5th International Workshop on Mechanisms of Vacuum Arcs
02-04/09/2015

Scanning Electron Microscopy tools for material characterization

Focus on EBSD for characterisation of dislocation structures

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CERN | EN ENGINEERING DEPARTMENT

Outline

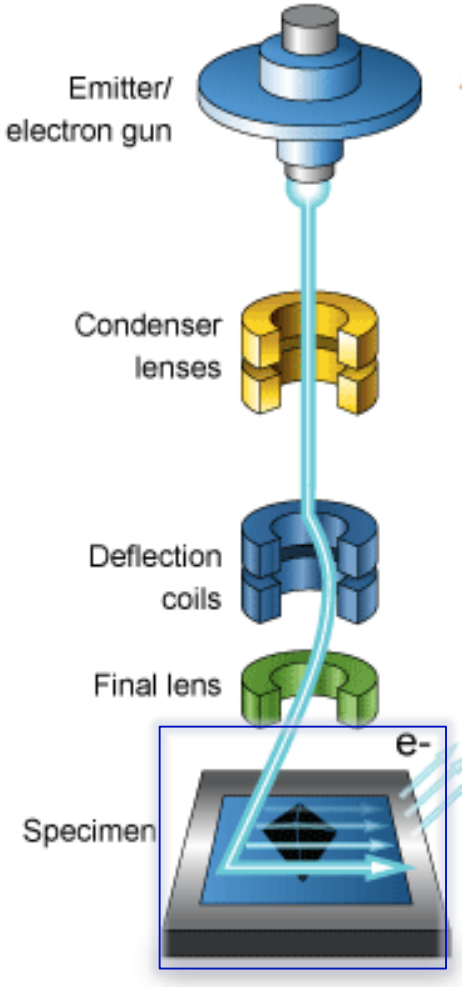
1. Introduction
   - SEM* principle
   - Response to incident electrons
2. Characterisation of dislocation structures
   - EBSD*
   - Link with dislocations
   - Misorientation criterion
   - Example (ferritic steel)
3. Conclusion
   - Microscopy at CERN for CLIC
   - SEM-FIB*

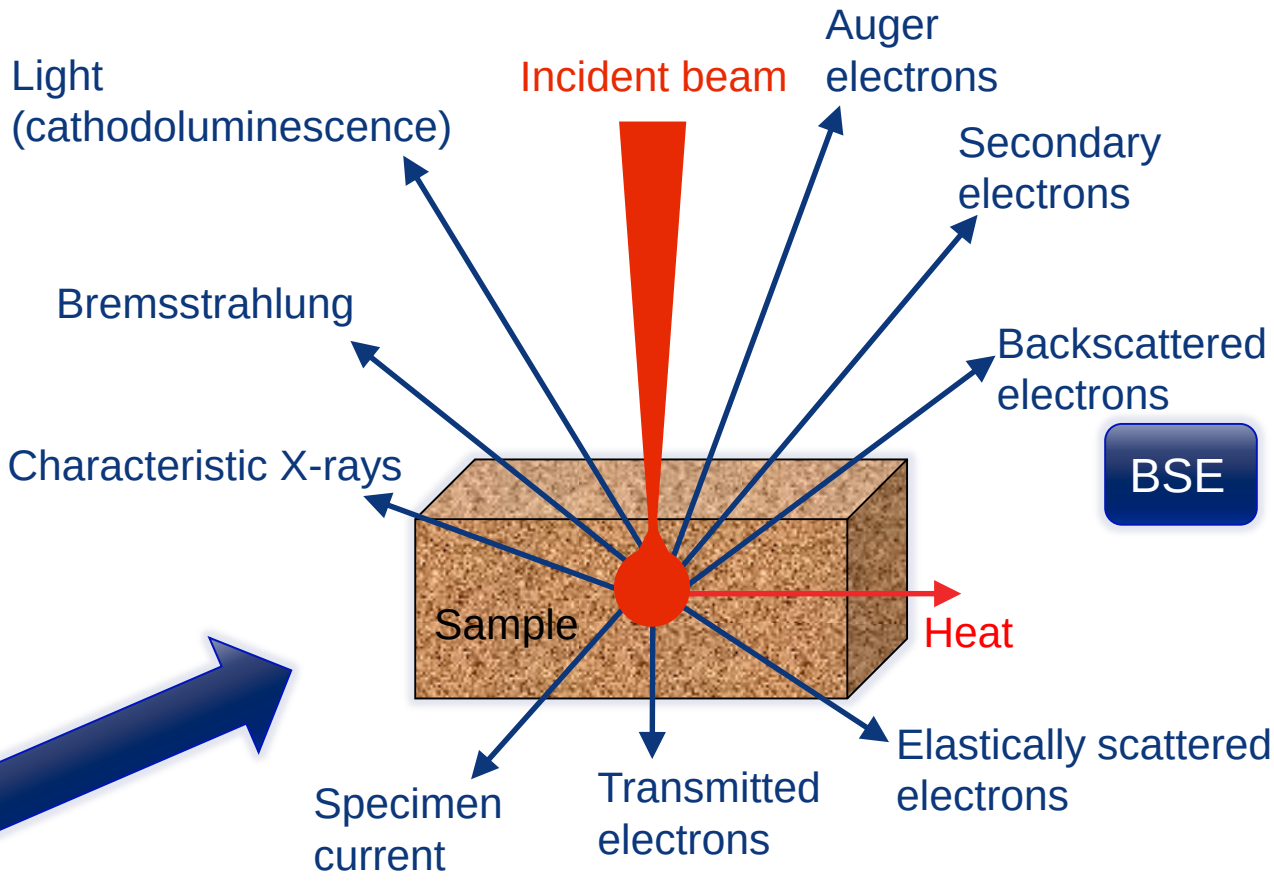
**SEM: Scanning Electron Microscope*
**EBSD: Electron BackScattered Diffraction*
**FIB: Focused Ion Beam*

