Analyses microstructurales et mécaniques des matériaux irradiés, perspectives offertes en diffraction de poudre sur la ligne chaude MARS

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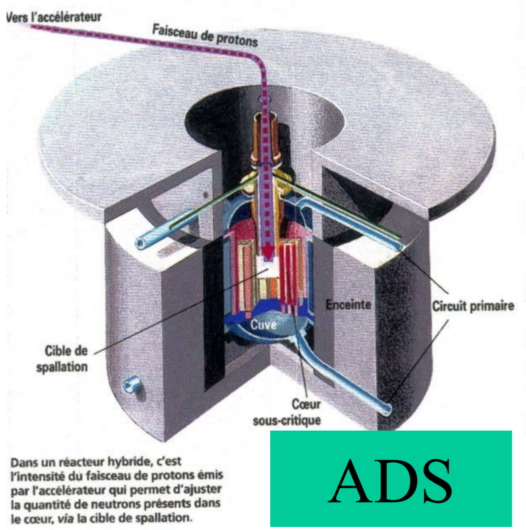
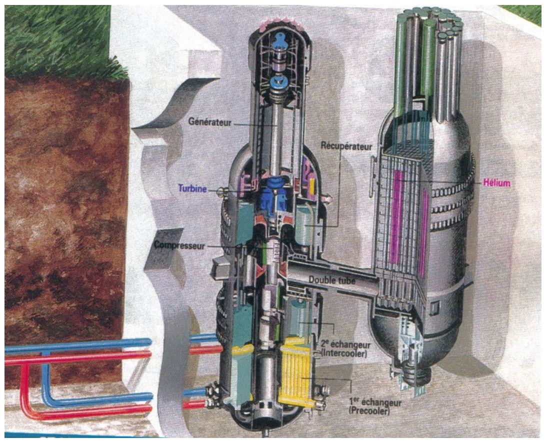
DEN scientific topics concerned by MARS beam line for irradiated materials on SOLEIL facility

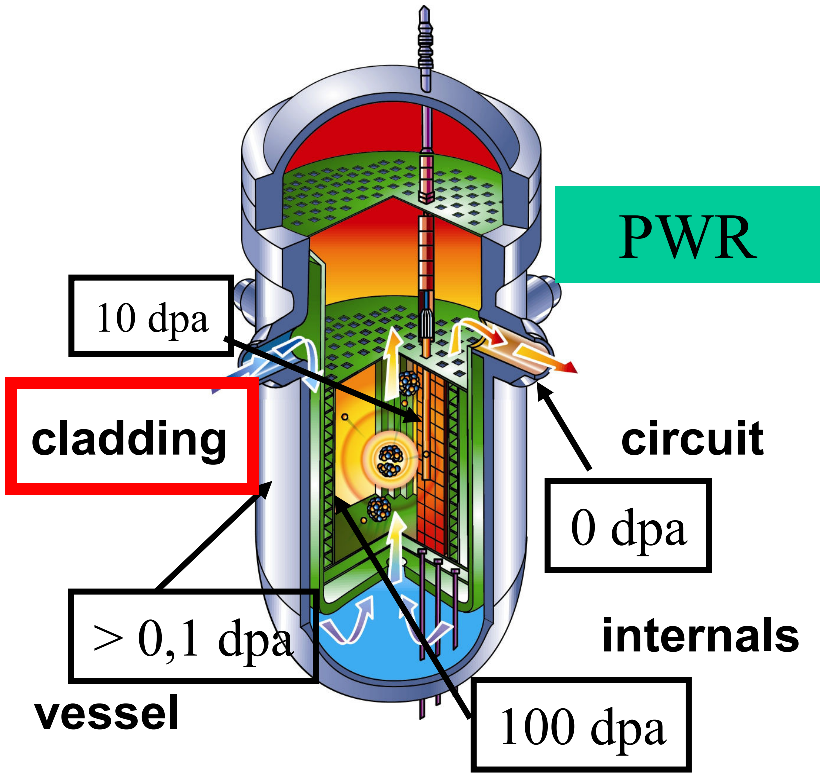
MARS beam line for the analysis of the Microstructure of irradiated alloys

Stress and strain fields determination for irradiated samples (micromechanical approach)

Complementary approach with: TEM, SANS, SEM







Applying MARS facility to structure materials studies

- Structure materials characterization, stress and strain field determination
  - Evolution of the microstructure under irradiation
  - Determination of the loops density and loops size due to irradiation
  - Analysis of the oxide layers formed during irradiation, stress state
  - Analysis of the evolution of ceramic materials for advanced reactors

- Few words about the beamline

(for details see poster B. Sitaud)

