4D experimental testing and simulations for statistical analysis of crystal plasticity in structural materials

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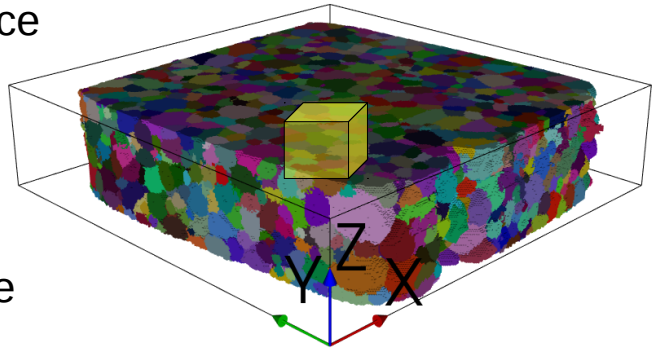
- PhD supervisors: PROUDHON Henry

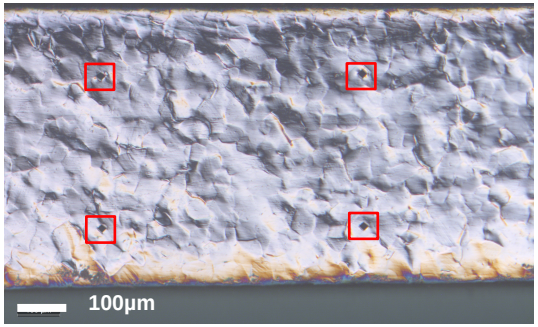
Agenda

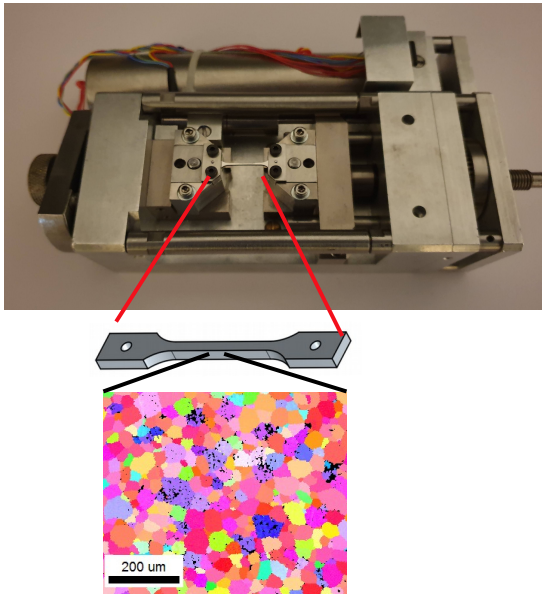
- Scope and Objective
- Previous status
- Multimodal experimental data :
  - Surface data: SEM in-situ campaign
  - Slip activity analysis
  - Volume data: DCT, 3DXRD
- Simulation data
  - Meshing improvement
  - Zset model : Mandel Crystal
- Achievements & Outlook

Previous status (10/07/20)

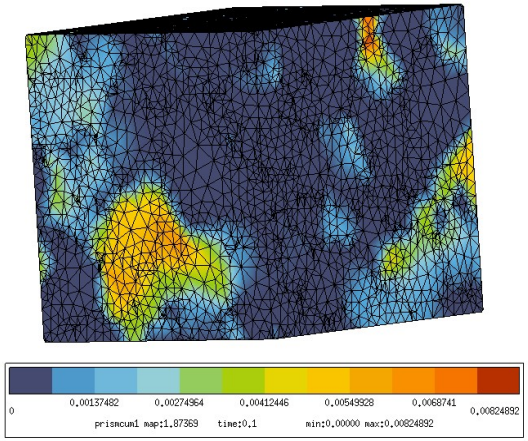
- **Material :** Commercially pure Ti (T40) Phase-α, HCP lattice
- **Digital twin -** 2020 PSICHE : 3D/4D DCT → 3200 grains
- **Multimodal data**:
  - Macroscopic tensile tests :
    Absence of slip bands, out of plane deformation
  - Strategy for SEM in-situ : focus on lattice curvature
- **Numerical data :** CPFE + FFT simulations on real data







EBSD (IPF – Z)
Grain size ~ 50µm



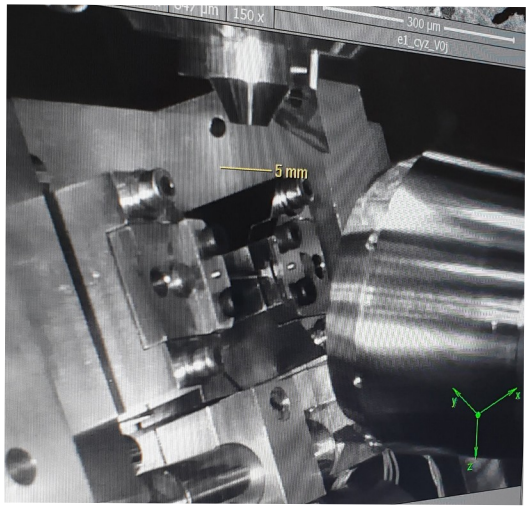
PSICHE - 300 grains, ε = 0.28 %
Zset – Accumulated prismatic activity,

