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DE LA RECHERCHE À L'INDUSTRIE



(1)



(2)



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

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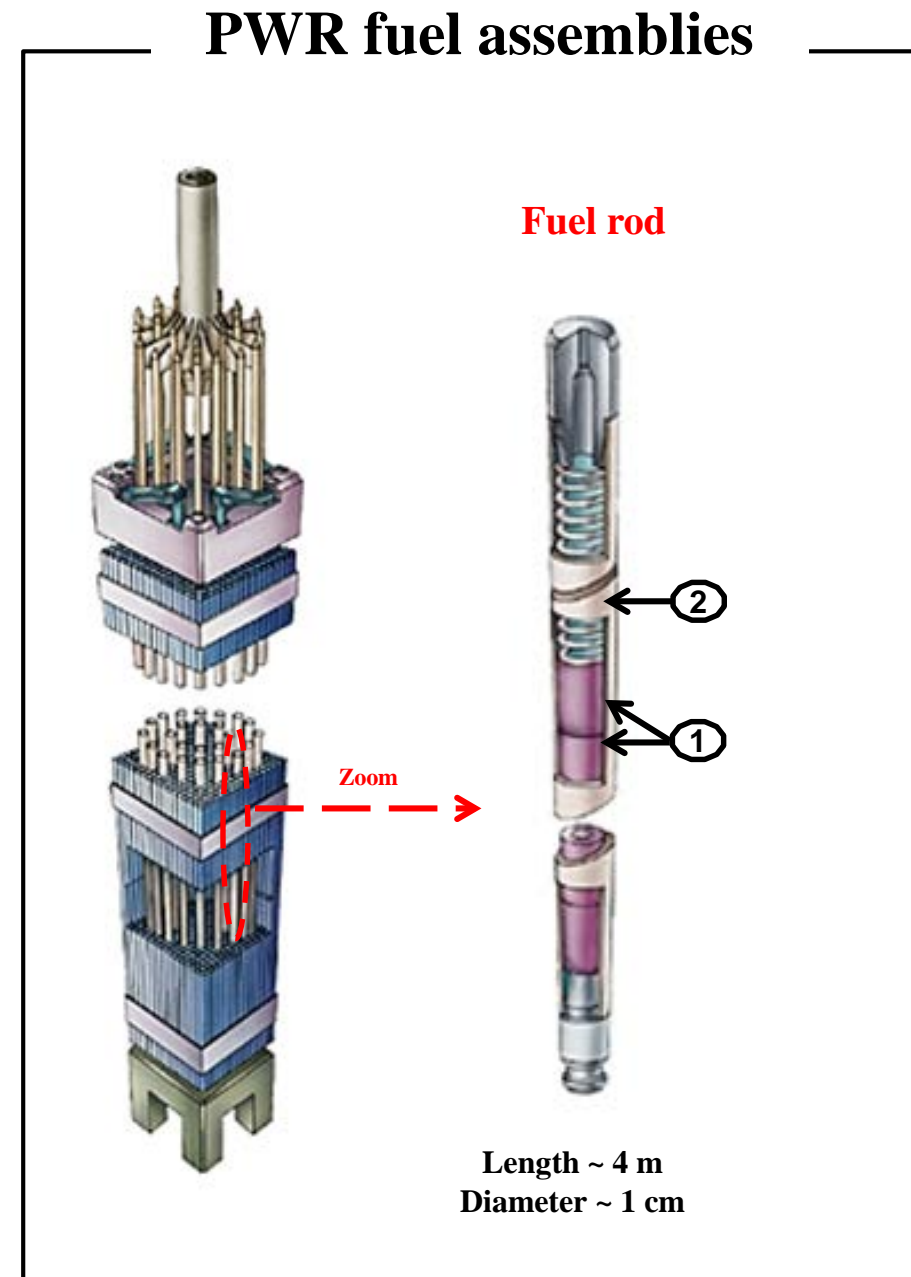
(1) CEA Cadarache, DEN/DEC/SFER/Laboratoire des Combustibles Uranium, 13108 Saint Paul Lez Durance, France

(2) LEM3, Ile du Saulcy, 57045 Metz, France

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

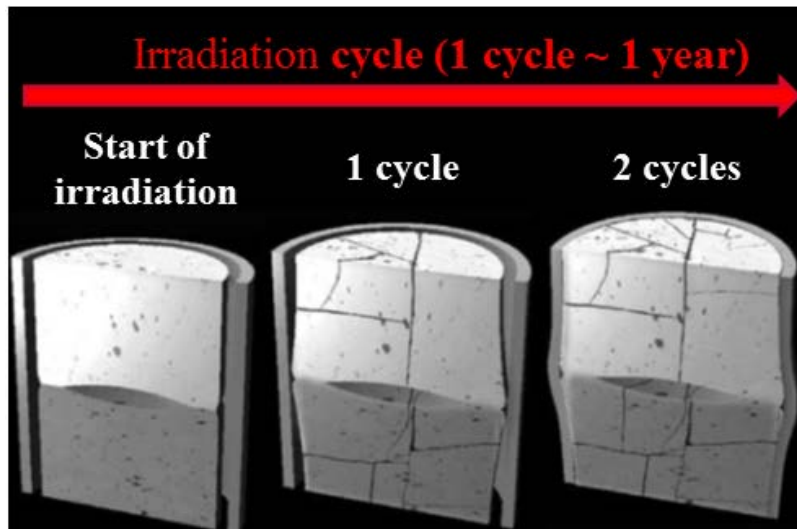
The fuel rods consist of:

- (1) UO_2 pellets piled up in
- (2) a zirconium alloy cladding tube cooled by pressurized water



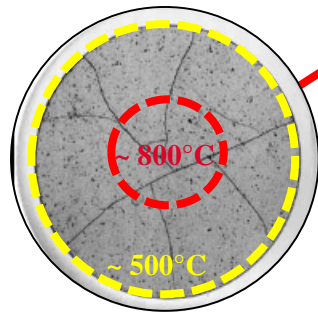
In service the UO_2 pellets undergo significant plastic deformation by creep

Nominal conditions



9,5 mm

Under irradiation:

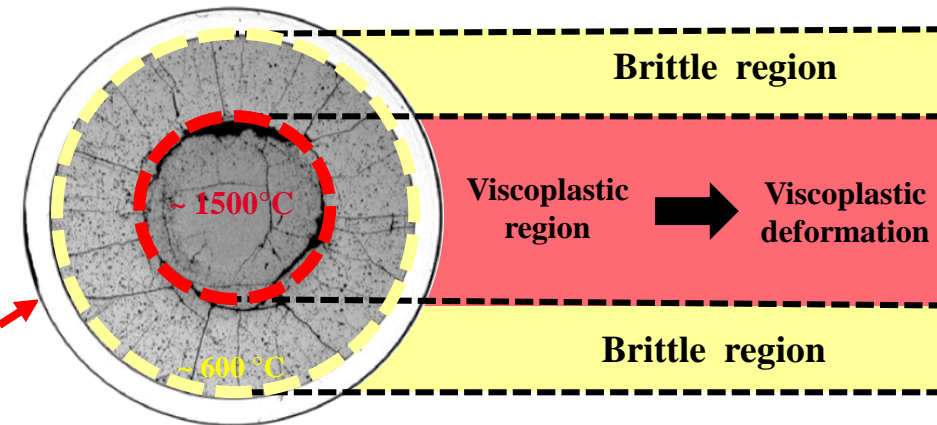


thermal gradient + fission products

→ 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

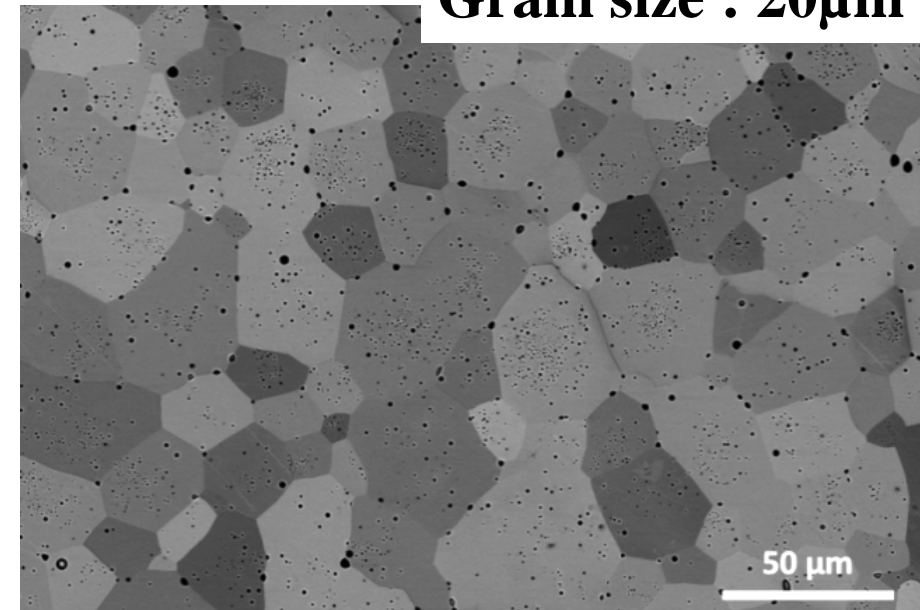
Grain size : $20\mu m$

Sintering conditions [1,2]

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

+

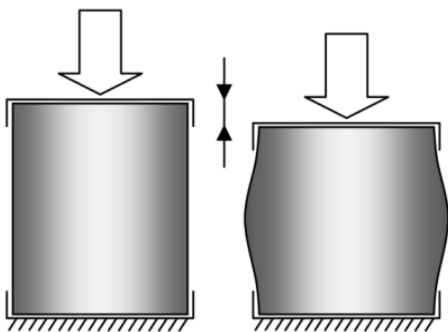
Sintering heat treatment at **$1900^\circ C$** for
4h, under an atmosphere of **Ar + 5% H_2**



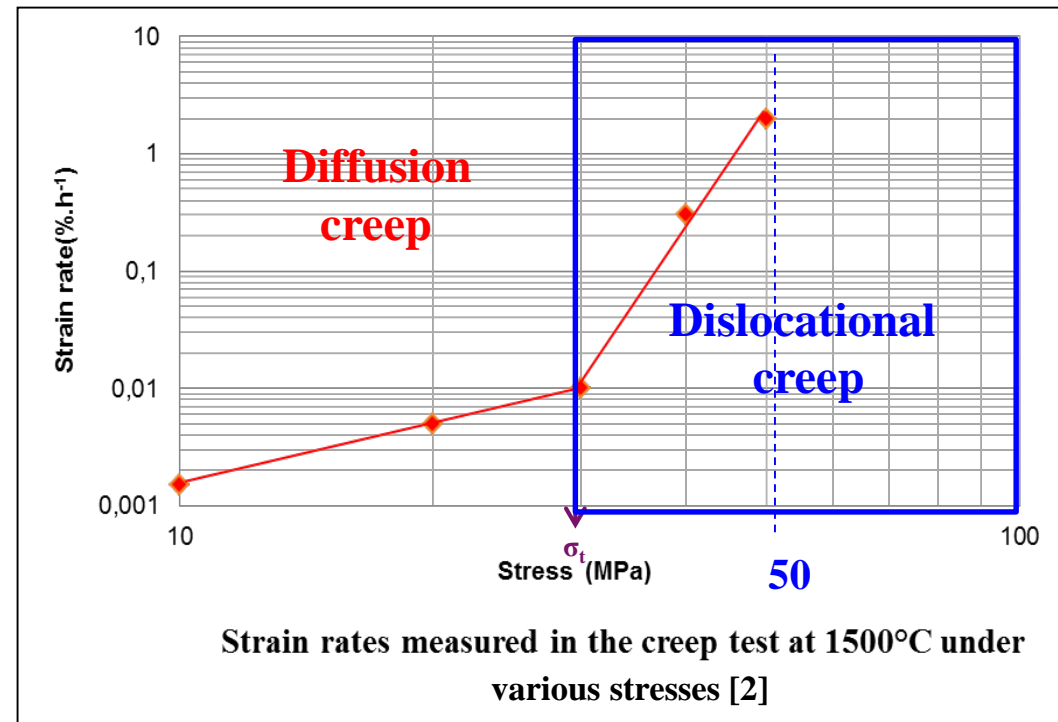
SEM micrograph (BSE mode) of an UO_2 sample

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

Our reference
sample



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

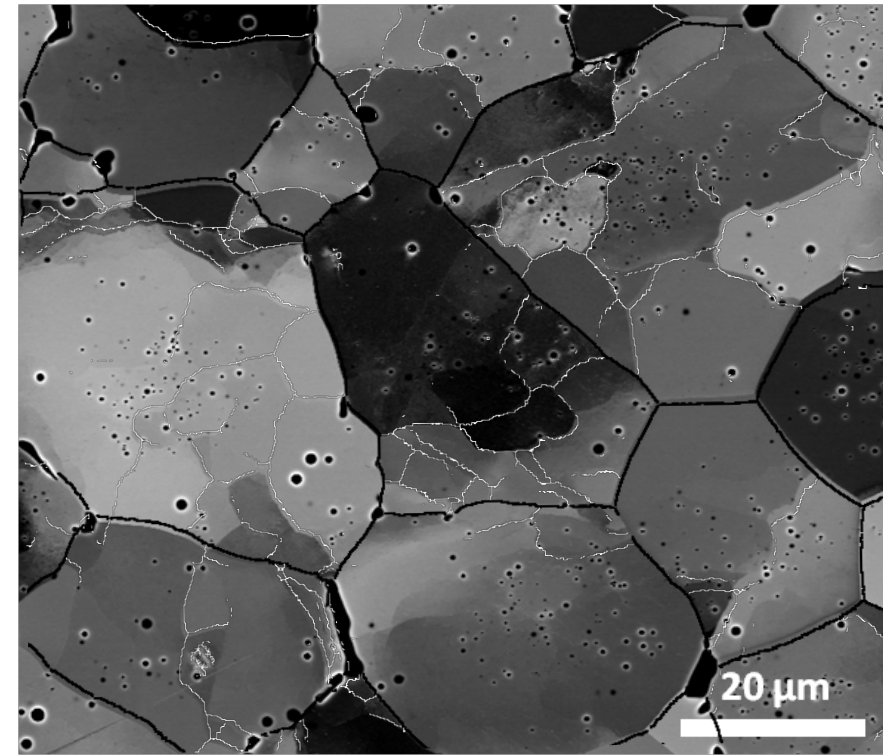


[1]. X. ILTIS, M. BEN SAADA, H. MANSOUR, N. GEY, A. HAZOTTE, N. MALOUFI, A new characterization approach for studying relationships between microstructure and creep damage mechanisms of uranium dioxide, J. of Nuc. Mat. 474 (2016).