The MARS beamline : a new multi-technique beamline for radioactive matter

Diffraction capabilities

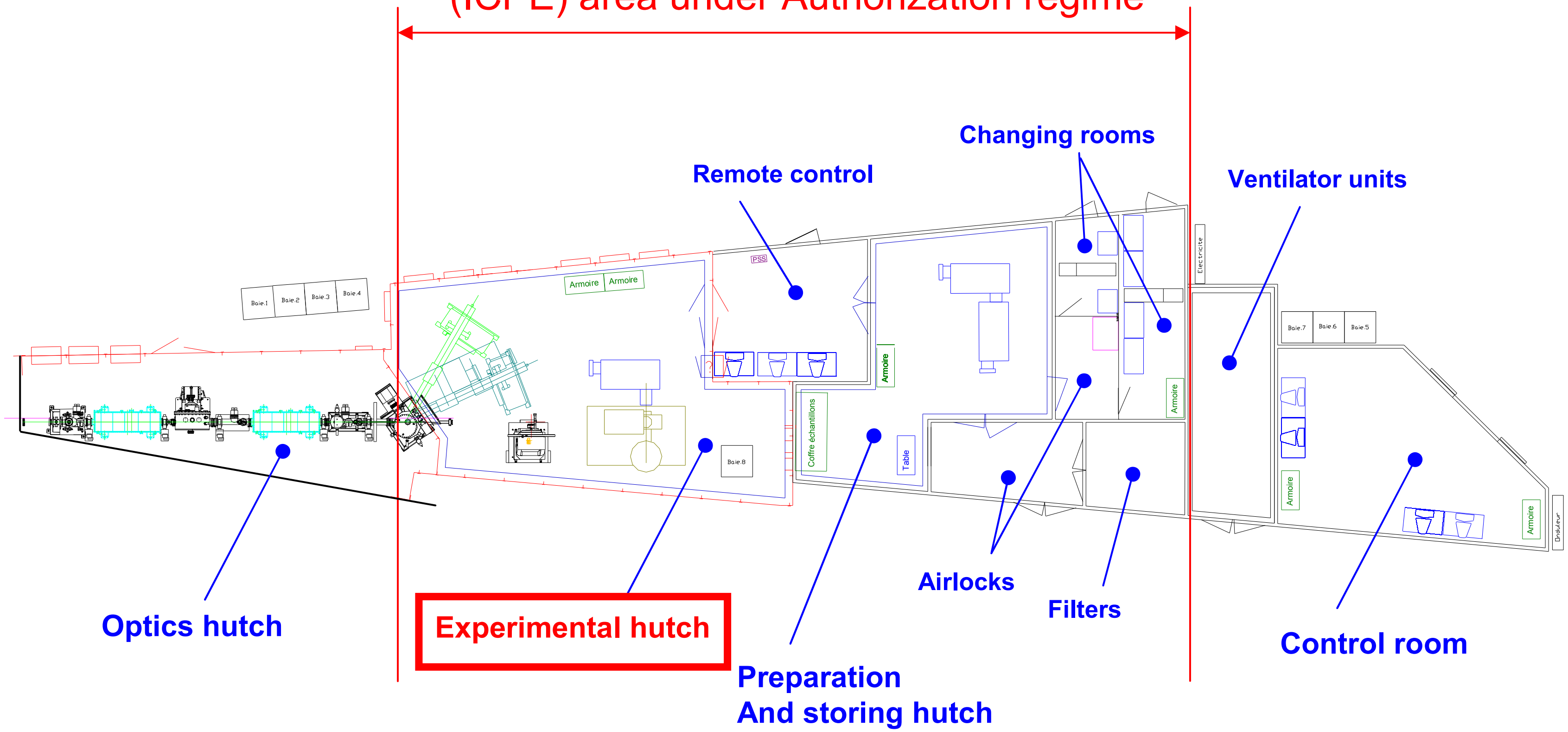
Main characteristics of the MARS beamline

Technical data:

