



**HAL**  
open science

## Sub-boundary characterization of crept uranium dioxide fuel pellets, by EBSD and ECCI

M. Ben Saada, H. Mansour, N. Maloufi, N. Gey, B. Beausir, X. Iltis

### ► To cite this version:

M. Ben Saada, H. Mansour, N. Maloufi, N. Gey, B. Beausir, et al.. Sub-boundary characterization of crept uranium dioxide fuel pellets, by EBSD and ECCI. CREEP 2015 - International Conference on Creep And Fracture Engineering Materials And Structure, May 2015, Toulouse, France. cea-02491623

**HAL Id: cea-02491623**

**<https://cea.hal.science/cea-02491623>**

Submitted on 26 Feb 2020

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

DE LA RECHERCHE À L'INDUSTRIE



(1)



(2)



www.cea.fr

## Sub-boundary characterization of crept uranium dioxide fuel pellets, by EBSD and ECCI

Mariem Ben Saada<sup>1,2</sup>

H. Mansour<sup>2</sup>, N. Maloufi<sup>2</sup>, N. Gey<sup>2</sup>, B. Beausir<sup>2</sup>, X. Iltis<sup>1</sup>

*Email: [Mariem.BENSAADA@cea.fr](mailto:Mariem.BENSAADA@cea.fr)*

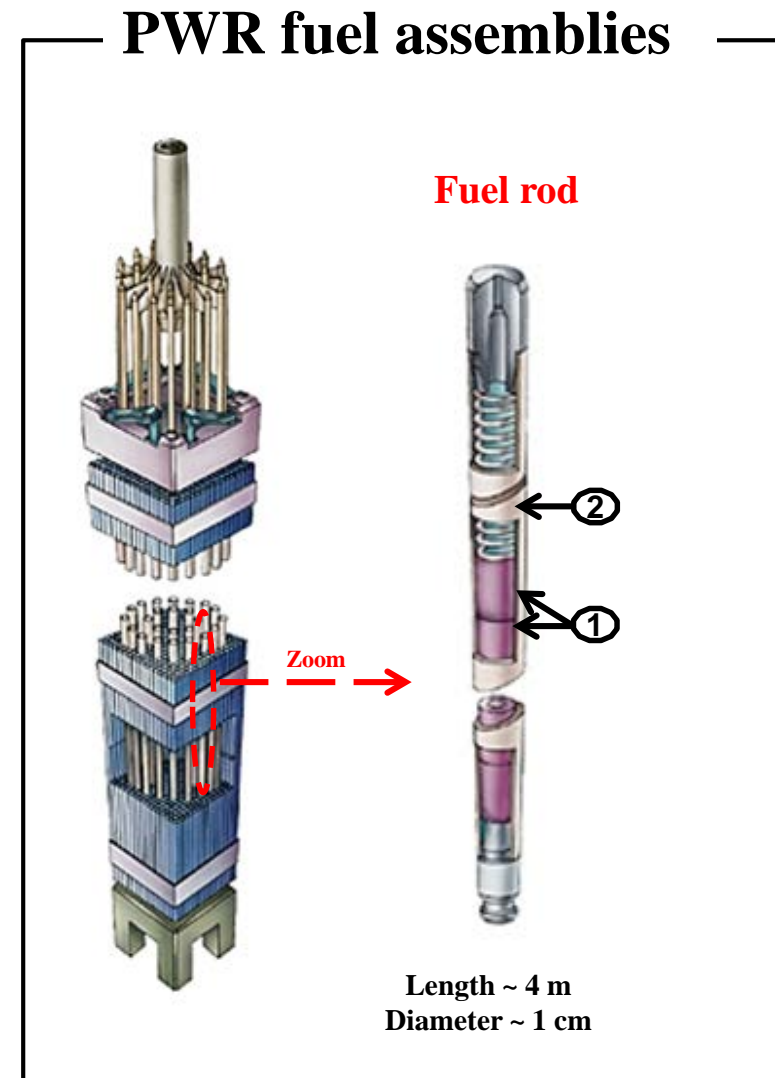
**EBSD: Electron BackScattered Diffraction**  
**ECCI: Electron Channeling Contrast Imaging**

Creep 2015, June 01– June 04, 2015, Toulouse, France

# Sintered Uranium Dioxide $\text{UO}_2$ pellets are commonly used in nuclear Pressurized Water Reactors (PWR)

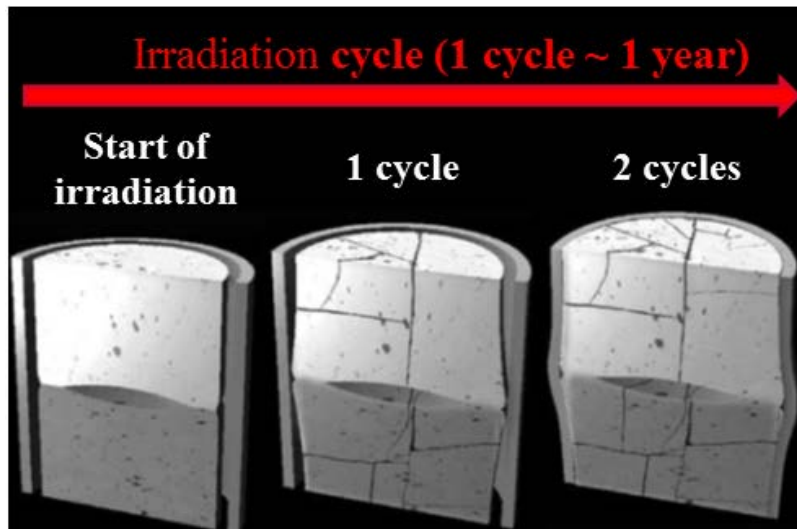
The fuel rods consist of:

- (1)  $\text{UO}_2$  pellets piled up in
- (2) a zirconium alloy cladding tube cooled by pressurized water



