Standard Electron Probe Microanalysis of Irradiated Fuel at PSI

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Contain of the presentation

- Presentation of our shielded Electron Micro-Probe (EPMA)
  - Short description of the machine
  - Main characteristics of the machine
- Description of the measurement procedures developed for the characterisation of irradiated fuel with our probe
  - X-Ray spectrum of a fuel specimen
  - Quantification difficulties
  - Typical measurement procedure

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The LWV Micro-Probe

CAMECA SXR-SX50 shielded instrument

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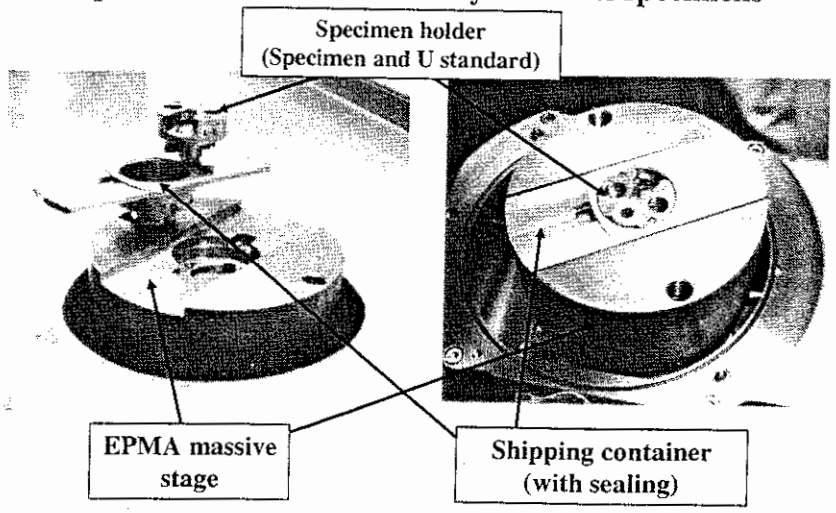
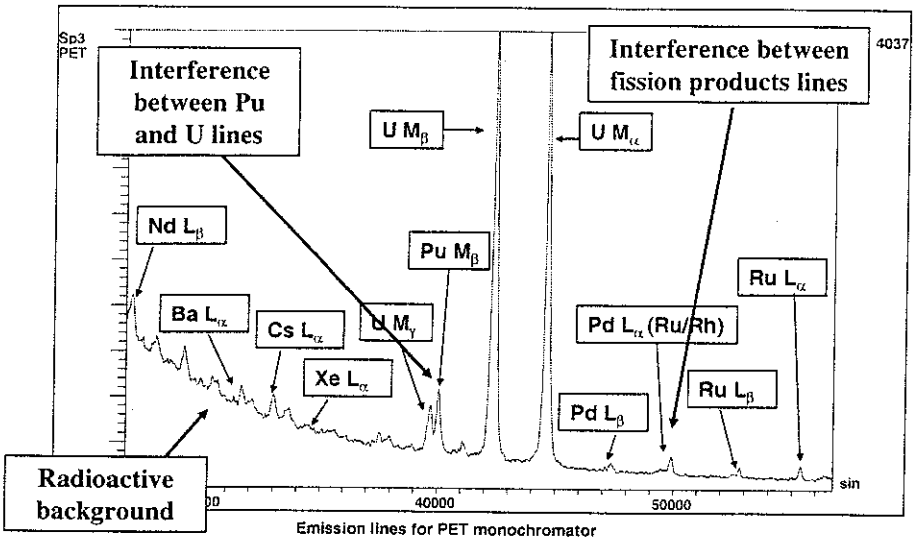
The LWV Micro-Probe