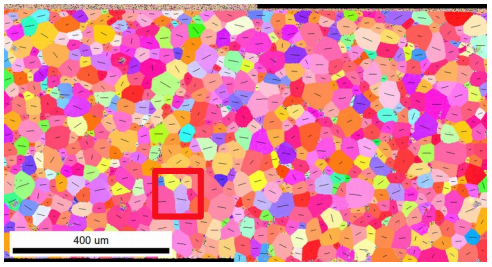
CPFE : Crystal Plasticity Finite Element
DCT : Diffraction Contrast Tomography
EBSD : Electron Back Scattered Diffraction

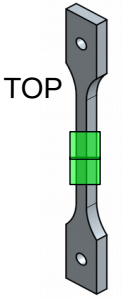
FFT = Fast Fourier Transform
HCP = Hexagonal Closed Pack
IPF = Inverse Pole Figure

Experimental data – SEM in-situ - EBSD

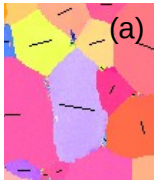
- **Objectives**: Validate integration ; familiarize with technique
- **Integration** : Space, weigth, tilt
- **Test** :
  - 2 load steps → εp = 2.36 %
  - EBSD (1µm/pixel) → Lattice curvature
  - Post mortem SE + optical observations
    - Out of plane deformation + **slip bands**
    - Interpretation : Better surface preparation

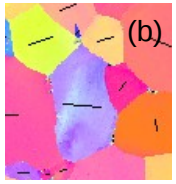




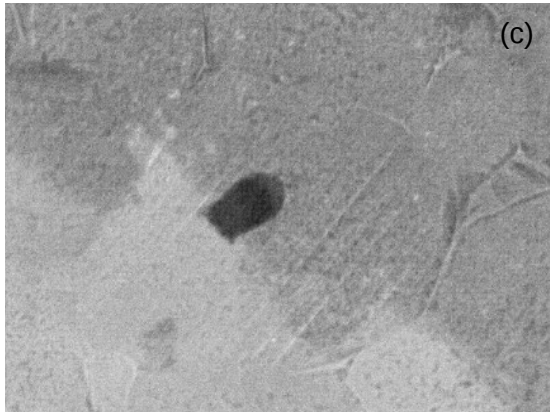


2 merged EBSD scans
IPF-Z (1µm resolution, 8h)

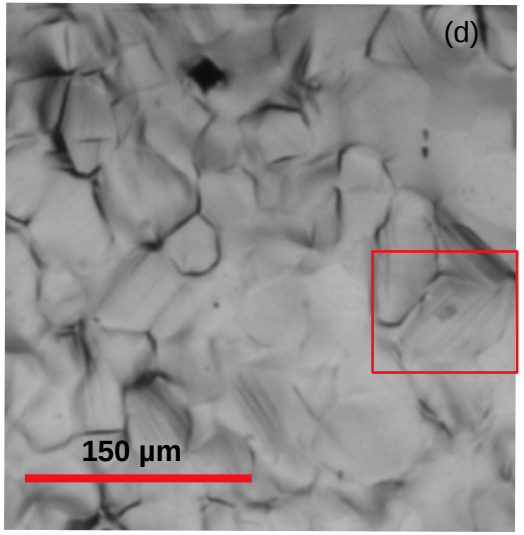




(a)Initial underformed state, (b) εp=2,36 %



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

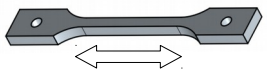


EBSD : Electron Back Scattered Diffraction
OPS : Oxyde Polishing Suspension

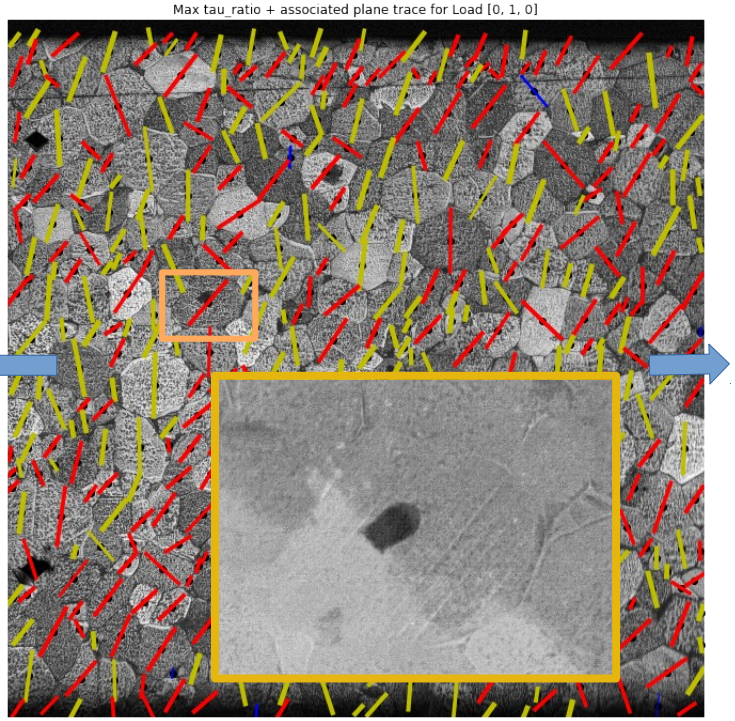
SE = Secondary Electron
SEM = Scanning Electronic Microscope