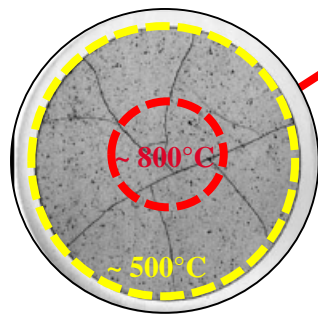
# In service the $UO_2$ pellets undergo significant plastic deformation by creep

## Nominal conditions



9,5 mm

**Under irradiation:**

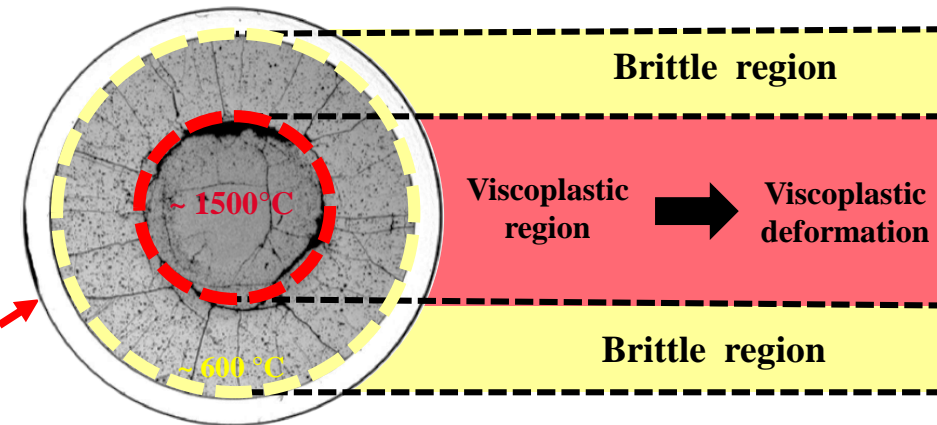


thermal gradient + fission products

→ Fuel swelling

→ Large stresses and cracks in the pellet

## Power transient



*Cracking in a  $UO_2$  pellet subjected to 2 annual PWR cycles, followed by 12 hours at a linear power of 430 W/cm.*

## Increasing power causes:

- An increase of pellet temperature up to about 1500-2000°C in its central part  
→ **Viscoplastic-deformation: Creep of the pellet**

➤ **To avoid cladding failure**

→ Increase the creep of the fuel (viscoplastic deformation)

# This creep process is partly reproduced by compression creep tests on non-irradiated $UO_2$ sintered samples

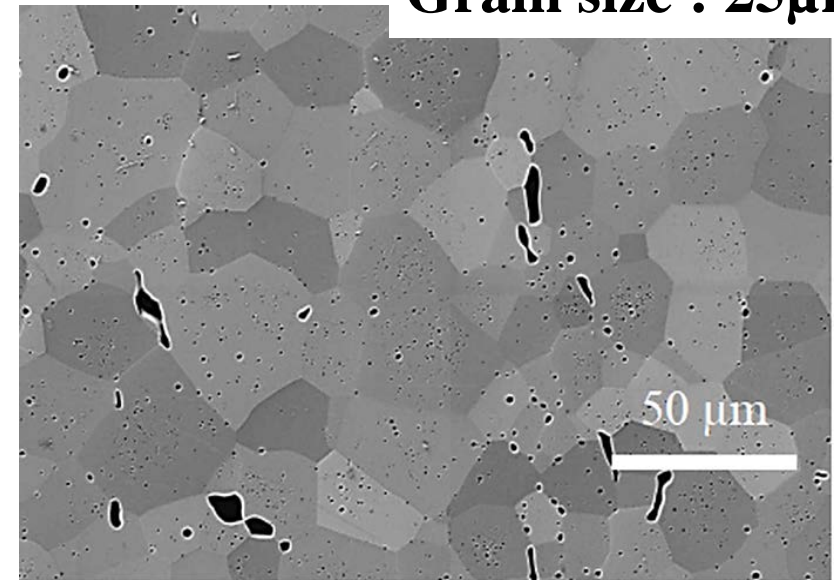
Grain size :  $25\mu m$

## Sintering conditions [1,2]

Uniaxial pressing of the powder  
under **400 MPa**

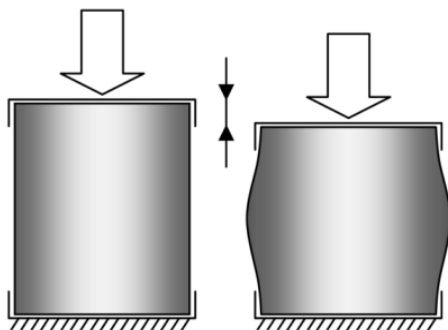
+

Sintering heat treatment at  **$1900^\circ C$**  for  
**4h**, under an atmosphere of **Ar + 5%  $H_2$**



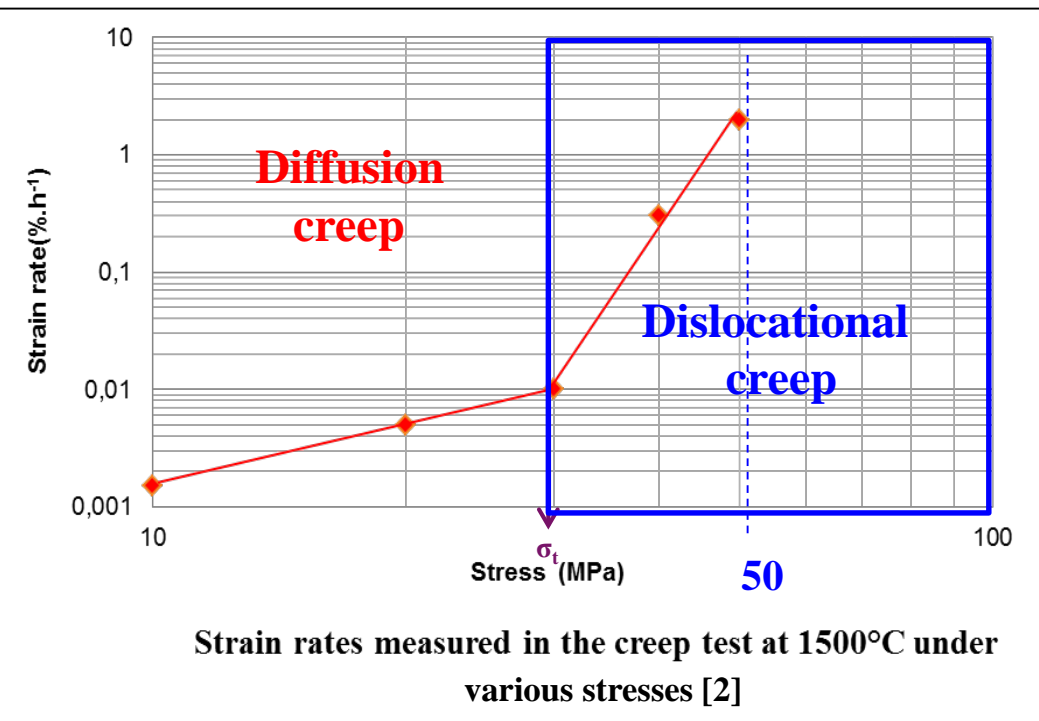
SEM micrograph (BSE mode) of an  $UO_2$  sample