Specimen holder for the analysis of fuel specimens



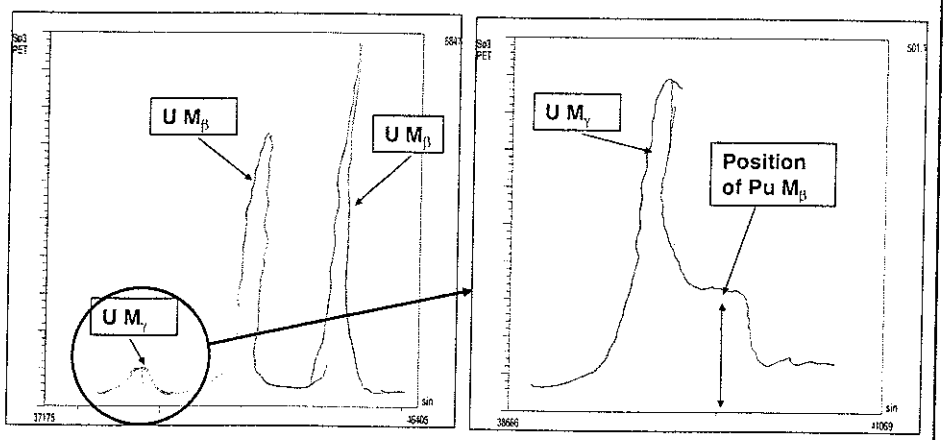
X-Ray spectrum of high burn-up MOX fuel



Emission lines for PET monochromator

X-Ray spectrum of UO_2 standard material

Influence of U on the measurement of Pu



(Instrument : CAMECA SXR-SX50; HV : 20keV, Current: 150 nA)

Emission lines for PET monochromator

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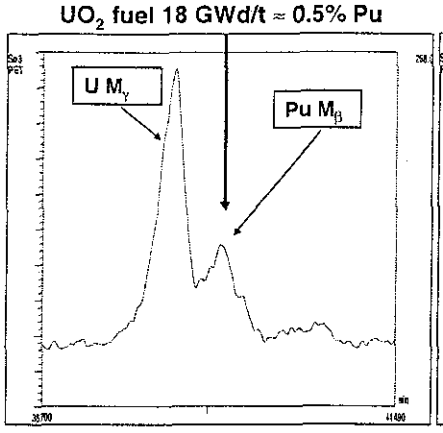
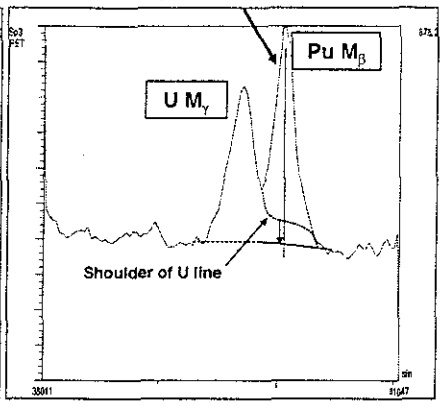
X-Ray spectrum of irradiated UO_2 and MOX fuel

Influence of U on the measurement of Pu

UO_2 fuel 18 GWd/t ≈ 0.5% Pu



MOX fuel, 59 GWd/t ≈ 5% Pu



(Instrument : CAMECA SXR-SX50; HV : 20keV, Current: 150 nA)

Emission lines for PET monochromator

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Spectrometer settings for fuel analysis

Element/ line	Crystal	Peak 10^3.sin Θ	Backgrounds (range in 10^3.sin Θ) and/or Slope factor [1]	Standard used [2]	Comments
U M_γ	PET	44660	- (1100 to 1200)/ + (1100 to 1200)	UO_2	(Interference from Pd)
Pu M_β	PET	40090	+ (400 to 500)/ 1.0 to 1.04	UO_2 or $(U,Pu)O_2$	Count rate of U M_β (x 1.05) or Pu M_β on $(U,Pu)O_2$; Interf. from U M_γ, Gd, (Pd)
Gd L_α	LIF	50880	- (700 to 800)/ + (700 to 800	(U,Gd)-oxide	Pur U-Gd fuel
O K_α	PC1	39840	- (1300 to 1800)/ + (1300 to 1800)	UO_2	-BG to be checked on highly active samples
Nd L_α	PET	27090	+(220 to 260)/ 1.02 to 1.06	NdF3	High radioactive BG (-BG) Interf. from Ba $L_{\beta 2,15}$ on +BG
Cs L_α	PET	33060	-(270 to 370)/ 0.94 to 1.00	CsI	Interference from U $L_{\beta 4}$ and on BG
Xe L_α	PET	34470	+(200 to 250)/1.05 or ±290 to ±400	Interpolation from CsI	Small Te and U interference