Post-processing – Slip activity analysis

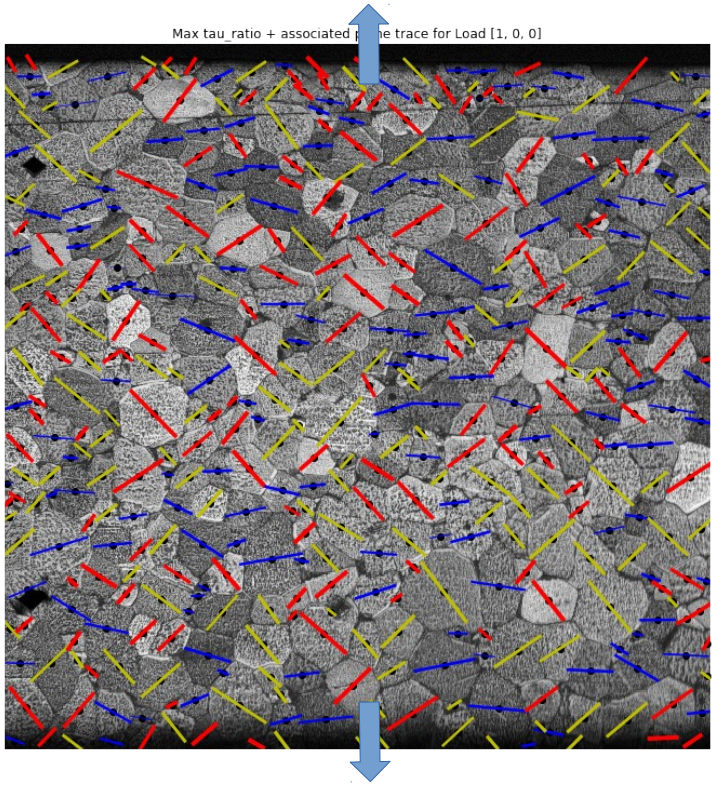
- Prediction of which slip system activated 1st in each grain with Pymicro



Rolling Direction (RD) = Load direction



- EBSD IQ + predicted slip traces (Real config)
- Good prediction for prismatic activity
- Low effective global slip activity

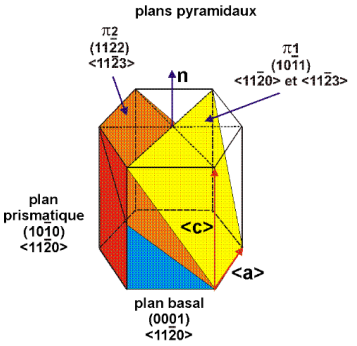


Prediction for transverse loading

Criterion : $Max\left(\frac{ms}{CRSS_0}\right)$

ms = Schmid factor
$CRSS_0$ = Critical Resolved Shear Stress

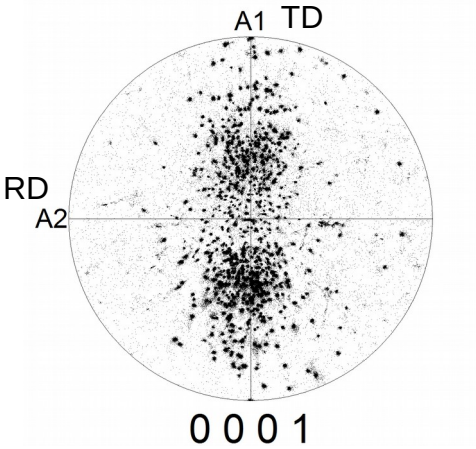
(Barkia et al, 2015) :
- CRSS_0_bas = 182 MPa
- CRSS_0_prism = 120 MPa
- CRSS_0_pyr1a = 149 MPa

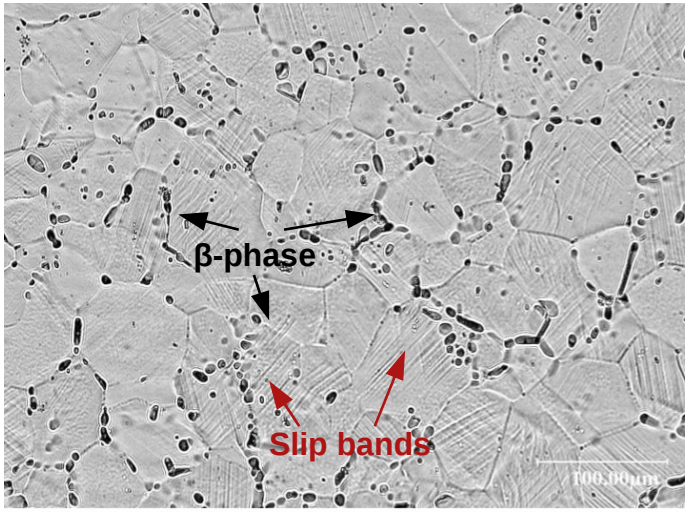


Basal
Prismatic
Pyramidal π1 <a>

Bibliography – Commercially Pure Ti

- T40 = Grade 2, T60 = Grade 4.
- Sheet or plate → characteristic texture
- Lütjering, 2007 :
  - O, Fe, grain size : Strengthening effect
  - O > 0.25%
    - Slip mode transition from wavy to planar
    - Decrease in sensitivity to twinning
- Barkia et al, 2015 :
  - SEM in-situ
  - Dominant mechanism at εp ~ 2 %
    in both grades = Dislocation slip (prismatic)
  - T60 : Reported β-phase at grain boundaries