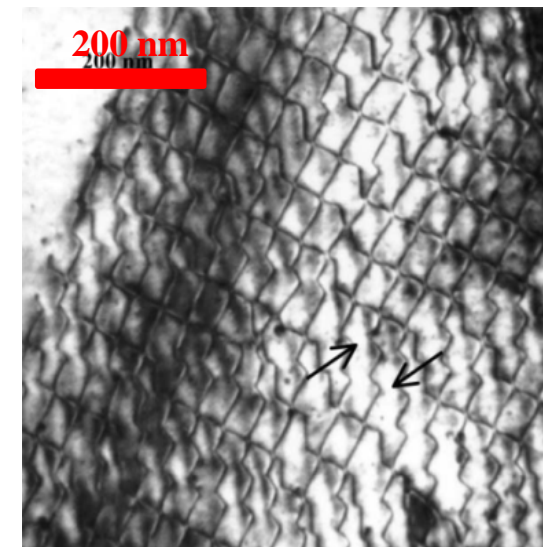
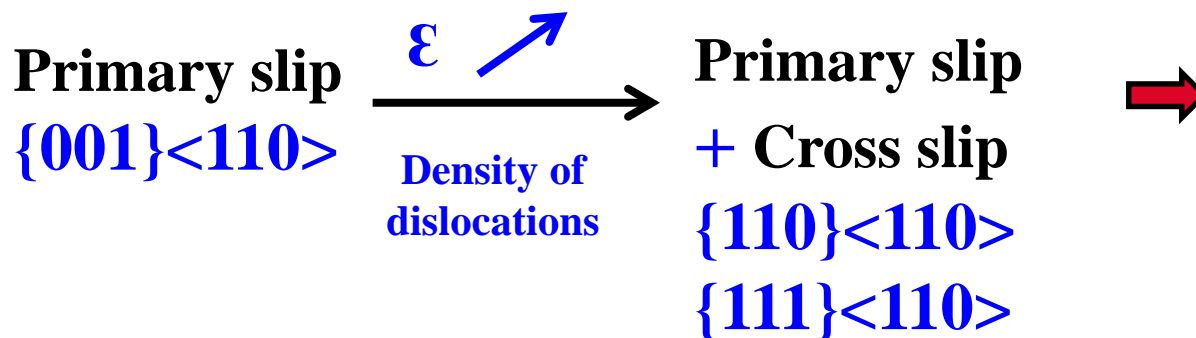
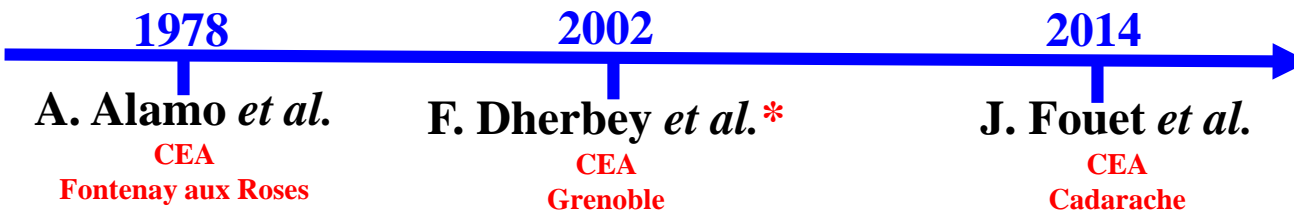
[2]. X. ILTIS, N. GEY, C. CAGNA, A. HAZOTTE, Ph. SORNAY, Microstructural evolution of uranium dioxide following compression creep tests : An EBSD and image analysis study, J. of Nuc. Mat. 456 (2015).

Creep damage

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



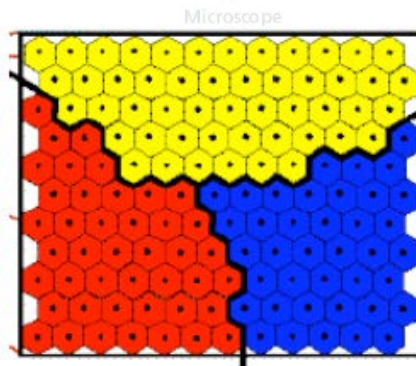
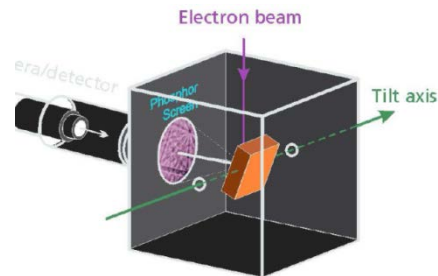
▪ **TEM studies**



Dislocation network*

EBSD

Electron BackScattered
Diffraction



Acquisition of Orientation map to:

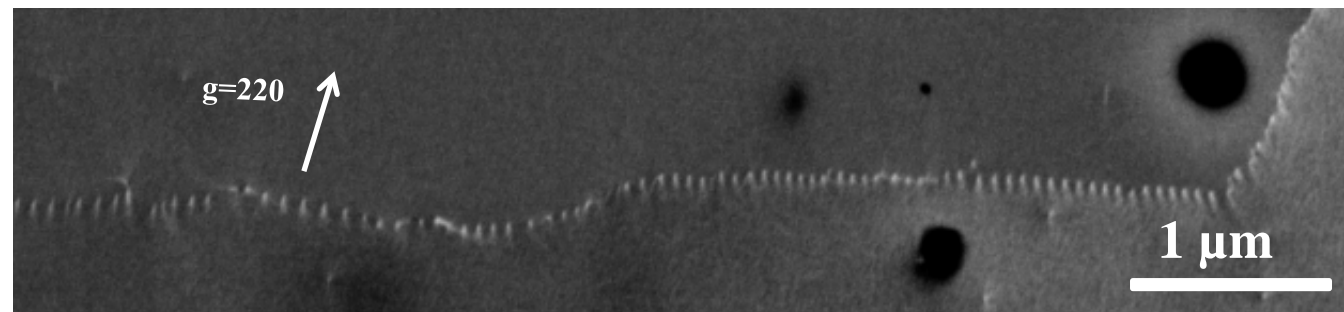
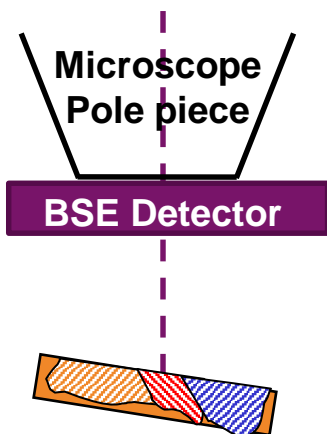
- Identify the low angle boundaries (sub-grains),
- Quantify the Geometrically Necessary Dislocations (GND)

