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DE LA RECHERCHE À L'INDUSTRIE

(1)

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LABORATOIRE D'ÉTUDE DES MICROSTRUCTURES ET DE MÉCANIQUE DES MATÉRIAUX

UNIVERSITÉ DE LORRAINE

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RMS Royal Microscopical Society

EBSD 2016

How advanced EBSD and Accurate-ECCI can be applied to quantify sub-boundaries induced during dislocational creep of UO_2

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EBSD 2016, 22 March-23 March , 2016, Manchester

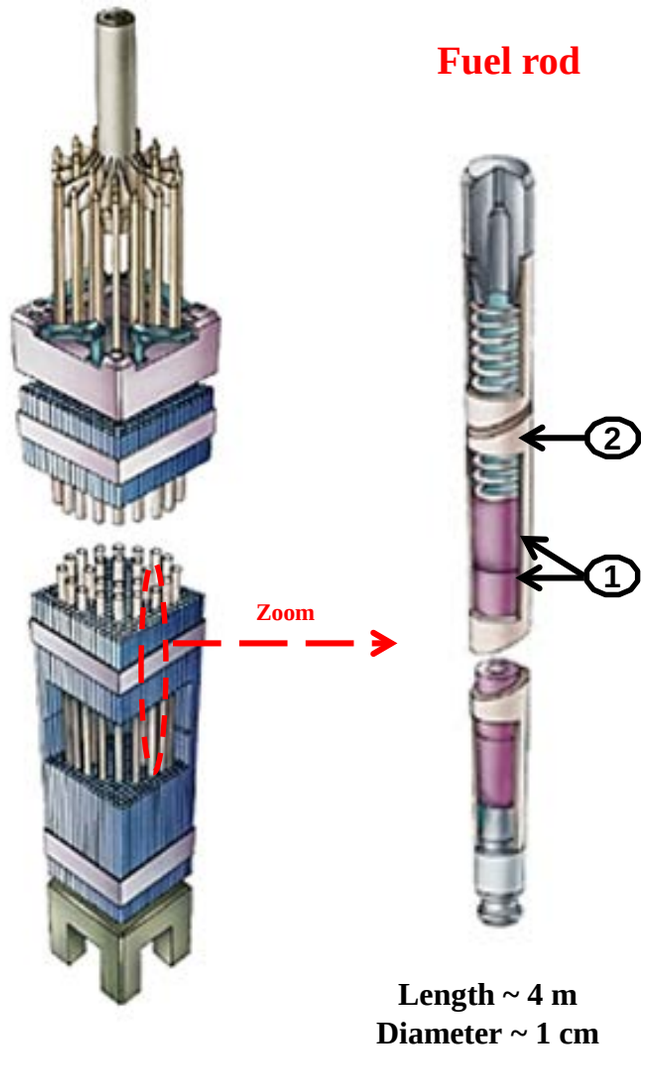
Sintered Uranium Dioxide UO_2 pellets are commonly used in nuclear Pressurized Water Reactors (PWR)

The fuel rods consist of:

(1) UO_2 pellets pilled up in

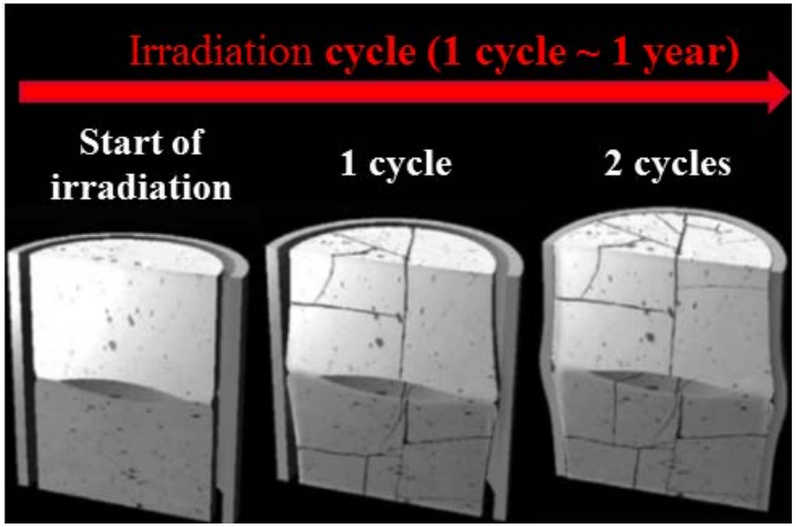
(2) a zirconium alloy cladding tube cooled by pressurized water

PWR fuel assemblies



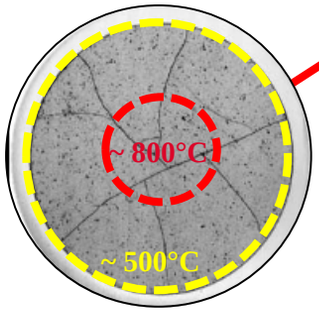
In service the UO_2 pellets undergo significant plastic deformation by creep

Nominal conditions



9,5 mm

Under irradiation:

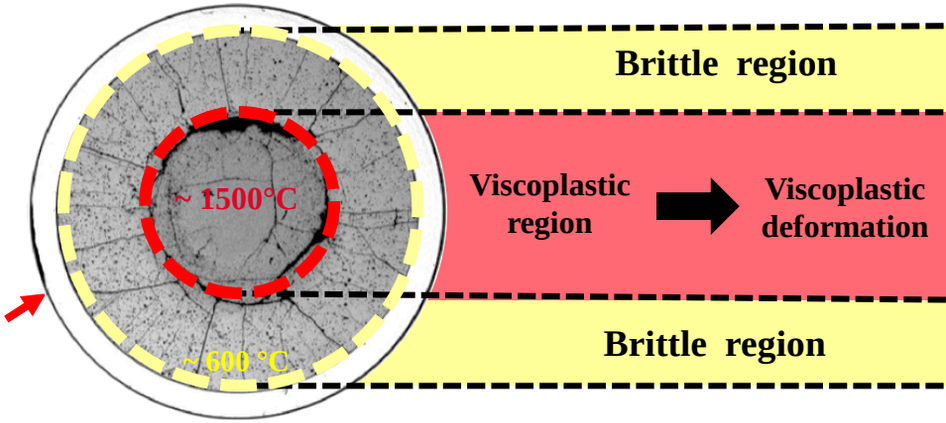


Temperature increase (pellet center)

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

Sintering conditions [1,2]

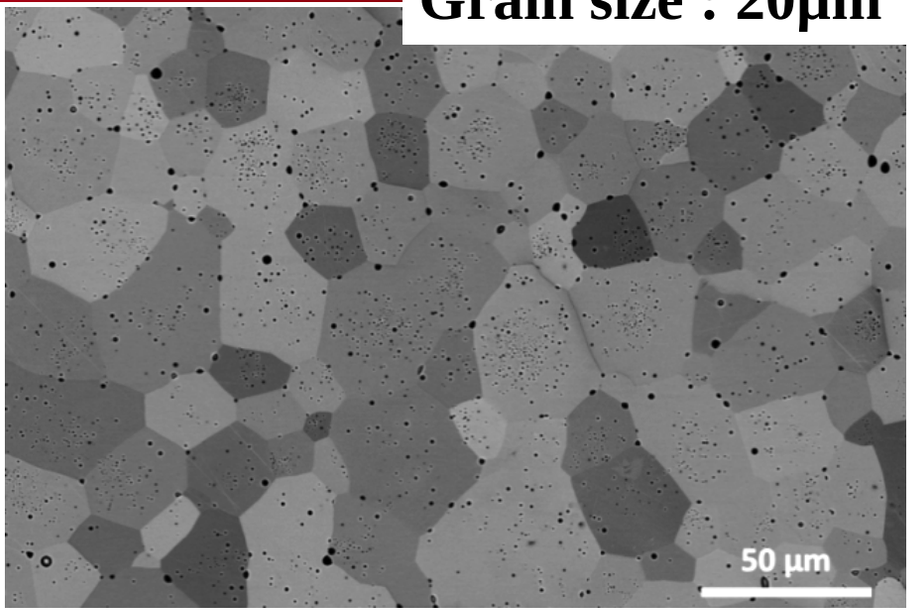
Uniaxial pressing of the powder under 400 MPa

