



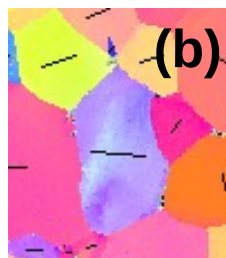
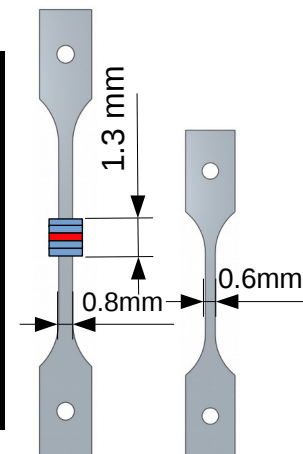
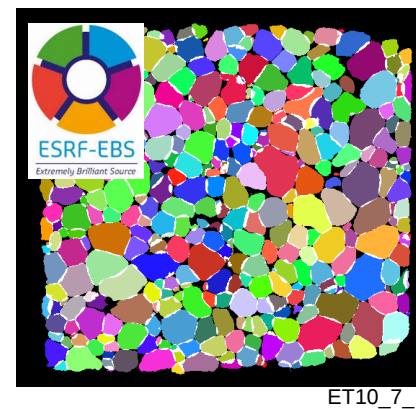
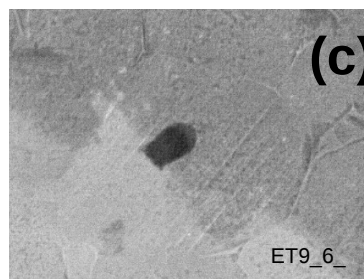
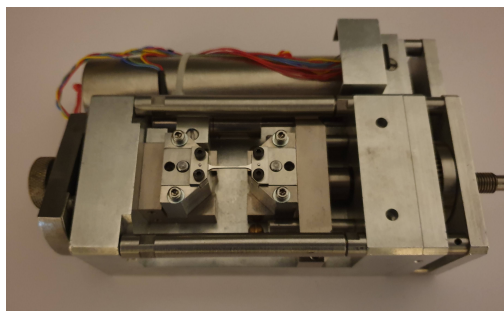
■ In situ multimodal experimental testing and simulations in volume for statistical analysis of crystal plasticity

PhD student : RIBART Clement / PhD supervisor: PROUDHON Henry
MINES ParisTech, PSL research university, Centre des Matériaux, Evry

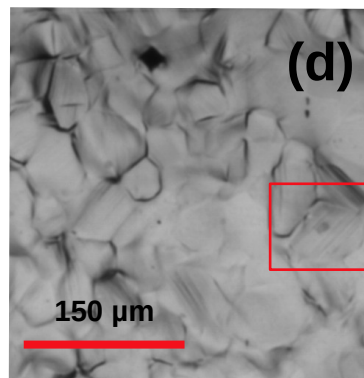
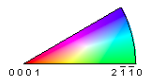
- Previous status
- Experimental data
 - Plasticity factors study
 - Synchrotron 3D DCT Reconstruction (ERSF EBS)
 - Synchrotron 3DXRD analysis (SOLEIL PSICHE)
 - Laboratory 3D DCT (Lund)
 - Metallurgy analysis
 - Samples preparation
- Outlook

Previous status (01/13/21)

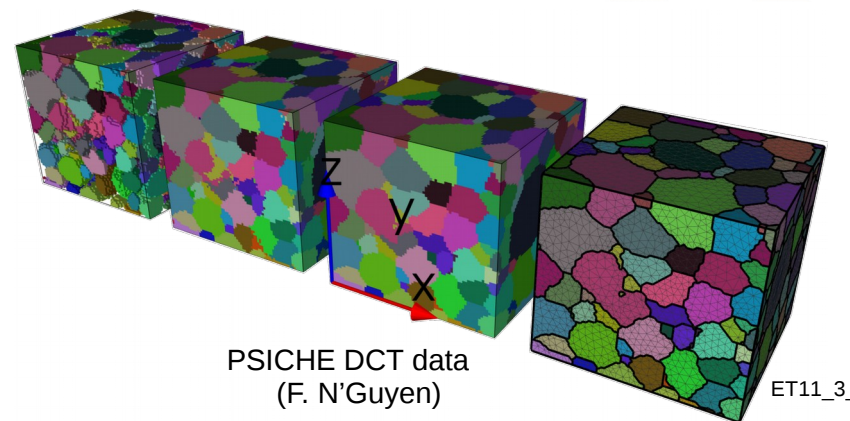
- Preliminary SEM in situ tests :EBSD : **OK** - DIC : **KO** - Low slip activity
- New 3D DCT scan (ESRF EBS) 4,000 grains/scan, 3min
- Adhoc optimized meshing protocol
- New batch of CP-Ti (geometry, orientations, grade)