## Compression creep tests at $1500^\circ C$ [1,2]



Our reference  
sample

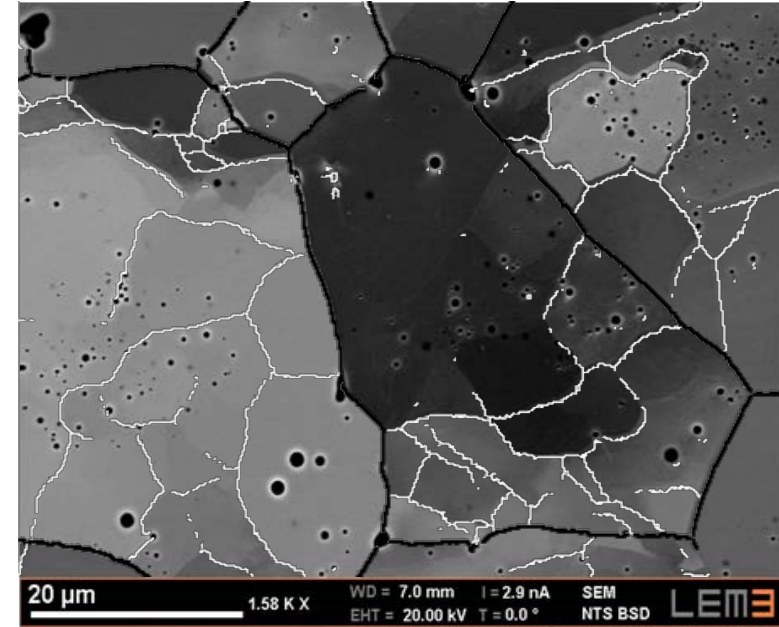
- **50 MPa**
- **test duration: 2.7h**
- **mean deformation: ~ 8%**



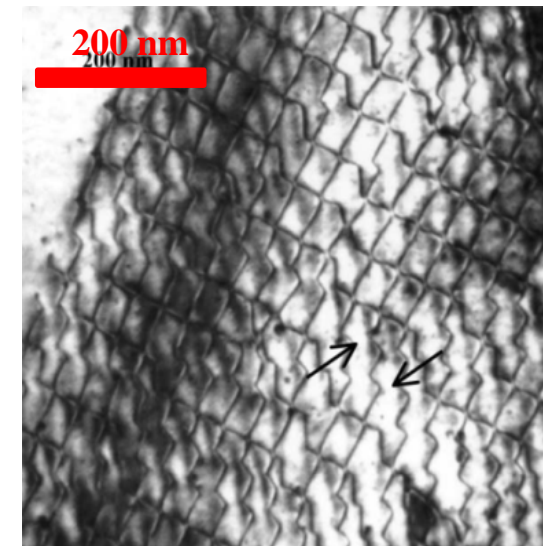
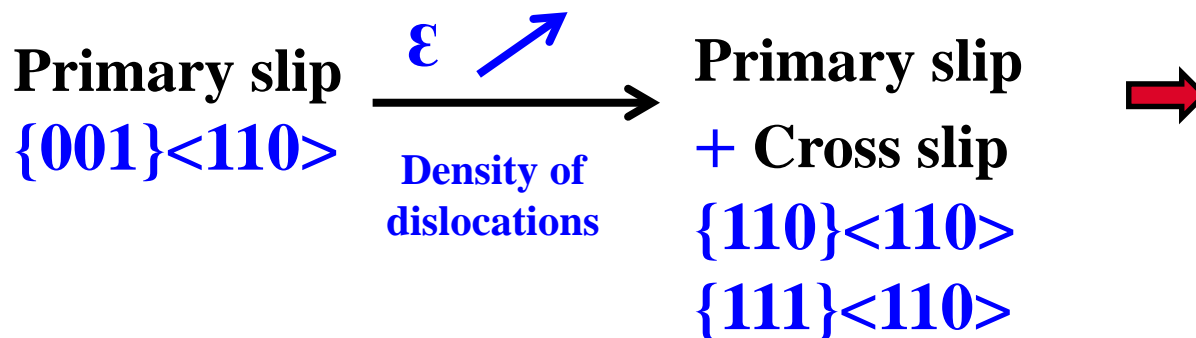
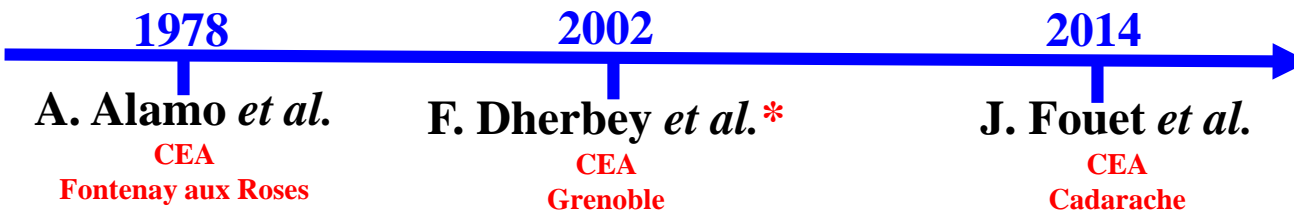