+

Sintering heat treatment at 1900°C for 4h, under an atmosphere of Ar + 5% H_2

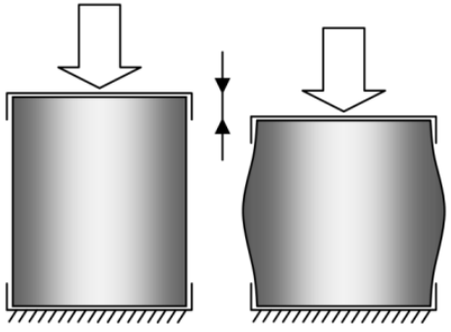
⟹

Grain size : 20µm



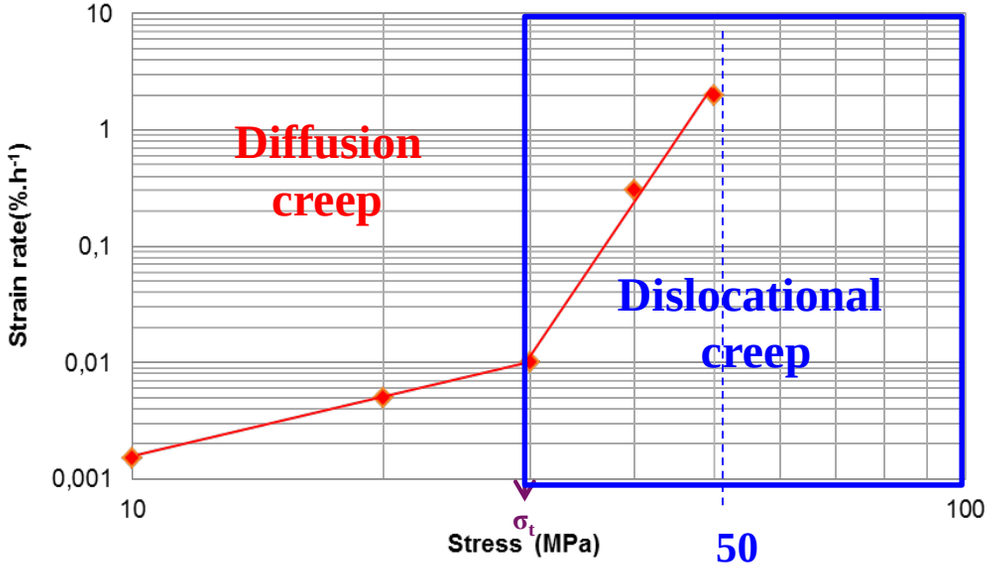
SEM micrograph (BSE mode) of an UO_2 sample

Compression creep tests at 1500°C [1,2]



Our reference sample

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



Strain rates measured in the creep test at 1500°C under various stresses [2]

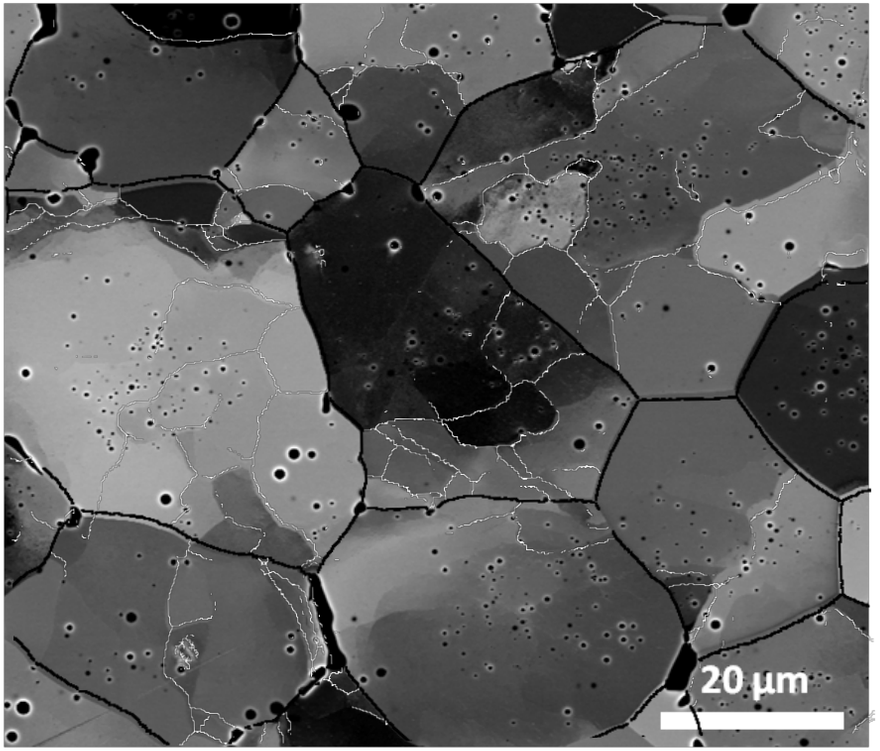
[1]. X. ILTIS, M. BEN SAADA, H. MANSOUR, N. GEY, A. HAZOTTE, N. MALOUFI, A new characterization approach for studying relationships between microstructure and creep damage mechanisms of uranium dioxide, J. of Nuc. Mat. 474 (2016).
[2]. X. ILTIS, N. GEY, C. CAGNA, A. HAZOTTE, Ph. SORNAY, Microstructural evolution of uranium dioxide following compression creep tests : An EBSD and image analysis study, J. of Nuc. Mat. 456 (2015).

Creep damage was previously evidenced by Scanning and Transmission Electron Microscopy (SEM and TEM)

Creep damage

- Intergranular cavities
- Sub-grains formation
- Dislocations density increase

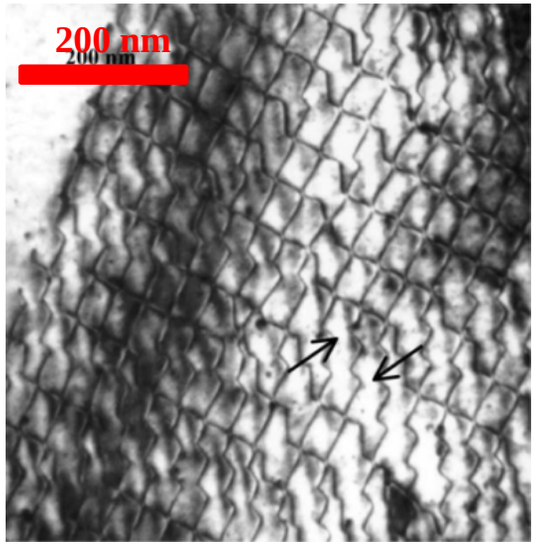
- SEM studies



- TEM studies

1978	2002	2014
A. Alamo *et al.*	F. Dherbey *et al.**	J. Fouet *et al.*
CEA Fontenay aux Roses	CEA Grenoble	CEA Cadarache

Primary slip {001}<110> $\xrightarrow[\text{Density of dislocations}]{\varepsilon \nearrow}$ Primary slip + Cross slip {110}<110> {111}<110>



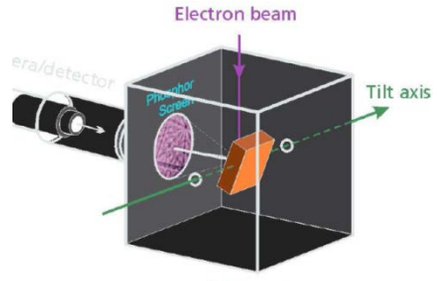
Dislocation network*

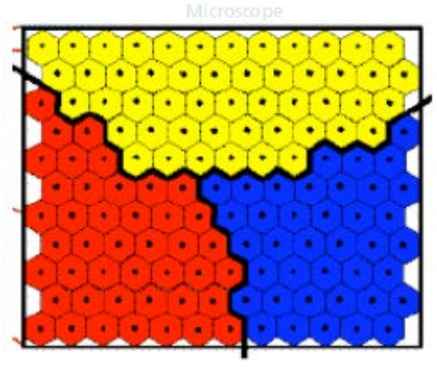
OUR AIM is to characterize the sub-boundaries created by dislocational creep in UO_2

