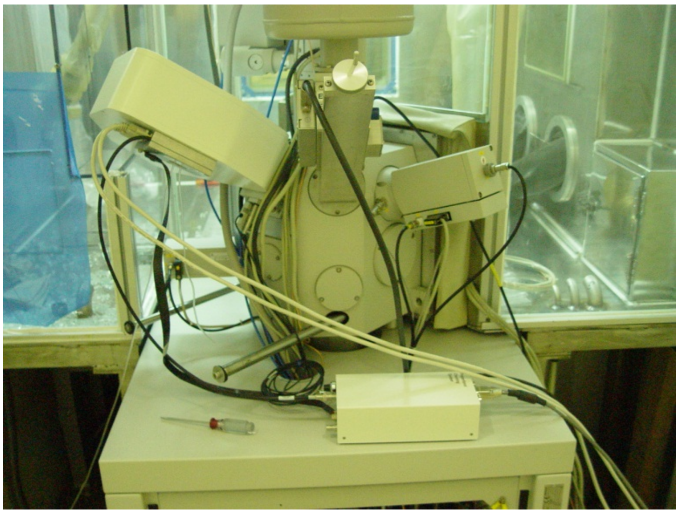


Fig 1. SEM of XL-30

Lead shielding of 17 cm surrounding the glovebox provides a safe protection against radiation with 25 cm lead glass window as figure 2. The glovebox has a docking port, two manipulator, radioactive sample storage, and three shielding doors. The docking port can be connected to a sample cask for the radioactive sample transfer to hotcell. The design of the glovebox was based on a radioactive level as table 1.

Table 1. Radioactive criteria of the glovebox

Pressurized Water Reactor Fuel Sample	
Burnup	50 GWd/tU
Decay time	3 years
Enrichment	5 wt% U-235
Radioactivity	7.4 GBq

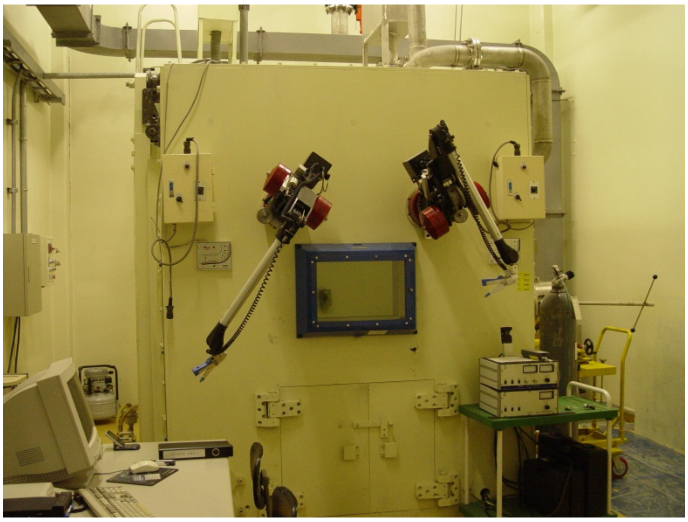


Fig 2. Shielded glovbox

3. WDS analysis

WDS has in a better position than EDS for analyzing a spent fuel sample. The X-ray detector of the WDS is a gas proportional counter, which has low detection efficiency for a high energy photon like gamma ray [1]. And also a straight path between a sample and a detector can be easily shielded to make the P/B ratio high. About 5 cm lead shielding minimized X-ray background counts due to gamma-ray emitted from the PWR spent fuel sample by 100 cps. It was a 0.5 mm thick disk-type sample with burnup 55 GWd/tU.
The electron beam stability is important for the quantitative analysis. It directly affects the measurement precision. To inspect the beam stability, a picoammeter (Keithley 6485 model) was installed. The beam current is measured in a Faraday cup and then the target is changed to the PWR spent fuel for the quantitative analysis. The beam current data can be used to evaluate effectiveness of WDS data. We developed WDS analysis program through the procedure of WDS-SEM analysis as figure 3.