BULK MATERIAL SEM

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

A-ECCI

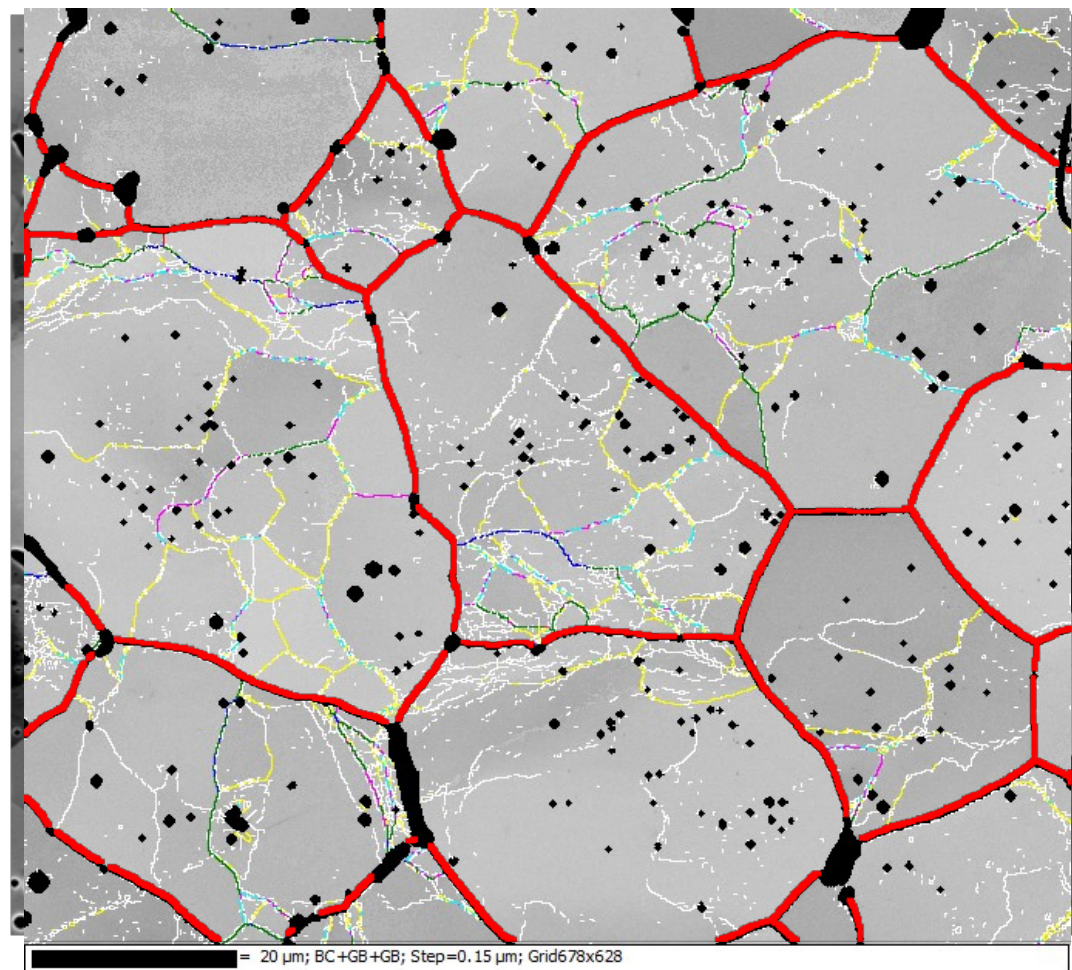
Acurrate Electron Channeling
Contrast Imaging



BSE image at high magnification in specific diffraction condition

Misorientation map

- ☹ The substructure is embedded in the **orientation noise**
- ☹ Voids are indexed with artificial misorientations with the neighborhood



BSE micrograph of an UO₂ sample tested at 1500°C, under 50 MPa



Optimize data acquisition and processing



Best angular resolution on the EBSD map

Steps:

1) Acquisition:

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

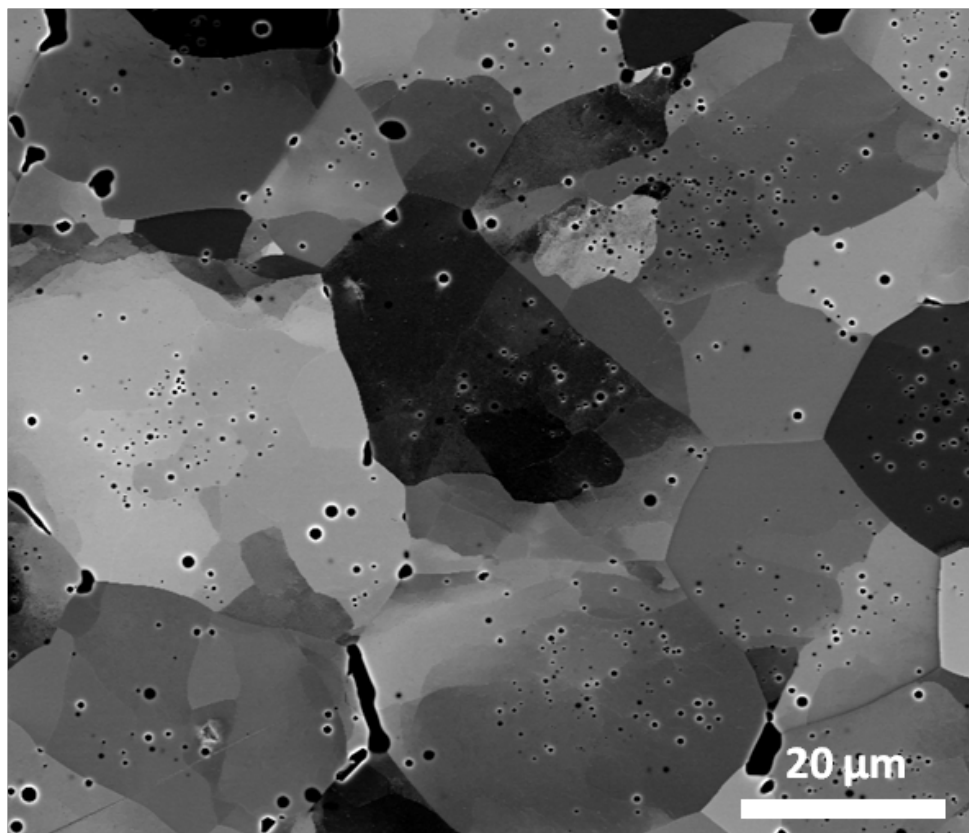
2) Data cleaning and filtering:

- 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

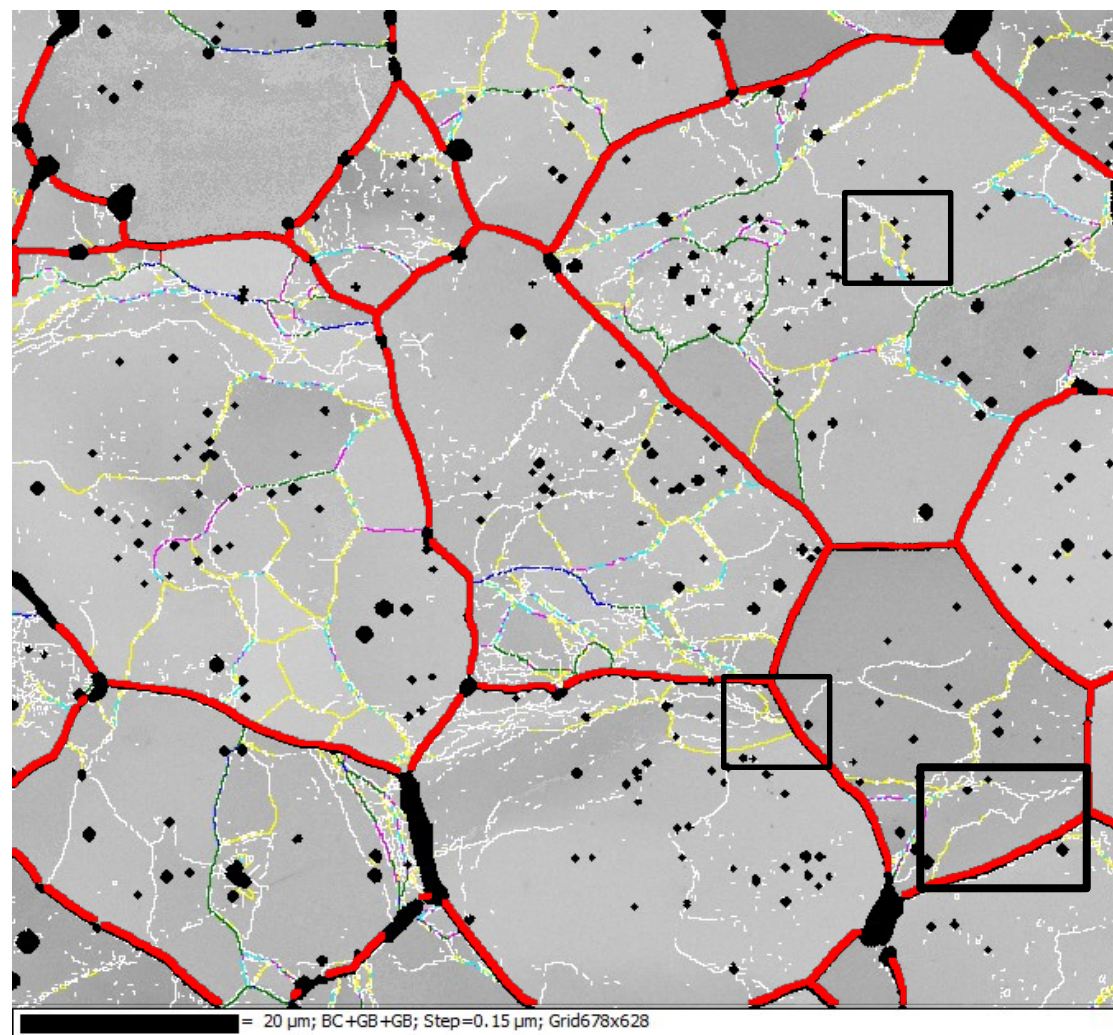
Misorientation map



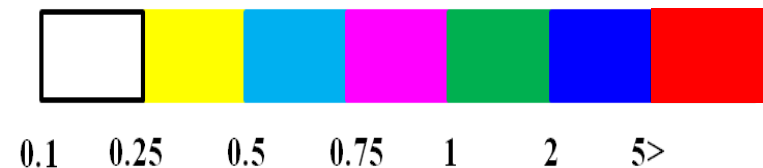
BSE micrograph of an UO_2 sample tested at 1500°C, under 50 MPa

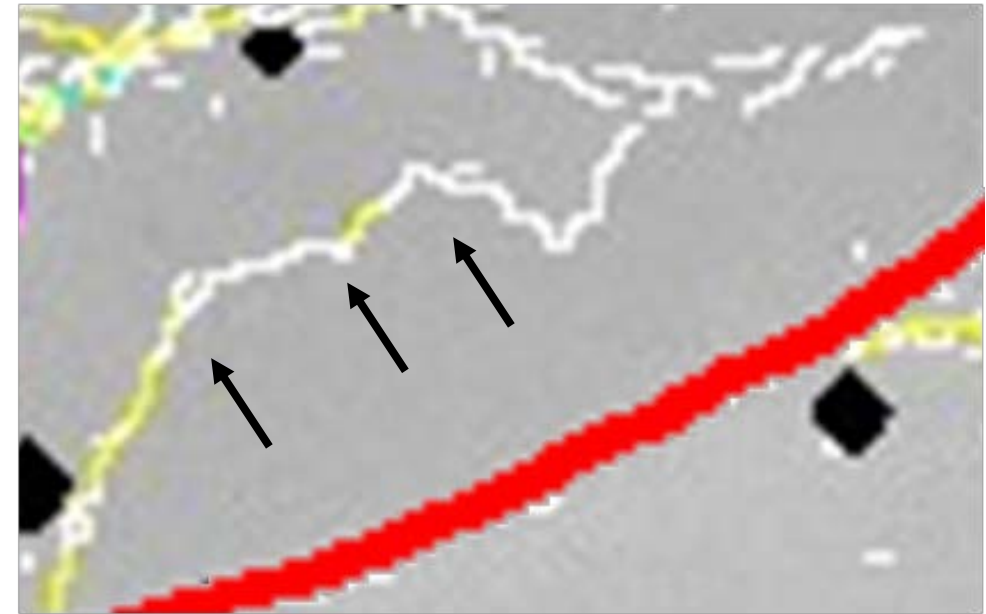
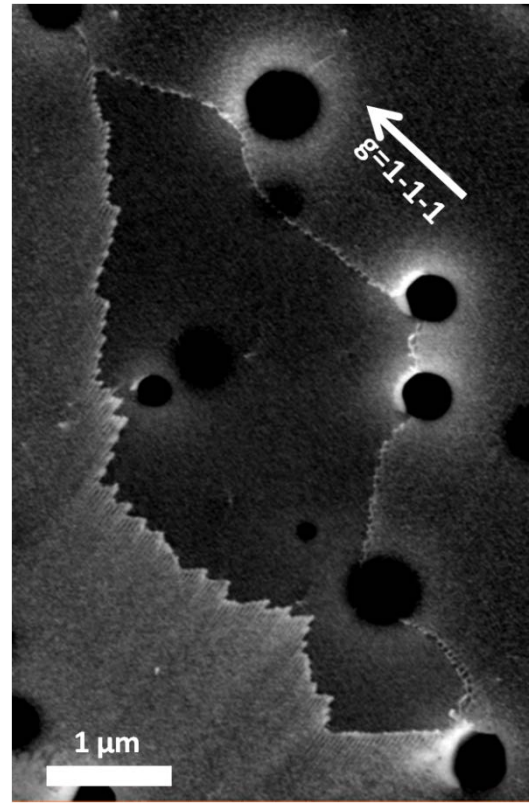
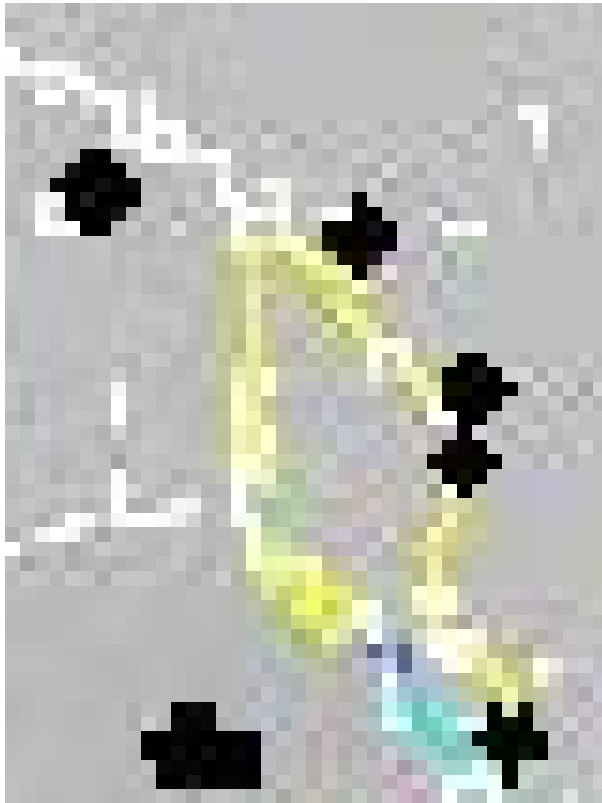
The deformation induced a network of low angle boundaries

