

Micro-Structure and Surface Analysis on Nuclear Fuel and Simulated Fuel

, , , , , ,

150

가 ,

EDS

Abstract

To study the irradiation behavior on nuclear material such as nuclear fuel and the structural material of reactor, Scanning Electron Microscope(SEM) with Energy Dispersive X-ray System(EDS) was installed inside the shielded glove box at PIE facility in KAERI and the license for using the SEM and shielded glove box was acquired. Therefore, the micro structure and surface analysis system for irradiated material was established. Before the examination on irradiated material, the surface micro-structures on unirradiated nuclear fuel and SIMFUEL were analyzed by using the shielded SEM and EDS. The optimum condition for operation was also established to minimize the radiation effect to the equipment and detectors.

1.

가 .

, , (grain)
(pore) , (grain boundary) bubble 가

bubble

Cs

, EDS

가
(SIMFUEL)

가

2.

가.

2가

PolyFast

(polishing)

(etching)

(coating)

PolyFast

EDS

(shielded glove box)

(detector)

가

(manipulator)

2nm

40

가 PHILIPS

XL-30

가

가

가

EDS 134eV 가 , 0.1 0.2 wt% EDAX Dx4i EDS 가

software

3.

1 ,

가

1 μm ,

2

1000

3 . intra inter

granula , 4

6 μm

5 6

10000 5 6

spot UO₂ EDS

7

8 . 가 25 30 keV ,

1/2 2/3 가 U-M U-L

x-ray가 peak U-M

x-ray , 3.35 keV ,

가 ,

1

88.46 % , 88.5 % ,

88.57 % . 3.2 % 가 ,

Table 1. Surface Quantitative Analysis of Fresh UO₂

Scan Method	Area			Window			Spot		
	1 _{st}	2 _{nd}	3 _{rd}	1 _{st}	2 _{nd}	3 _{rd}	1 _{st}	2 _{nd}	3 _{rd}
U(wt%)	88.60	88.11	88.67	88.50	88.20	88.84	88.54	88.23	88.95
O(wt%)	11.40	11.89	11.33	11.50	11.80	11.16	11.46	11.77	11.05

88.15 % , 0.35 % , 0.40 % , 0.48 %

4.

가
 ,
 ,
 EDS
 peak
 EDS , 2.4 keV Pd, Mo, Zr U, 2.8 keV Mo, Ru, Rh Pd
 x-ray EDS 135 eV
 peak EPMA
 10 eV 가가
 2 , EPMA EDS
 , EPMA 가
 PPM EDS 0.1
 0.2 w% ,
 0.0% EPMA

Table 2. Comparison Between Chemical Analysis, EPMA and EDS for SIMFUEL

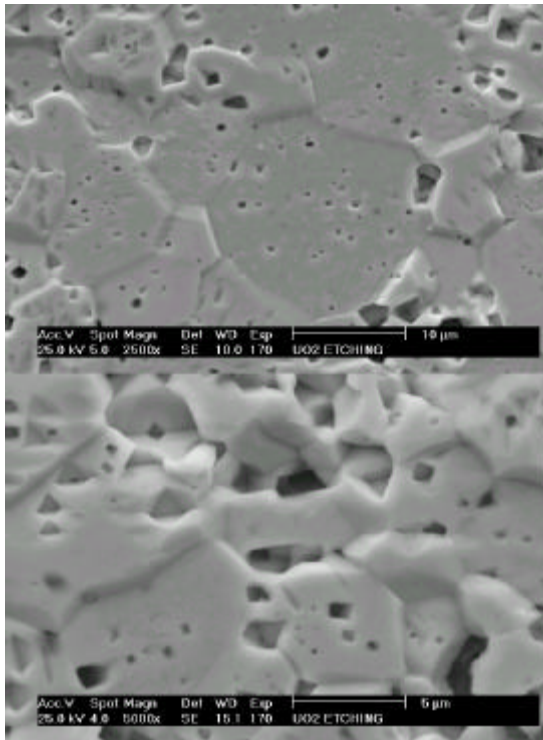
		Zr	Mo	Ru	Pd	Ba	La	Ce	Pr	Nd	Sm	Sr	Y	Rh	Te	O	U
(wt%)		0.422	0.392	0.269	0.187	0.218	0.143	0.278	0.131	0.476	0.101	0.084	0.052	0.049	0.058	11.515	86.625
	EPMA	0.397	0.449	0.239	-	0.203	0.155	0.233	-	0.48	-	0.076	0.05	0.073	-	-	-
	EDS	0.19	0.48	1.62	1.69	-	-	-	0.24	1.03	0.17	0.71	-	-	-	11.43	82.44

5.