## Creep damage

- Intergranular cavities
  - Sub-grains formation
  - Dislocations density increase
- **SEM studies**



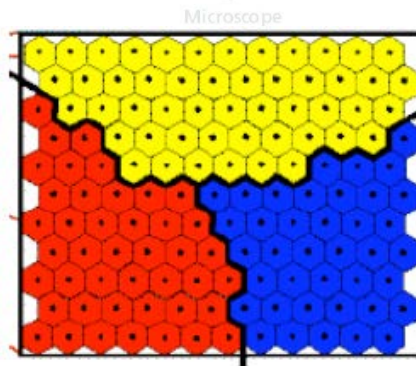
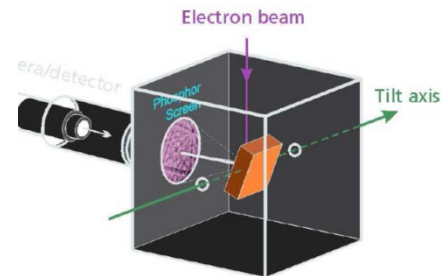
▪ **TEM studies**



**Dislocation network\***

## EBSD

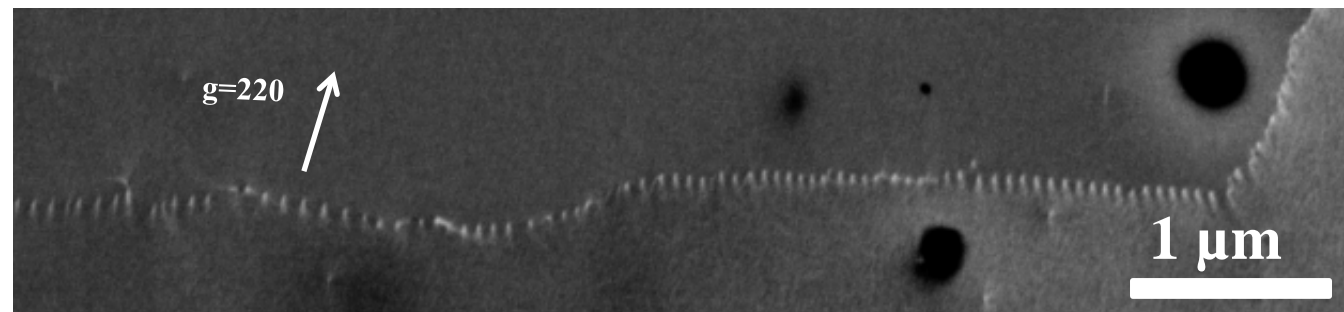
Electron BackScattered  
Diffraction



Acquisition of Orientation map to:

- Identify the low angle boundaries (sub-grains),
- Quantify the Geometrically Necessary Dislocations (GND)

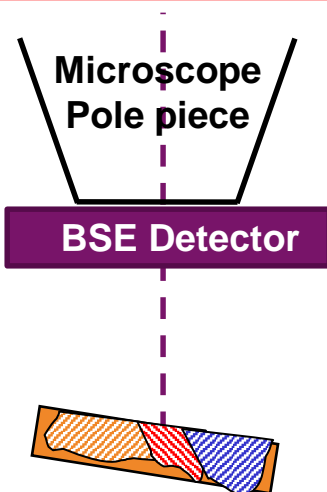
To identify crystallographic defects like dislocations and their arrangement in sub-grains boundaries [4]



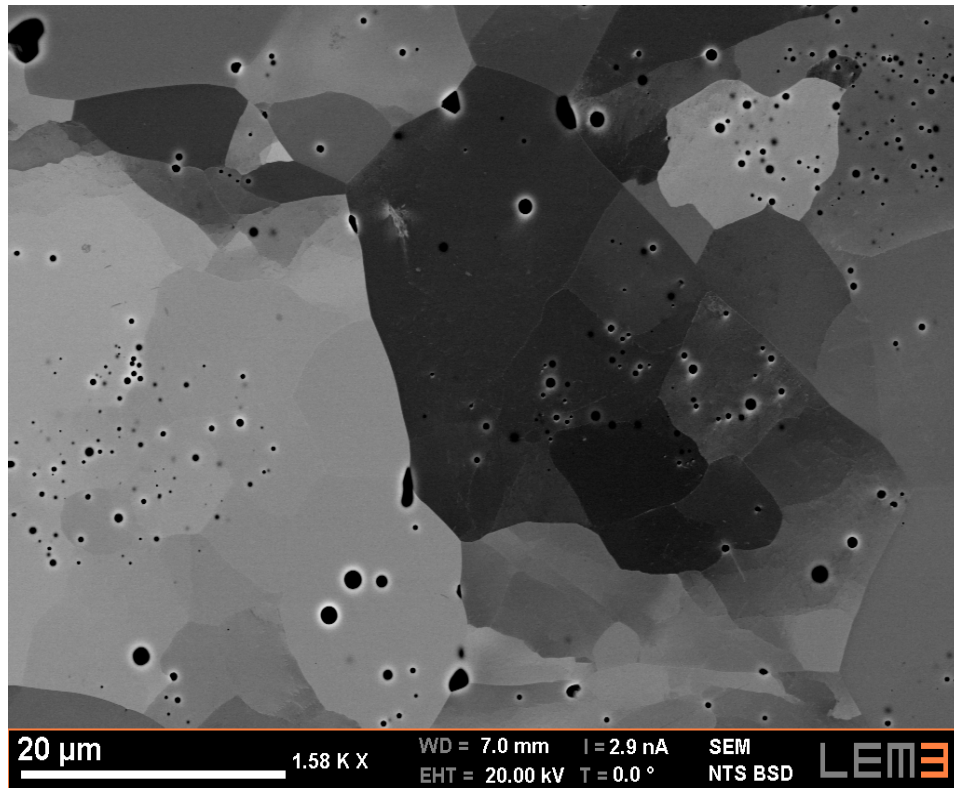
BSE image at high magnification in specific diffraction condition