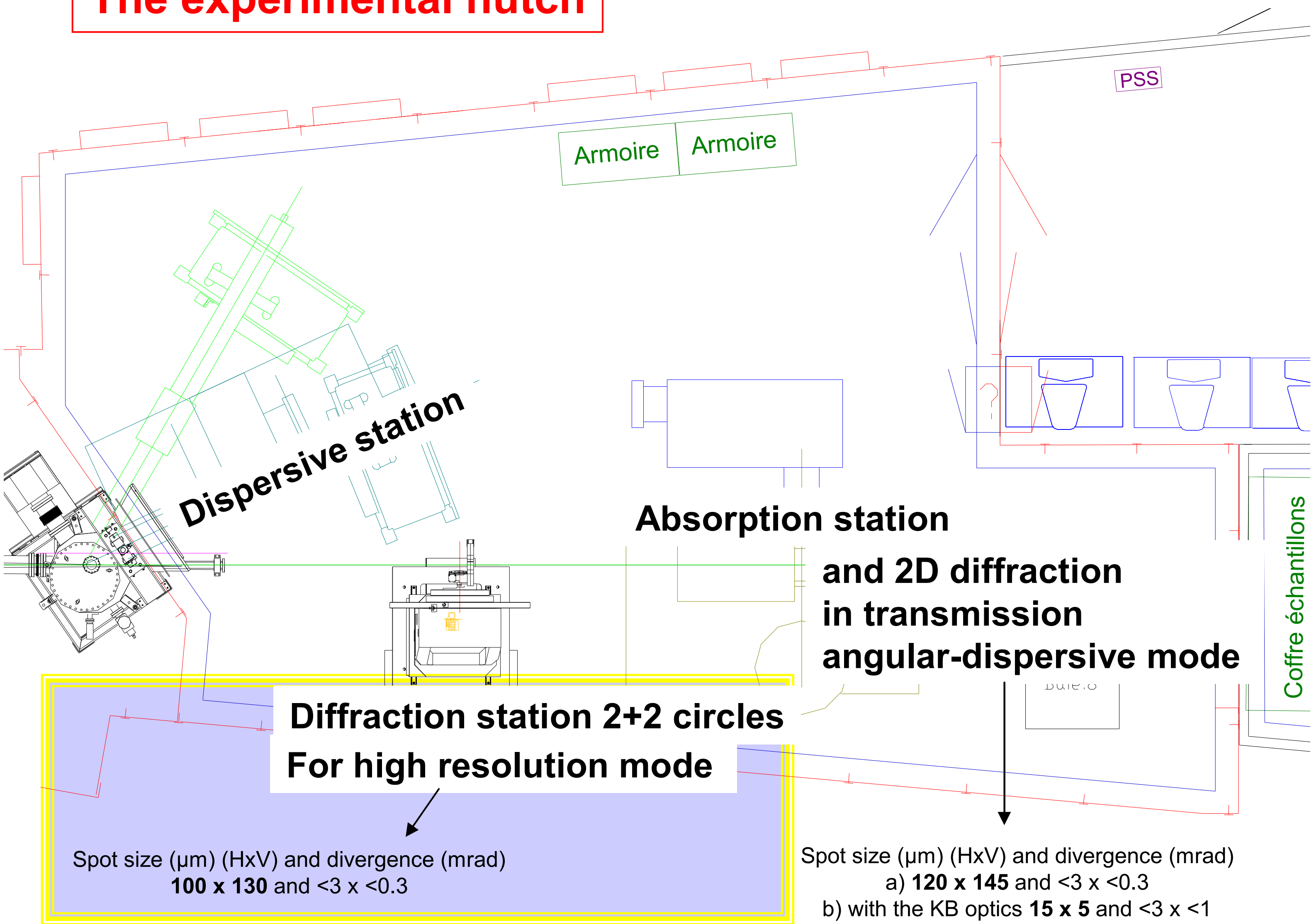
Energy range	Between 3.5 to 36 keV (focused beam)
Energy Resolution	$dE/E < 3.\ 10^{-4}$
Source	Bending Magnet , horizontal acceptance 3 mrad
Optics	Innovative combination of two long mirrors and two monochromators (DCM and SCM)
Sample Environment	Glove boxes Cryostats Electrochemical cell furnaces High pressure cells (Diamond anvil-type) Special shielded containments Multi barrier confinements
Beam size at sample	From 150x150 μm^2 to 5x5 μm^2 depending on the optical configuration (FWHM)
Flux on sample	About 4.10^{+12} Phot/s @ 10 keV (beam current 500 mA)

Layout of the Mars beamline (exit port C03-1)

"Installation Classée pour la Protection de l'Environnement" (ICPE) area under Authorization regime



The experimental hutch



Spot size (µm) (HxV) and divergence (mrad)
100 x 130 and <3 x <0.3

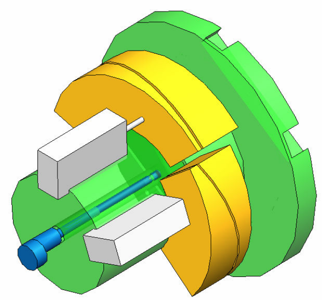
Spot size (µm) (HxV) and divergence (mrad)
a) **120 x 145** and <3 x <0.3
b) with the KB optics **15 x 5** and <3 x <1

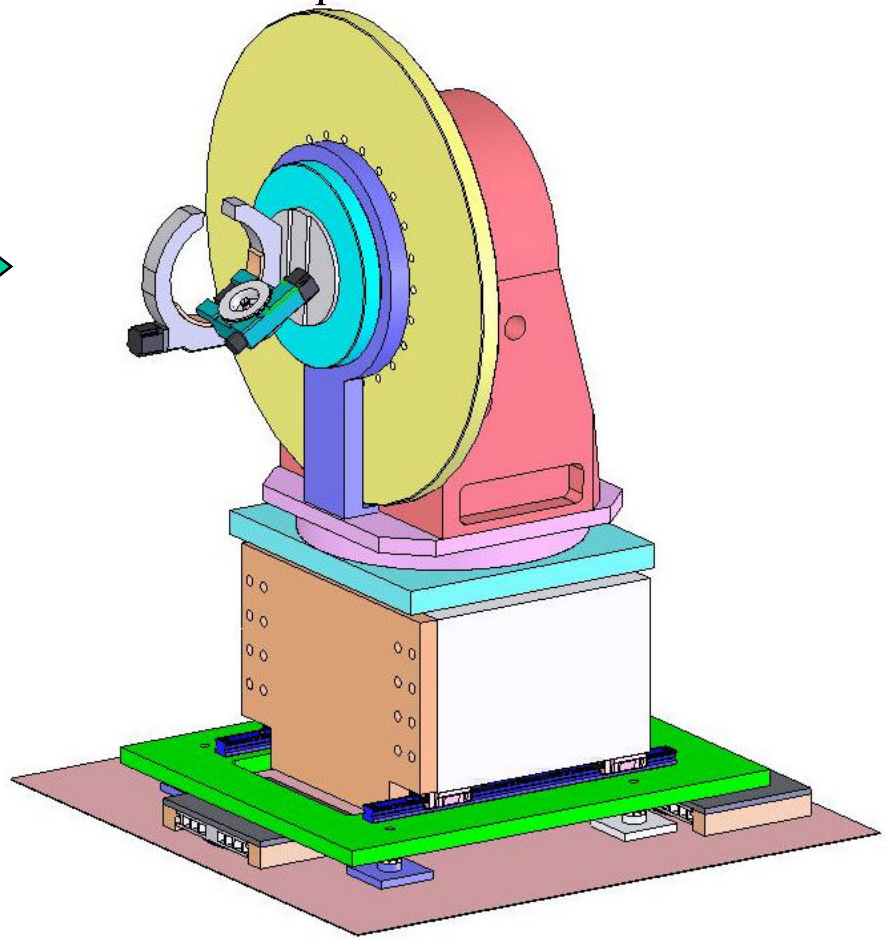
The High resolution diffraction station (2+2 circles)