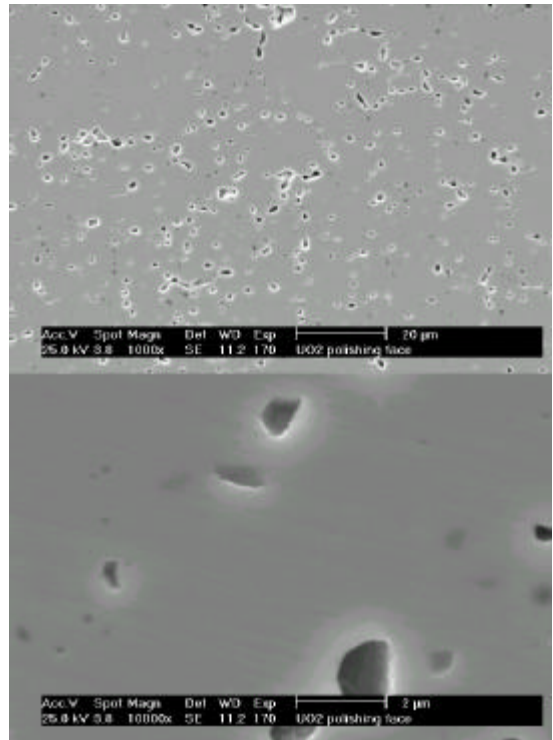
SEM EDX
 ,
 가 , 가
 , 가
 가
 EPMA

*

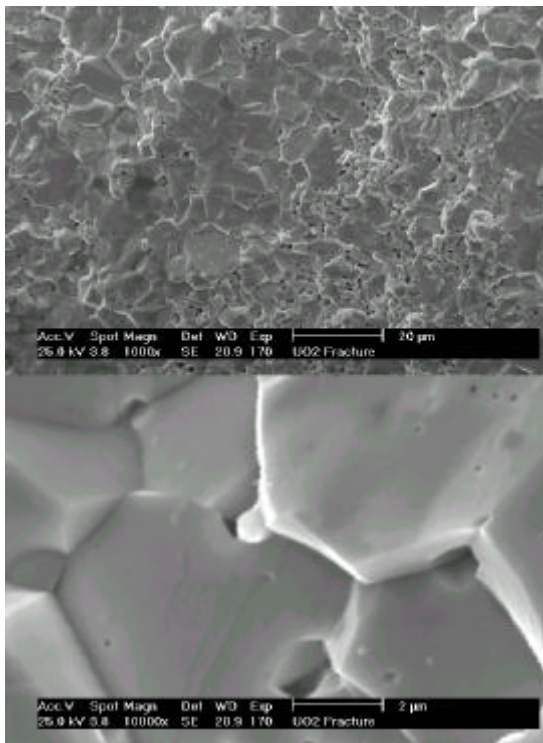
- [1] J. R. Gibson, D. N. Braski, "Scanning Electron Microscope Facility for Examination of Radioactive Materials", ORNL/TM-9451, 1985.
- [2] PNL, "Shielded Analytical Instruments for Characterization of Highly Radioactive Materials", PNL-5862, 1986.
- [3] BNL, "A Fully Shielded & Analytical Scanning Electron Microscope for the Examination of Radioactive Materials", EMAG-91, 1991
- [4] INEL, "Operation of a Scanning Electron Microscope in a Hot Cell", DE88-006775, 1987.
- [5] SRL, "Contained Scanning Electron Microscope Facility for Examining Radioactive Materials", DE86-012928, 1986.



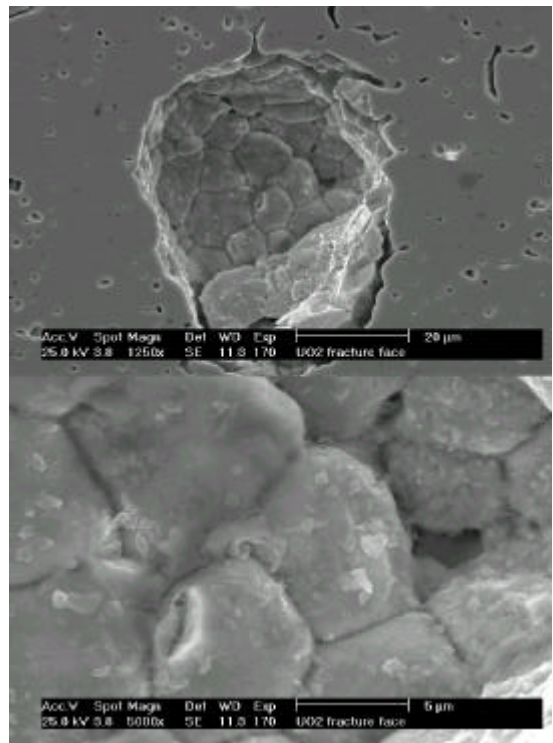
1. Etching Surface of Fresh UO₂.



