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 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.
- 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.
2. Shielded SEM

- 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 glovebox
  - Signal and electrical line extension
  - Extension of chamber door open length
2. Shielded SEM

- Lead shielding of 17 cm surrounding the glovebox provides a safe protection against radiation with 25 cm lead glass window.
- 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 the hotcell.

Radioactivity limit of the glovebox

<table>
<thead>
<tr>
<th>Pressurized Water Reactor Fuel Sample</th>
<th></th>
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</thead>
<tbody>
<tr>
<td>Burnup</td>
<td>50 GWd/tU</td>
</tr>
<tr>
<td>Decay time</td>
<td>3 years</td>
</tr>
<tr>
<td>Enrichment</td>
<td>5 wt% U-235</td>
</tr>
<tr>
<td>Radioactivity</td>
<td>7.4 GBq</td>
</tr>
</tbody>
</table>
3. PIE Procedure

Fuel Assembly (in pool):
- Transportation
- Receiving
- Unloading
- Storage
- Pool Side Examination
  - Visual Inspection
  - Dimensional Measurement
  - Gamma Scanning

Fuel Rod (in hot cell):
- Dismantling
- Cutting
- Non-Destructive Examination
  - Visual Inspection
  - Eddy-Current Test
  - Profilometry
  - X-ray Radiography
  - Internal P Measure.
  - Fission Gas Analysis

Specimen (in hot cell):
- Storage
- Evaluation
  - Failure mechanism
  - Fuel integrity
  - In-core behavior
- Destructive Examination
  - Metallurgy
  - Ceramography
  - Micro-Gamma Scan.
  - Densimetry
  - Burnup/H Analysis
  - SEM/EPMA; Chem.Anal.
4. SEM analysis {RIM structure}

- The fractography of fuel RIM had been carried out as the research of high burnup fuel performance.
- RIM structure: a few hundred nanometer polyhedral and round subgrain, about one micrometer sized pore
4. SEM analysis {CRUD}

- The CRUD morphology has been analyzed in order to measure thickness, grain size and composition of CRUD.
- Radially sectioned cladding samples were prepared for the thickness measurement of CRUD.
- Taping samples were prepared for the composition analysis for CRUD.
4. SEM analysis  \{Post-Irradiation Annealing\}

• The post irradiation annealing test of a spent fuel had been carried out through conforming grain separation and precipitates.
4. SEM analysis (Backscattered Electron Image)

- Most of the total BSE signal is due to Z-contrast effects.
- In crystalline materials, a contribution is also made from the interaction between primary electrons and the crystal structure of the target.
- BSE images for SF can be used for
  - Pore morphology
  - Grain morphology
  - Hydride morphology
4. SEM analysis \{WDS-SEM\}

- Two factors of WDS-SEM analysis for the spent fuel
  - Electron beam stability
  - Background increase due to gamma-ray
4. SEM analysis \{SEM-WDS\}

- Preliminary test for the spent fuel

<table>
<thead>
<tr>
<th>Element</th>
<th>X-ray line</th>
<th>Energy, keV</th>
<th>Crystal</th>
<th>Bragg angle</th>
<th>Counts per second</th>
<th>Non-shield</th>
<th>3 cm Lead</th>
<th>5 cm Lead</th>
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</thead>
<tbody>
<tr>
<td>Nd</td>
<td>Lα1</td>
<td>5.229</td>
<td>LiF</td>
<td>36.07</td>
<td>1173</td>
<td>224</td>
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<tr>
<td>U</td>
<td>Mα1</td>
<td>3.171</td>
<td>PET</td>
<td>26.57</td>
<td>751</td>
<td>143</td>
<td>87</td>
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<tr>
<td>Pu</td>
<td>Mβ1</td>
<td>3.531</td>
<td>PET</td>
<td>25.06</td>
<td>694</td>
<td>181</td>
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<tr>
<td>Xe</td>
<td>Lα1</td>
<td>4.109</td>
<td>PET</td>
<td>23.68</td>
<td>1001</td>
<td>239</td>
<td>133</td>
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</table>

<table>
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<th>No.</th>
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<th>3</th>
<th>4</th>
<th>5</th>
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<tbody>
<tr>
<td>Element</td>
<td>U</td>
<td>Mn</td>
<td>U</td>
<td>Mn</td>
<td>Mn - Gamma*</td>
</tr>
<tr>
<td>n</td>
<td>1</td>
<td>1</td>
<td>3</td>
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<td>t</td>
<td>60</td>
<td>600</td>
<td>60</td>
<td>120</td>
<td>120</td>
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<tr>
<td>P</td>
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<td>14298710</td>
<td>163868</td>
<td>2859742</td>
<td>2883742</td>
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<tr>
<td>P/B</td>
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<td>40.51</td>
<td>24.47</td>
<td>40.51</td>
<td>24.52</td>
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<tr>
<td>DL, ppm</td>
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<td>6</td>
<td>122</td>
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</table>
4. SEM analysis \{SEM-WDS\}

- Standardless quantitative analysis for major fission products

<table>
<thead>
<tr>
<th>Sample</th>
<th>X-ray line</th>
<th>$\varphi(\rho_z)$ emit Area (g/cm²)</th>
<th>ZA</th>
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<tbody>
<tr>
<td>Spent Fuel</td>
<td>Nd La</td>
<td>1.14E-03</td>
<td>0.768</td>
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<tr>
<td></td>
<td>Xe La</td>
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<td>0.688</td>
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<tr>
<td>Nd</td>
<td>Nd La</td>
<td>1.49E-03</td>
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<tr>
<td>Xe</td>
<td>Xe La</td>
<td>1.56E-03</td>
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</tr>
<tr>
<td>NdF₃</td>
<td>Nd La</td>
<td>1.42E-03</td>
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</table>
6. 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.
• We are considering introducing the new shielded SEM of advanced technologies.