EBSD
Electron BackScattered Diffraction

BULK MATERIAL SEM

A-ECCI
Acurrate Electron Channeling Contrast Imaging

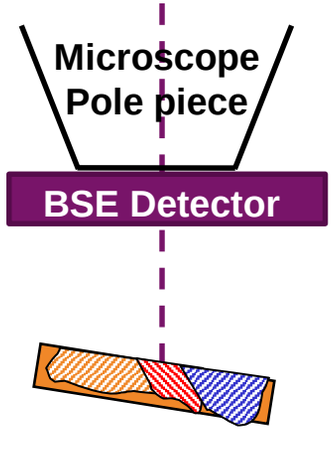


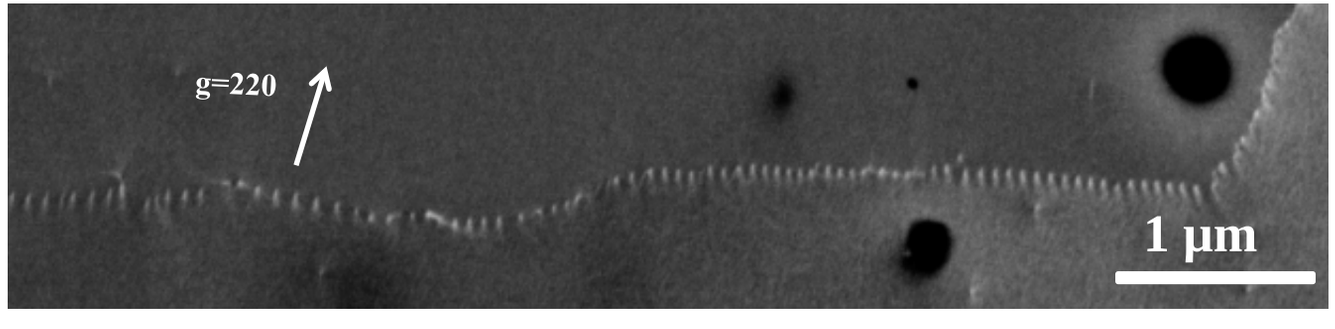


Acquisition of Orientation map to:

- **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





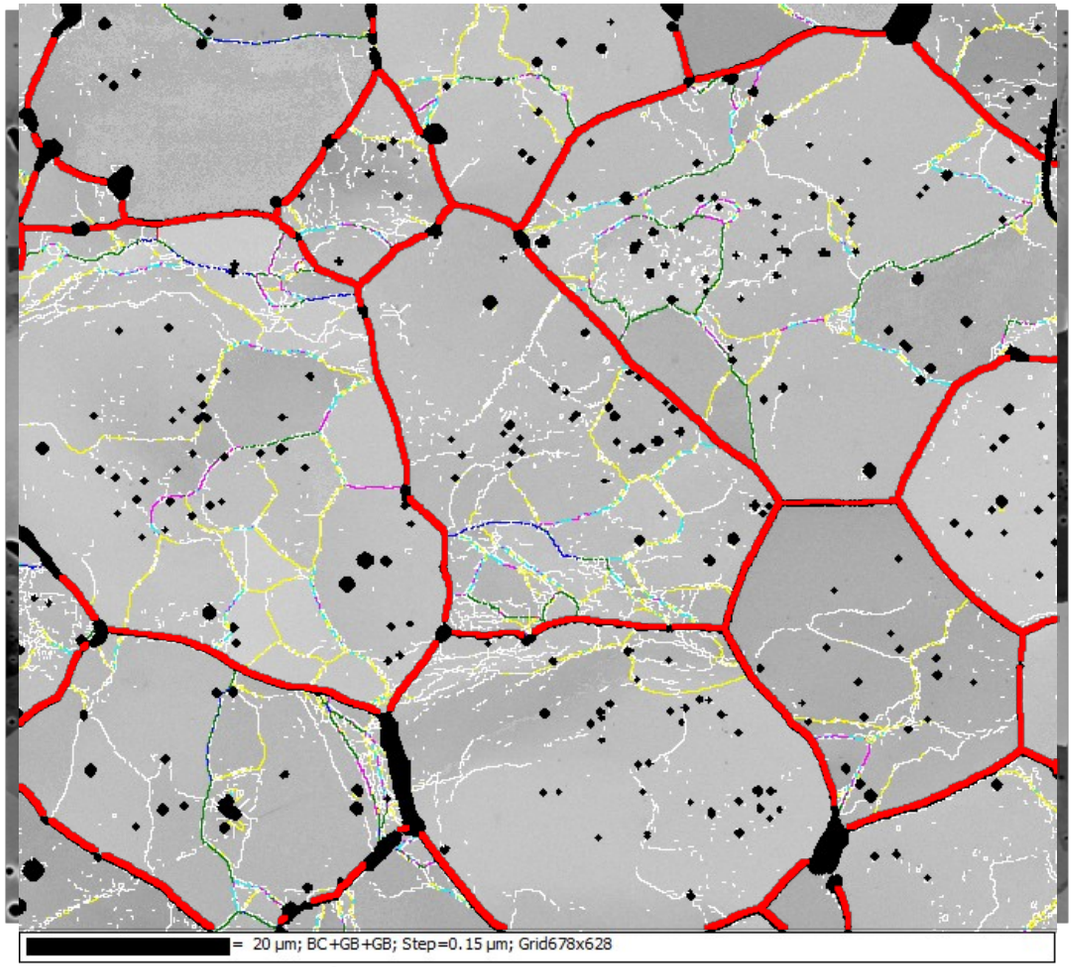
BSE image at high magnification in specific diffraction condition

RESULTS: EBSD analysis

Misorientation map

☹ The substructure is embedded in the **orientation noise**

☹ Voids are indexed with artificial misorientations with the neighborhood



BSE micrograph of an UO_2 sample tested at 1500°C, under 50 MPa

Misorientation angle Θ
Color code

Optimize data acquisition and processing

⮟

Best angular resolution on the EBSD map

Steps:

1) <u>Acquisition:</u>

- Camera setting: Binning ⬊
- Indexing pattern: improved angular resolution mode in Aztec*

2) <u>Data cleaning and filtering:</u>

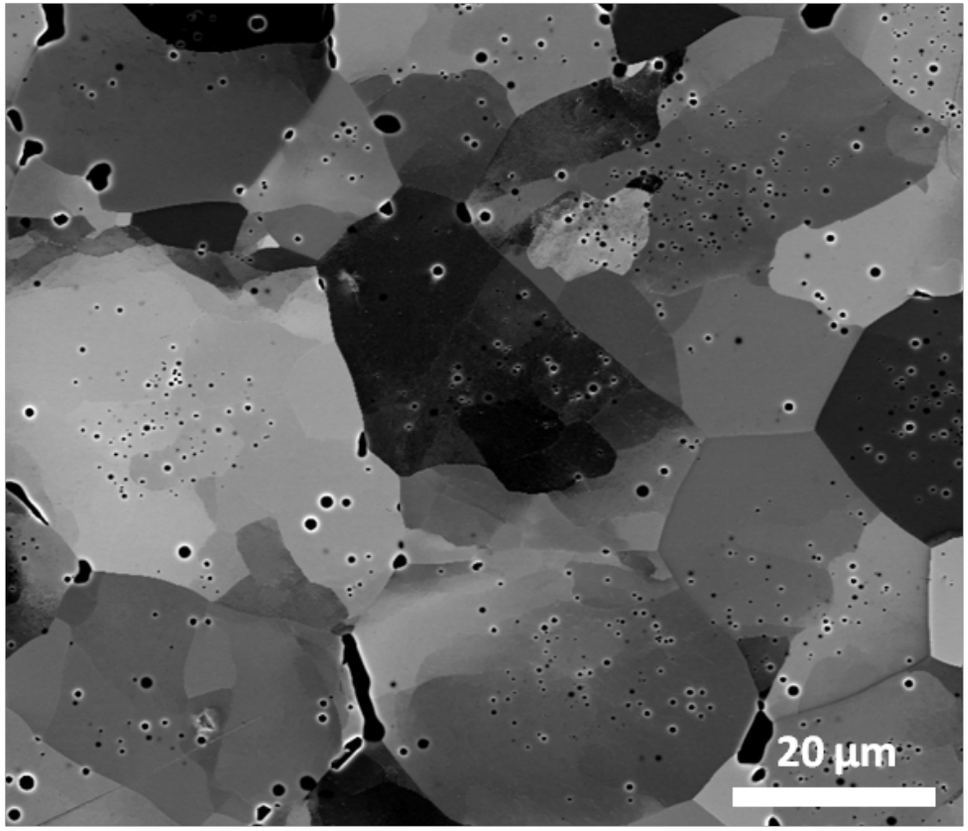
• Bande Slope index was used to identify pixels indexed over voids and set them to non indexed