**BULK MATERIAL SEM**

**ECCI**  
Electron Channeling Contrast  
Imaging [3]





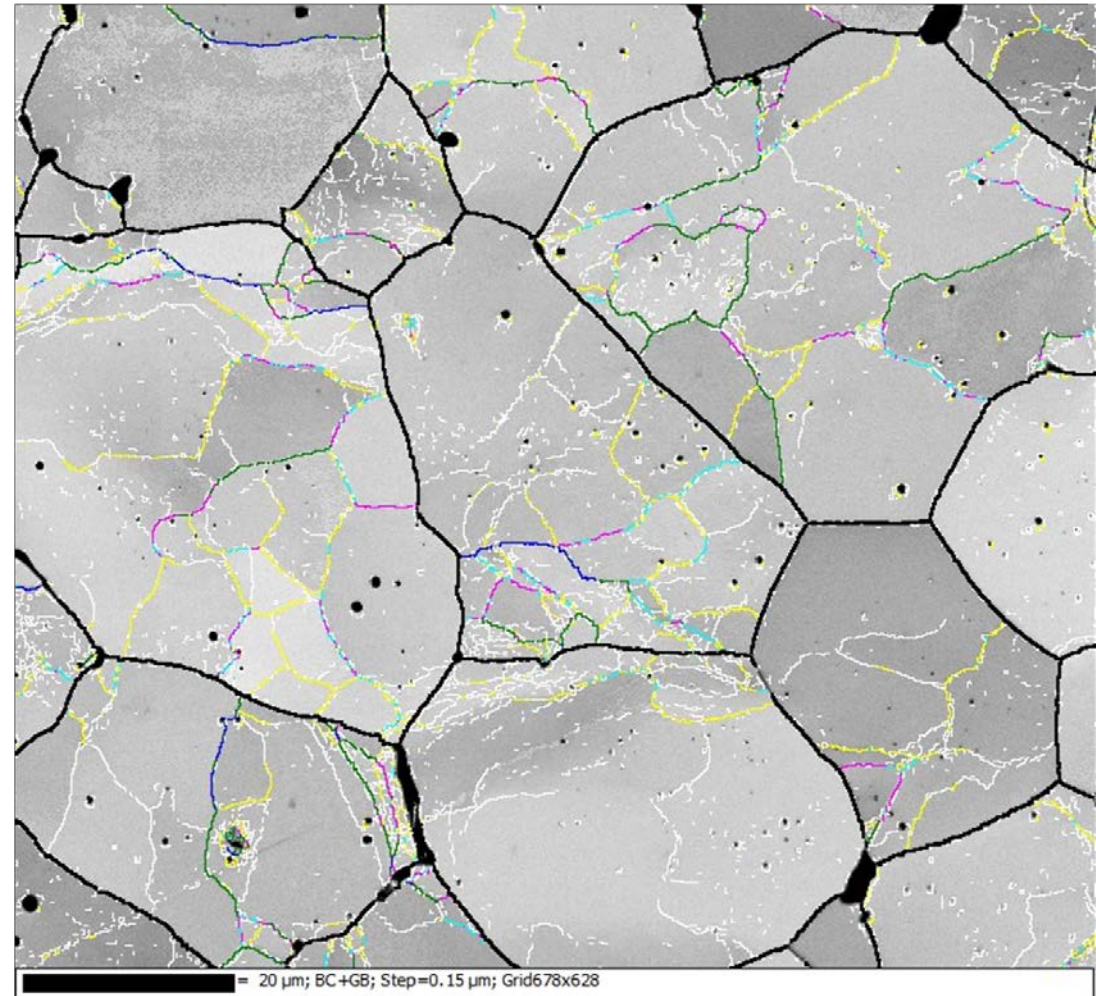


BSE micrograph of an  $UO_2$  sample tested at  $1500^\circ C$ , under 50 MPa

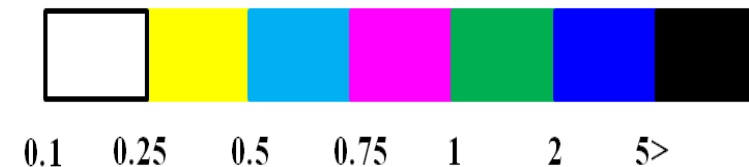
The deformation induced a network of low angle boundaries

EBSD resolves low angle boundaries revealed by misorientations down to  $0.1^\circ$  !