Spectrometer settings for fuel analysis

Element/ line	Crystal	Peak 10^3.sin Θ	Backgrounds (range in 10^3.sin Θ) and/or Slope factor [1]	Standard used [2]	Comments
Zr L_α	PET	69435	- (800 to 1000)/ + (800 to 1000)	Zr, Zircaloy, ZrO_2	Interference from Gd on BG
Ba L_α	PET	31725	asymmetric BG or slope	BaF_2	Small U interference on peak. Interference on +BG (U, Xe L_β) Interference on -BG smaller
Ru L_α	PET	55400	asymmetric BG or slope	Rh, (Ru)	Interference from Tc L_β on BG
Tc L_α	PET	58480	- (500 to 600)/ + (500 to 600)	Interpolation from Mo/Ru	Small interference from $CeL_\alpha 2$. Interf.: -BG (U, Mo), +BG (Mo)
Mo L_α	PET	61790	- (700 to 850)/ + (700 to 850)	Mo	Small interference from Zr L_γ
Pd L_β	PET	47400	- (1000 to 1100)/ + (1000 to 1100)	Pd	Interference from Ru/Rh and U on Pd L_α and its -BG (from U)

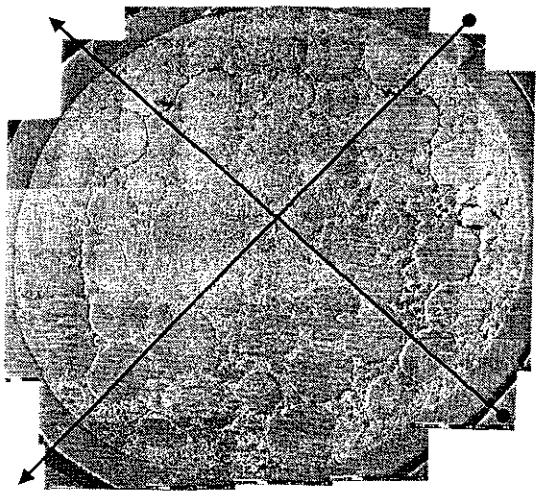
[1] Background (BG) selection beside the peak (-BG/+BG) through spectrum analyses (depending on sample composition and radioactive background).

[2] Spectrometer settings for standards normally the same as for the sample to be measured.

Fuel analysis with EPMA - Macrography

Cross section of a high burn-up sphere-pac MOX fuel (NOK-M308)

Mosaic of low magnification SE images



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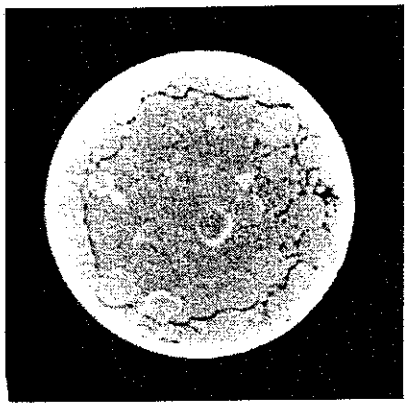
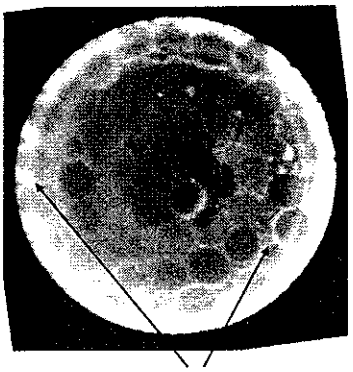
Fuel analysis with EPMA - Autoradiography

Cross section of a high burn-up sphere-pac MOX fuel
(NOK-M308)