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



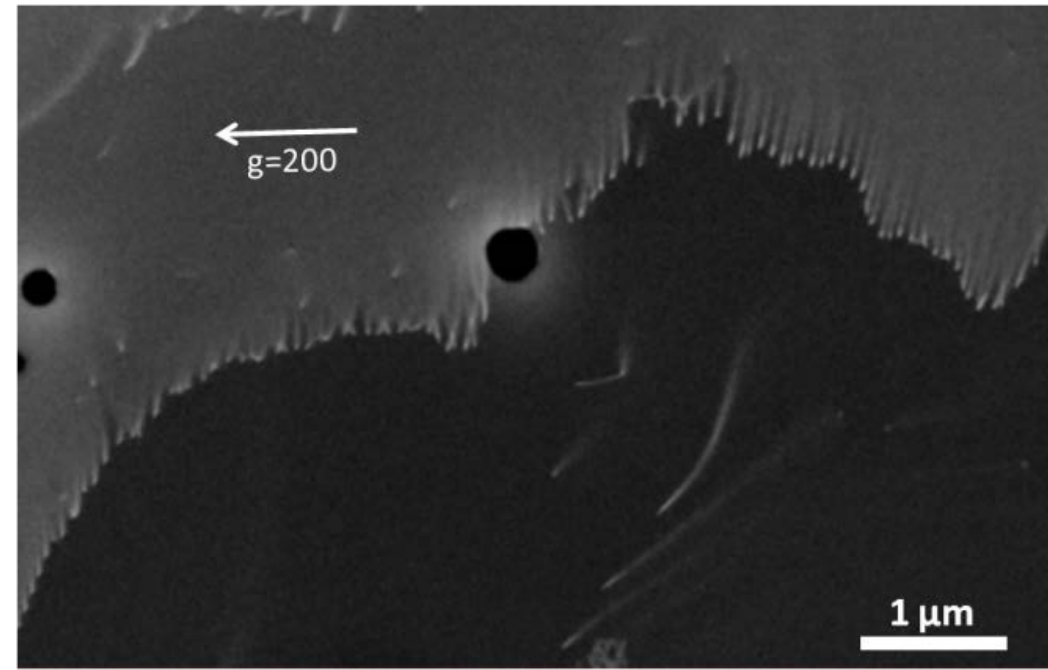
Misorientation angle Θ
Color code



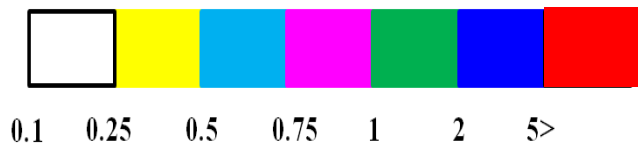


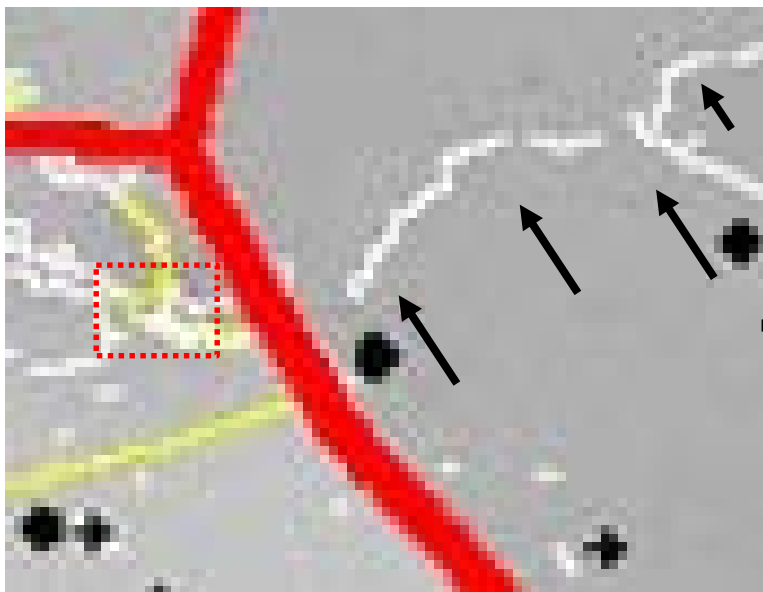
$\Delta\theta \in [0.1^\circ \text{ to } 0.5^\circ]$

