

Application of FE-SEM to the measurement of U, Pu, Am in the irradiated MA-MOX fuel



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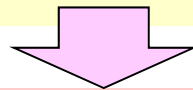
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Objective of application of FE-SEM

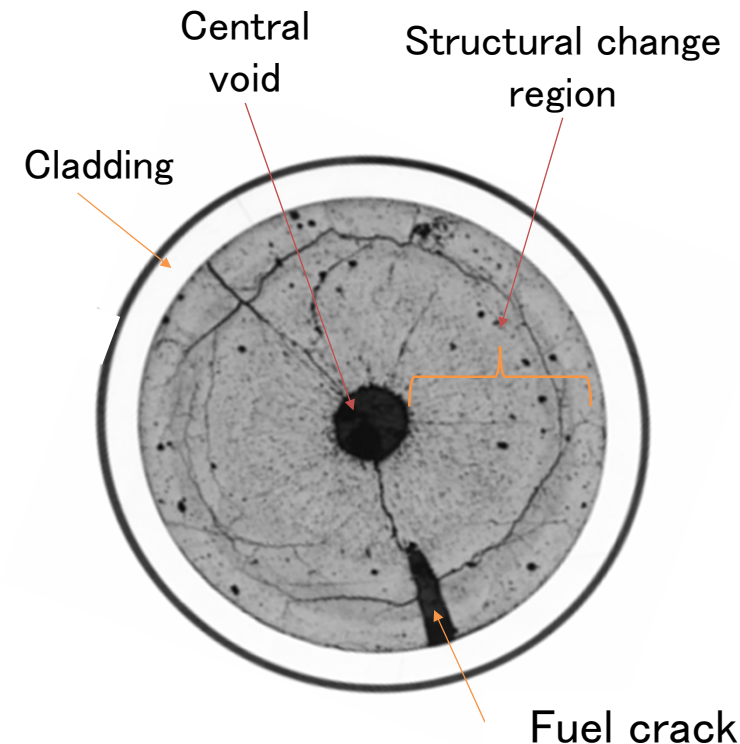
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During irradiation of MOX fuel containing minor actinides(MA) in a fast reactor, two changes take place because of a radial temperature gradient:

- 1. Microstructure**
- 2. Distributions of actinide elements**



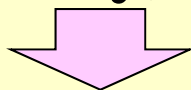
FE-SEM was developed for the examinations of above two changes.



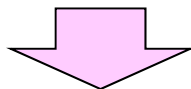
Typical cross section image of fuel specimen irradiated in a fast reactor.

Irradiated fuel specimens

:high radioactivity, α -particles emitting



- Prevent leakage of radioactive materials
- Protect operators and the instruments from radiation



It is necessary to modify PIE instruments when irradiated fuel specimens are examined.

The installed FE-SEM

: JEOL JSM-7001F

This FE-SEM uses thermal Schottky type field emission gun.

- ✓ High resolution observations (3.0nm at 15kV)
- ✓ Electron beam current is high stably

The FE-SEM is equipped with WDX (Oxford Instruments INCA WAVE) and EDX(Oxford Instruments INCA X-act) for surface elemental analysis.

- 1) To prevent leakage of radioactive materials, the instrument was attached to a remote control air-tight specimen transfer unit between a shielded hot cell and the FE-SEM.**
- 2) To protect operators and the instruments from radiation, the FE-SEM was installed in a lead shield box and the control unit was separately located outside the box.**

Outline of the FE-SEM installation and modification (4)

(Specimen transfer unit)

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- Electric-powered transfer unit of the radioactive specimen (maximum size 20mm diameter, 10mm height)
- Airtight boundary is achieved by O-ring seals.
- It is able to remove the irradiated fuel specimen manually if this unit have troubles.