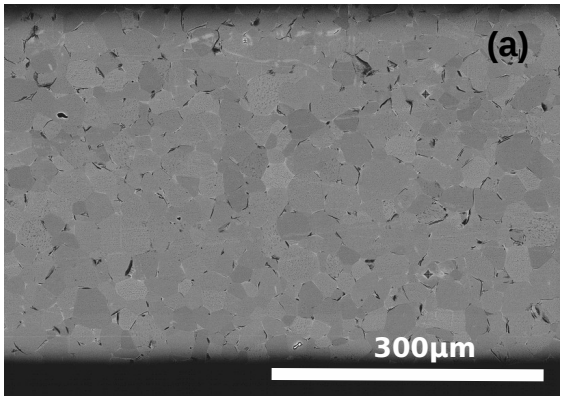
Barkia - T60 (εp ~ 1.7 %)

Experimental data - Metallurgy

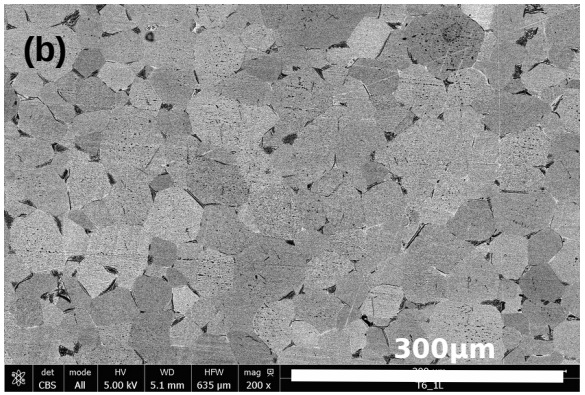
- Material certificates before heat treatment

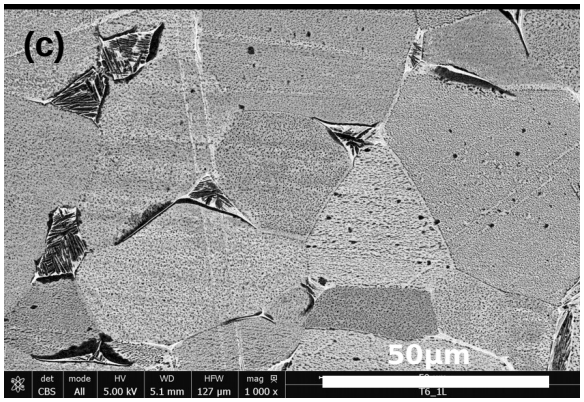
Weight %	Fe	C	O	N
T40 - BIGMECA	**0.14**	0.005	**0.08**	0.008
T40 - Barkia	**0.034**	0.004	**0.16**	0.003
T60 - BIGMECA	**0.12**	0.007	**0.33**	0.002
T60 - Barkia	**0.17**	0.007	**0.32**	0.002

- Preparation : Heat Treatment → Electropolishing → Kroll attack
- EDS chemical analysis : same proportion of rich Fe phases at grain boundaries



(a) T40 SEM BSE (ET10_3)
855°C, 17h, Argon 10L/min



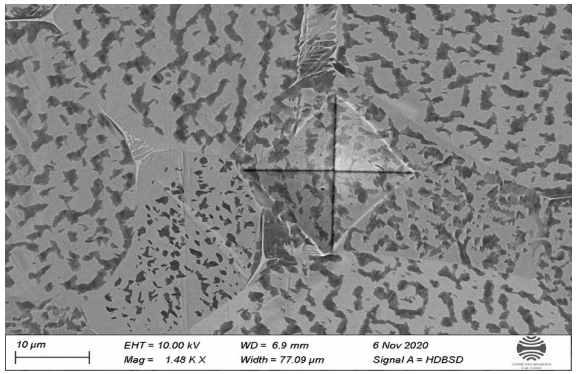


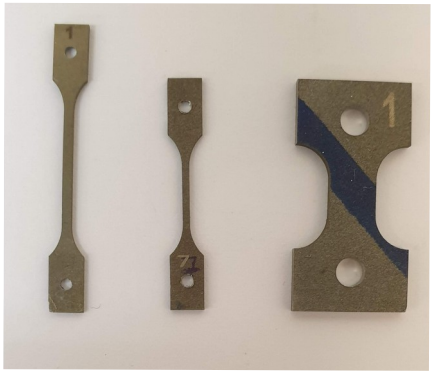
(b) and (c)T60 _SEM BSE (6_T1L)
855°C, 24h, Argon 10L/min

BSE = Back Scattered Electrons
EDS = Energy Dispersive Spectroscopy
SEM = Scanning Electronic Microscope

Other factors influencing plasticity

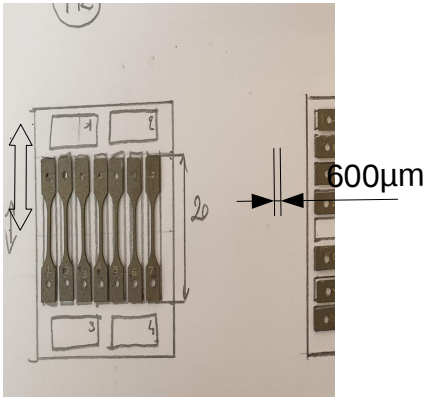
- Processing history
- Grain size
- Load direction
- Strain rate
- Surface preparation?
- Geometry?