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

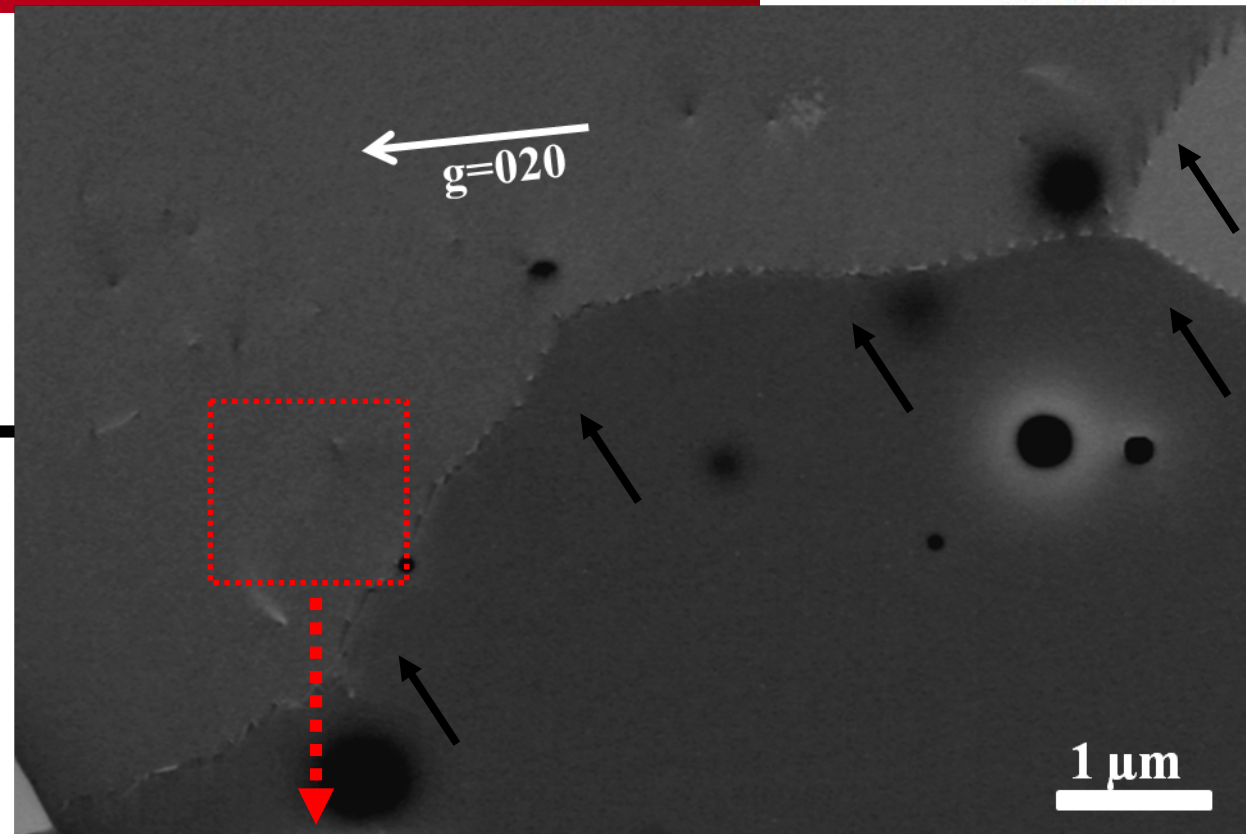


Misorientation angle θ
Color code

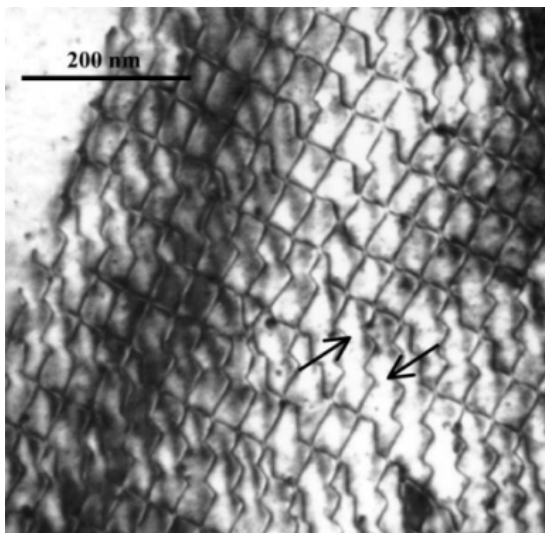




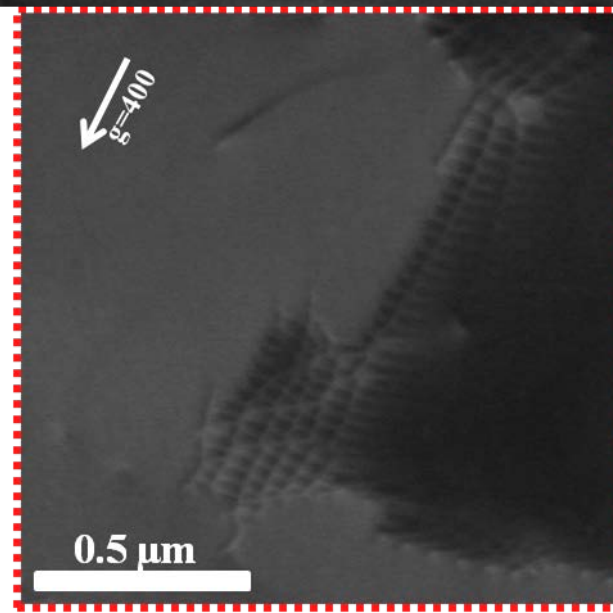
ECCI



Previous TEM results



=



**Dislocations
Network**

F. Dherbey *et al.* 2002

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: $\rho_{\text{total}} = \frac{\|\alpha_{ij}\|}{|b|}$

