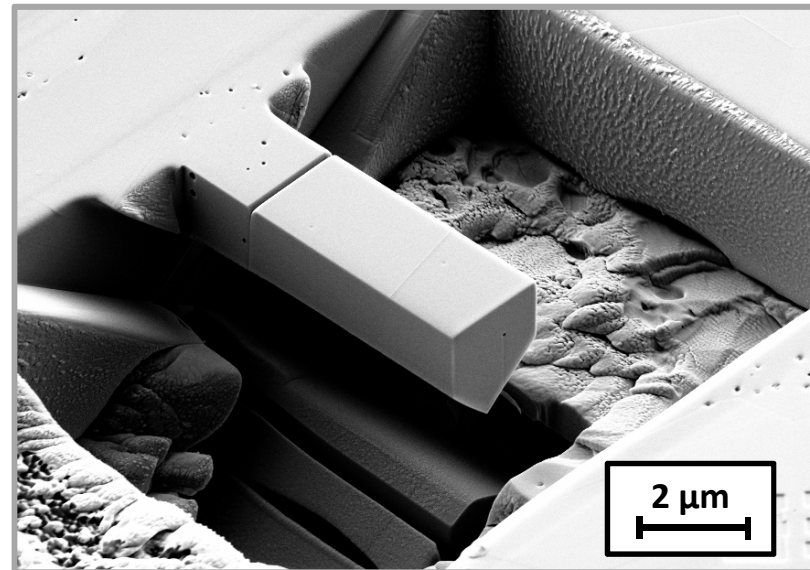


EUROMAT 2017

18-22/09/2017

*Mechanical properties at micro-scale applied
to nuclear fuel model material*



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I. Context

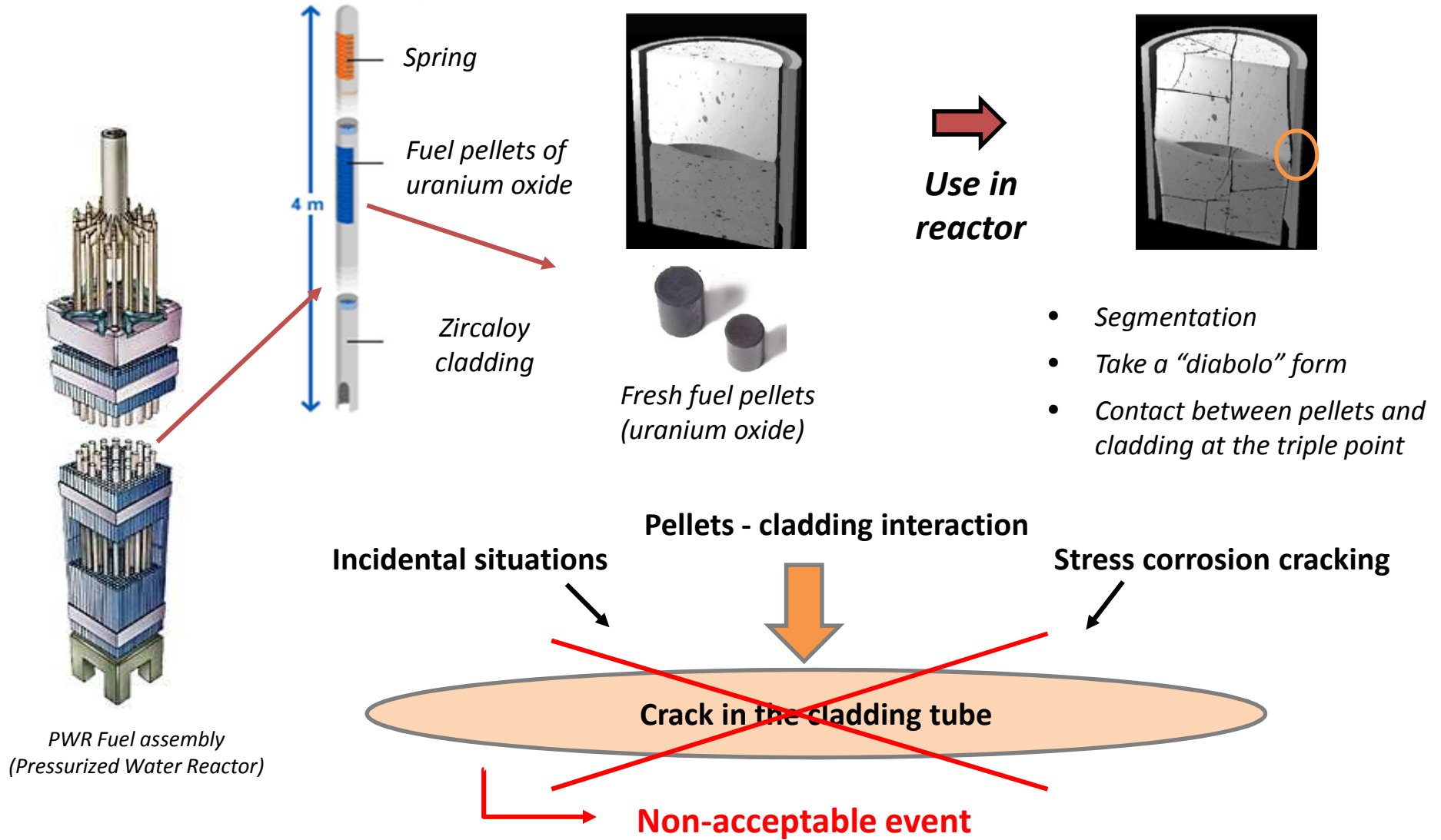
- 1. *The nuclear fuel and its modeling***
- 2. *Mechanical properties at the micro-scale***
- 3. *Strategy and goal***