Introduction

- SEM principle
- Response to incident electrons

SEM principle, response to incident electrons





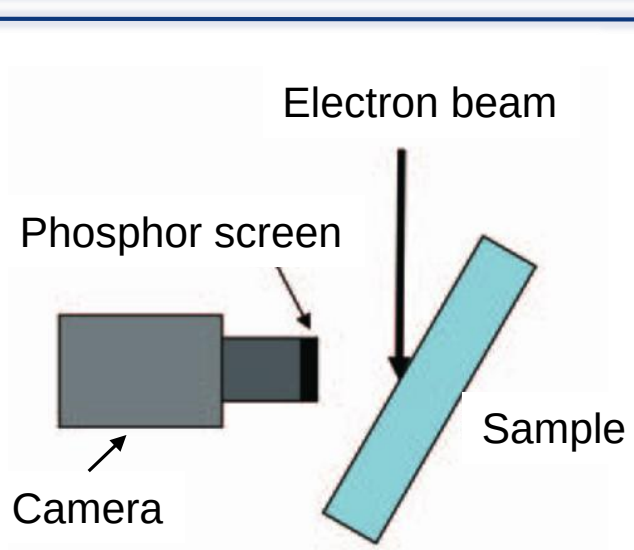
Ref: AMMRF

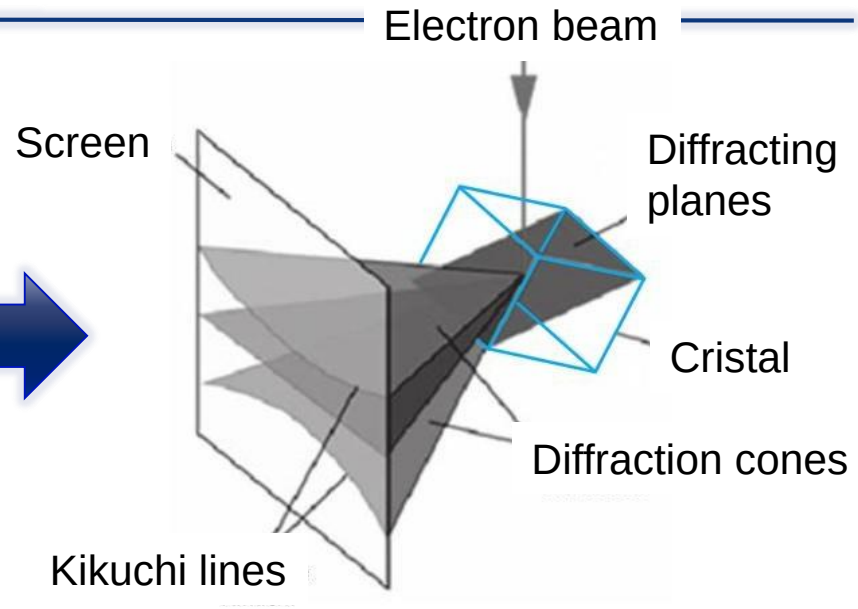
Characterisation of dislocation structures

- EBSD principle
- Link with dislocations
- Introduction of a misorientation criteria, example of KAM*
- EBSD and TEM*, between complementarity and substitution
- Case of a ferritic steel