• Kuwahara filter was applied to reduce the orientation noise**

*K. Thomsen , N.H. Schmidt , A.Bewick , K. Larsen and J. Goulden, Improving the Accuracy of Orientation Measurements using EBSD, Oxford note

** F.J. Humphreys, P.S. Bate, P.J. Hurley, Orientation averaging of electron backscattered diffraction data, J. Microsc. 201 (2001) 50–58

RESULTS: EBSD analysis

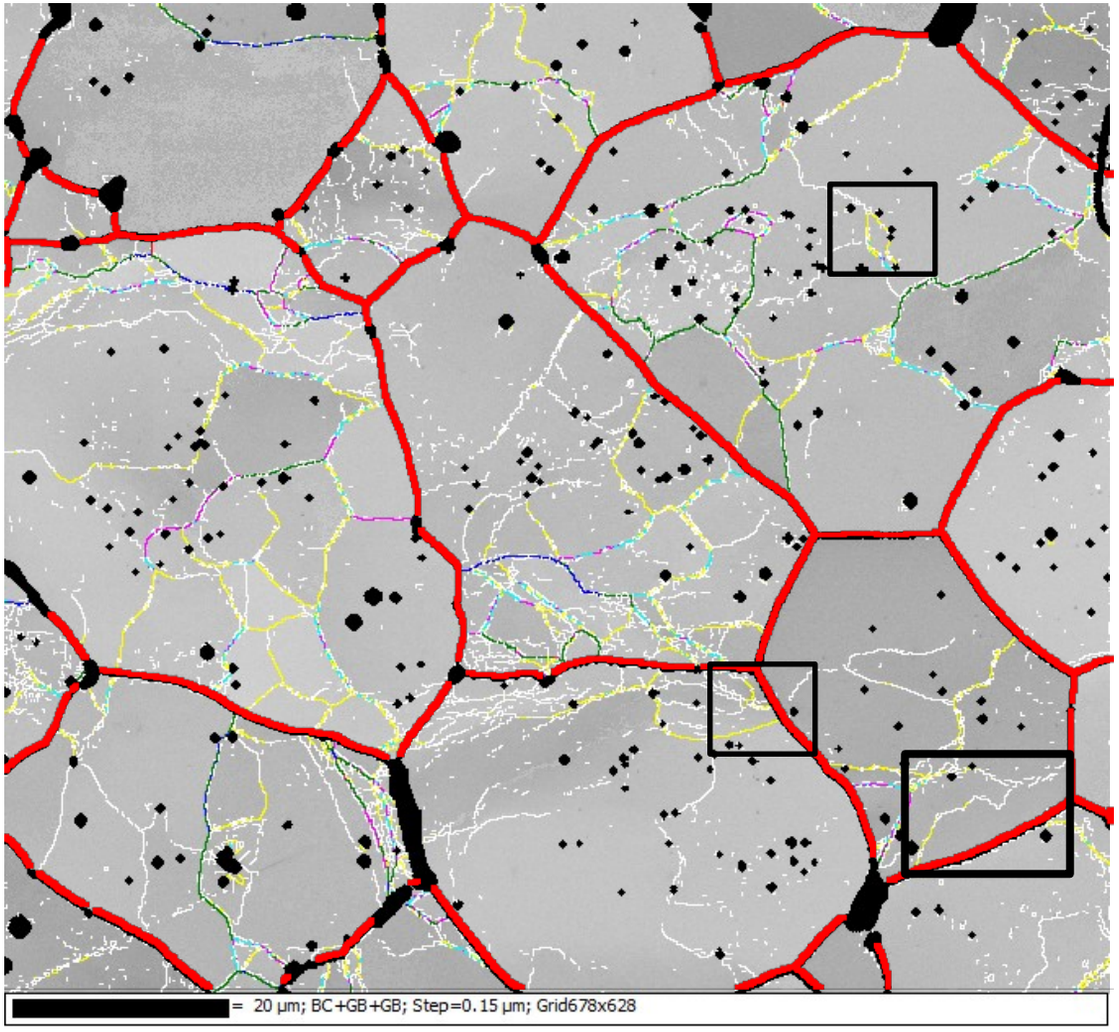


BSE micrograph of an UO_2 sample tested at 1500°C, under 50 MPa

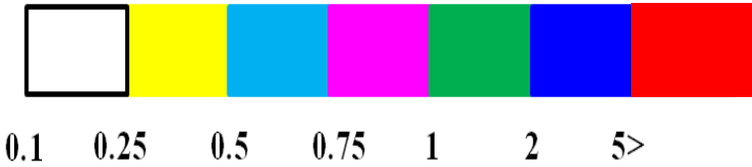
The deformation induced a network of low angle boundaries

EBSD resolves low angle boundaries with misorientations down to 0.1° !