II. Numerical & Experimental approach

- 1. *Numerical modeling***
- 2. *Specimen preparation***
- 3. *Mechanical tests***

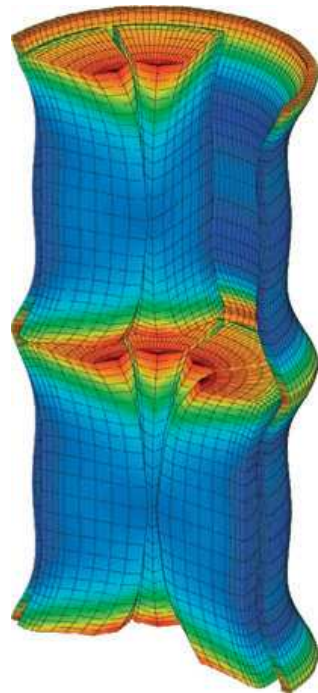
III. Results : *fracture toughness of 8Y-FSZ grains*

IV. Conclusion and perspectives



Good understanding of its behavior is needed

➔ Numerical modeling



Modeling of stress location in Zircaloy cladding and fuel pellets (Pressurized Water Reactor)

Fuel modeling :

- Viscoplasticity in the core
- **Brittle on the periphery**

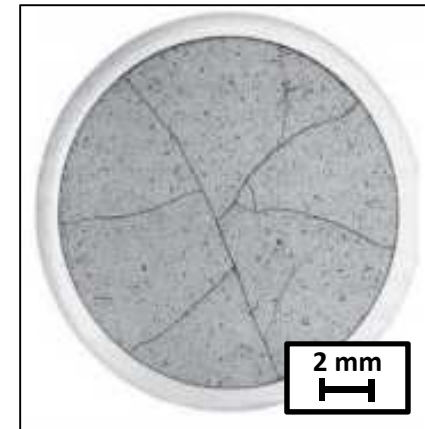
When does a crack appear ?
How does it propagate ?

➔ Experimental values are still needed

➔ Need for fracture toughness (K_{IC}) values

Irradiated fuel

➤ Cracked



Sectional optical micrograph of an irradiated fuel pellet

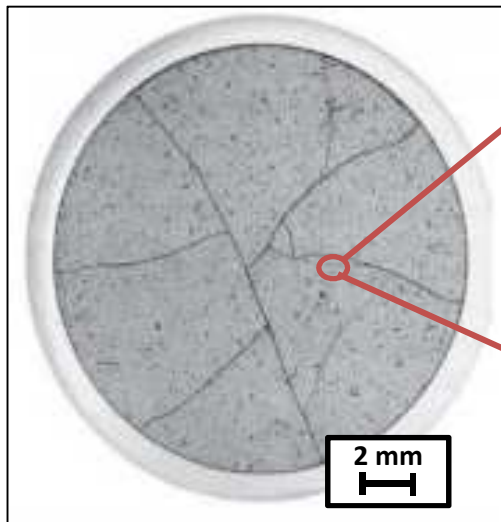
Impossible to machine a standard specimen

