



JOINT RESEARCH CENTRE
EUROPEAN COMMISSION



EUROPEAN WORKING GROUP

on

“HOT LABORATORIES AND REMOTE HANDLING”

Karlsruhe-Germany. October 13-15, 1999

**An instrumental method to investigate the diffusion of traces
elements in zirconium oxide layers and zircaloy cladding materials**

M. Betti, L. Aldave de las Heras, L.O. Actis-Dato, T. Gouder, E.H. Toscano



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Institute for
Transuranium Elements
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An instrumental method to investigate the diffusion of traces elements in zirconium oxide layers and zircaloy cladding materials

- *Introduction to the subject*
 - *Experimental Descriptions*
 - *Some significant applications and results*
 - *Conclusions*
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Zircaloy Cladding Corrosion

ZIRCALLOY CLADDING



PWR Reactor Conditions
(300°C, 150 Bar)
LiOH, H₃BO₃ coolant additives

EXTERNAL CORROSION

- (ZrO₂ LAYER > 100 μm)
- < MECHANICAL PROPERTIES
- < HEAT TRANSFER TO COOLING SYSTEM



Analytical Techniques

GDMS analysis

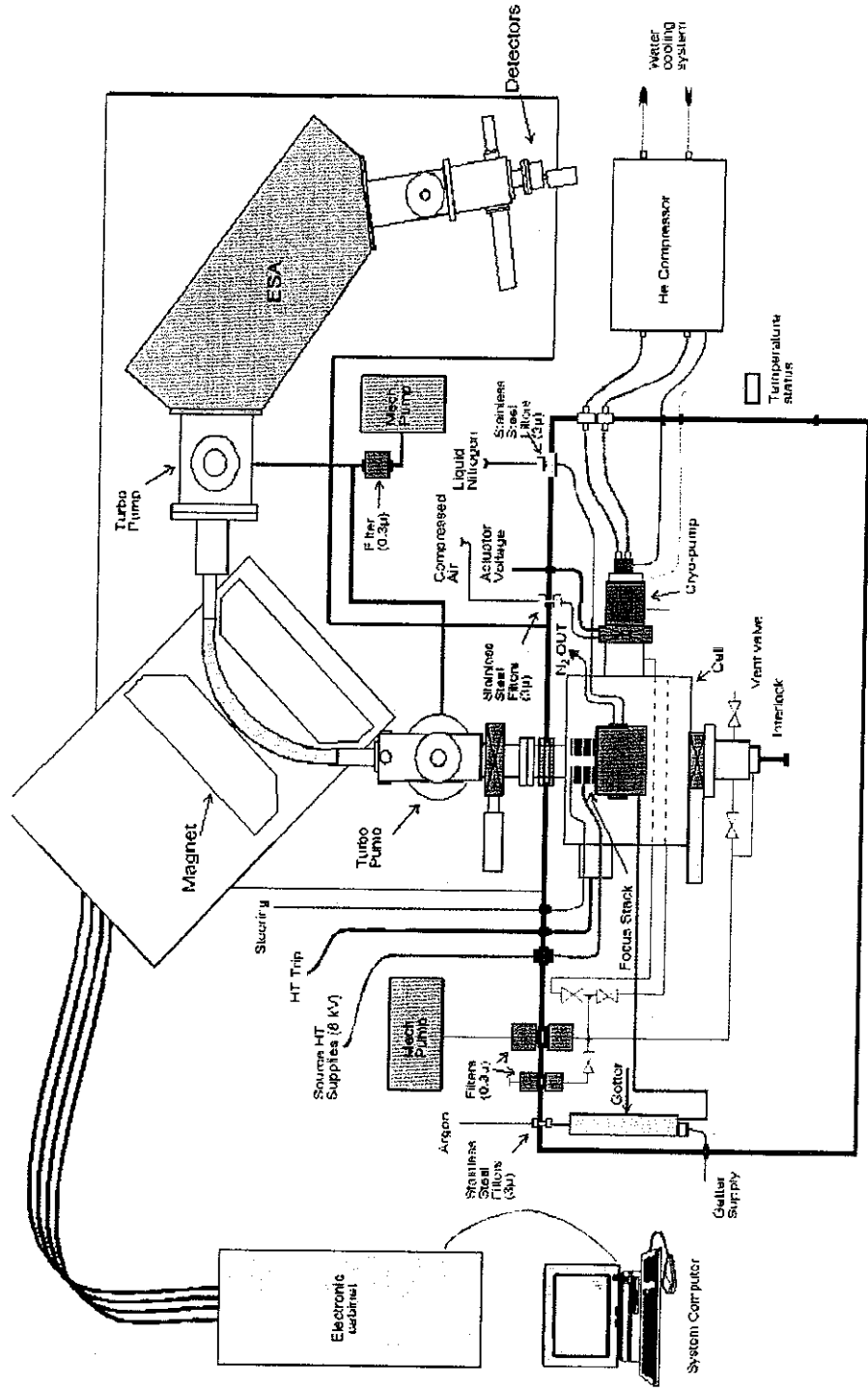
- Element concentration (Zr internal standard, RSF's)
- Depth profiles (major elements, traces)
- Diffusion of impurities (oxide layer, metal)