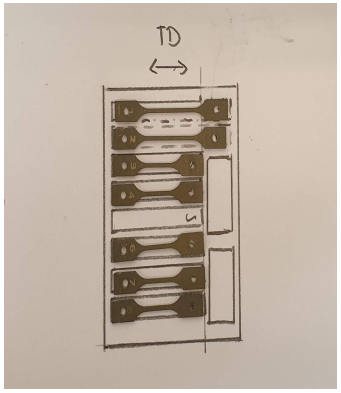




SEM BSE : Natural speckle
EDS → Local hardening during polishing

➡ Machining of new samples

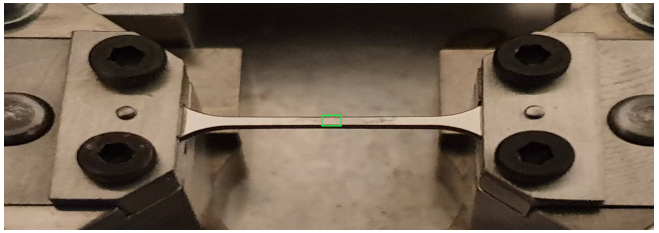


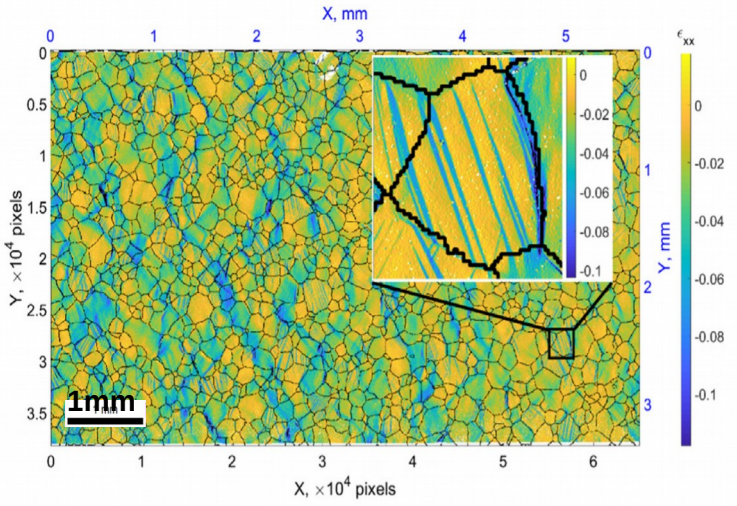


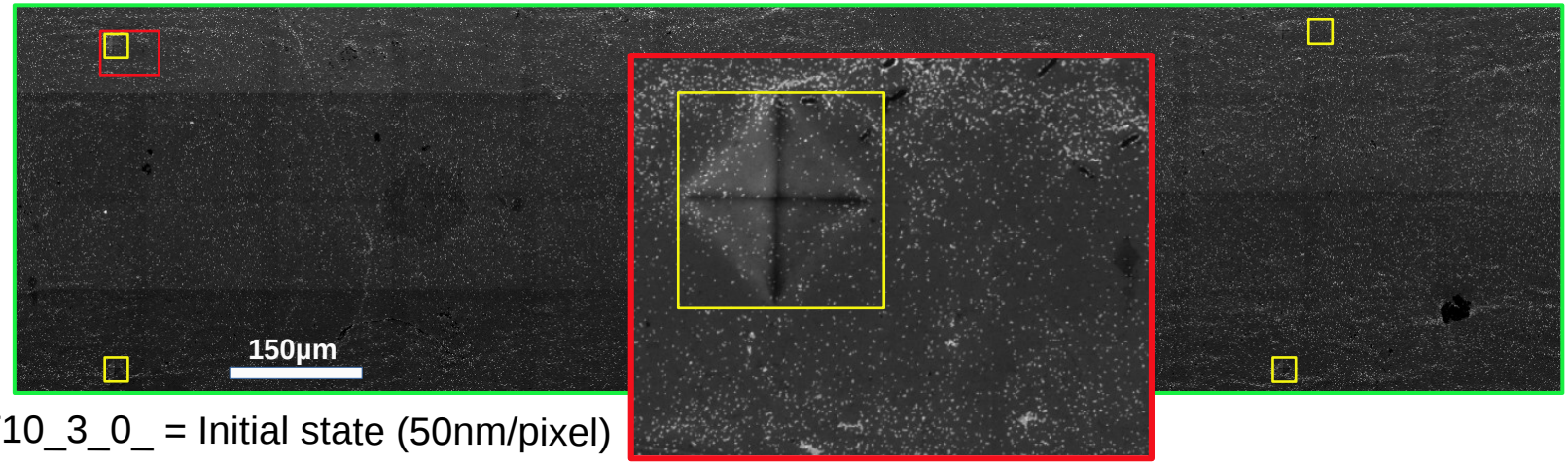
BSE =Back Scattered Electrons
EDS = Energy Dispersive Spectroscopy
SEM = Scanning Electronic Microscope