Fresh nuclear fuel

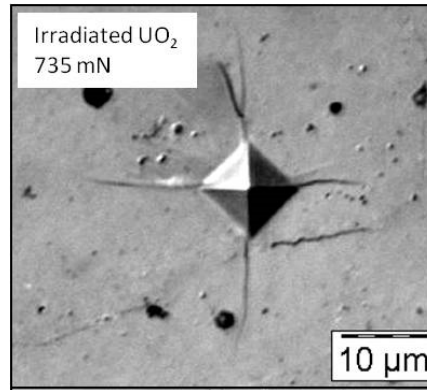
➤ OK

Testing the material at the micro-scale

- To avoid cracks and pores
- To measure the « true » mechanical properties



Cross-sectional optical micrograph of irradiated fuel pellet



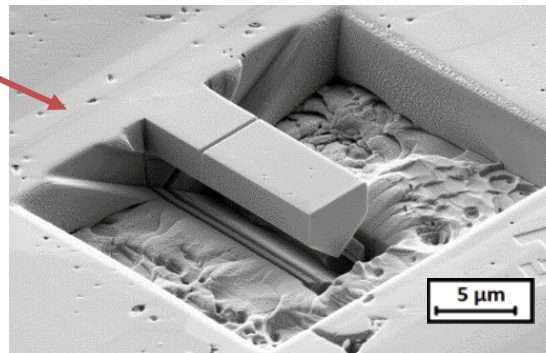
Vickers indentation test on irradiated UO_2

Indentation

- Handy solution
 - Studied before (N. Leroux; Post-doc)
- A calibration is needed

Micro-cantilever

1st step : Set up and validate the method



Notched μ -cantilever for fracture toughness measurement (ZrO_2)

- Closer to a bending test
- Fabrication with FIB/SEM
- Mechanical testing with a nano-indenter
- Modeling is necessary

Set up a μ -cantilever testing method

Specifications :

- Size : $\approx 10 \mu\text{m}$ length
few μm high and width
- Pentagonal section

	UO ₂ (Fresh)	ZrO ₂ FSZ
Crystal structure	Fm $\bar{3}$ m	Fm $\bar{3}$ m
Lattice parameter (nm)	0,547	0,517
Grain size (μm)	≈ 12	≈ 10
Density (g.cm ⁻³)	10,97	6,1
Porosity	$\approx 5 \%$	$\approx 7 \%$
Elastic constants (macroscopic)	E = 194 GPa Nu = 0,30	E = 160 GPa Nu = 0,30
Tensile strength (25°C) (MPa)	$\approx 100-150$	≈ 180
Fracture toughness (MPa.m ^{0,5})	≈ 1	$\approx 3,5$

Validate the method on a material with :

Known properties

Properties close to UO₂

8Y-FSZ

Cubic Zirconia with 8mol% Y

Tests on specific crystallographic planes

Study :

Fracture toughness determination of 8Y-FSZ planes with a μ -cantilever method

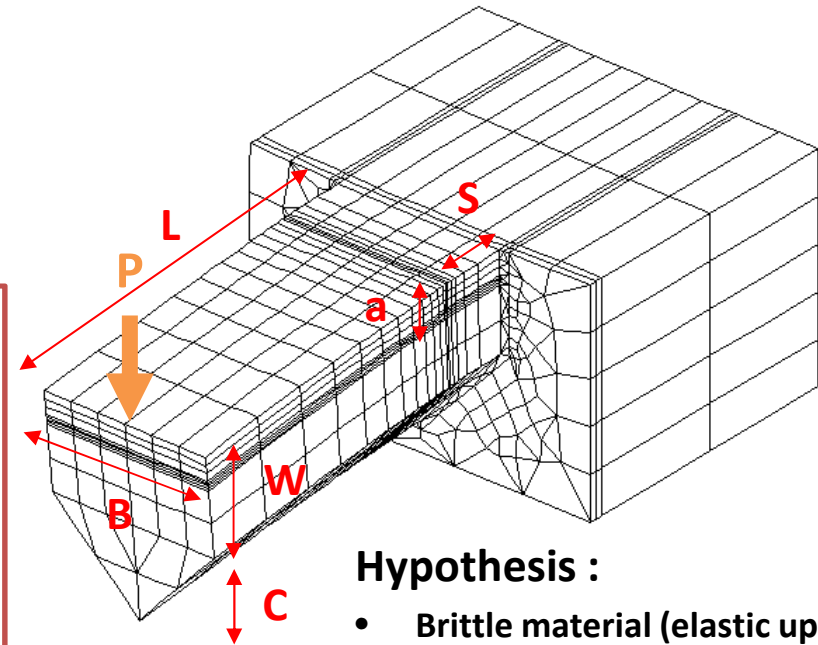
Numerical modeling : why ?