(a) Initial underformed state
(b) $\epsilon_p = 2,36\%$

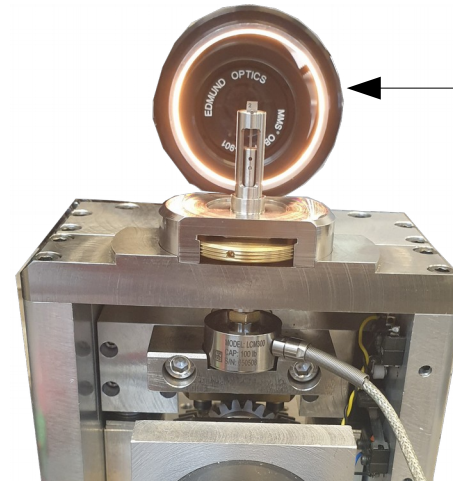
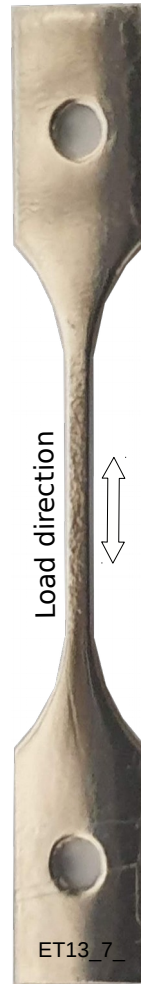


(c) SE-SEM (126nm resolution)
(d) Optical microscope

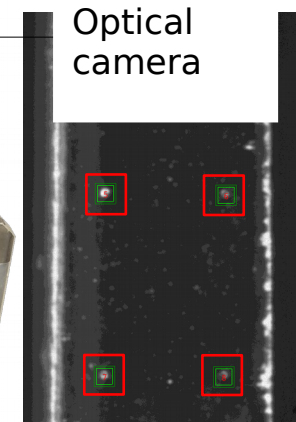


Plasticity factors study

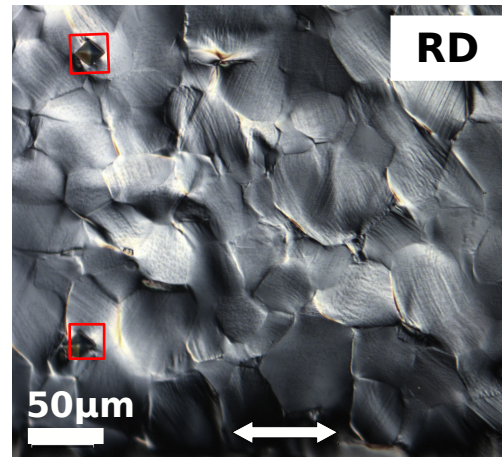
Samples	ET12_3_	ET13_7_
Material	T40	
Heat Treatment	855° C, 24h	
Initial section (caliper)	0,31 mm ²	0,34 mm ²
Polishing OPS	28h	28h
Load direction	RD	TD
Cross-head speed	2 μm/s	
L0 (μ-indents)	1,000 μm	
Force max	100N	125 N
ϵ_{TOTAL}	3,5 %	3,6%
ϵ_p	3.3 %	3,3 %
σ_{MAX}	323 MPa	368 MPa
Slip activity (EBSD + Schmid)	Prismatic	??
E homogenized	?? GPa	?? GPa