Robust 2-circle diffractometer (max weight 75 kg) for shielded sample holders

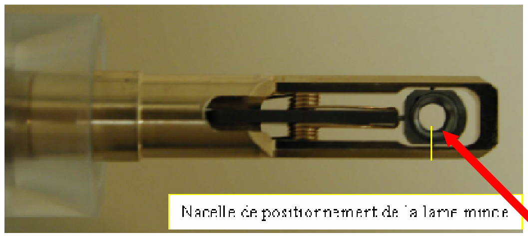
Shielding thickness (~8 cm) calculated for a dose rate ~1.6 10^7 Bq for stainless steel irradiated

Shielding sample holders





•Design based on the use of a TEM specimen holder
•Sample: thin foil before thinning



Φ=3mm
thickness ~200 µm

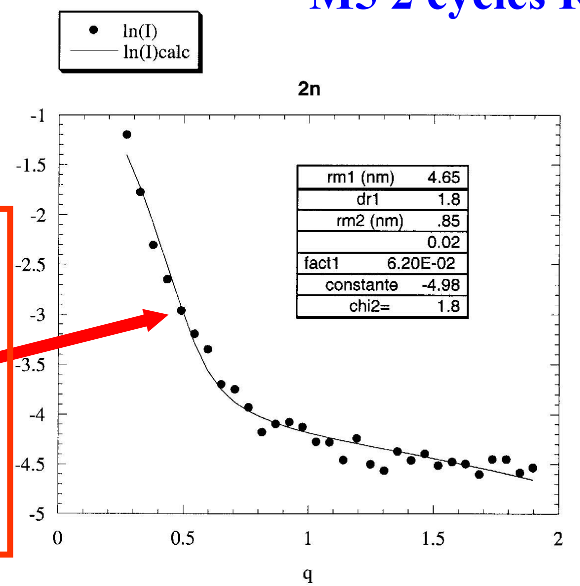
Evolution of the microstructure under irradiation

x-ray analyses of the different phases , determination of the crystallographic structure. EXAFS analysis in the case of the solid solution

Example : Evolution of Zr alloys (M5) under irradiation

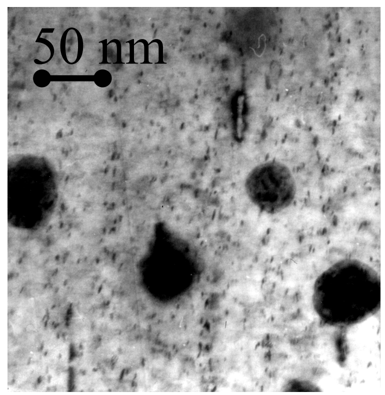
SANS and MET : **βNb radiation enhanced precipitation** ⟹ XRD analyses of the precipitation (Nb effect on solid solution, EXAFS)

Exploitation : Ln(I) = f(q)



M5 2 cycles REP (aiguilles β-Nb)

2 cycles



l = 6,6 nm
d = $8{,}4 \times 10^{21}\ m^{-3}$

At small angles : after irradiation, new particles supposed to be βNb with a density in the range of $1 \times 10^{22}\ m^{-3}$ and size ~ 10 nm (same range as TEM)

LLB, CEA-CNRS, M-H Mathon

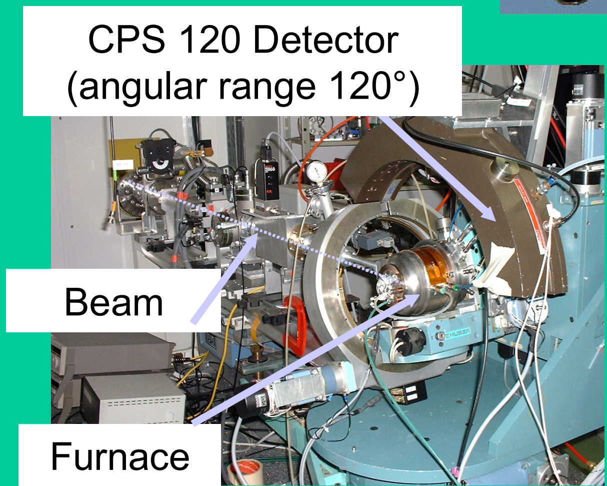
Analysis of the oxide layers formed during irradiation

Phases identification and stress state determination using x-ray diffraction, oxide scales formed on the proton beam window, analyses at different depth using grazing incidence, and/or large range of wave length.