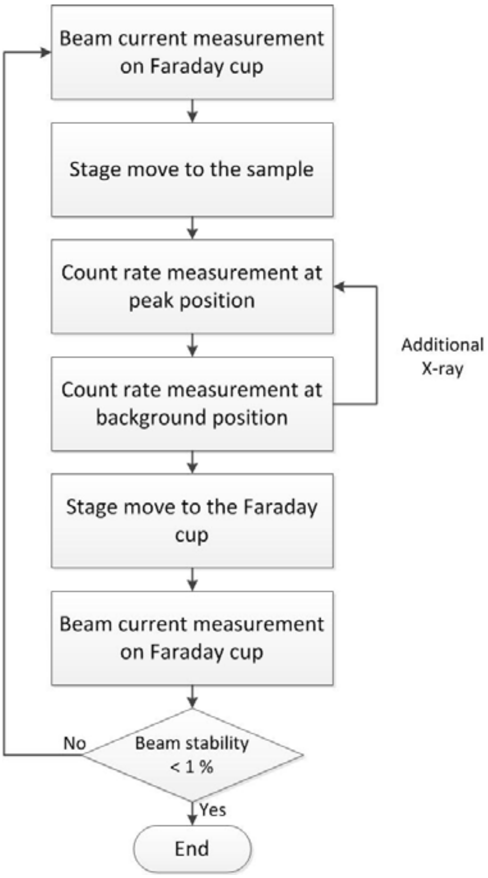


Fig 3. Flow chart of WDS analysis procedure.

The program consists of a main view, a beam current monitor, and a COM port monitor. The main view has the functions of inspecting the WDS and beam current data, showing a graphical chart for the synchronized data of the count rate and current data, evaluating the peak count rate normalized by a beam current and determining the effectiveness of the data. The beam current monitor has the functions of setting up the picoammeter, which measures the beam current, and confirming the values of the current. The COM port monitor shows information from the WDS such as the command and measurement data. The text in the window of the COM port monitor can be dragged and copied to a text editor.

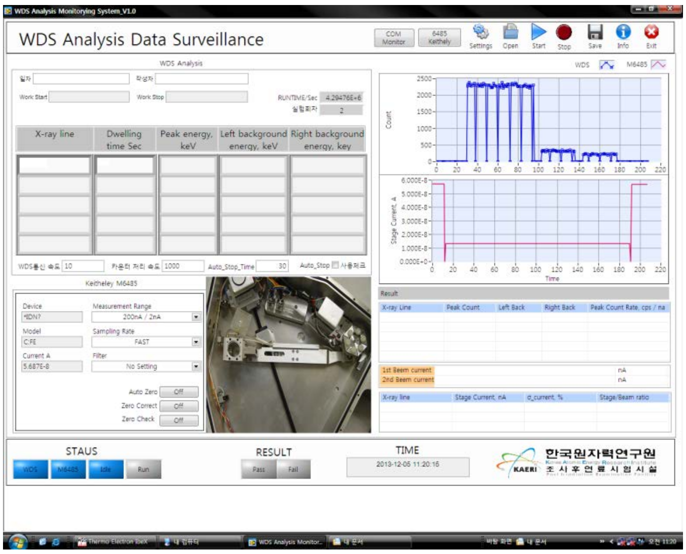
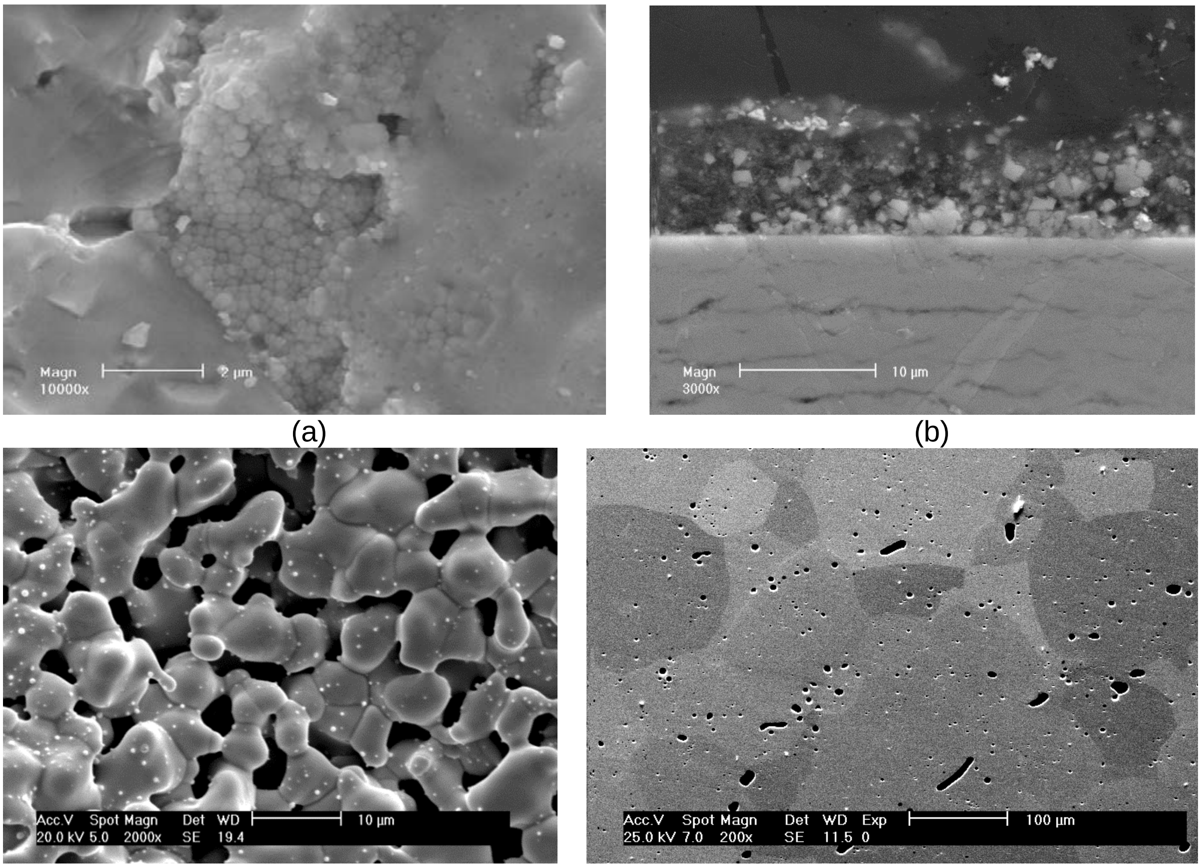