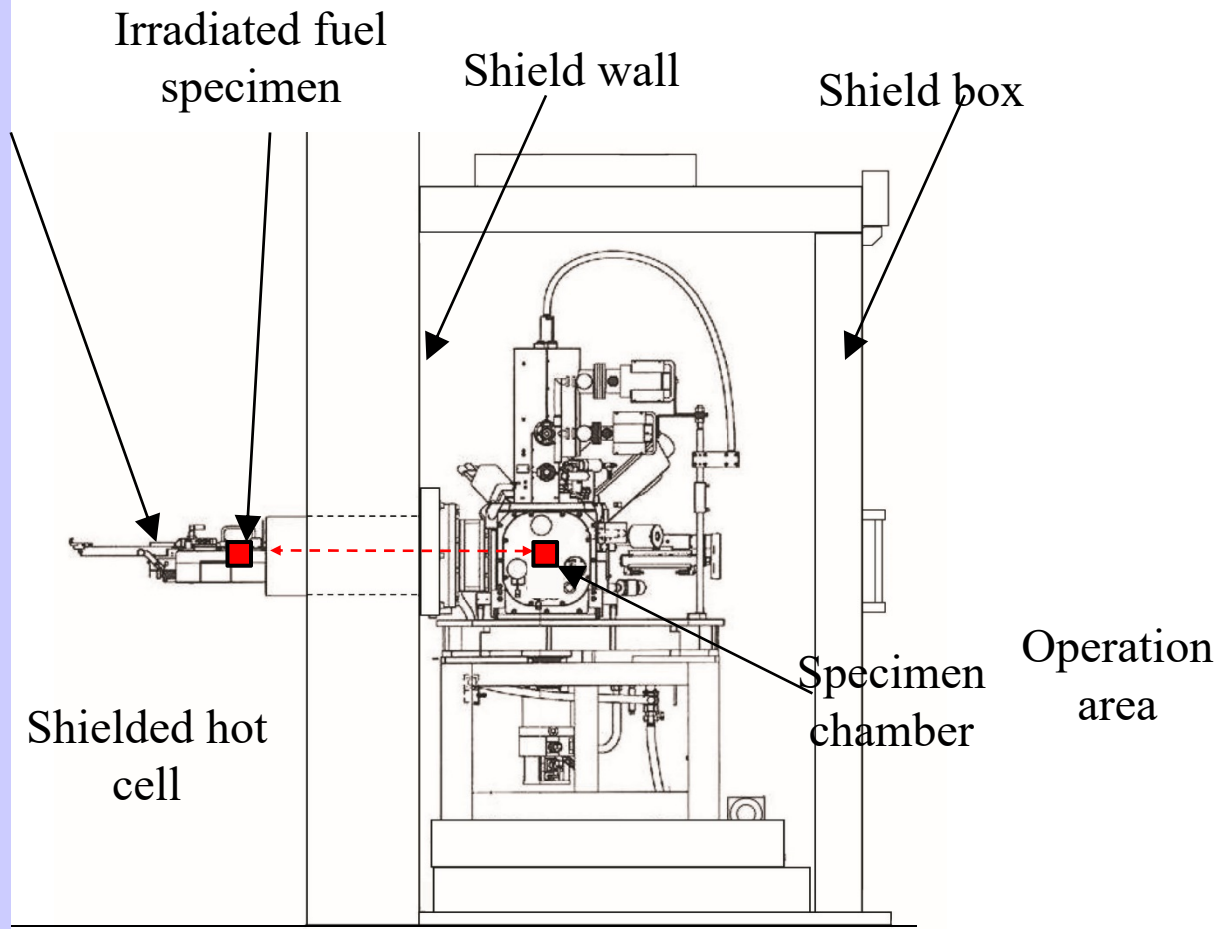
Photo of the specimen transfer unit in the shielded hot cell.

Outline of the FE-SEM installation and modification (5)

(Specimen transfer unit)

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- Electric-powered transfer unit of the radioactive specimen (maximum size 20mm diameter, 10mm height)
- Airtight boundary is achieved by O-ring seals.
- It is able to remove the irradiated fuel specimen manually if this unit have troubles.



Schematic diagram of the modified FE-SEM.

Outline of the FE-SEM installation and modification (6)

(Lead shield box)

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- The thickness of the lead shield box walls is about 120mm.
- Doors are attached for maintenance of FE-SEM.
- To prevent exposure operators, these doors are locked when the radiation dose inside the shield box is over $100\mu\text{Sv/h}$.



Photo of the modified FE-SEM and lead shield box.

Outline of the FE-SEM installation and modification (7)

(Separately control unit of the FE-SEM)

- The acceleration voltage, magnification, astigmatism, and specimen stage position can be changed from outside the shield box.
- A CCD camera is installed in the specimen chamber of the FE-SEM to monitor the operation of specimen exchange.



Photo of the modified FE-SEM and shielded box.

Result 1: Dosage rate during specimen transfers and examinations

■ In order to study the structural change and distribution change of MAs, the mixed oxide fuel doped with MAs (MA-MOX fuel) is irradiated in the fast reactor JOYO.

→ Cut, polished and coated with carbon in the shielded hot cell.

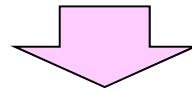
▪ Maximum specimen dosage rate of these specimens was 16mSv/h.

Specimen holder
(Mounted irradiated
fuel Specimen)