- To calculate fracture toughness
- To adjust cantilever dimensions
- To be able to adapt to :
 - New materials (plasticity...)
 - New geometries (chevron notch...)
- To optimize the test



FEM analysis (Castem® (CEA, France))

→ Modeling of the cantilever's test



Hypothesis :

- Brittle material (elastic up to fracture)
- Notch = perfectly sharp

1st step : notch's depth = a

P applied → Elastic energy E_1 calculated

2nd step : notch's depth = $a + da$

P applied → Elastic energy E_2 calculated

Energy release rate :

$$G = \frac{E_1 - E_2}{B \cdot da}$$

Fracture toughness :

$$K_{IC} = \sqrt{G \cdot E'}$$

Finally we need :

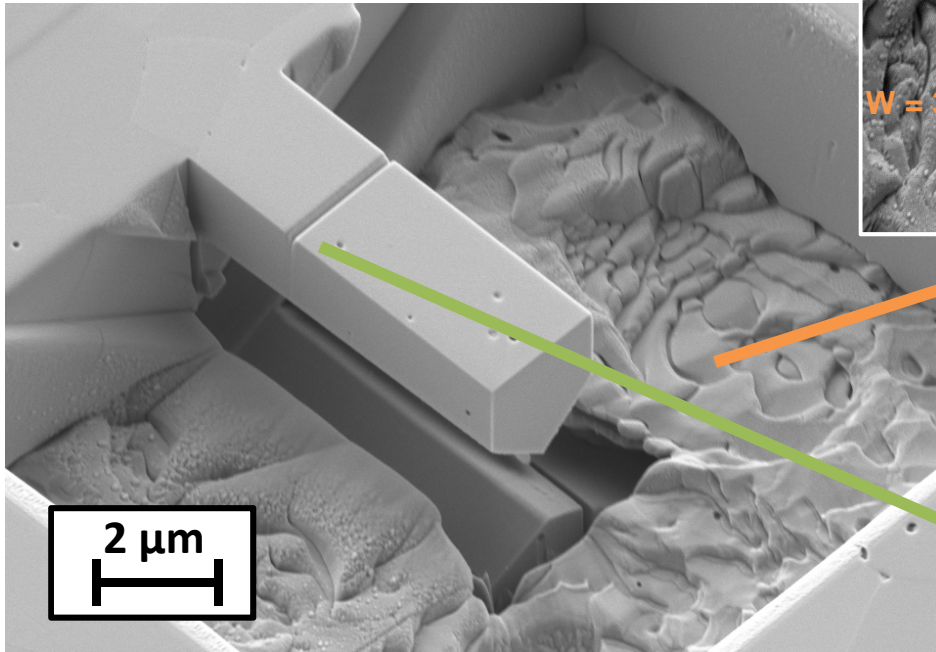


- Fracture load