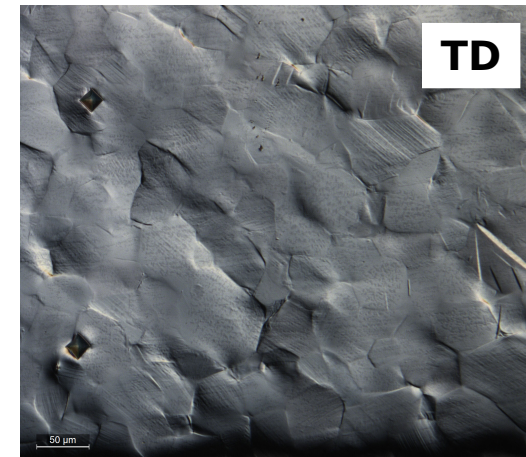
Bulky test rig



Optical camera



RD



TD

Load direction

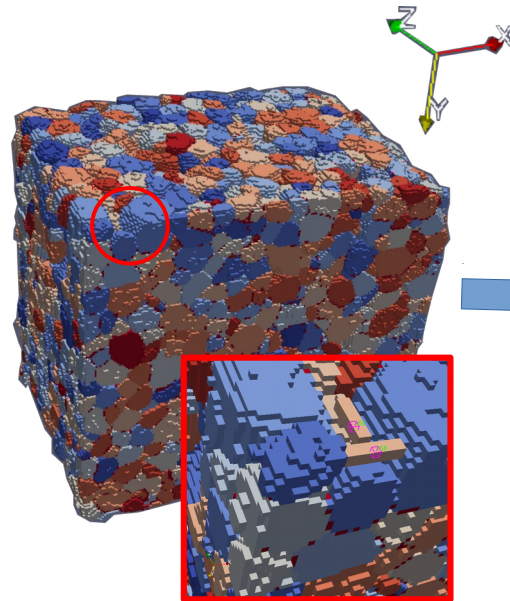
Synchrotron DCT reconstruction (ESRF)



1 scan 4,000 grains



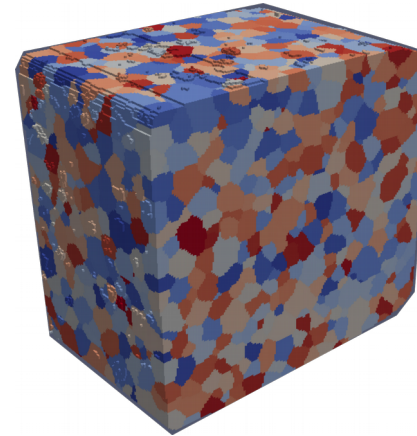
DCT Rec (ESRF)



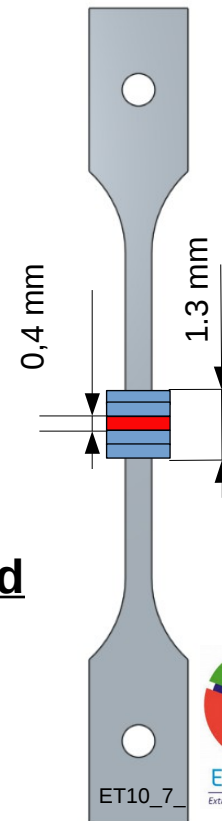
BIGMECA platform
Pymicro



Digital twin

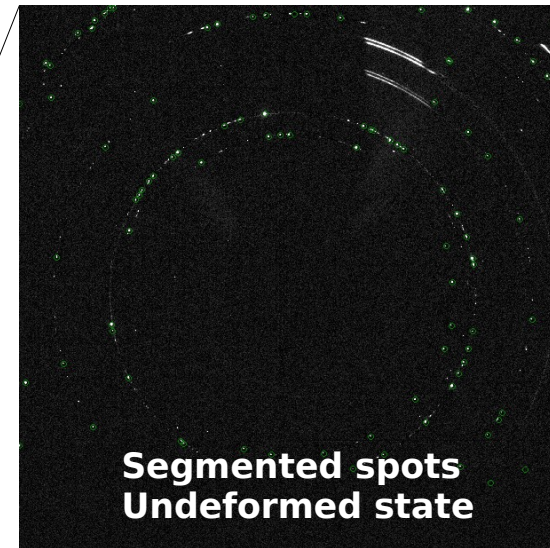
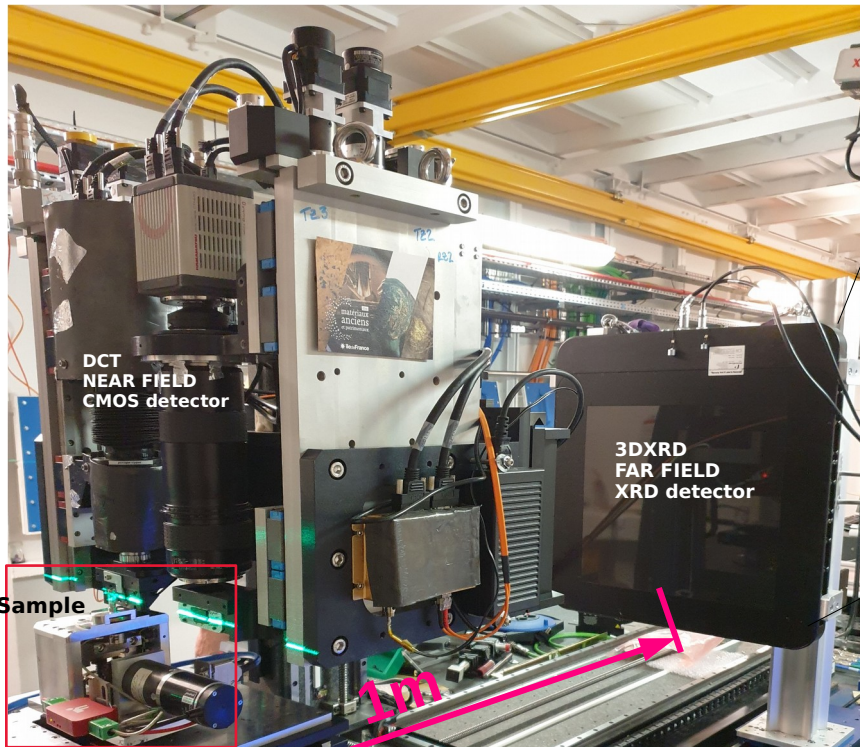