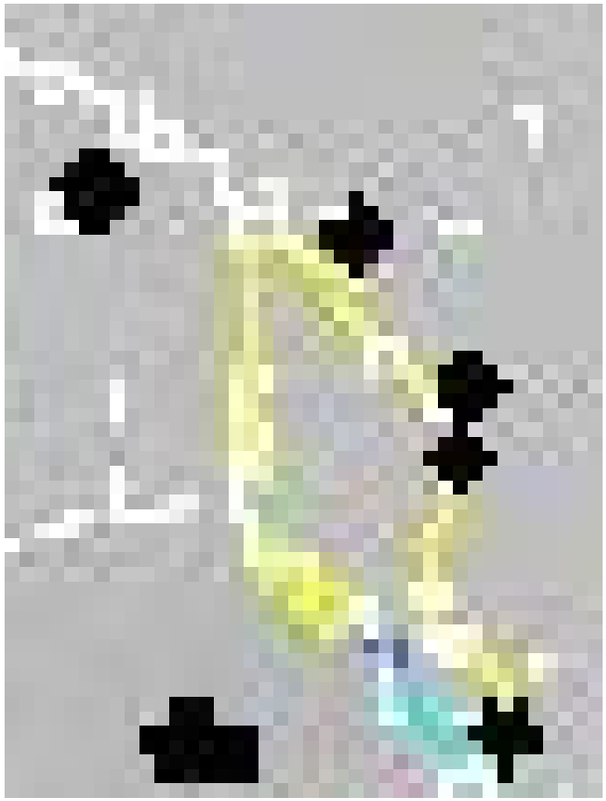
Misorientation map

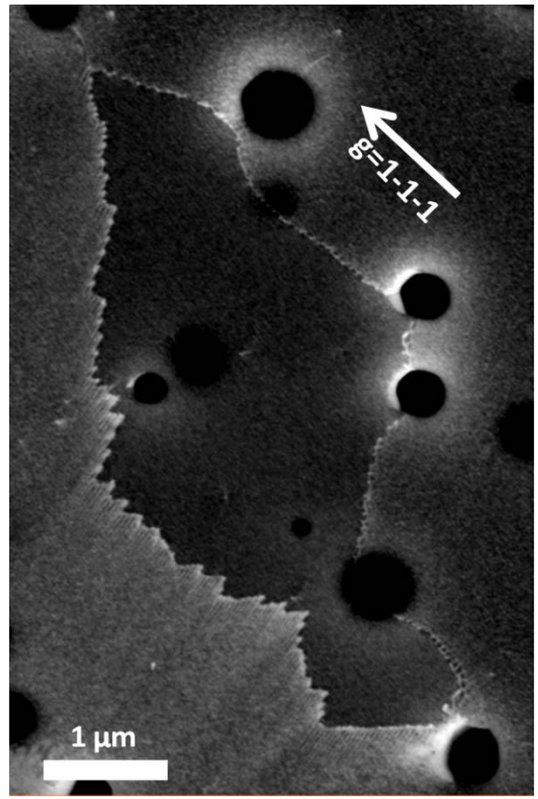


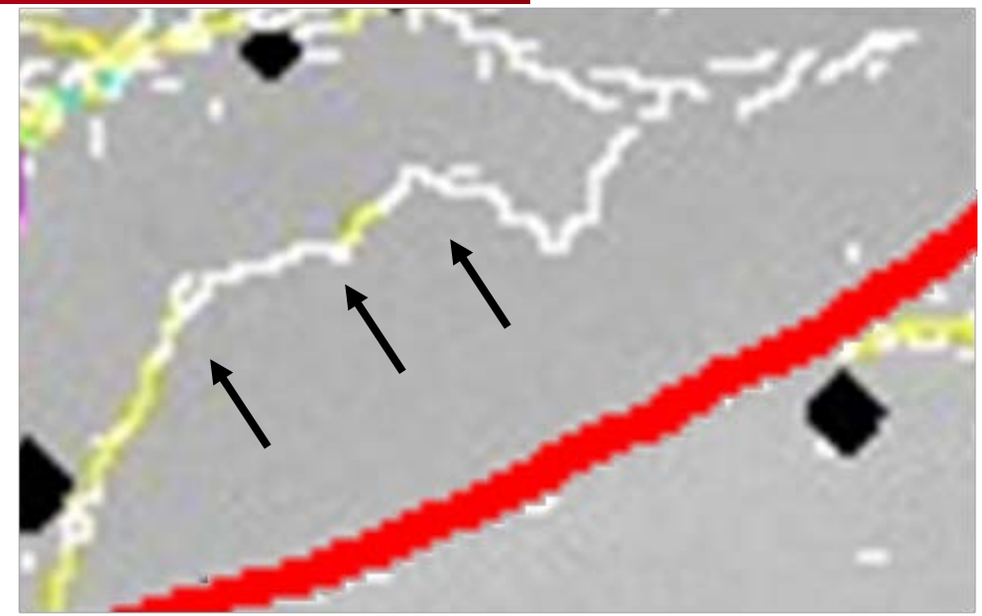
Misorientation angle Θ
Color code



Sub-boundaries with low misorientation angles confirmed by ECCI

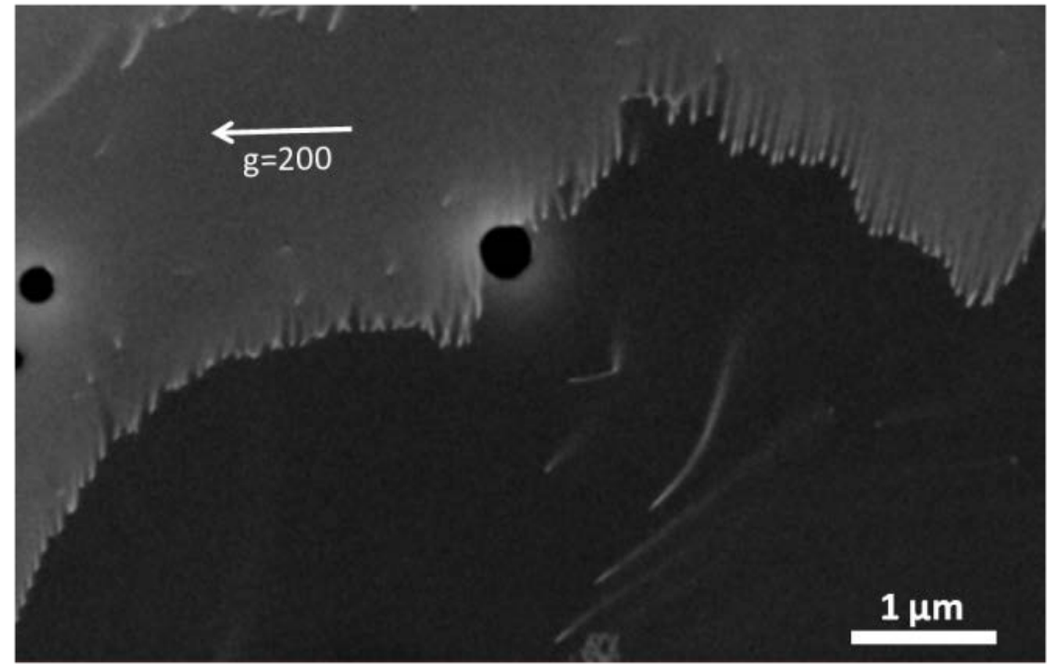




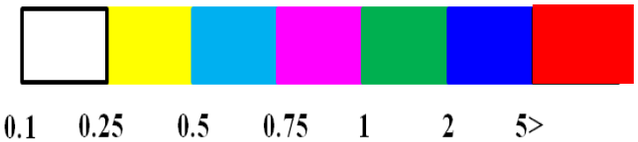


$\Delta\theta \in [0.1° \text{ to } 0.5°]$

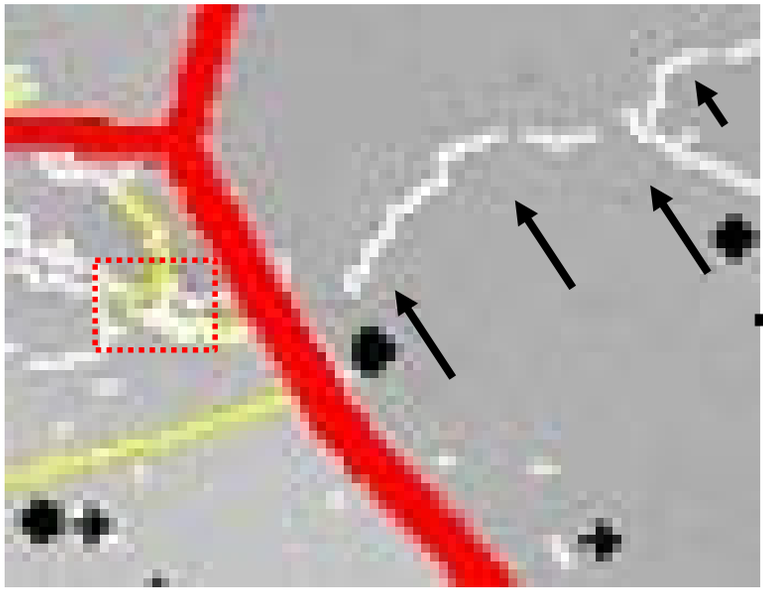
$\Delta\theta \in [0.25° \text{ to } 0.5°]$