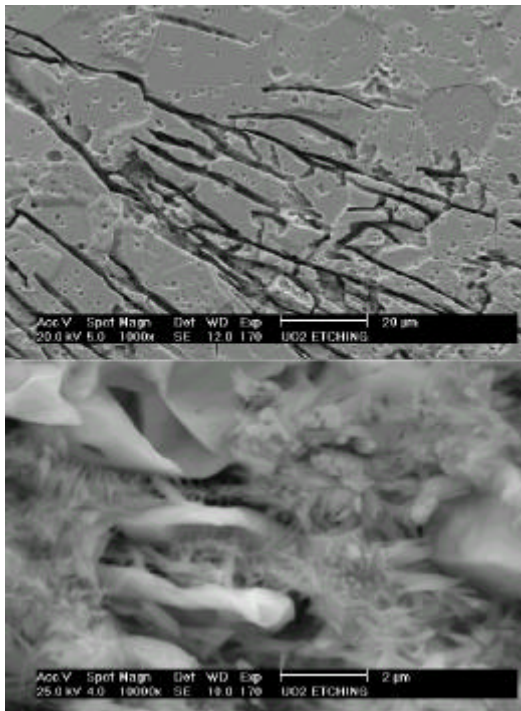
2. Polishing Surface of Fresh UO₂.



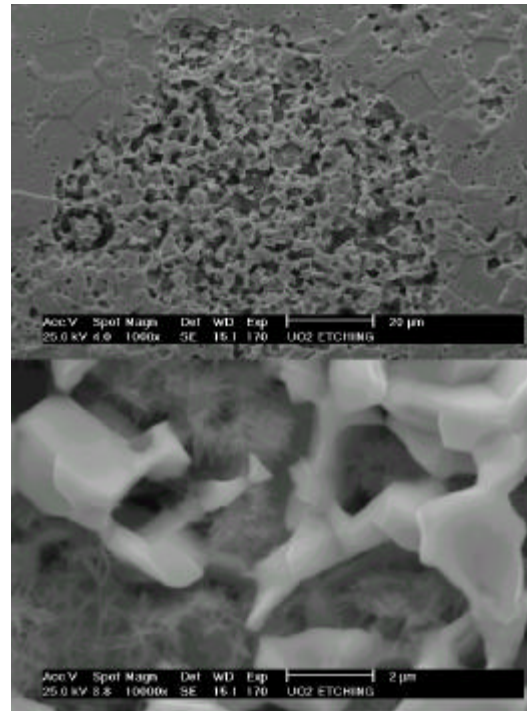
3. Fracture Surface of Fresh UO₂.



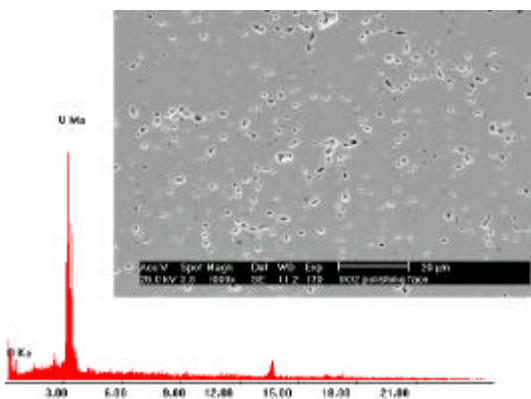
4. Grain Shape of Fracture Surface.



5. Coarse Shape without Forming Grain inside Micro-crack.



6. Coarse Shape without Forming Grain inside Concentrated Pore.

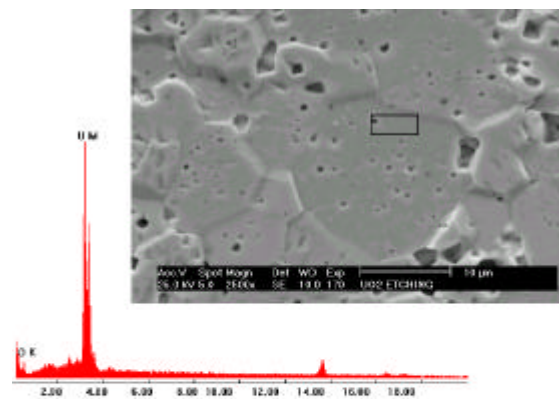


UO2 Polishing Surface, Carbon coating, Target : Total Area

Acquisition Time : 15:21:21 Date : 4-Aug-99
 EDAX ZAF Quantification (Standardless)
 Element Normalized
 Elem Wt% At% K-Ratio Z A F