Ready for meshing and simulation (FEM, FFT)



Acknowledgment :
Wolfgang LUDWIG (ESRF),
Aldo MARANO (CDM)

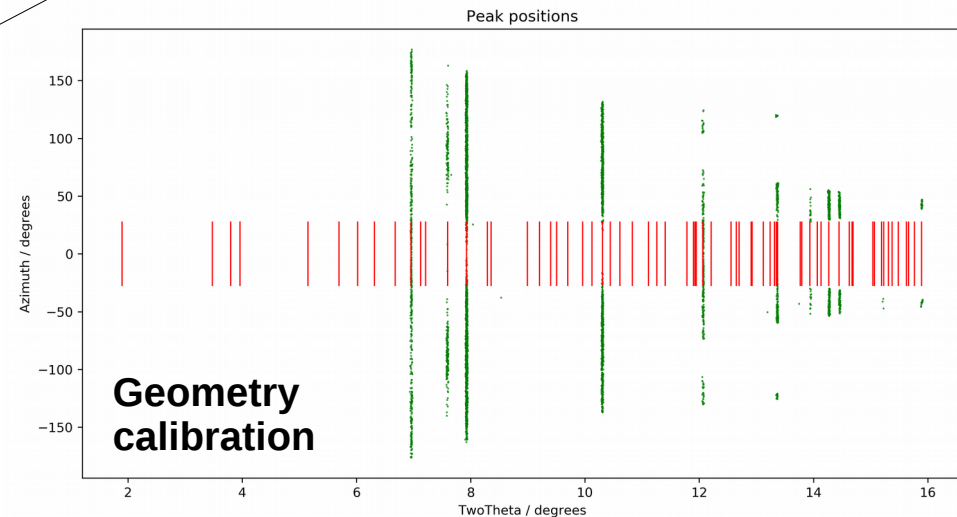
Synchrotron 3DXRD analysis (PSICHE)



FABLE tool
(ESRF)