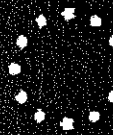
Profilometer DekTak 8000

- Crater Shape: Depth resolution
 - Crater Depth: Sputter rate, metal-oxide interface
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Glow Discharge Mass Spectrometer installed in glove box

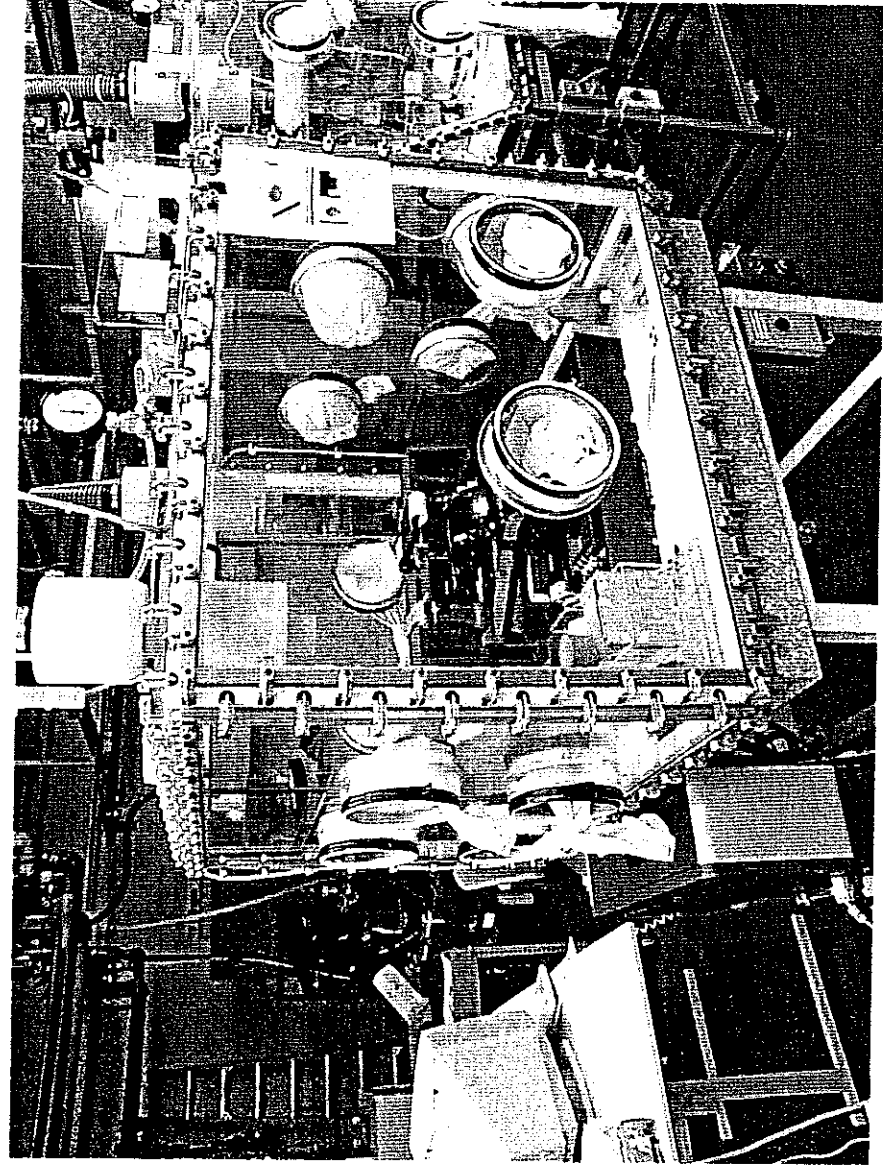