- Dimensions of the beam and the notch

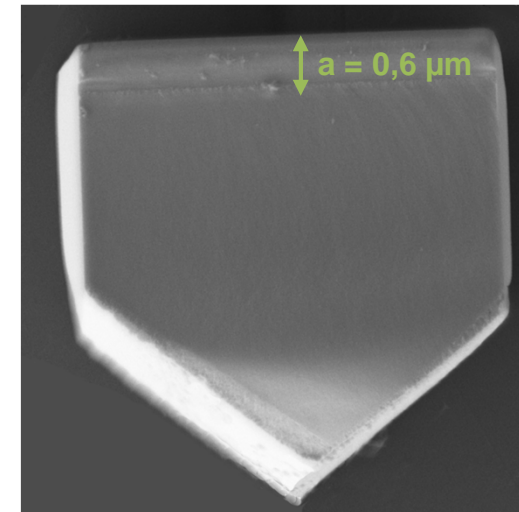
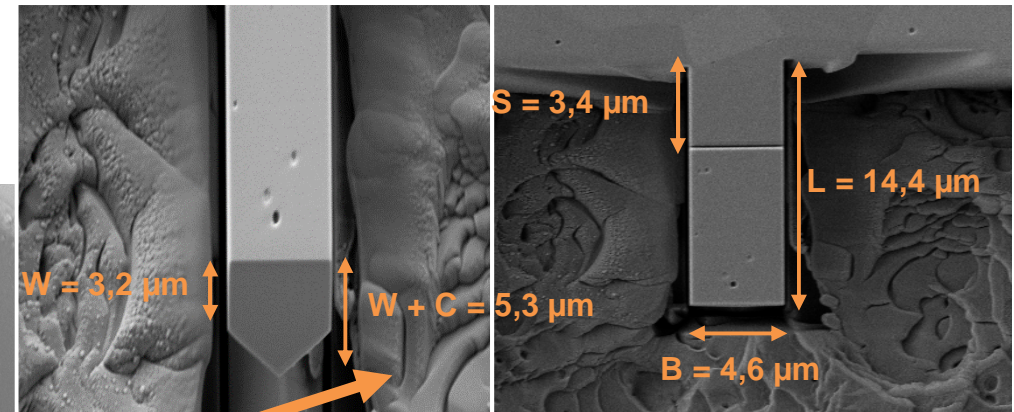
FIB milling with 30 kV Ga⁺ ions

- Zeiss Nvision 40 (Oberkochen, Germany)

Dimensional measurements



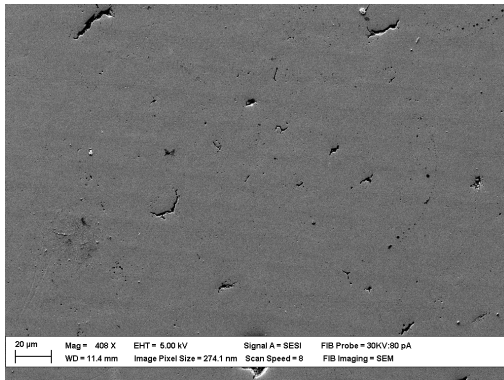
SEM micrograph of a notched cantilever in 8Y-FSZ



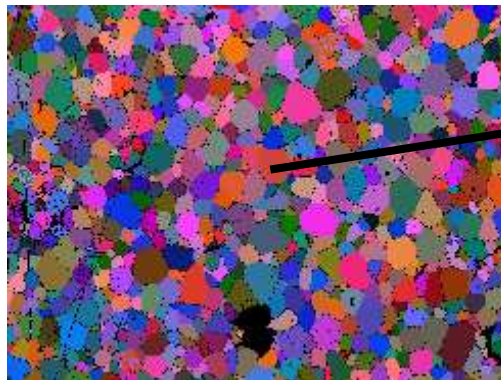
Depth measurement

- After cantilever's break

Selection of crystallographic-planes

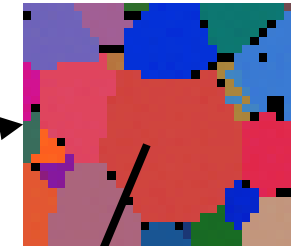


SEM picture of the mapped area

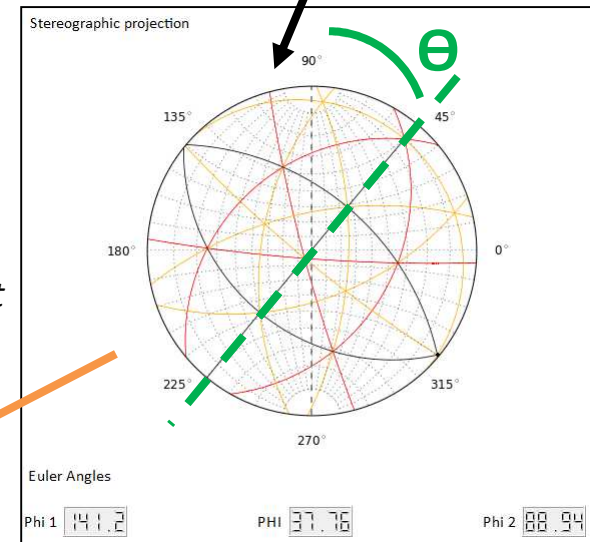


EBSD map

Selecting a suitable grain



Doing the notch at the beginning helps to avoid any edge effect : the notch is straight