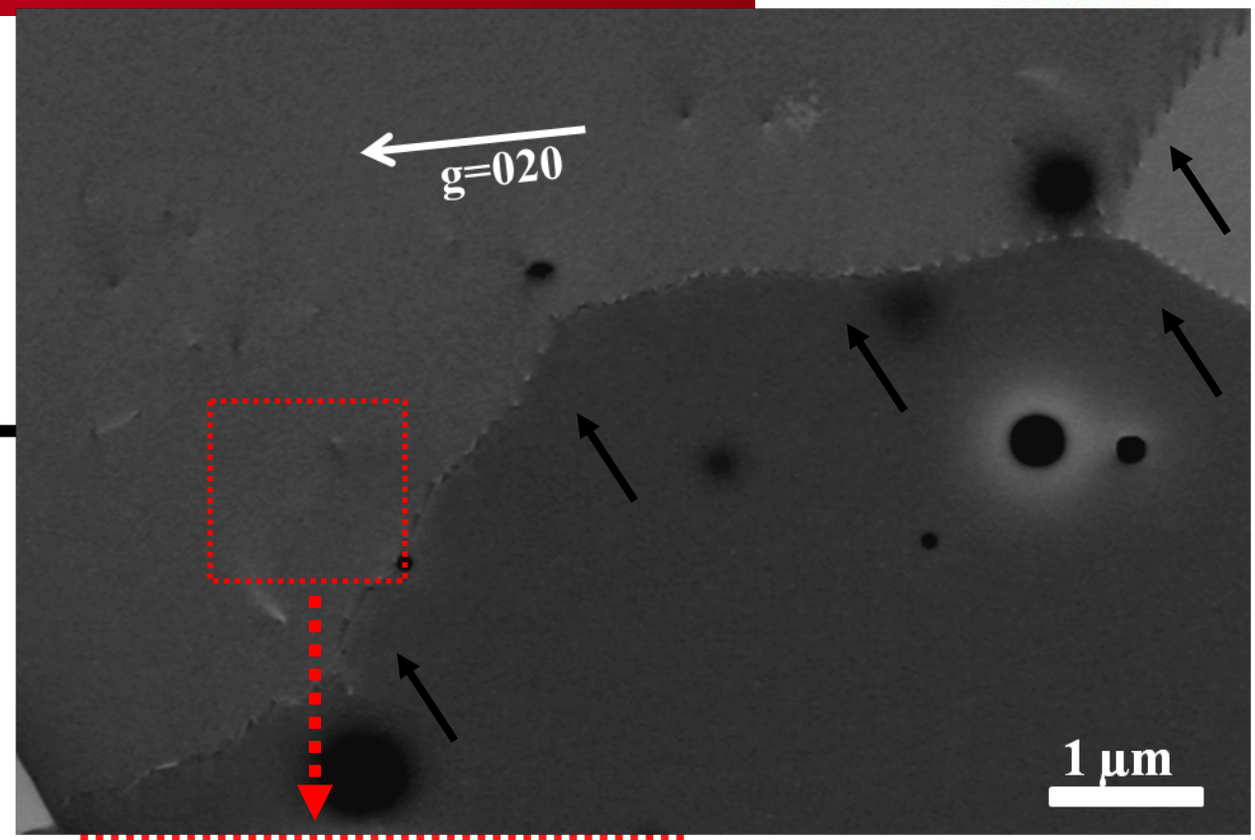
Misorientation angle Θ
Color code



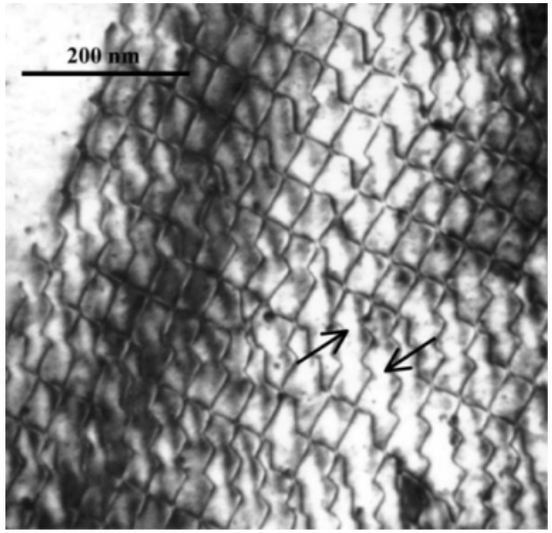
Sub-boundaries with low misorientation angles confirmed by ECCI



ECCI

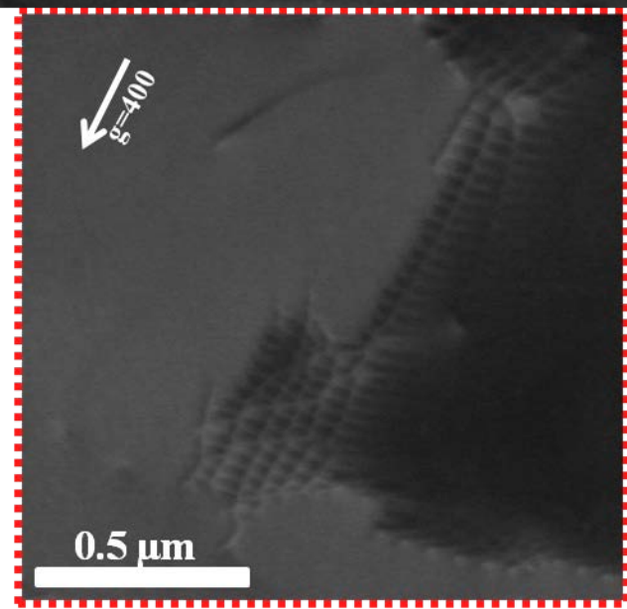


Previous TEM results



F. Dherbey *et al. 2002*

=



Dislocations Network

The GNDs: Geometrically Necessary Dislocations

The lattice curvature measured by EBSD can be used to evaluate the local density of GNDs :

From the disorientation between two neighboring points : $\Delta\theta_i$

separated spatially by Δx_j,

the lattice curvature: $K_{ij} = \frac{\Delta\theta_i}{\Delta x_j}$

Dislocation density tensor [Nye]:

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

and the total GND's density: $\rho_{total} = \frac{\|\alpha_{ij}\|}{|b|}$

b=a/2<110>

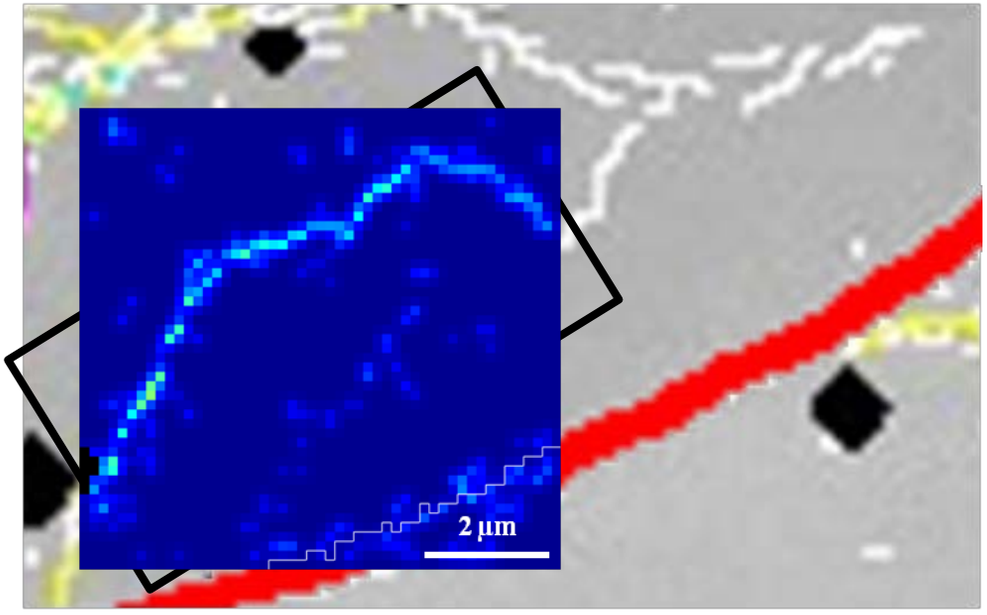
LEM3 Software

This software is developped by Benoit Beausir and Jean-Jacques Fundenberger

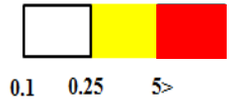
Laboratoire d'Etude des Microstructures et de Mécanique des Matériaux, Université de Lorraine/CNRS, Ile du Saulcy, 57045 Metz Cedex 01, France