Experimental data – SEM in-situ - DIC

- **Objective**: Perform Large FOV SEM nano-DIC (Chen et Daly, 2018)
- **Sample preparation :**
  Nano particules (NP) deposition (Krammer et Daly, 2011, 2013)





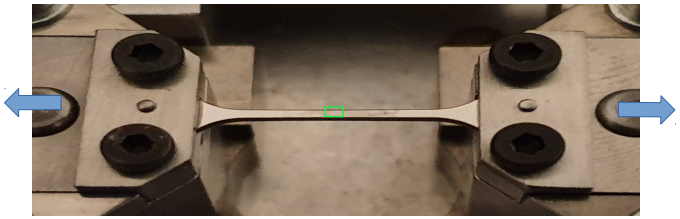


ET10_3_0_ = Initial state (50nm/pixel)

DIC : Digital Image Correlation
FOV : Field Of View

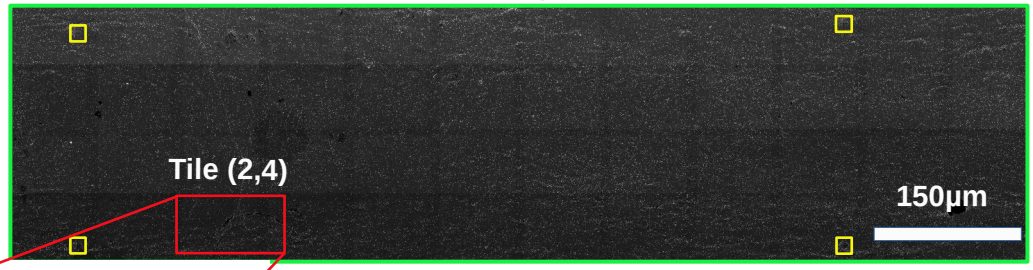
SE = Secondary Electron
SEM = Scanning Electronic Microscope