Example 1 : In service corrosion of Zr alloys cladding tube for PWR

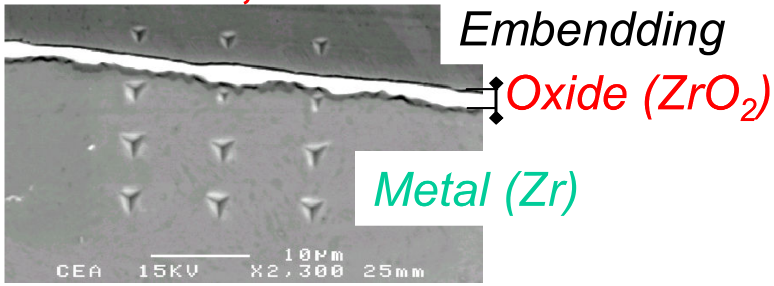
Cladding tube

$\Phi_{ext.}$ = 9,5 mm
ép. = 0,6 mm

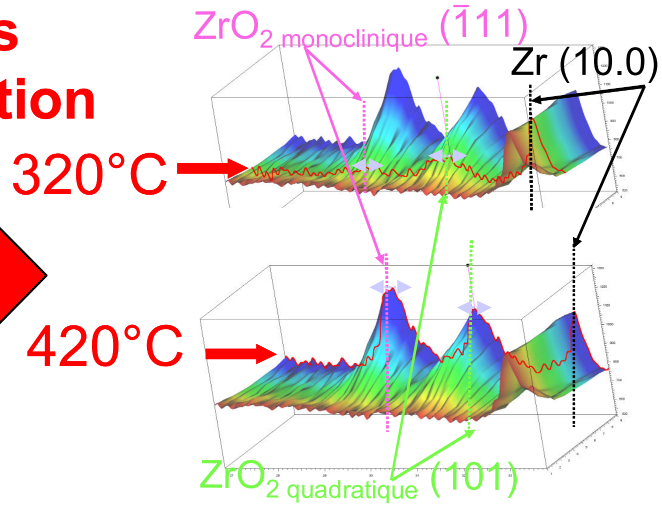


Device : H10 beam line (x-ray diffraction)

After oxidation, cross section



Phases identification



LURE, H10 M. Gailhanou, D. Thiaudière, P. Goudeau LMP

Example 2 : Gradients of residual stresses in Zr alloys tubes after high temperature oxidation and quenching (LOCA)

CCLRC

After a critical oxidation level (i.e. for long oxidation times), the tube can failed upon quenching.

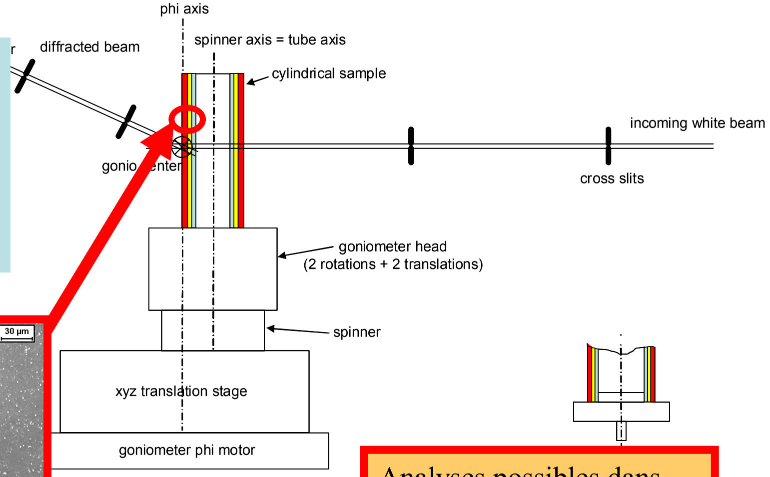
This phenomenon is certainly promoted by internal stresses due to the different thermal expansion coefficient of the three main layers and to the phase transition and oxidation stages.

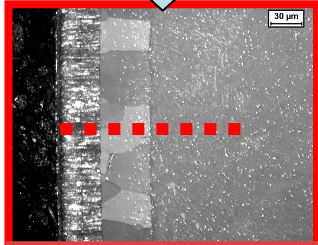
Have a good estimate of the **stress state** especially in the α(O) layer supposed to be the more brittle one

SRS, M. Golsham, LPMTM O. Castelnau,

Expérience de faisabilité en faisceau blanc sur la ligne 16.3 (M Golsham)

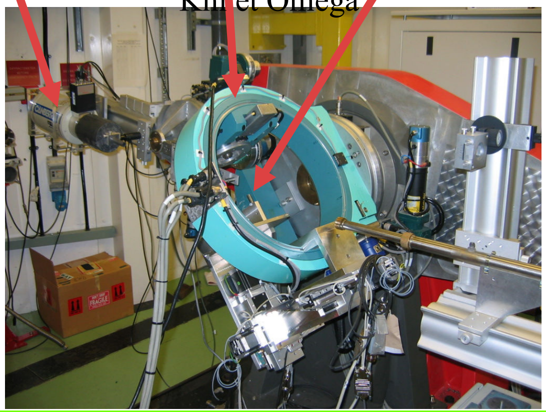
Objectif : déterminer les contraintes internes dans les 3 couches : ZrO_2, Zr-O, Zr(ex-β)
Valider par équilibre des contraintes





Test sur Zy-4, ECR 6%

Analyses possibles dans les trois couches (localement à proximité des interfaces).