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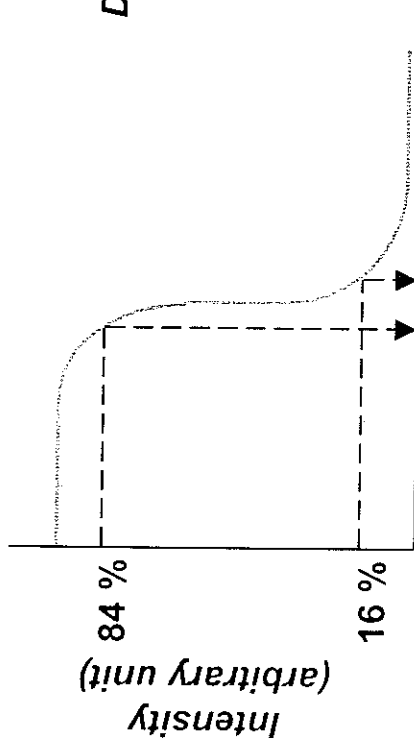
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Profilometer Dektak 8000 installed in glove box



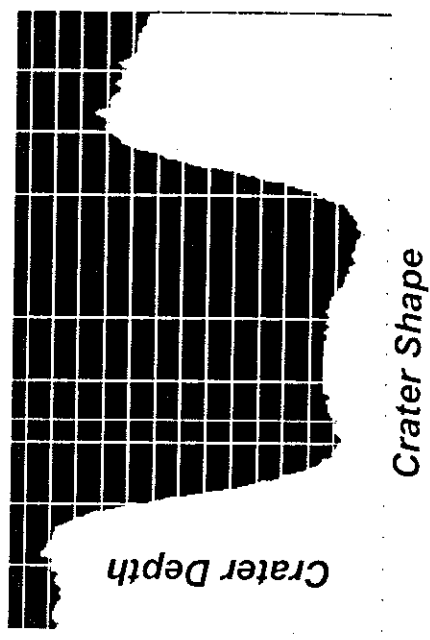
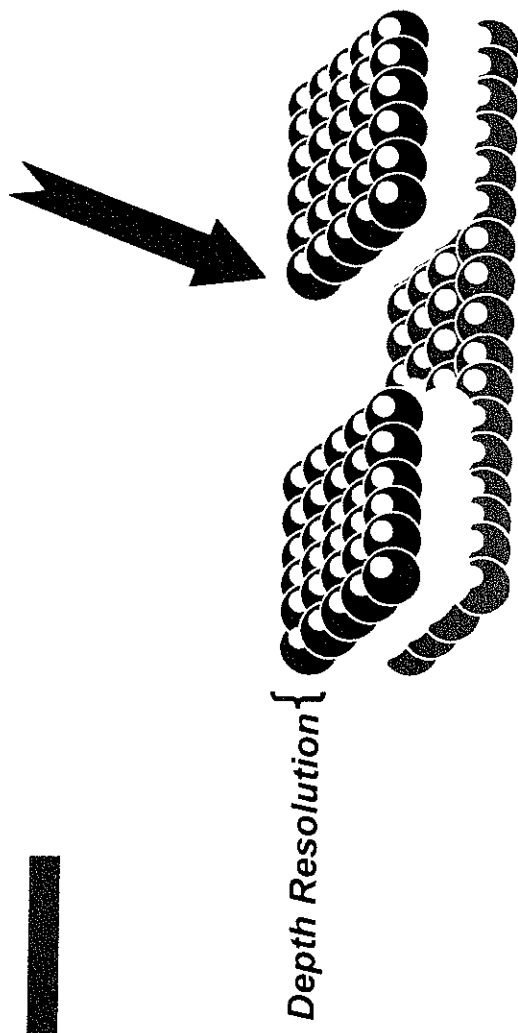
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Profile Parameters