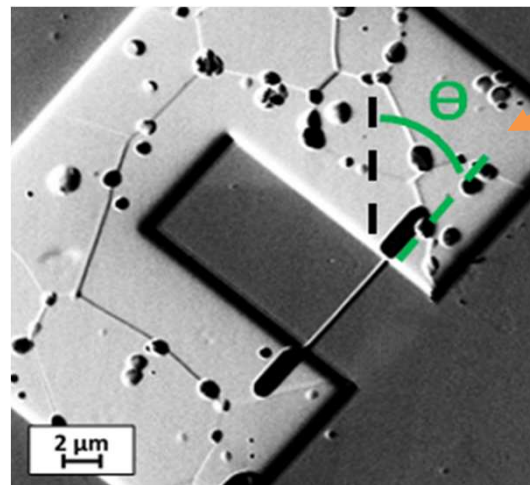
Stereographic projection

→ Targeted plane is normal to the sample's surface

1. Notch fabrication in the selected plan
2. Beam fabrication around the notch

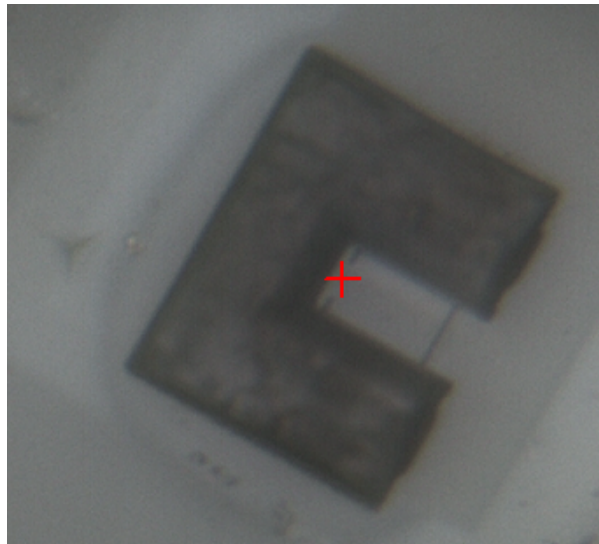
Precautions are necessary !

→ Follow the shape of the grain during the milling process



Fracture load using an ex-situ nano-indenter

- Agilent Technologies G200 (Santa Clara, California, USA)



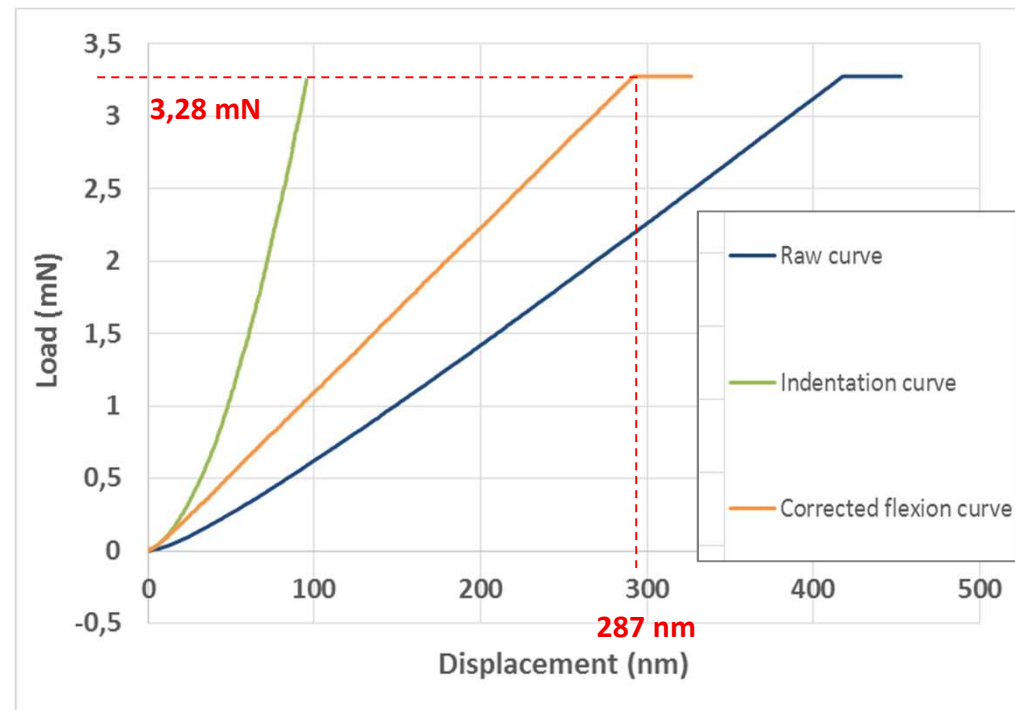
Optical micrograph of a targeted cantilever