Manipulator in
Shielded hot cell



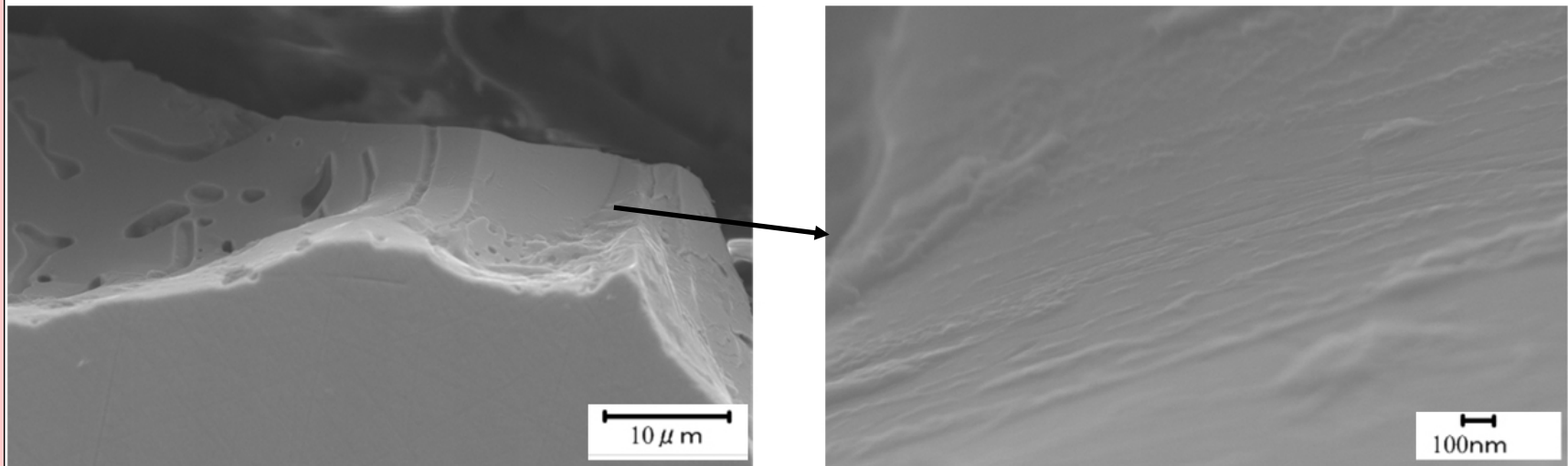
Photo of the specimen holder
in the shielded hot cell



During their transfers and examinations, the dose rate outside of the shield box was under $1\mu\text{Sv/h}$ (the dose limit of the operation area is $20\mu\text{Sv/h}$). The air-tightness of the shielded hot cell and the FE-SEM were maintained.

Results 2: the detail observations

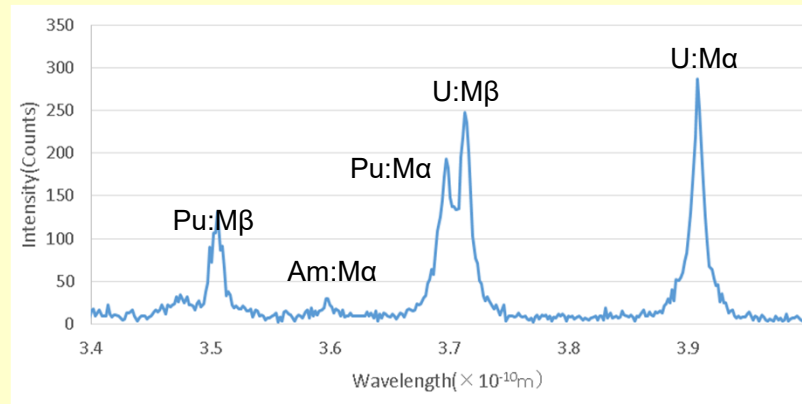
The images of the fuel specimen were taken at high magnification (2000X, 50000X) by the FE-SEM. It was possible to observe the face of grain boundary in detail (Dark area is crack area).



Secondary electron images of the MA-MOX fuel specimen surface
(Accelerating voltage, 15kV)

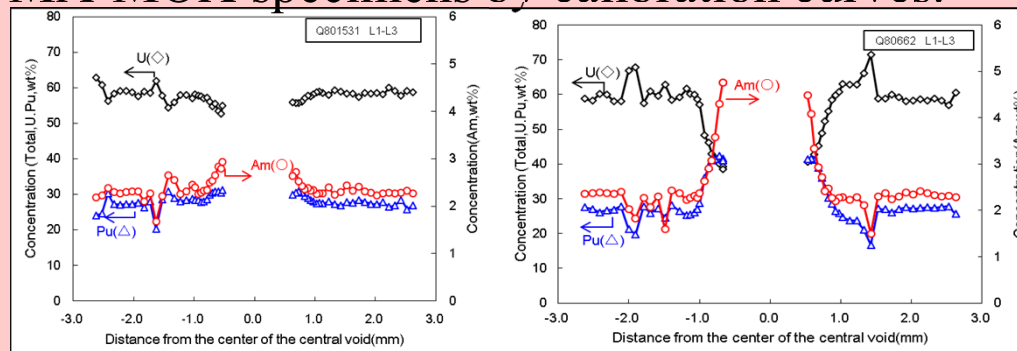
Result 3: the elemental analyses

The characteristic X-ray peaks of U, Pu and Am are successfully detected.



The element analysis result of the fuel specimen by WDX (beam current, about 30nA; accelerating voltage, 20kV)

The quantitative analyses of U, Pu and Am were obtained along the radial direction of the irradiated MA-MOX specimens by calibration curves.



The distribution changes of Pu and Am in high O/M specimens (right figure) are larger in the comparison with other specimens (left figure).

Conclusions

- **The newly modified FE-SEM installed in hot laboratory.**
- **The FE-SEM could carry out both observations of micro structures and elemental analyses of irradiated fuel specimen surfaces.**
- **It is able to grasp the changes of the microstructures and the quantitative changes of U, Pu and Am along the radial direction.**
- **The technique has the great advantage of being able to evaluate irradiated MA-MOX fuels in detail.**

- Thank you very much for your kind attention.