Interface's Width Δt
Sputter time (s)
Depth Z (μm) ΔZ

Determination of the interface's width



(flat bottom, perpendicular crater walls)



Analysed Samples

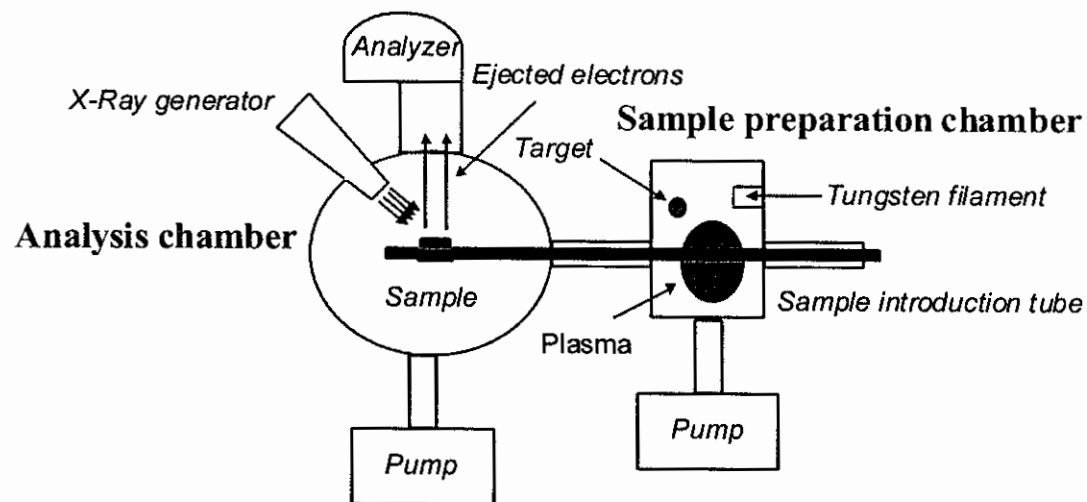
- Zr alloy (Zr alloy platelets)
- ZrO₂ (Zirconia platelets)
- ZrO₂-Zr alloy AUTOCLAVE SAMPLES
- Lithium Doped Zirconia (REACTIVE SPUTTER DEPOSITION)
- ZrO₂-Zr alloy CLADDING IRRADIATED SAMPLES



Reactive Sputter Deposition coupled with XPS

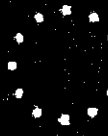
Production of ZrO_2 layers

- ZrO_2 Layer on Si
- ZrO_2 Layer doped with Li on Si



X-Ray Photoelectron Spectroscopy
(XPS)

Reactive Sputter Deposition



CONCLUSIONS

- *GDMs allow to follow impurities towards ZrO_2 layers*
 - *GDMs are adapted to in-depth analysis up to 100 μm*
 - *GDMs give isotopic information about irradiated samples*
 - *Analysis with the profilometer gives important additional information's*
 - *Investigations are performed using GDMs to understand the diffusion properties of coolant additives in irradiated samples*
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