Fig 4. Main view of the WDS analysis program

4. Experience

For about 15 years, the shielded SEM has applied to the various nuclear fuel research fields. In 2003, the fractography of fuel RIM was carried out as the research of high burnup fuel performance. Since 2004, the CRUD morphology has been analyzed in order to measure crud thickness, grain size and composition. Also, the shielded SEM has played an important role on the annealing test of a spent fuel through conforming grain separation and precipitates. For recent years, the SEM has been used for the researches of new developed fuel of KAERI like dual cooled annular fuel, large grain UO2 fuel and doped UO2 fuel. Figure 5(d) shows a polishing surface of a doped UO2 fuel sample as grain orientation contrast image.



(a) (b) (c) (d)

Fig 5. SEM images (a) RIM fractography (b) CRUD morphology (c) annealing test (d) doped UO2 fuel

Recently, WDS analysis techniques suitable for the spent fuel sample are being arranged. The WDS analysis has carried out under the test condition: accelerated voltage of 25 kV, spot size of 9, beam current of about 200 nA, beam stability of below 1 % and carbon coating. The detectability limits were evaluated on fresh doped UO2 pellets as table 2.

No.	1	2	3	4	5
Element	U	Mn	U	Mn	Mn - Gamma*
n	1	1	3	3	3
t	60	600	60	120	120
P	163868	14298710	163868	2859742	2883742
P/B	24.47	40.51	24.47	40.51	24.52
DL, ppm	212	6	122	16	21

where, n is the number of times, t is the dwelling time, P is the peak count, P/B is the peak to background ratio and DL is the detectability limit.

5. Conclusion

For about 15 years, the shielded SEM at PIEF of KAERI has applied to the various nuclear research fields: commercial PWR fuel and new developed nuclear fuel of KOREA. We have plant to improve the measurement techniques of WDS-SEM and carry out the full scale analysis for the small amount fission products of the PWR spent fuel and the irradiated fuel pellet in the HANARO research reactor by the shielded SEM. And also, we are considering introducing the new shielded SEM of advanced technologies.

6. References

[1] Glenn F. Knoll, Radiation Detection and Measurement 3rd edition, p.186, John Wiley & Sons(2002)