## Misorientation map



Misorientation angle  $\Theta$   
Color code





*From the disorientation between two neighboring points :  $\Delta\theta$*

*separated spatially by  $\Delta x$ ,*

*the lattice curvature:  $k = \frac{\Delta\theta}{\Delta x}$*

*and the GND's density:  $\rho = \frac{k}{b}$*

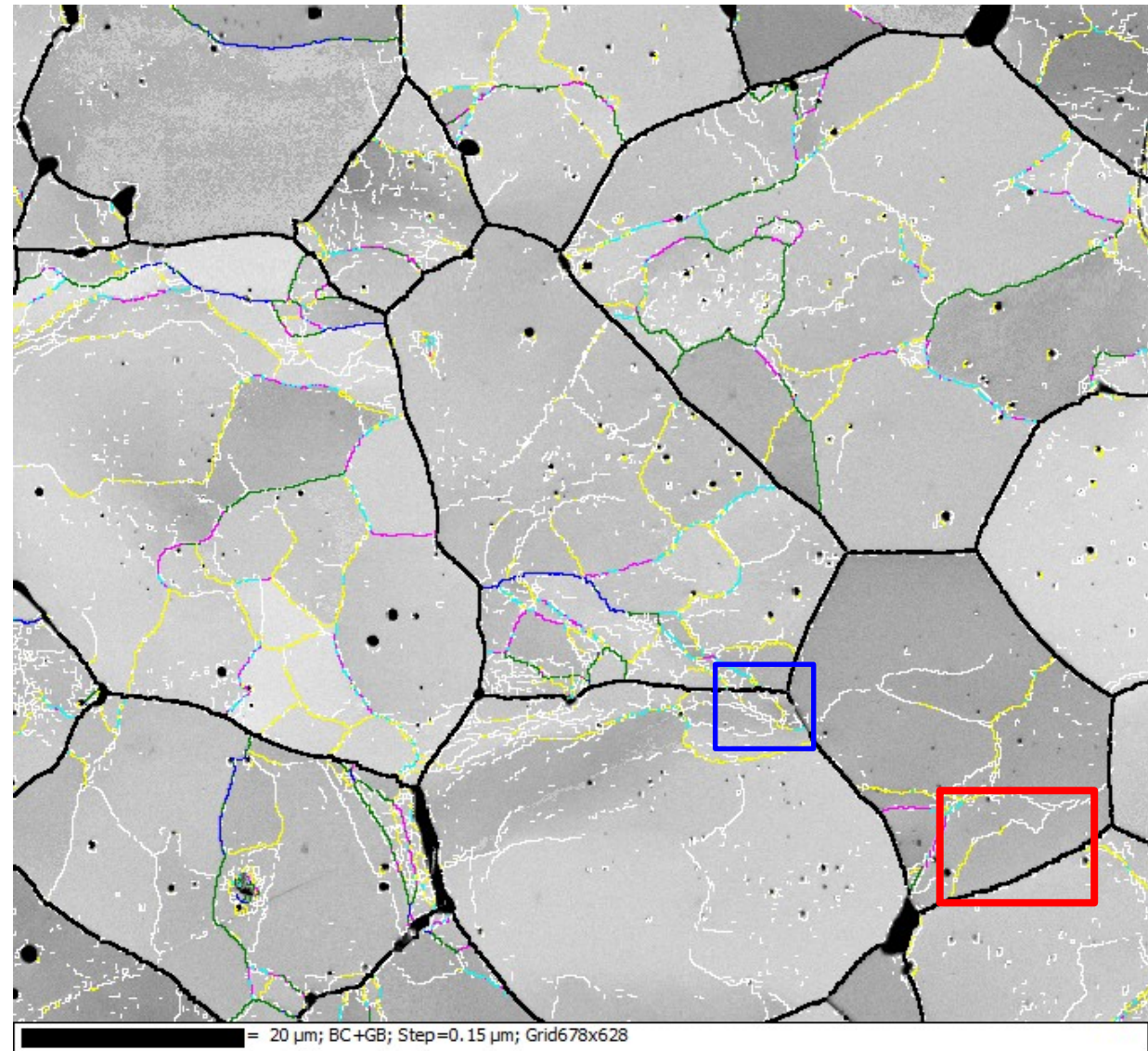
*Per EBSD step unit*

$$b = a/2 \langle 110 \rangle$$

**Nye dislocation density tensor**

$$\alpha = \begin{bmatrix} ? & a_{12} & a_{13} \\ a_{21} & ? & a_{23} \\ ? & ? & a_{33} \end{bmatrix}$$