Experimental data – SEM in-situ - DIC

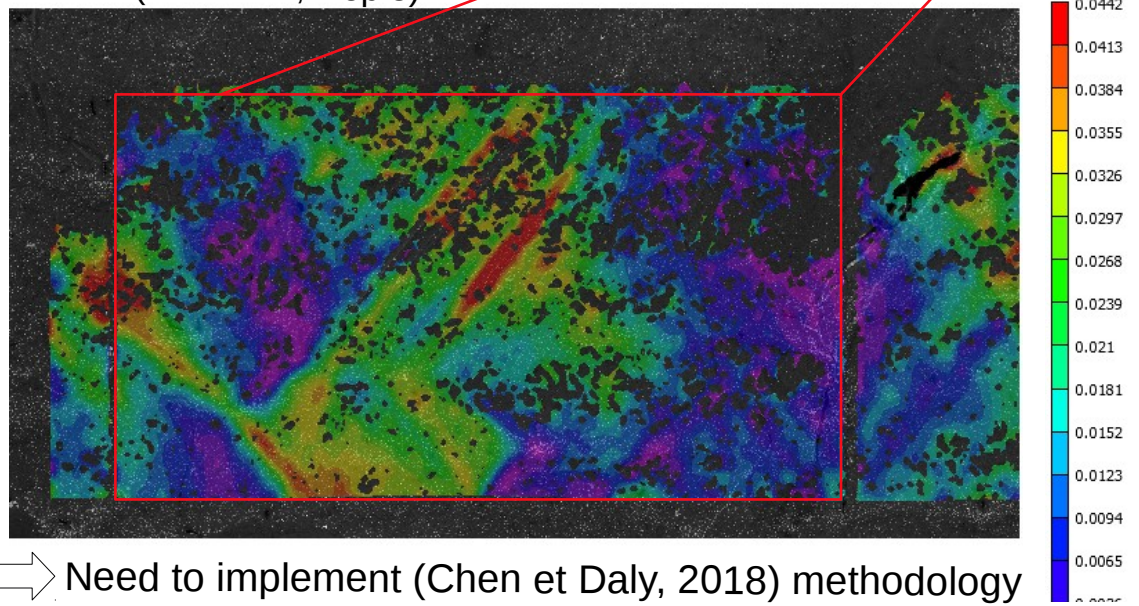


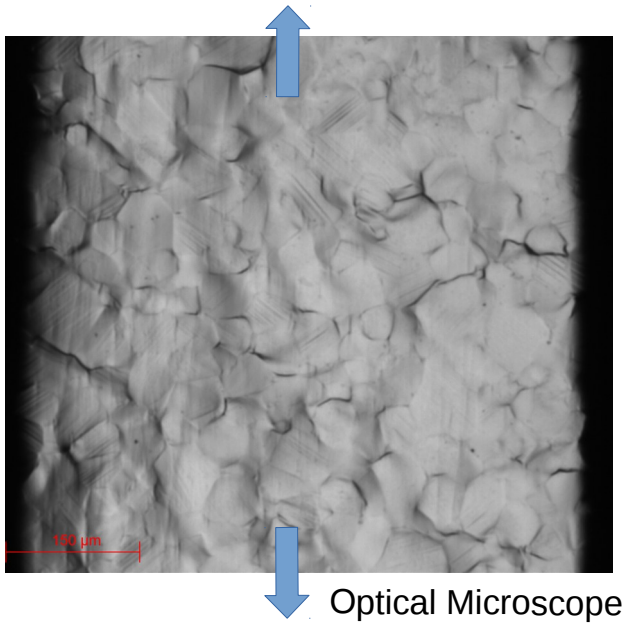
Interrupted in-situ : 2 load steps →εp=2.25%

Magnification x847, 3,000x2,250 pixel/tile, 50nm/pixel
44 tiles → Total scan duration/step : 1h



VIC 2D (subset 13, step 3)





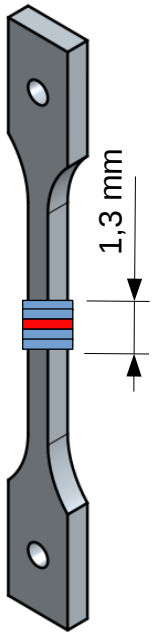
Optical Microscope

⇨ Need to implement (Chen et Daly, 2018) methodology

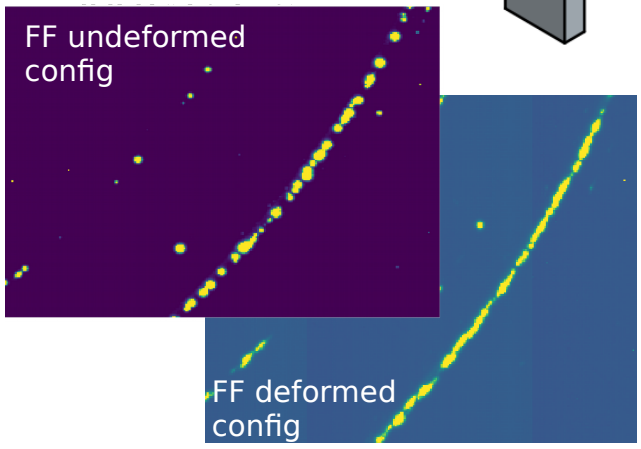
DIC : Digital Image Correlation

Experimental data – Volume data

- **DCT** (Near Field):
  - ESRF EBS : DCT 3D - December 2020
    - 5 scans, 300µm height, 50µm overlap
    - Resolution 1.22µm
    - Total volume ~ 0.65x0,65x1.3 mm
    - time/scan = 3min !
    - Input for GENCI project
  - 3D LabDCT : January 2021
  - PSICHE campaign : July 2021



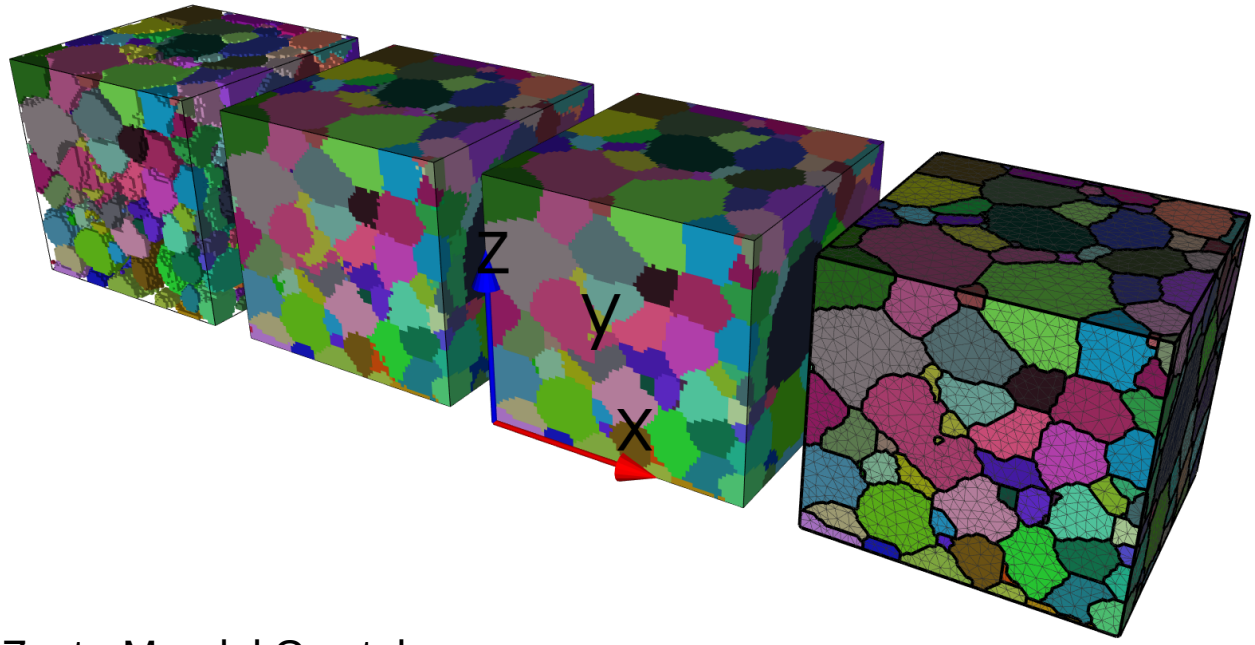
- **3DXRD** (Far Field - FF) :
  - Lattice curvature → Elastic stress tensor →Dislocations density
  - ESRF supported tool : FABLE GrainSpotter