**KAM:Kernel Average Misorientation*
**TEM:Transmission Electron Microscope*

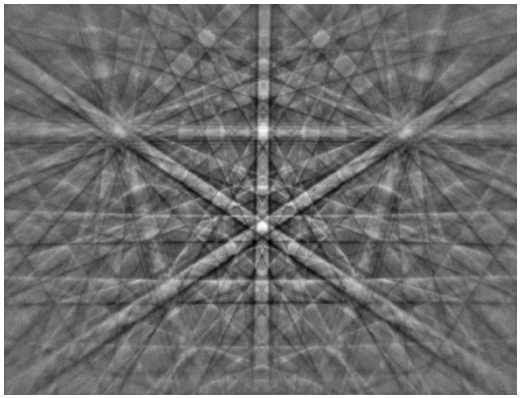
EBSD principle (1/2)





Ref:[BAU10]

- ✓Diffraction of back scattered electrons
- ✓Following Bragg condition
- ✓Diffraction cones
- ✓Kikuchi lines
- ✓Diffraction pattern

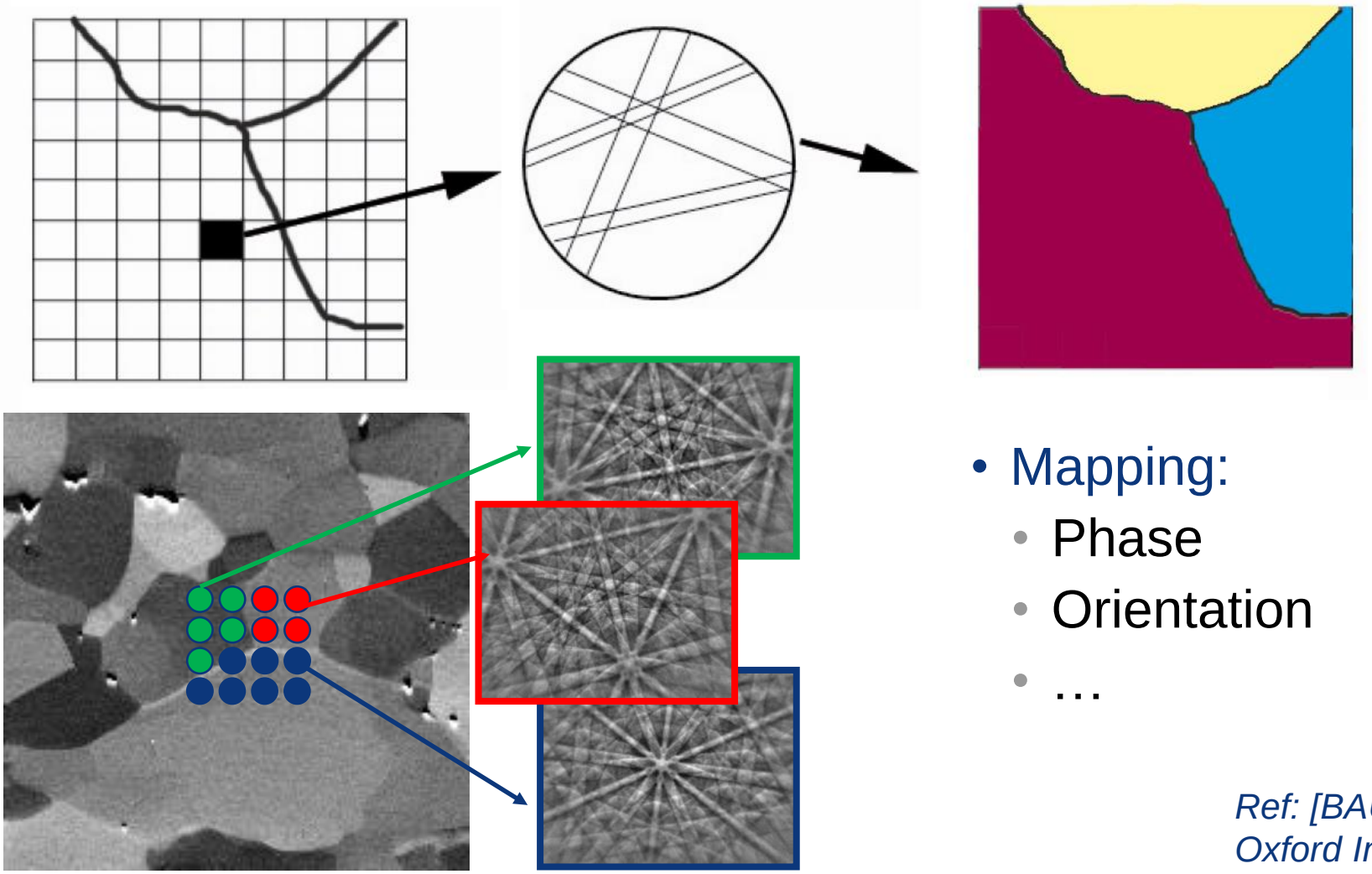


EBSD principle (2/2)