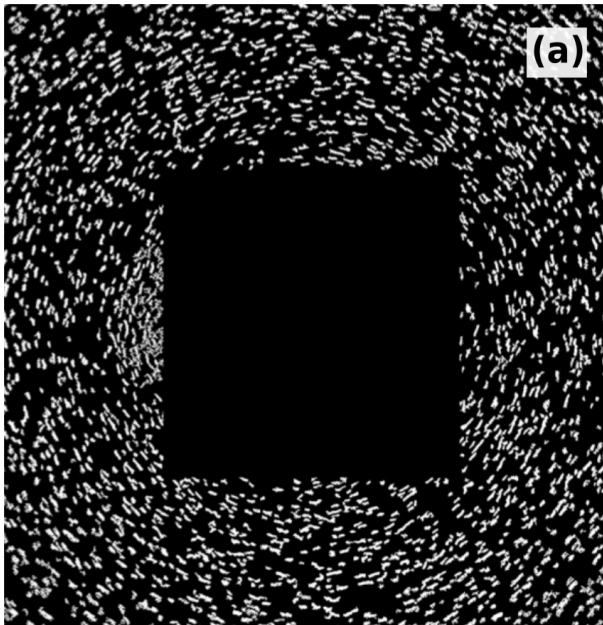
Sample

- ➔ Average elastic strain tensor
- ➔ Lattice curvature evolution

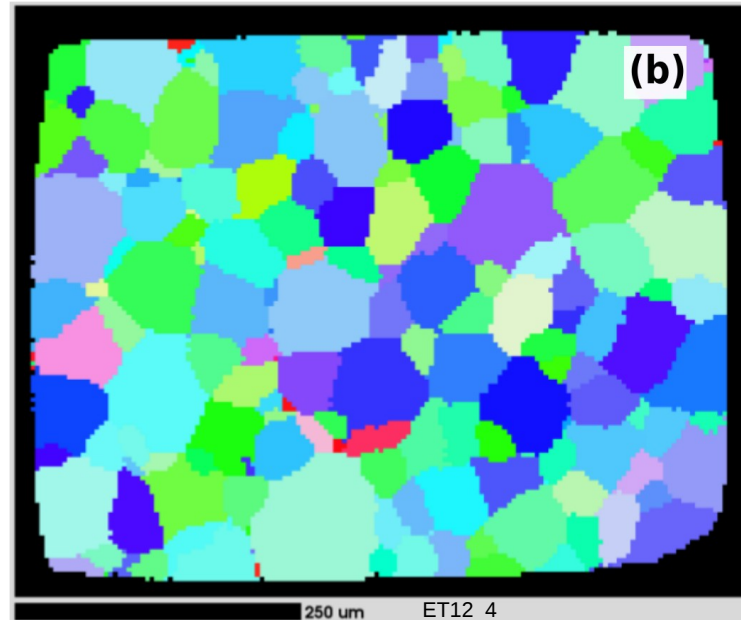


Laboratory 3D DCT

- New sample sent for 3D LabDCT at **Lund University**
- Polychromatic conical beam
- Consistent reconstruction
- **Ready for EBSD in situ = Hybrid multimodal data**



(a) Lab DCT detector
1 image/201 - resolution 4 μ m



(b) Reconstructed grain map slice with
GrainMapper (Xnovotech)

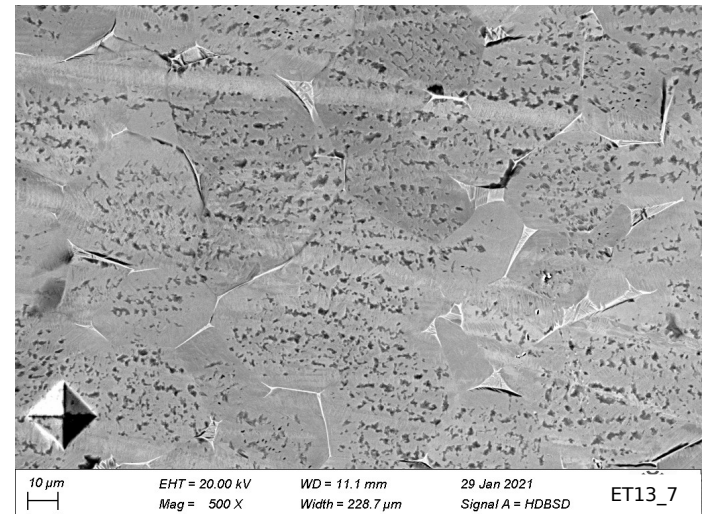
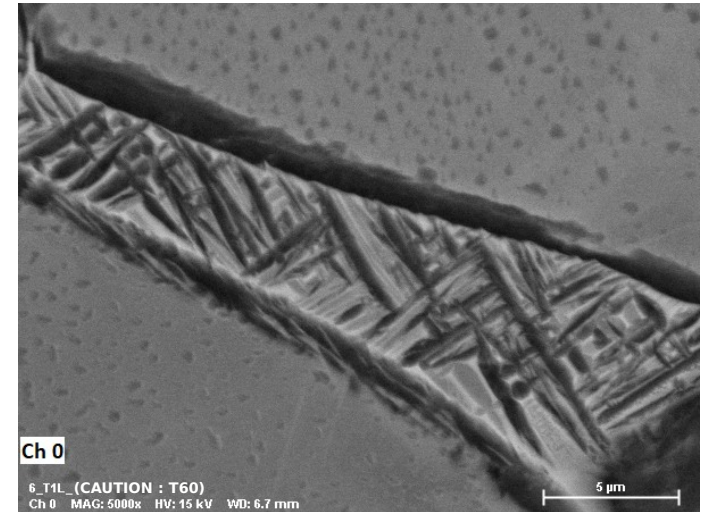
Acknowledgment :
Stephen HALL (Lund)