Target with an optical microscope :

- Main source of error

Correction of the indentation is performed

- Subtraction of indentation curve to the raw curve



Linear load-displacement curve

- Elastic behavior up to fracture

Fracture toughness measurements of crystallographic planes (8Y-FSZ)

- No significant differences between two families of planes

Planes	{100}	{111}
K_{IC} (MPa.m ^{0,5})	1,51 ± 0,14	1,60 ± 0,19
Number of beams	5	9

Comparison with other sources (high variability of results)

- Results are in good agreement with planes fracture toughness
 - Macroscopic value is more than twice microscopic value (single grain)
- ➔ *Probably grain boundaries and pores enhance fracture resistance*

Planes	{100}	{100}	{111}	macroscopic
K_{IC} (MPa.m ^{0,5})	1,9 ± 0,1	1,3	1,48 ± 0,07	3,0 ± 0,1
Author	<i>Pajares and al. (1988)</i>	<i>Morscher and al. (1991)</i>	<i>Stanescu and al. (1992)</i>	<i>Soulacroix (CEA) (2012)</i>

Initial aim of the work :

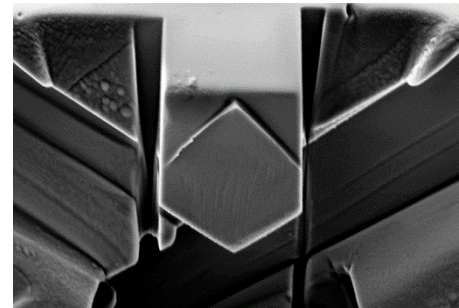
- **Set up a method of fracture properties measurements on FIB prepared specimens**
 - Allows measurements of fracture toughness of a single grain in a bulk material
- **Validate this method on 8Y-FSZ**
 - Validated by comparison with experiments reported on single crystals

Improvements :

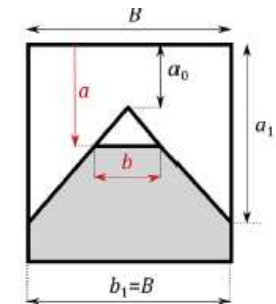
- **Chevron notch**
 - Propagation of the crack before the cantilever breaks [1]
 - Reduction of ion beam effects [2]

Optimization issues

- Preparation is more difficult
- Load to fracture is very low
- No proof of crack propagation



SEM micrograph of a chevron notch on a broken cantilever



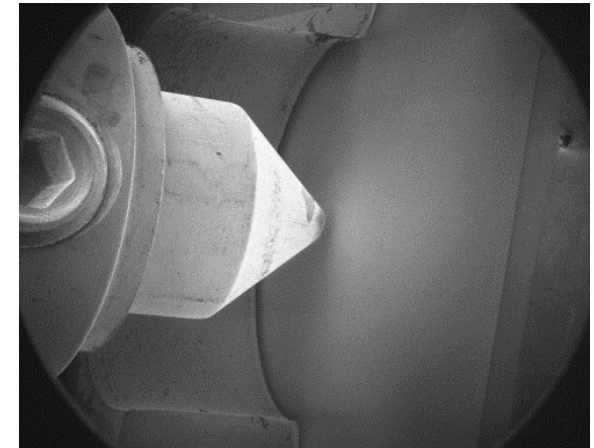
Chevron notch in a cantilever

[1] M. G. Mueller, V. Pejchal, G. Zagar, A. Singh, M. Cantoni, and A. Mortensen, *Acta Mater.*, vol. 86, pp. 385–395, 2015.