 O K 11.89 66.75 0.0143 1.2922 0.0933 1.0000
 U M 88.11 33.25 0.8637 0.9225 1.0626 1.0000
 Total 100.00 100.00

7. Surface Qualitative and Quantitative Analysis under Polishing Condition.

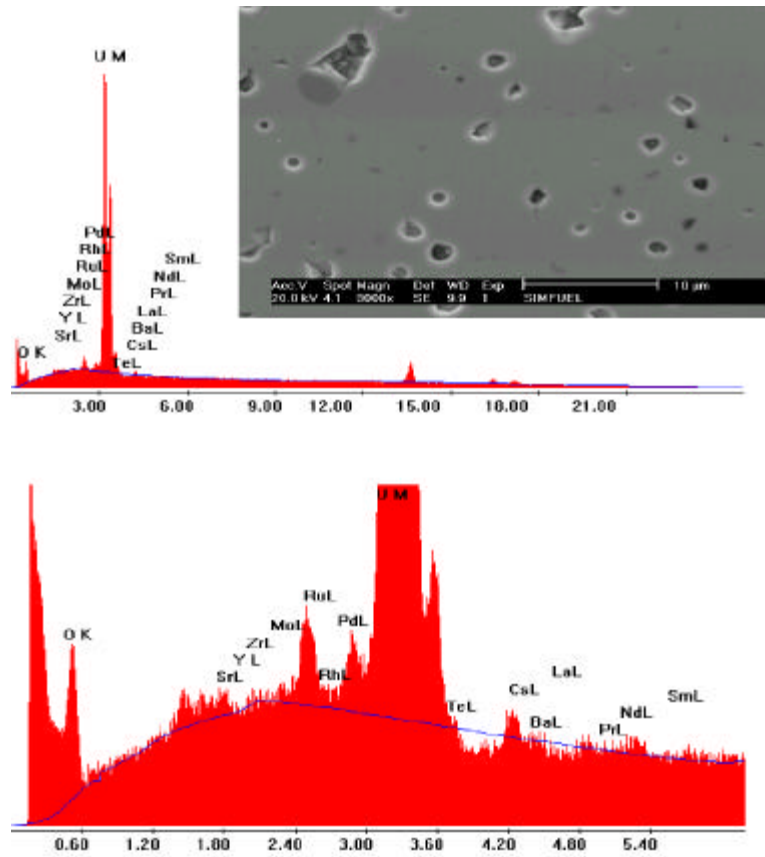


UO2 Etching Surface, Gold coating, Target : Window Area

Acquisition Time : 17:01:17 Date : 26-Aug-99
 EDAX ZAF Quantification (Standardless)
 Element Normalized
 Elem Wt% At% K-Ratio Z A F

 O K 11.50 65.92 0.0138 1.2947 0.0928 1.0000
 U M 88.50 34.08 0.8680 0.9247 1.0607 1.0000
 Total 100.00 100.00

8. Surface Qualitative and Quantitative Analysis under Etching Condition.



9. Surface Qualitative Analysis of SIMFUEL by EDS.

Acquisition Time : 20:41:40 Date : 4-Oct-99

Elem	Wt %	At %	K-Ratio	Z	A	F
OK	11.43	63.91	0.0139	1.2888	0.0944	1.0000
SrL	0.71	0.73	0.0035	1.0231	0.4761	1.0007
YL	0.00	0.00	0.0000	1.0341	0.5205	1.0008
ZrL	0.19	0.18	0.0011	1.0330	0.5612	1.0010
MoL	0.48	0.45	0.0033	1.0413	0.6511	1.0010
RuL	1.62	1.44	0.0127	1.0639	0.7355	1.0001
RhL	0.00	0.00	0.0000	1.0697	0.7753	1.0001
PdL	1.69	1.42	0.0145	1.0549	0.8136	1.0002
UM	82.44	30.98	0.7901	0.9179	1.0442	1.0000
TeL	0.00	0.00	0.0000	0.9706	0.5647	1.0004
CsL	0.00	0.00	0.0000	0.9813	0.6687	1.0004
BaL	0.00	0.00	0.0000	0.9666	0.6196	1.0001
LaL	0.00	0.00	0.0000	0.9728	0.6544	1.0001
PrL	0.24	0.15	0.0017	0.9936	0.7184	1.0000
NdL	1.03	0.64	0.0074	0.9879	0.7255	1.0000
SmL	0.17	0.10	0.0013	0.9810	0.7792	1.0000
Total	100.00	100.00				

10. Surface Quantitative Analysis of SIMFUEL by EDS.