α-autoradiography



β/γ-autoradiography



Fission produces

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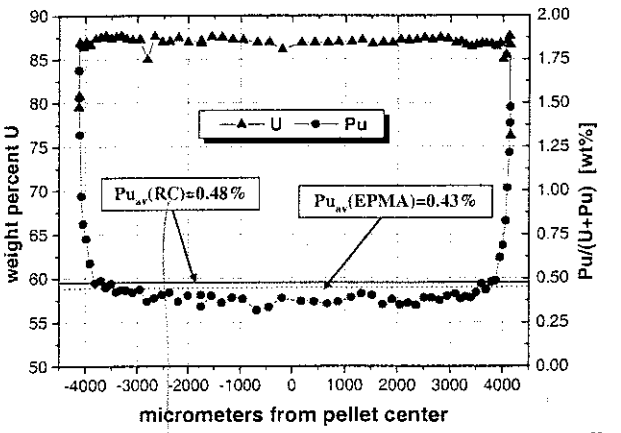
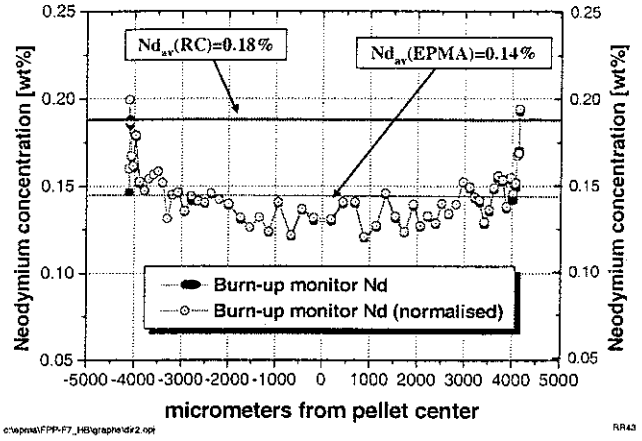
Fuel analysis with EPMA - U and Pu distributions in UO_2

UO_2 pellet fuel with 18GWd/t burn-up



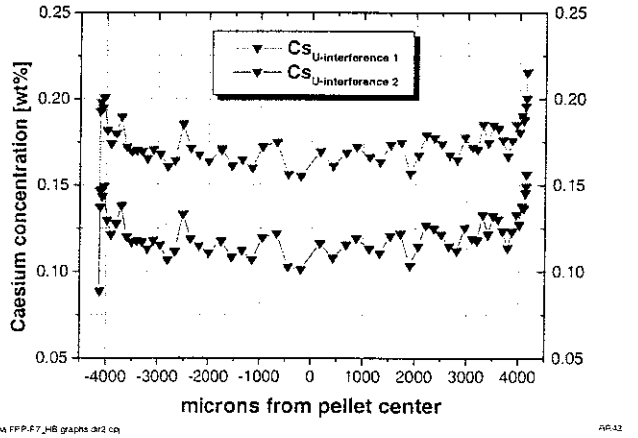
Fuel analysis with EPMA - Nd distribution in UO_2

UO_2 pellet fuel with 18GWd/t burn up



Fuel analysis with EPMA - Cs distribution in UO_2

UO_2 pellet fuel with 18GWd/t burn up



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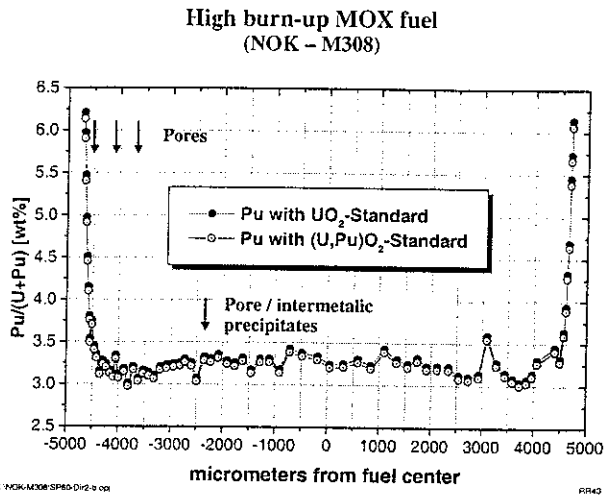
Fuel analysis with EPMA - Pu distribution in MOX