3DXRD = 3D X-ray Diffraction
DCT = Diffraction Contrast Tomography
EBS = Extra Brilliant Source

Simulation data

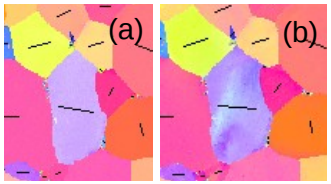
- Improved mesh
- Alignement of coordinate systems of digital twin and meshing
- Sub volume of PSICHE data ~ 300 grains :

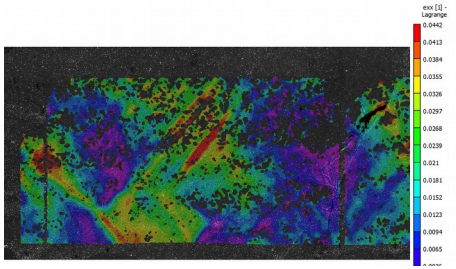


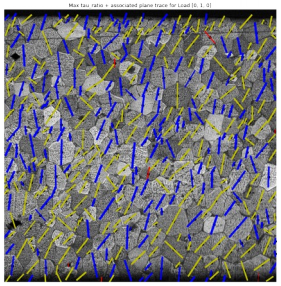
- Model : Zset - Mandel Crystal :
  - Large deformation formalism → Access to lattice curvature
  - Limitations : Runge Kutta (acceptable for εp=2,5 %), 1 slip family/simulation

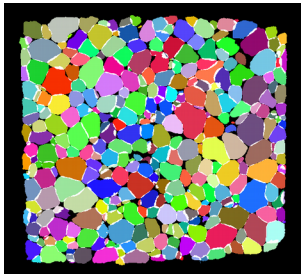
Achievements

- **Experimental data:**
  - SEM in-situ campaigns :
    - Methodology validation : in-situ EBSD + SEM-DIC
    - Observation of slip bands on T40 surface
    - Prediction of slip activity (Schmid Factor)
  - DCT ESRF : New data for digital twins

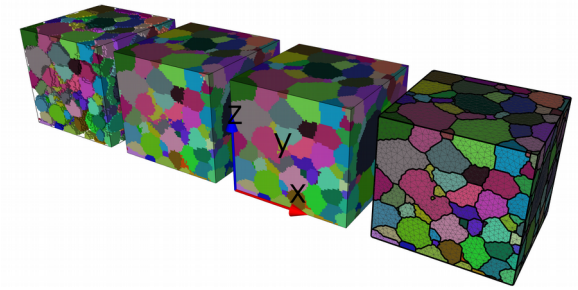






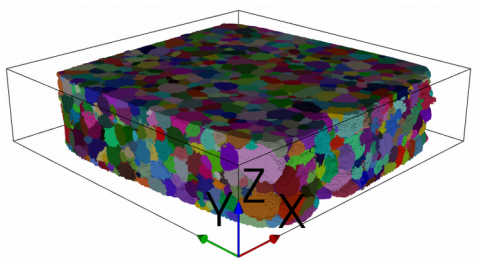


- **Simulation Data :**
  - Improvement of meshing
  - Alignement of multimodal coordinate systems

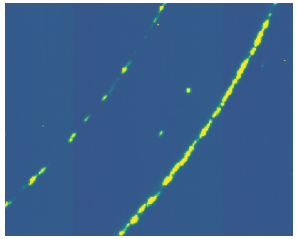


Outlook

- **Digital twin:**
  - Complete reconstructions (~10,000 grains)
  - Update 6D DCT Rec algorithm for PSICHE

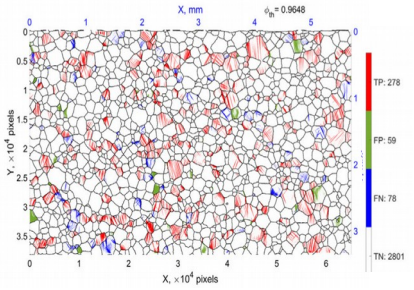


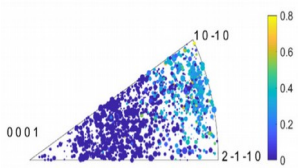
- **Experimental data:**
  - Conclude on parameters influencing deformation
  - Train on 3DXRD GrainSpotter
  - Scan 1 sample with 3D Lab DCT
  - Prepare for next PSICHE campaign



- **Simulation Data :**
  - Perform simulations on complete DCT volumes (GENCI)

- **Statistical learning:**
  - Extract physical data from structured dataset
  - Statistical analysis of plasticity mechanims





THANK YOU FOR YOUR ATTENTION.

ANY QUESTIONS ?