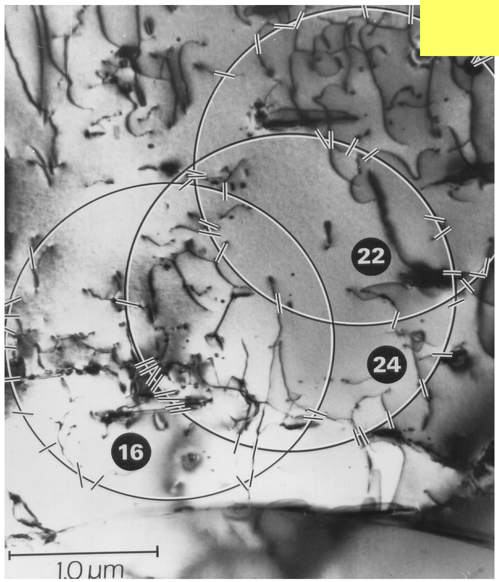
Determination of the loops density and loops size due to irradiation

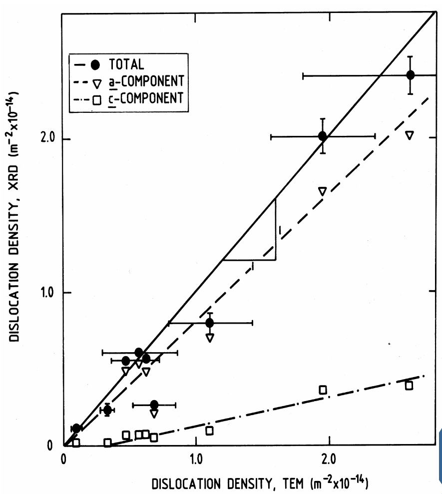
Evolution of the loops density for different diffracting planes using x-ray diffraction, broadening of x-ray diffraction peaks. Anisotropy of irradiation effects : strengthening, growth…, interactions between irradiation defects and deformation slip systems, in situ annealing and deformation

Example 1 : CANDU pressure tube deformation under irradiation

Method to Measure Dislocation Density → Growth / Irradiation creep

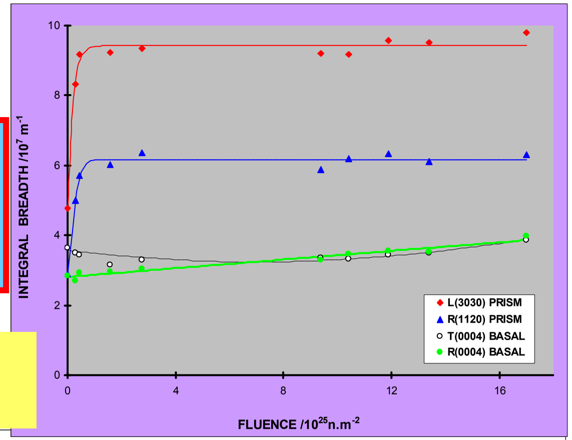
TEM

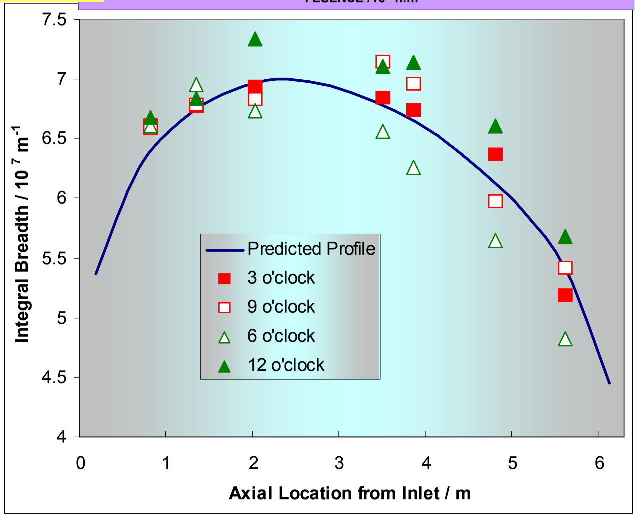




AECL, M. Griffith

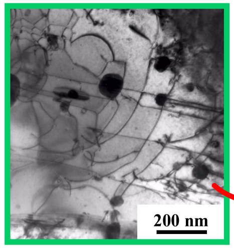
XRD



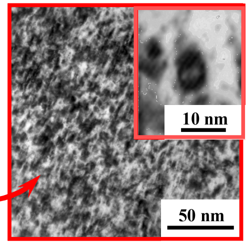


Example 2 : Internal stresses analyses for irradiated Zr alloys

Channeling for irradiated Zr alloys => strong heterogenity of the deformation (similar behaviour for steels)