- **Precipitates at grain boundaries**

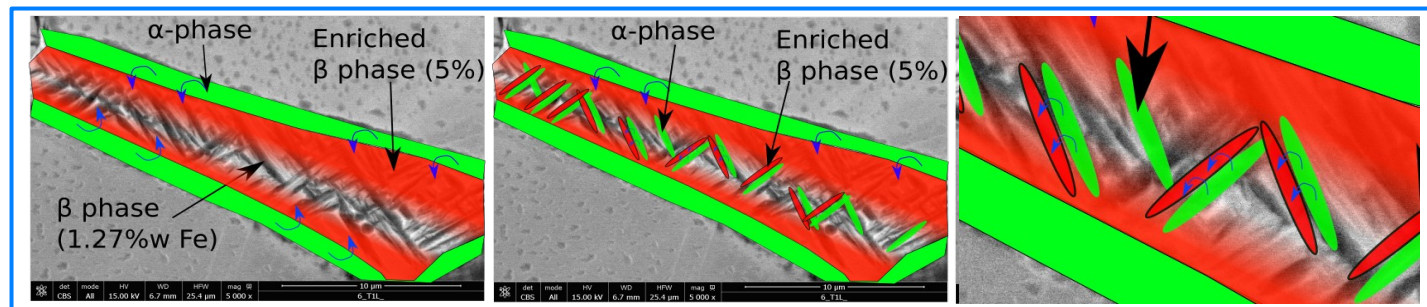
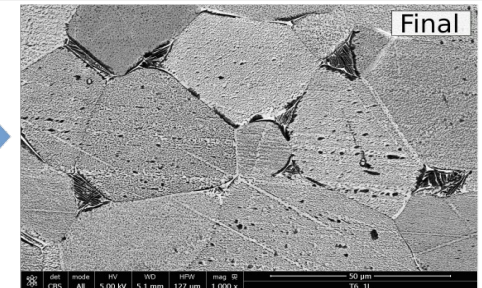
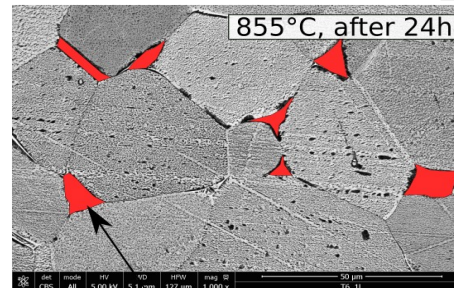
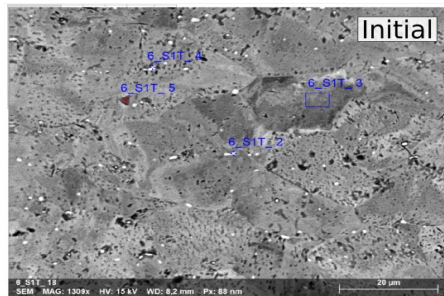
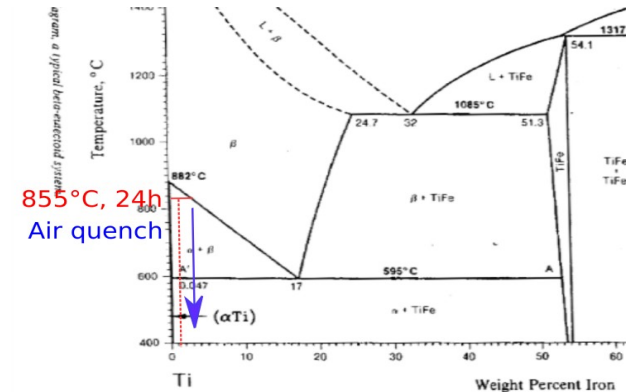
- Heat treatment
855°C, 24h, Argon 10L/min

- **Parasitic speckle**

- Grain orientation dependent
- AFM measurement (@ LMS) : on-going
- First measurement : 15nm deep recess
- Chemical attack during OPS ?