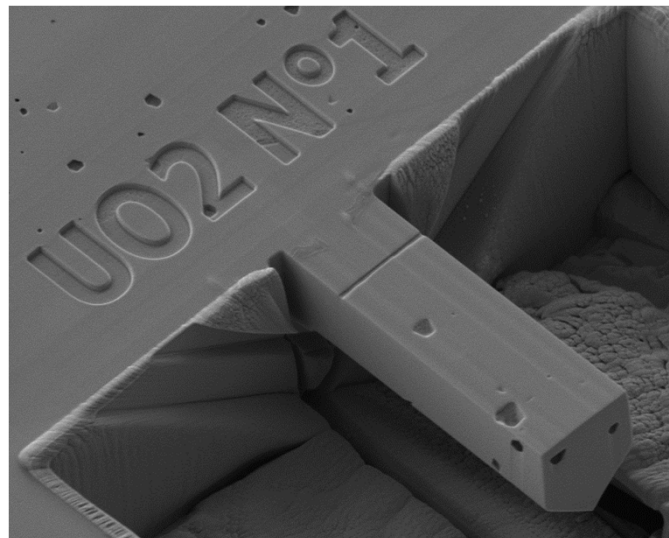
[2] A. D. Norton, S. Falco, N. Young, J. Severs, and R. I. Todd, *J. Eur. Ceram. Soc.*, vol. 35, no. 16, pp. 4521–4533, 2015.

Use of equipment dedicated to nuclear fuels

- **Microscope FIB/SEM Zeiss Auriga 40 (Oberkochen, Germany)**
 - Nuclearized : protection against radiations
- **In-situ nano-indenter CSM NHT² (Peseux, Switzerland)**
 - Preparation and testing of specimens in SEM



Vision at SEM during an indentation loading



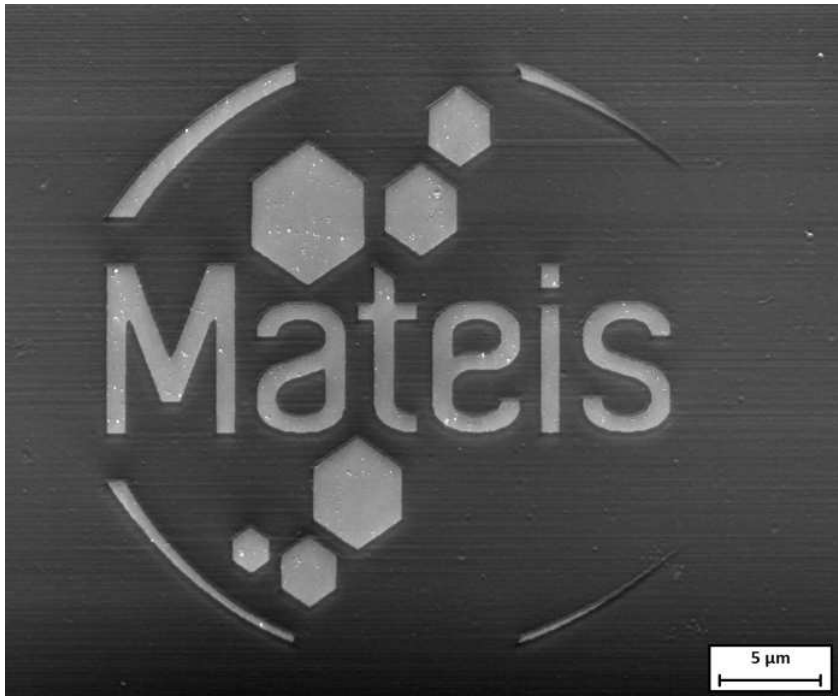
SEM micrograph of a notched cantilever in fresh UO₂

Measurements on nuclear fuels

- **Apply the cantilever method (straight notch)**
 - On different types of fuels (fresh and irradiated)
 - On fuels with different combustion rates



Thank you for your attention



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