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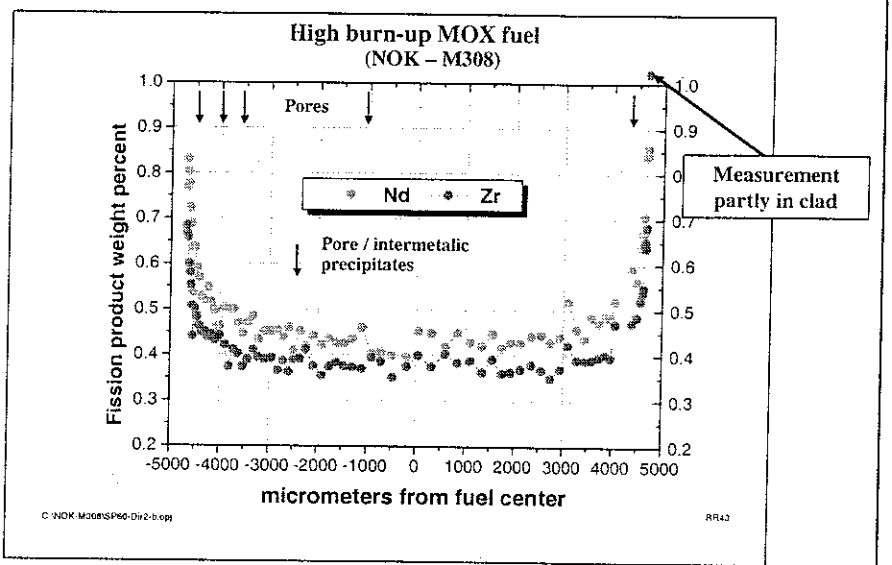
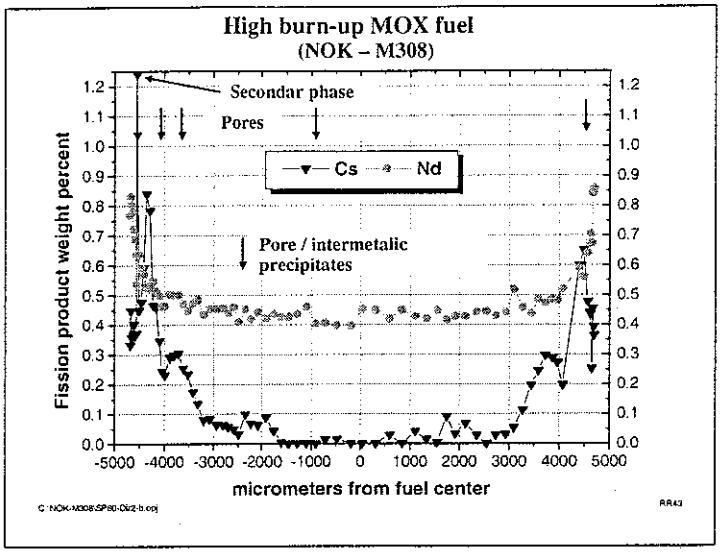
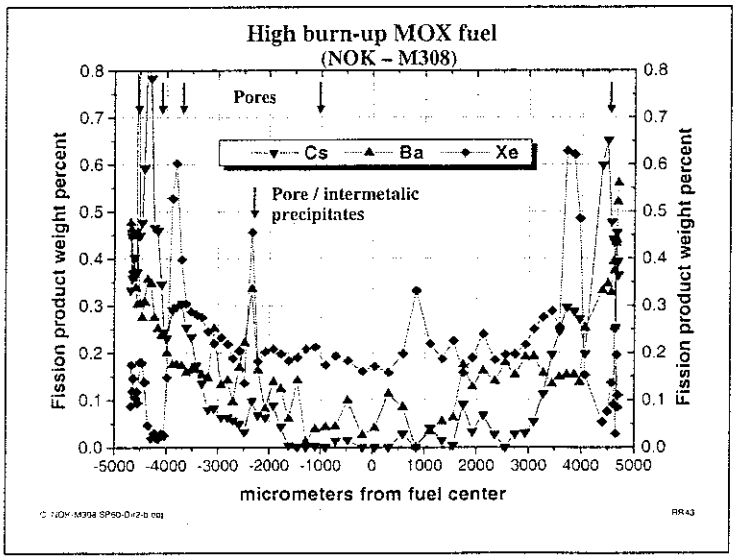
Fuel analysis with EPMA – Nd and Zr distributions in MOX



Fuel analysis with EPMA – Cs and Nd distributions in MOX



Fuel analysis with EPMA – Cs, Ba and Xe distributions in MOX



Conclusions

- EPMA is a very powerful technique for the characterisation and the analysis of high burn-up fuel specimen
- The measurement and analysis procedures must be carefully set up and checked in order to insure a good quantification of the measurements