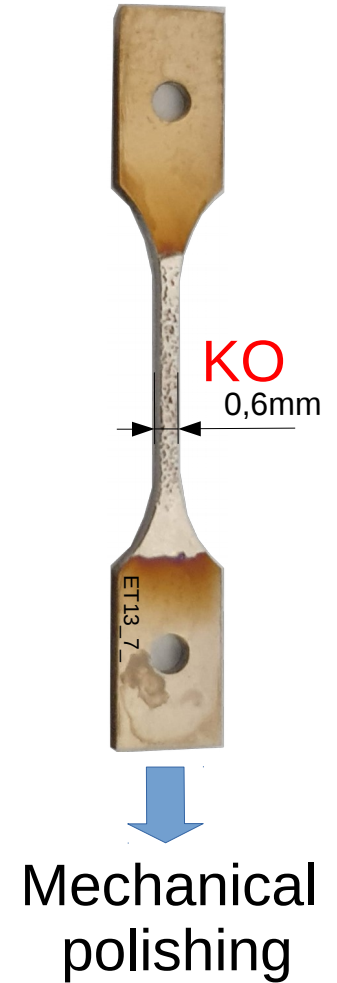
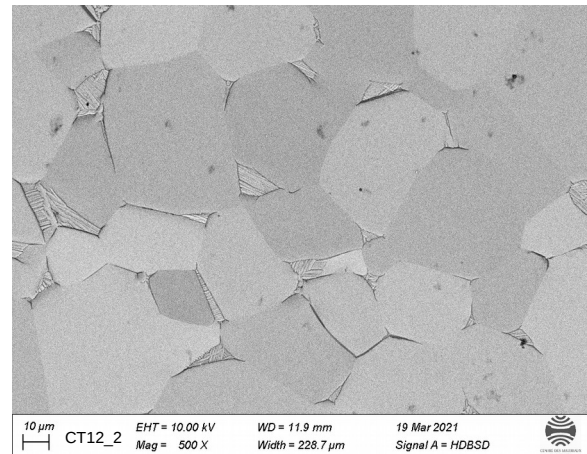
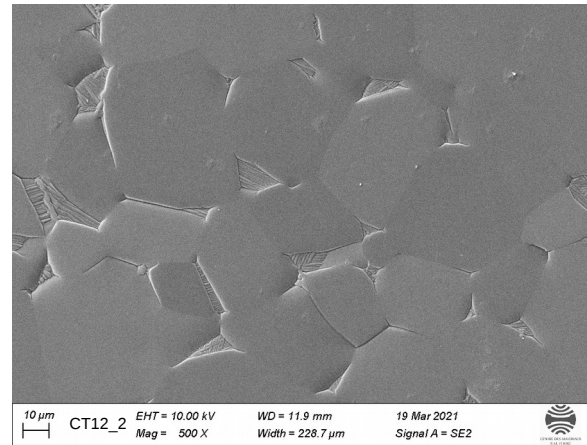
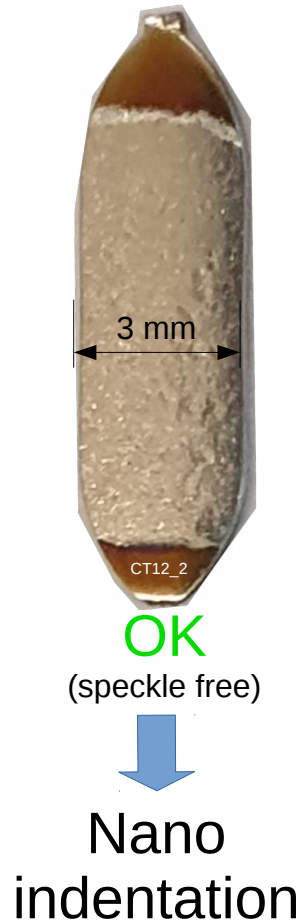
- **Precipitates at grain boundaries**
 - Heat treatment : 855°C, 24h, Argon 10L/min
 - Fe diffusion coefficient = $10^{-12} \text{m}^2/\text{s}$
→ Homogeneous β in GBs after 24h
- **Action** : Local EBSD
(index all possible phases : α , β , TiFe)



**Acknowledgment : Yvon MILLET (TIMET),
Elizabeth GAUTIER (Mines Nancy),
Vladimir ESIN (CDM)**

Samples preparation

- Electropolishing @ LMS



Acknowledgment :
Alexandre TANGUY (LMS)