RESULTS: Sub-boundaries and GNDs quantification

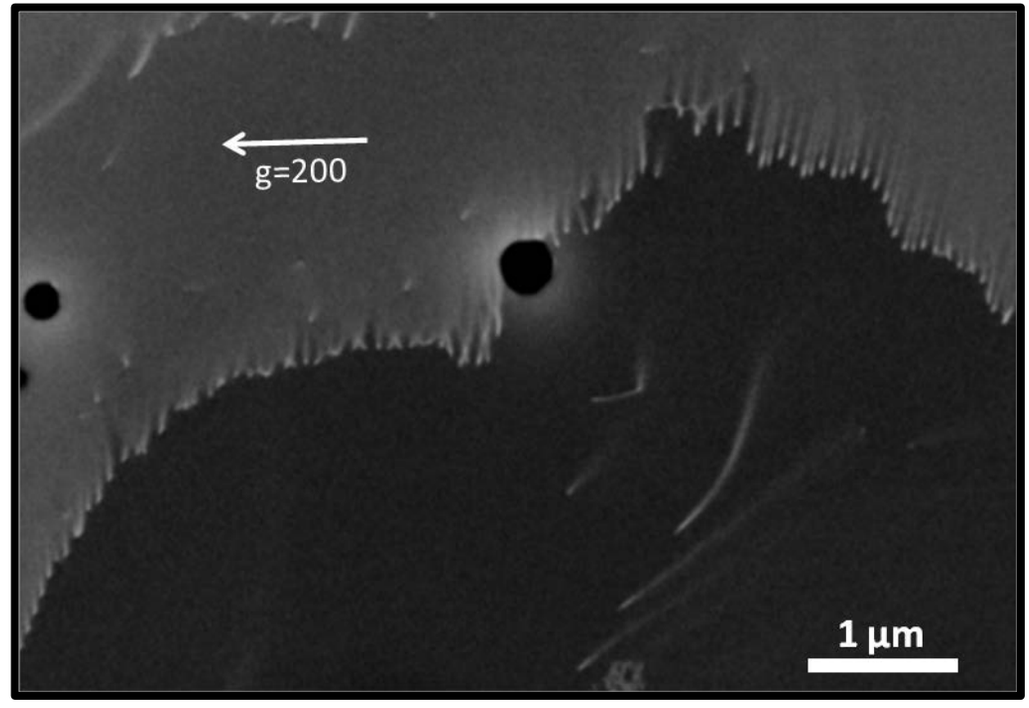
EBSD: GND evaluation from lattice curvature



$\Delta\theta \in [0.1° \text{ to } 0.5°]$



ECCI: Dislocations arrangement in low angle boundaries



ρ_{GNDs}(min/max) = [1.47- 12.3]x 10^{13} m^{-2}

$\rho_{ECCI/average}$= 8x$10^{13}$$m^{-2}$

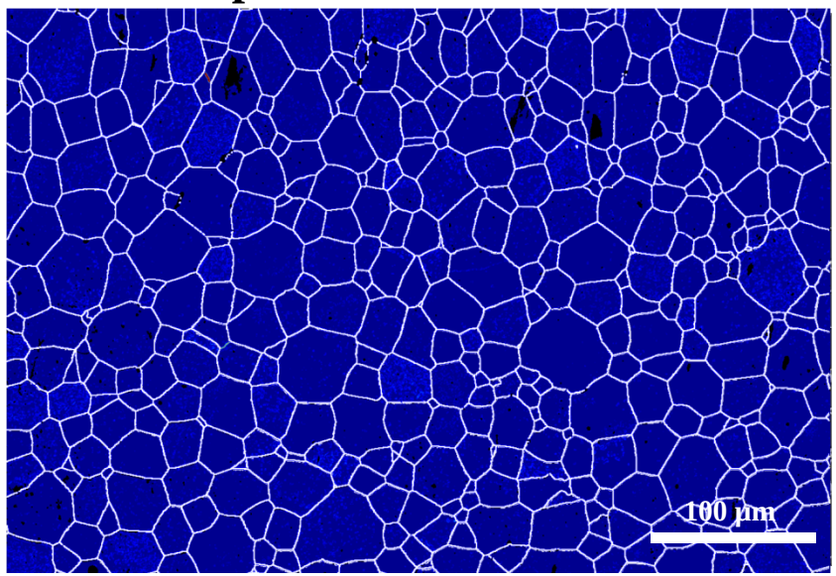
Δx_j= 150nm

0e0 1.29x$10^{14}$$m^{-2}$

RESULTS: Sub-boundaries and GNDs quantification at large scale

EBSD (after GND filtering)

As sintered sample: not deformed

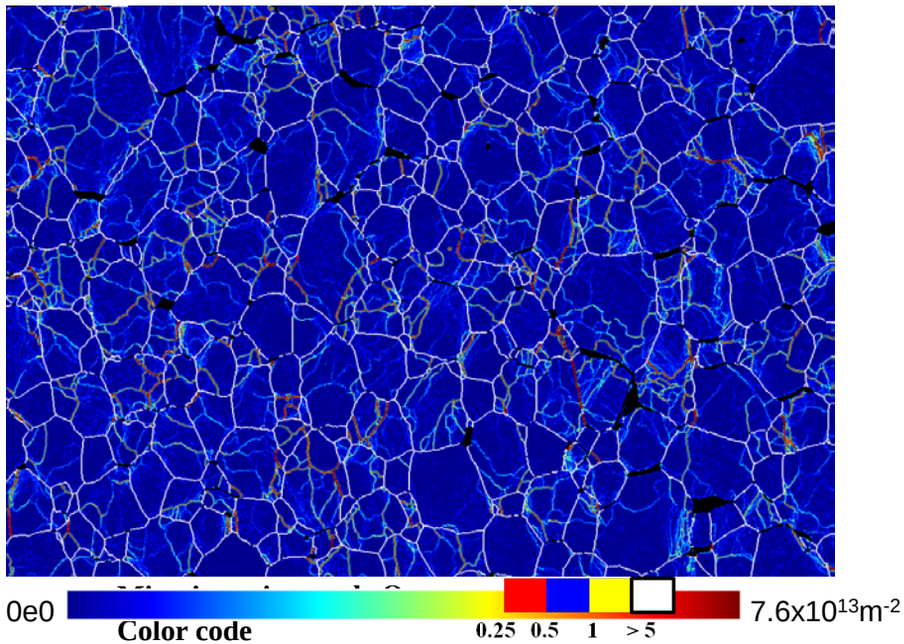


$\rho_{GNDs} = 6x\ 10^{11}\ m^{-2}$

This represents the spurious GND density induced by the noise

10 time higher

Deformed sample at 8% in the dislocational creep regime



$\rho_{GNDs} = 6.45x\ 10^{12}\ m^{-2}$

$\Delta x_j = 400nm$

CONCLUSION: EBSD and ECCI are powerful tools to analyse creep damage in UO_2

- EBSD: Quantification of low angle boundaries down to 0.1°,

  Their evolution with the creep deformation,

  Evaluation of the GNds

- ECCI: Imaging of the dislocations in the low angle boundaries

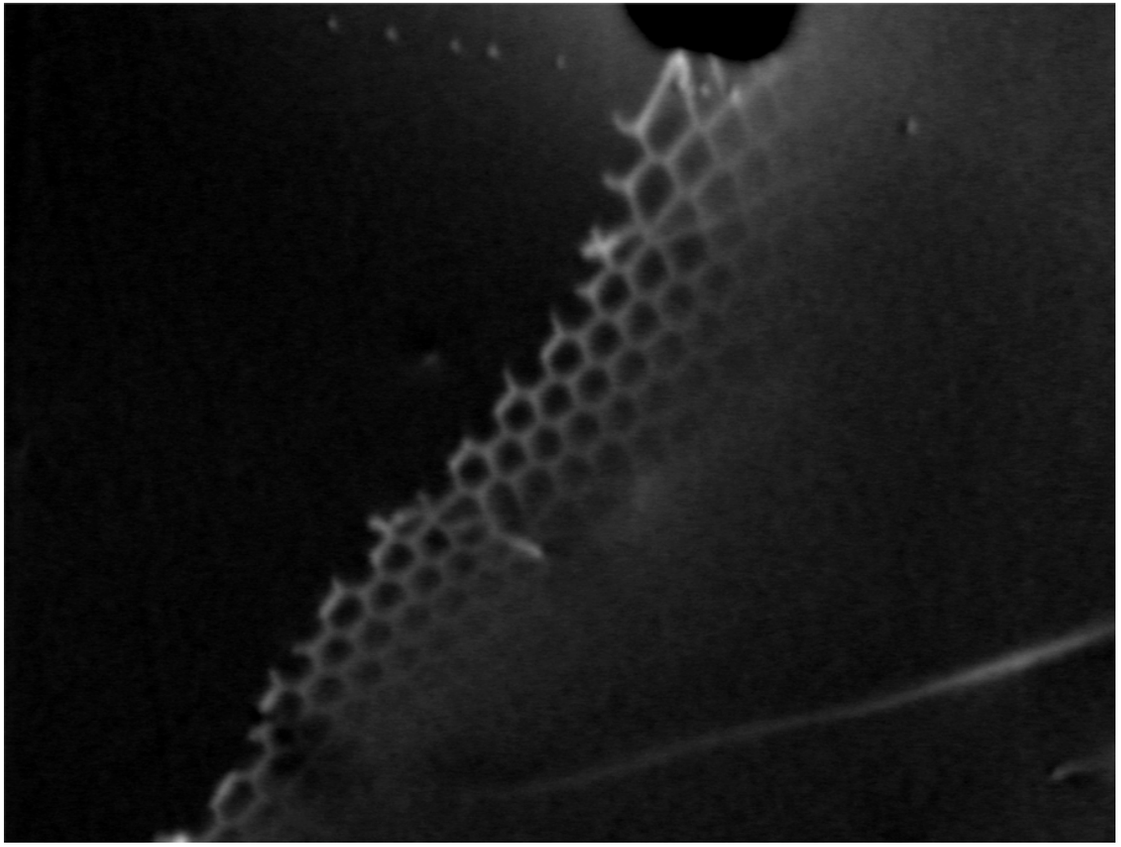
  with a TEM quality image but in bulk material,