LEM3 Software

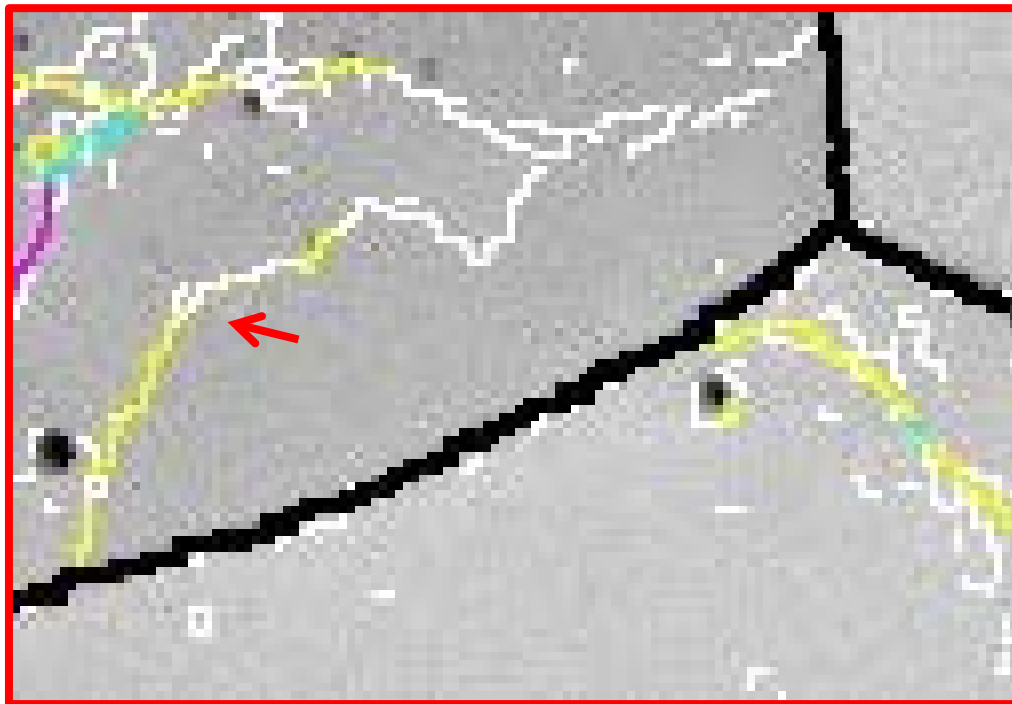


Misorientation angle  $\theta$   
color code



## Sub-grain n°1

**EBSD: GND evaluation from lattice curvature**



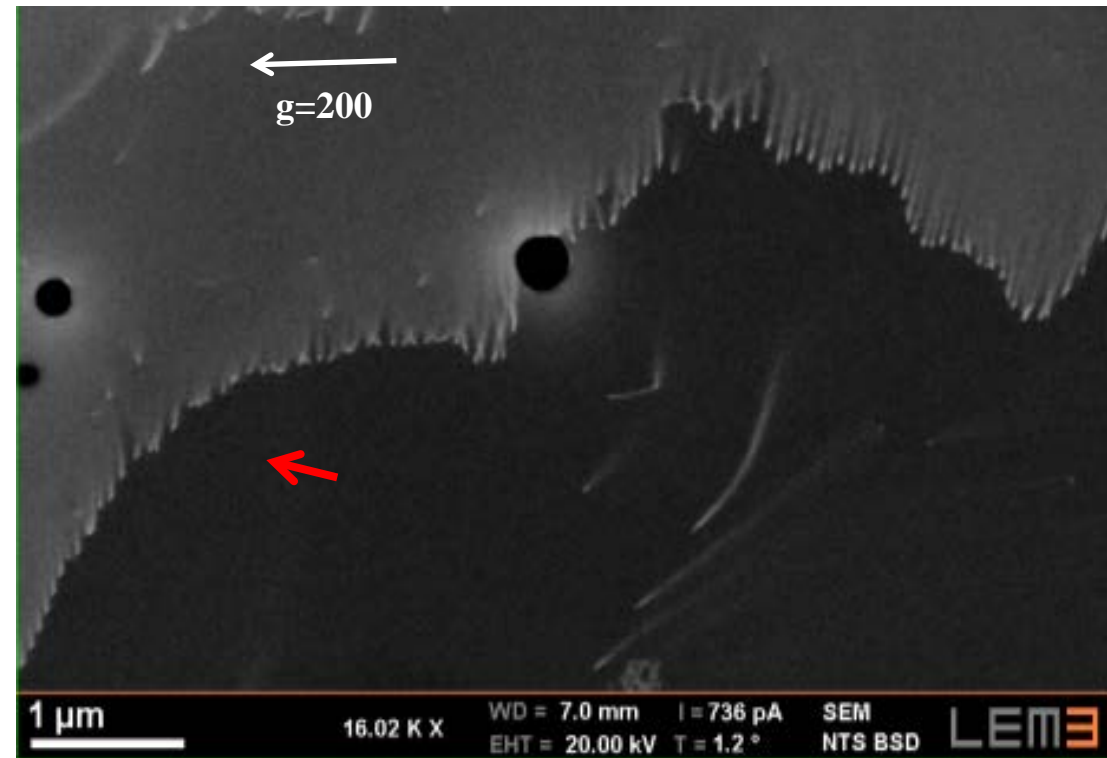
GNDs density

0.1 0.25 >5

$$\Delta\theta \in [0.1^\circ \text{ to } 0.3^\circ]$$

$$\rho_{\text{EBSD}} = k/b = 3 \cdot 10^{13} \text{ to } 9 \cdot 10^{13} \text{ m}^{-2}$$

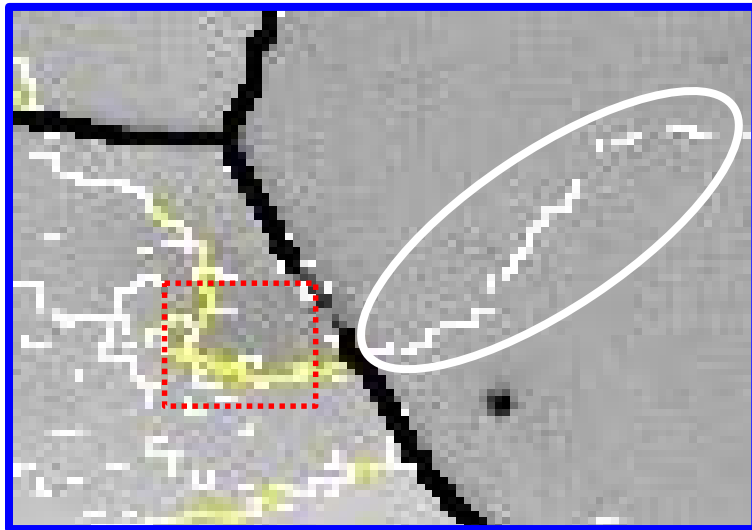
**ECCI: Dislocations arrangement in low angle boundaries**



$$\rho_{\text{ECCI}} = 8 \cdot 10^{13} \text{ m}^{-2}$$



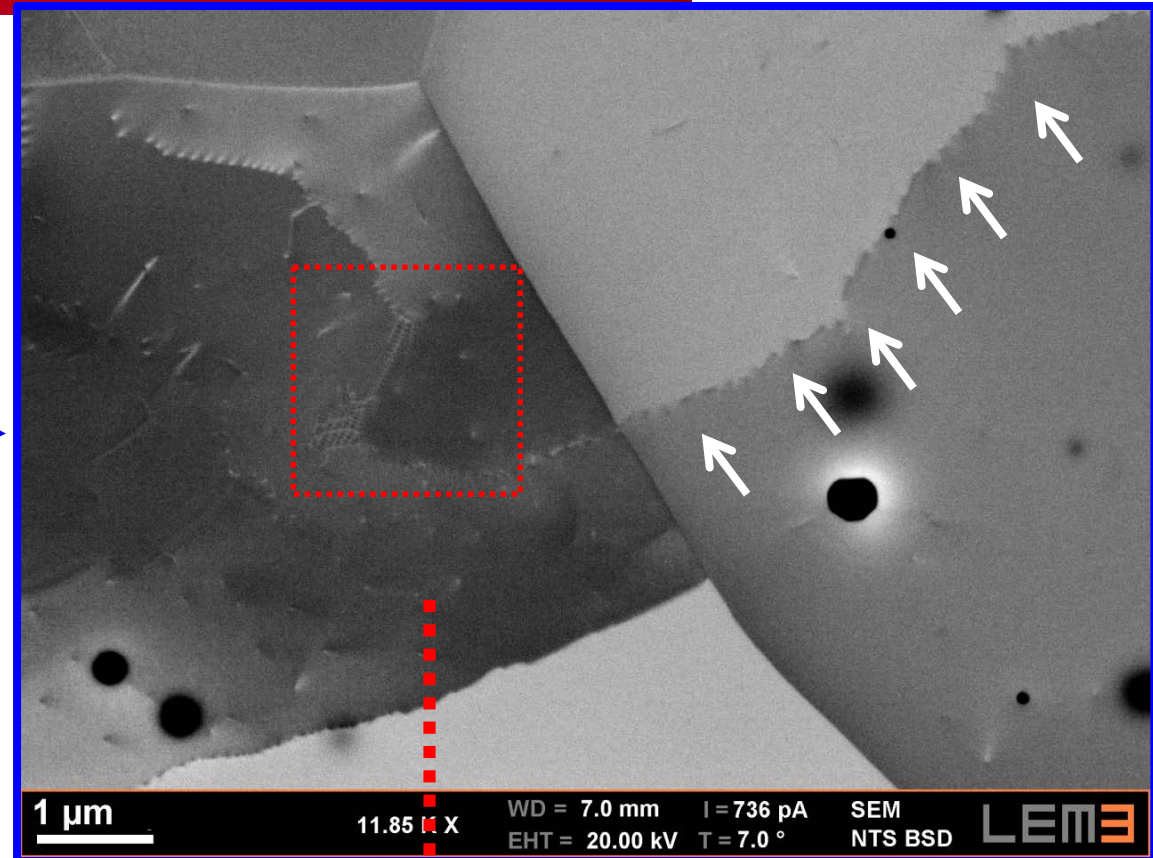
## Sub-grain n°2:



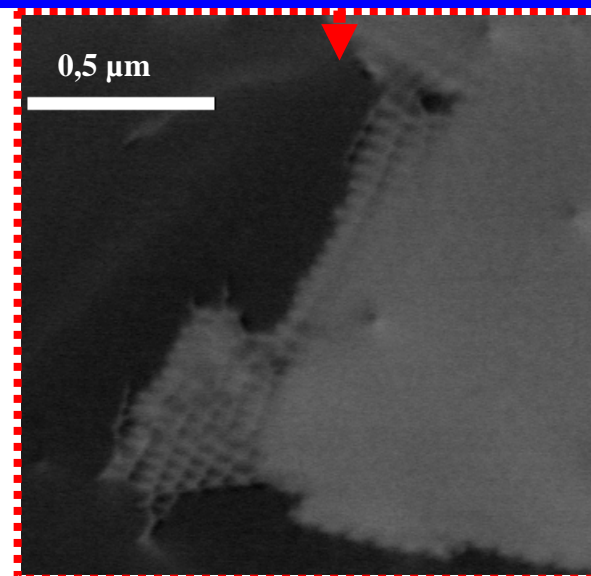
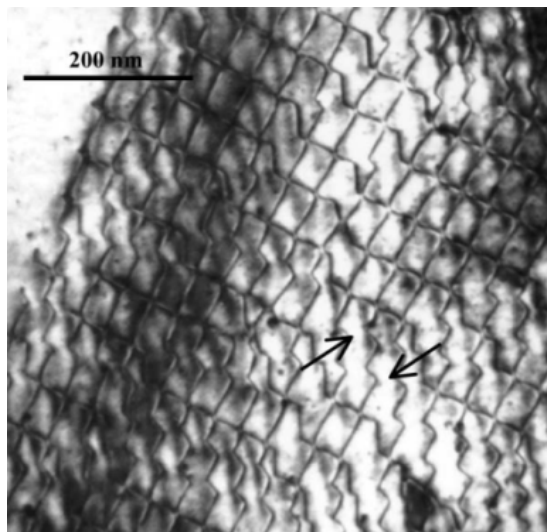
Misorientation angle  $\theta$   
color code



ECCI



## Previous TEM result



**Dislocation Network  
shown by ECCI**



- **EBSD: Quantification of low angle boundaries down to  $0.1^\circ$ ,**  
**Their evolution with the creep deformation,**  
**Evaluation of the dislocation density**
- **ECCI: Imaging of the dislocations in the low angle boundaries**  
**with a TEM quality image but in bulk material,**  
**Analyse their arrangement in hexagonal network,**  
**Deduce the dislocation density**

**In coming work, we plan to use these techniques to understand  
the deformation mechanisms in bulk  $\text{UO}_2$  material**

1 $\mu\text{m}$



# Thank you for your attention

## REFERENCES:

- [1]. Z. EL OMARI, H. MANSOUR, M. BEN SAADA, N. GEY, N. MALOUFI, A. HAZOTTE, X. ILTIS, Etude des liens entre la microstructure et les mécanismes d'endommagement par fluage du dioxyde d'uranium, *Récents Progrès en Génie des Procédés* 107 (2015) pp. 3.14-1 à 3.14-8.
- [2]. X. ILTIS, N. GEY, C. CAGNA, A. HAZOTTE, Ph. SORNAY, Microstructural evolution of uranium dioxide following compression creep tests : An EBSD and image analysis study, *Journal of Nuclear Materials* 456 (2015) pp. 426-435.
- [3]. H. MANSOUR, J. GUYON, M.A. CRIMP, N. GEY, B. BEAUSIR, N. MALOUFI, Accurate electron contrast analysis of dislocations in fine grained bulk materials, *Scripta Materiala* 84-85 (2014) pp. 11-14.
- [4]. H. MANSOUR, M. BEN SAADA, N. MALOUFI, N. GEY, X. ILTIS, Sub-boundary characterization of crept uranium dioxide fuel pellets, by EBSD and ECCI, *CREEP 2015*, May 31 – June 4, Toulouse, France.



This software is developed by [Benoît Beausir](#) and [Jean-Jacques Fundenberger](#)

LEM3  
LABORATOIRE D'ÉTUDE DES MICROSTRUCTURES  
ET DE MÉCANIQUE  
DES MATÉRIAUX

UNIVERSITÉ  
DE LORRAINE

cnrs

Laboratoire d'Etude des Microstructures et de Mécanique des Matériaux,  
Université de Lorraine/CNRS, Ile du Saulcy, 57045 Metz Cedex 01, France