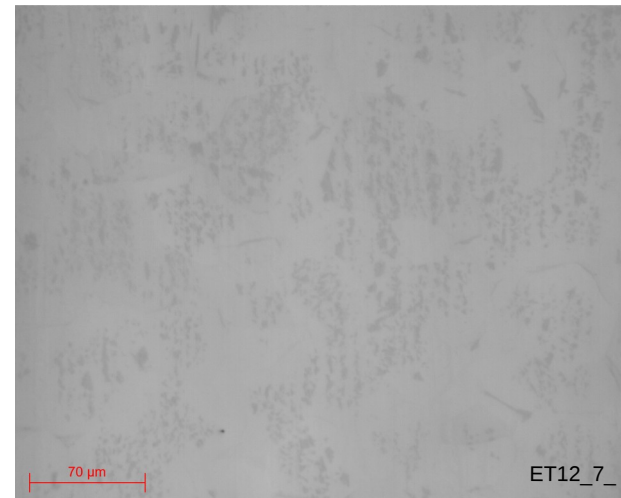
Samples preparation

- **Parasitic speckle** : New attempt to eliminate
- No improvement . To be repeated (non ideal conditions)

Solution	OP-S Non dry	Eposil M	Eposil M 11	MasterMet
Manufacturer	Struers	Verder/ATM	Verder/ATM	Buelher
Particules size	250 nm	60 nm	60nm	60nm
pH	9-10	9.5	11 (hydrogen water)	10



93g support

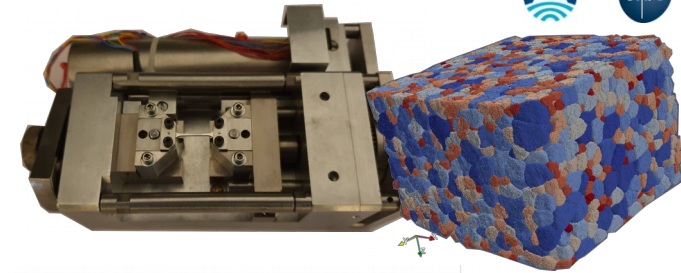


Result after 20h vibratory polishing -
Eposil M (+50 % water) - Optical LEICA

Acknowledgment : Daniel GALY (Safran)

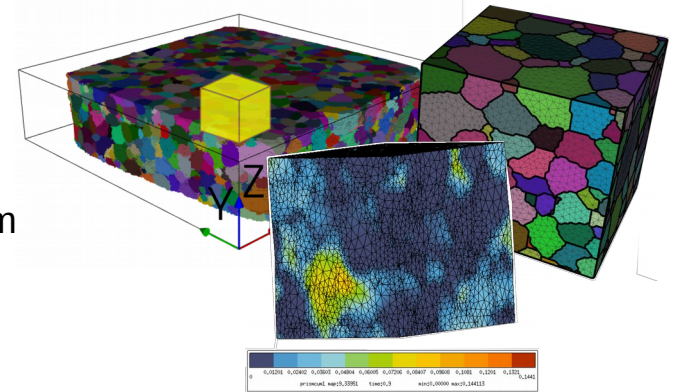
- **Experimental data:**

- Optimize and enrich continuously multimodal dataset :
 - EBSD in situ on samples with initial DCT
 - New synchrotrons beamtime (PSICHE – July)
 - Opportunity : SEM-DIC (nano particules @ LMS)



- **Simulation Data :**

- Enrich dataset : Continuum crystal plasticity simulations on digital twins (~4,000 grains)
- CPFEM - Zset : Mandel crystal large deformation formalism

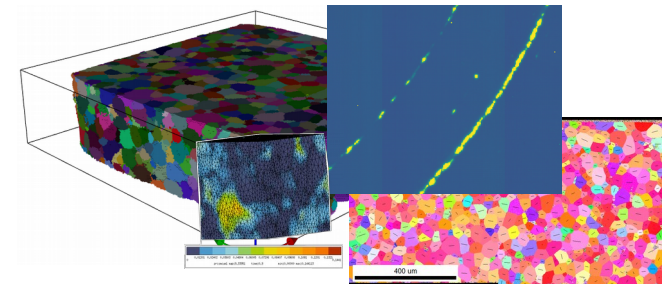


- **Data unification:**

- Consolidate modalities to prepare ground for statistical analysis (leverage BIGMECA data platform)

- **Statistical learning :**

- Extract physical data from images and volumes.
- Perform statistical analysis of plasticity mechanisms.



THANK YOU FOR YOUR ATTENTION