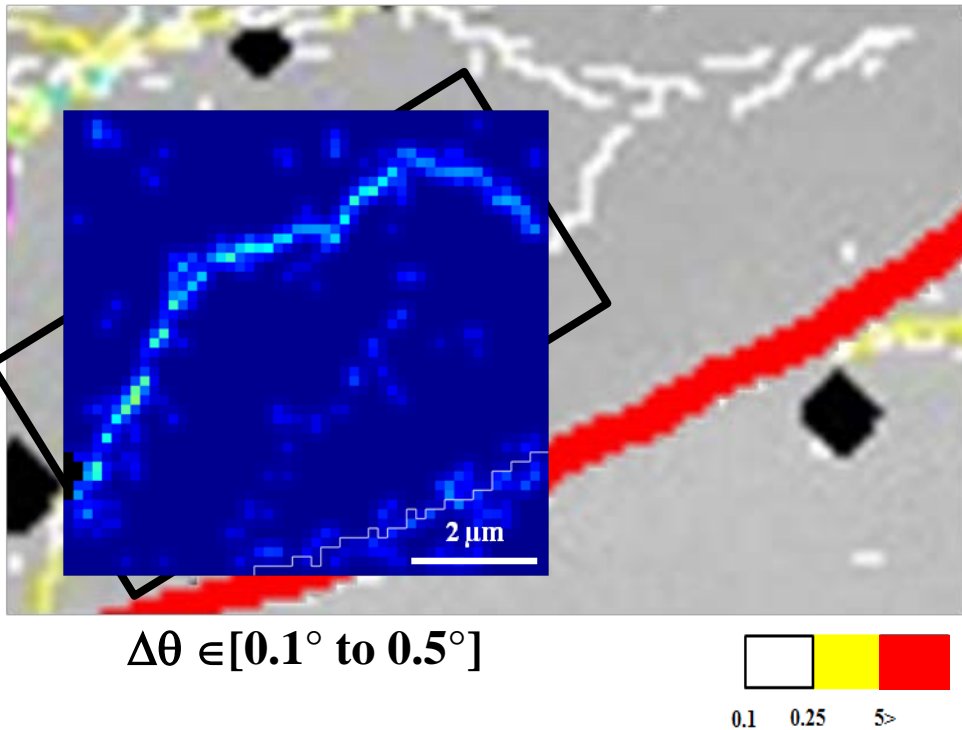
LEM3
Software



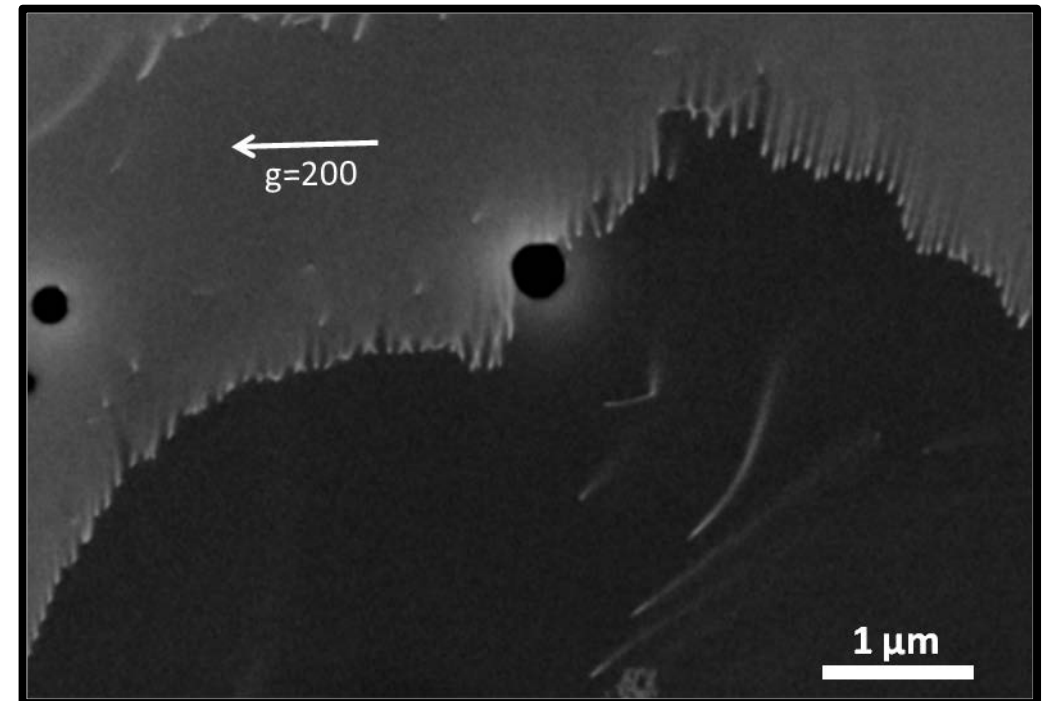
$b = a/2 \langle 110 \rangle$

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

EBSD: GND evaluation from lattice curvature



ECCI: Dislocations arrangement in low angle boundaries



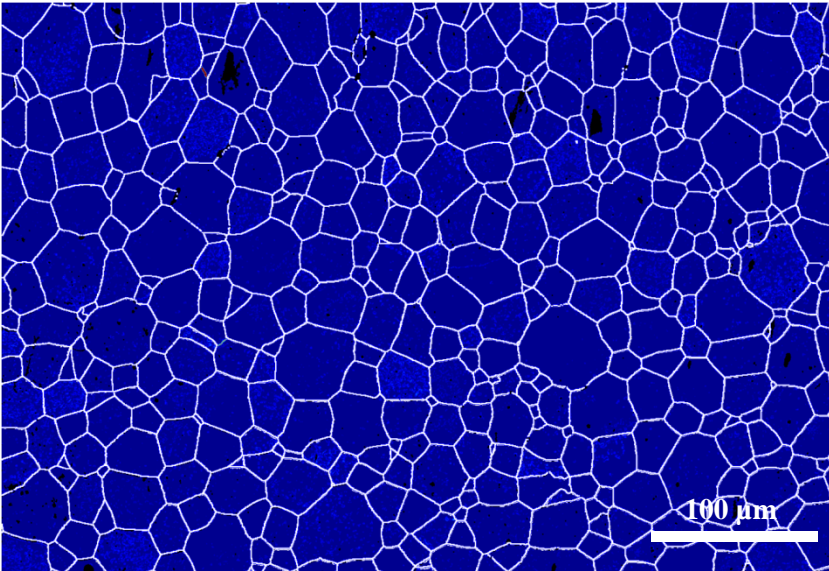
$$\rho_{\text{GNDs}}(\text{min/max}) = [1.47 - 12.3] \times 10^{13} \text{ m}^{-2}$$

$$\Delta x_j = 150 \text{ nm}$$

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

EBSD (GNDs filtering)

As sintered sample: not deformed

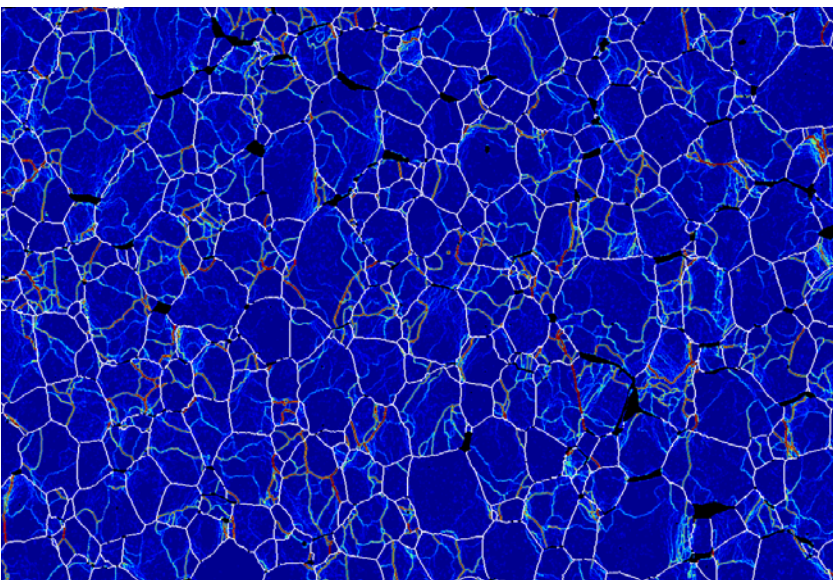


$$\rho_{\text{GNDs}} = 6 \times 10^{11} \text{ m}^{-2}$$

This represents the **spurious** GND density induced by the noise

10 time higher

Deformed sample at 8% in the dislocational creep regime



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

$\Delta x_j = 400 \text{ nm}$



- **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