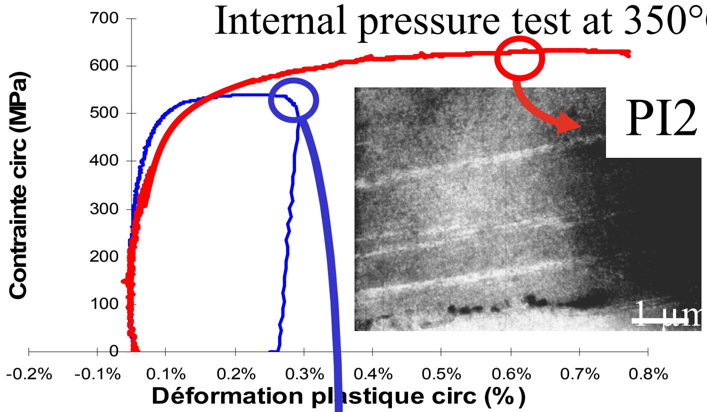


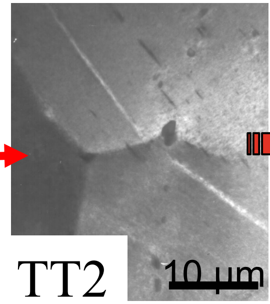
Non irradié



Irradié

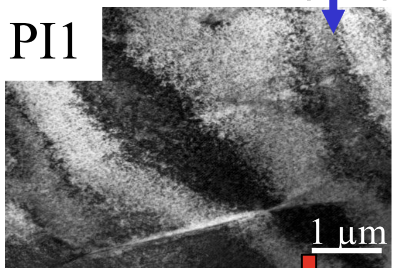
Internal pressure test at 350°C





B channels propagation

Channeling = local softening
Σ increases but channels for Ep=0.2%



XRD analyses

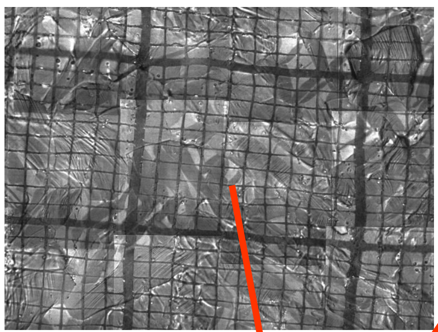
Plastic strain localization ⇨ **internal stress**
⇨ local softening balanced by internal stress

CEA/SRMA F. Onimus Thesis

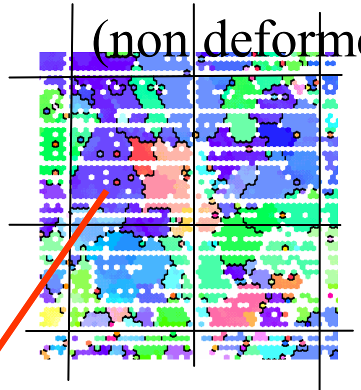
Example 3 : Single grain analysis of the plastic behavior of a polycrystalline Zr alloy

Single grain Microdiffraction plastic deformation

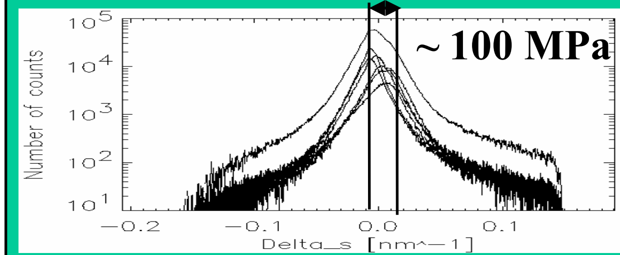
Grain size 30 µm ~ spatial resolution



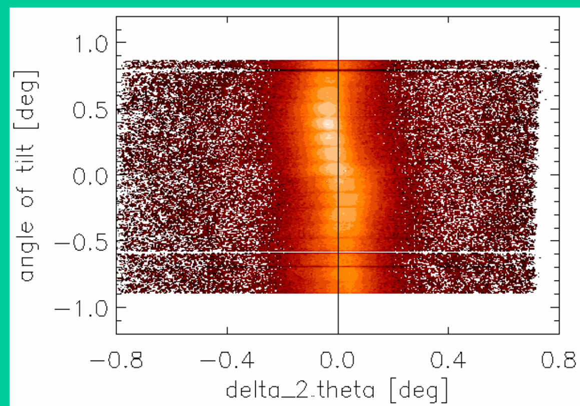
Microstructure



EBSD map



Plan (-2114)



High level of intragranular stress fluctuation

Orientation determined => microdiffraction
glide system activity – strain heterogenities:
deformation bands, damages…

ESRF,M. Drakopoulos, LPMTM, O. Castelnau, T. Ungar

Analysis of the structural evolutions under irradiation of ceramic materials for advanced reactors

Example : advanced materials for Gen. IV

Few data on irradiation behaviour

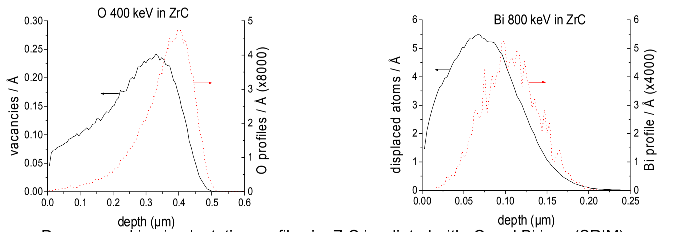
=>simulation of the neutron damage with irradiation by slow heavy ions (main damage due to displacement cascades)