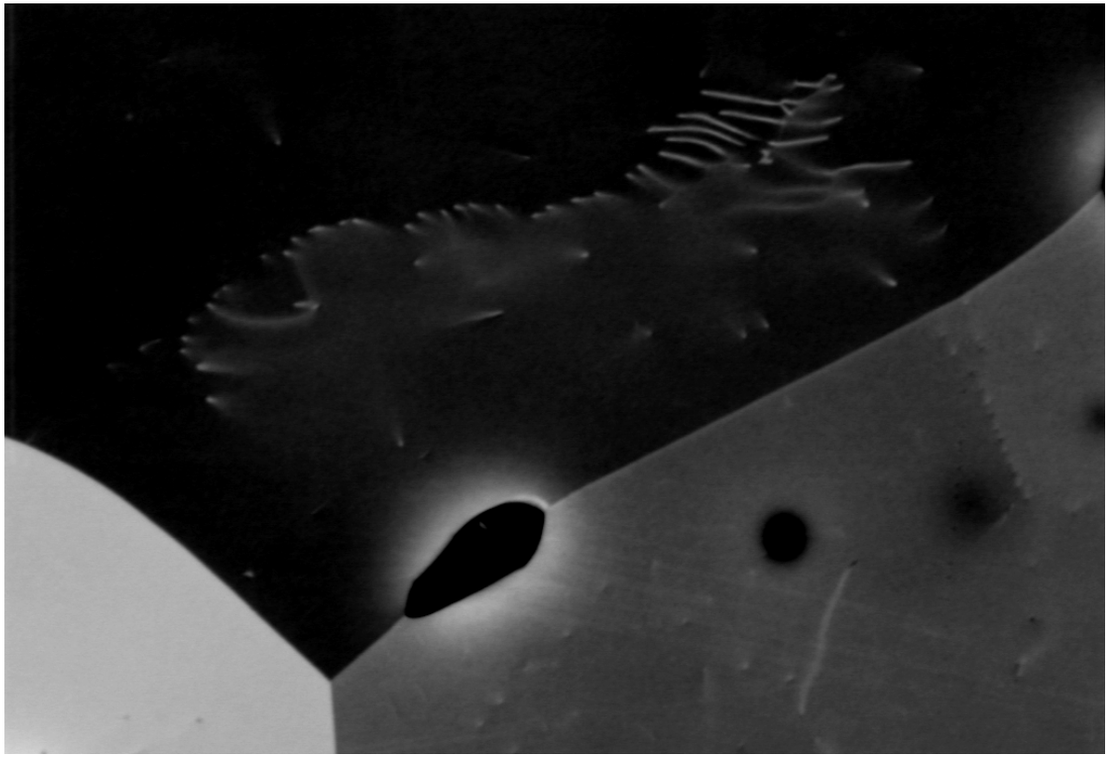
  Analyse their arrangement in networks (Hexagonal, square…) ,

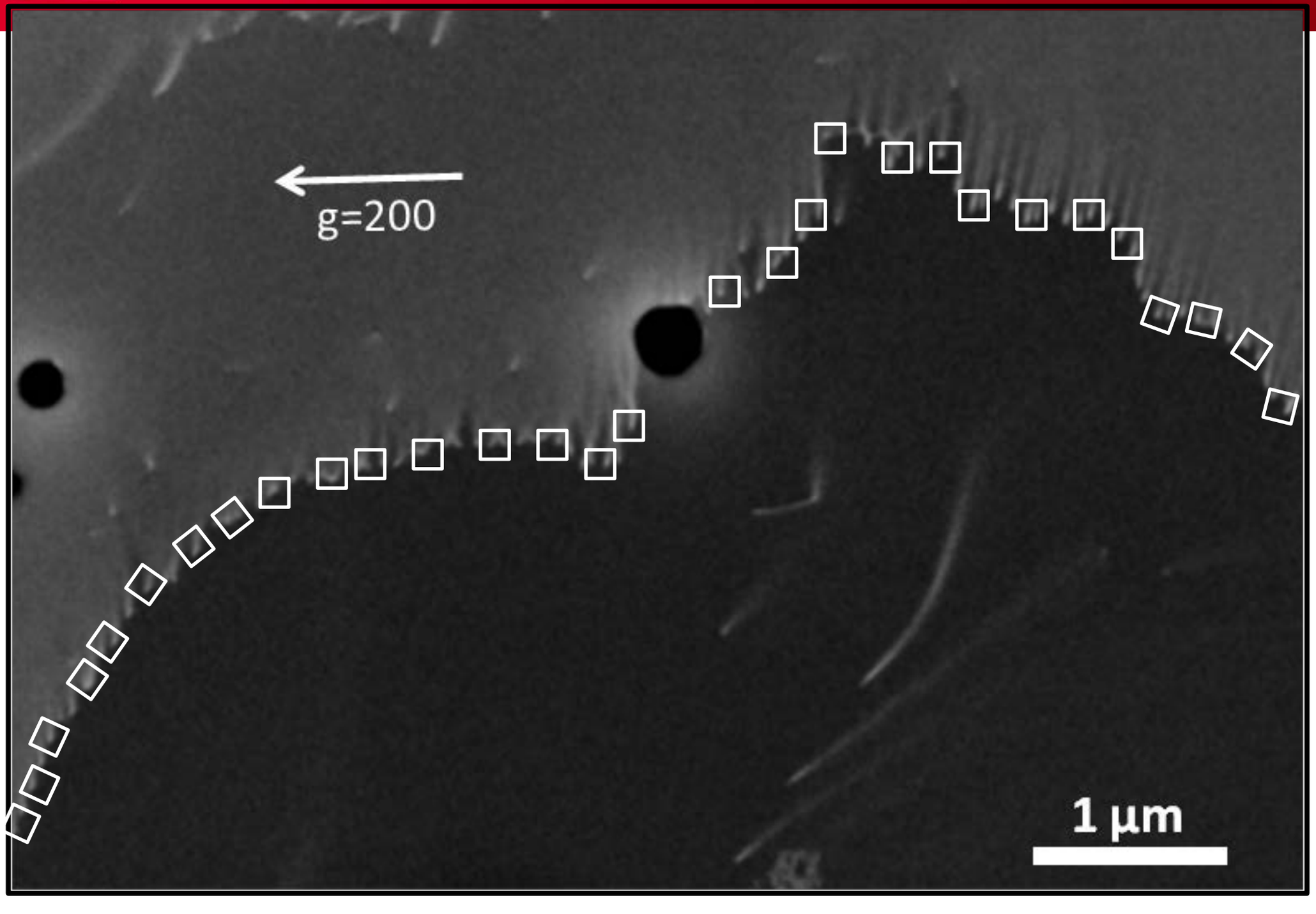
  Deduce the dislocation density

On coming work, we applied EBSD/ECCI to quantify the evolution of microstructures and sub-boundaries for different creep conditions

Thank you for your attention



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1 µm