Method:

Selection of the ions (implantations at IN2P3 Lyon):

Bi 800 keV : close cascades (recovering)

O 400 keV : separated cascades

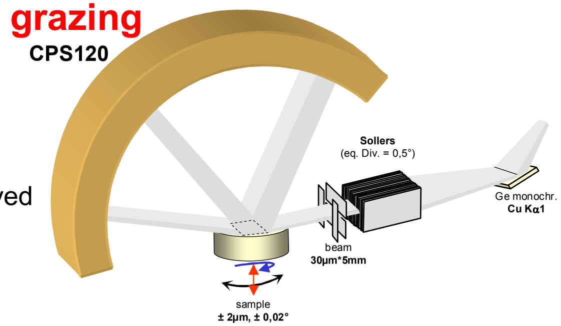


Damage and ion implantation profiles in ZrC irradiated with O and Bi ions (SRIM)

Analysis with grazing X-rays

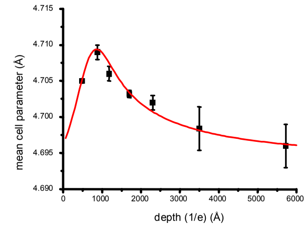
(damage thickness 1000-3000 Å)

facility on an improved CPS120 basis

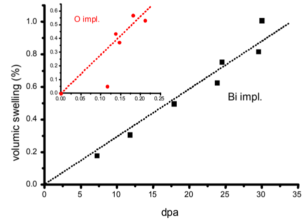


Preliminary results: ZrC and TiC implanted at room temperature

- no phase change (surstructures)
- cell parameter variation in the damaged volume
- linear swelling with fluence, compatible with results obtained in nuclear reactors at low temperature
- important disorder , mainly on carbon sub-network



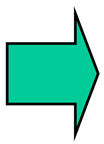
paramètre cristallin moyen de ZrC implanté Bi 800 keV, $5.10^{15}/cm^2$



gonflement de ZrC en fonction de l'endommagement

Further studies:

- **influence of the temperature**:
- annealing after irradiation (furnace, in situ)
- implantations at high temperature (600-1000°C)
- **Neutron irradiation**



characterization of the damage:

- TEM (extended defects),
- XRD(surstructures),
- EXAFS (point defects identification)

ECP/CNRS, G. Baldinozzi, CEA/SRMA D. Siméone, D. Gosset

Etude de combustibles irradiés DEN/CAD/DEC :

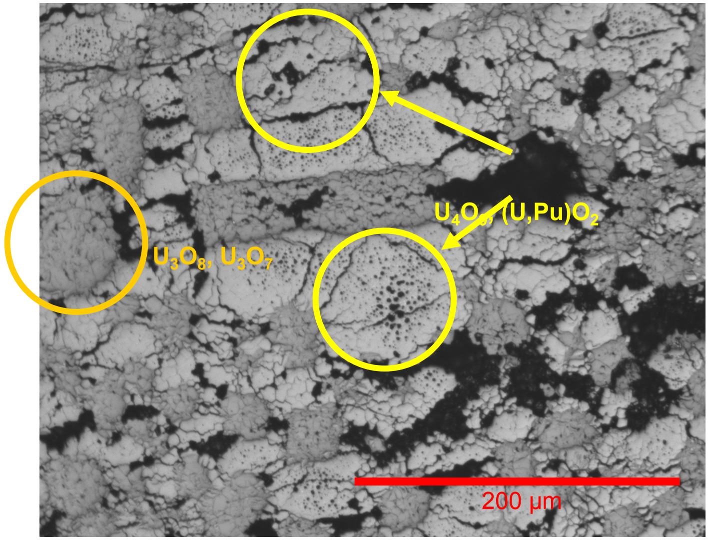
- Combustibles et cibles de transmutation (UOx, MOX, ECRIX …)
- Irradiation (REP, Phénix, réacteurs expérimentaux)

Etude structurale, μstructurale et mécanique de combustible irradié :

- XRD, XRF, XAS
- Analyses de contraintes (micro-macro)

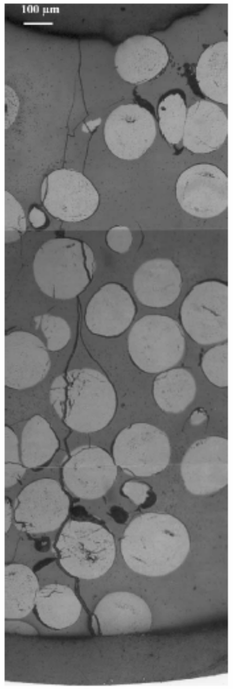
Apport majeur des technologies μfaisceau

Spécificité des combustibles hétérogènes :

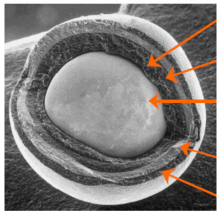


MOX

cible
de transmutation



Particule
HTR



Combustible
UMo-Al