Sample

Diffraction pattern

Mapping



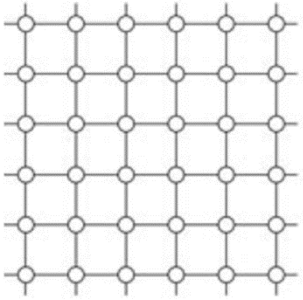
- Mapping:
  - Phase
  - Orientation
  - …

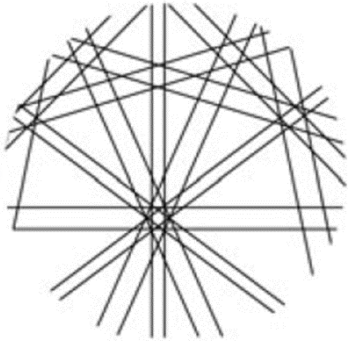
Ref: [BAU10]
Oxford Instruments

2. Dislocation structures

Link with dislocations

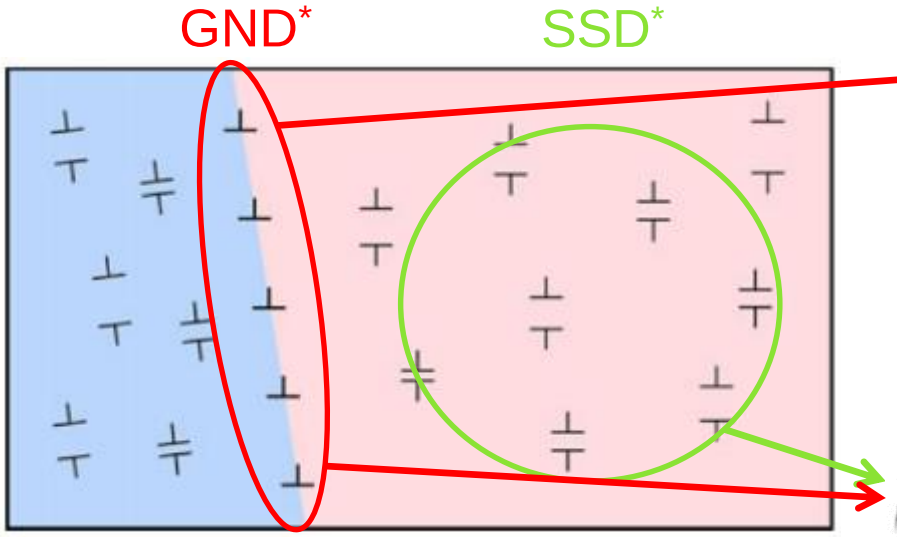
Perfect crystal

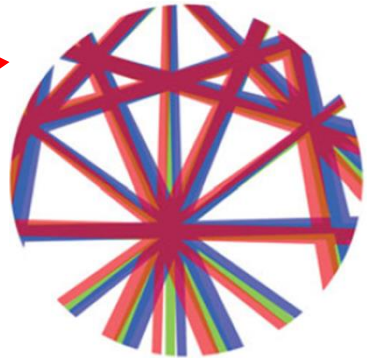




Theoretical diffraction pattern

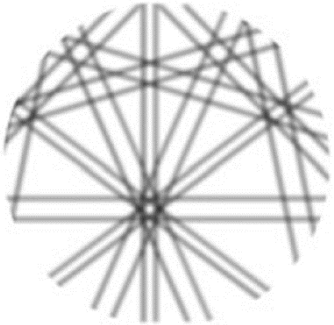
Influence of dislocations on the diffraction pattern





Local misorientation

➔ Rotation of the pattern



➔Contrast and sharpness decrease

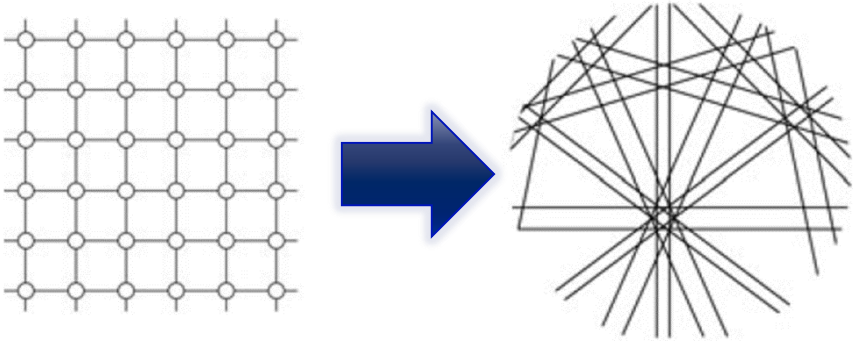
*GND : Geomatrically Necessary Dislocations

*SSD: Statistically Stored Dislocations

Ref: [WRI11]

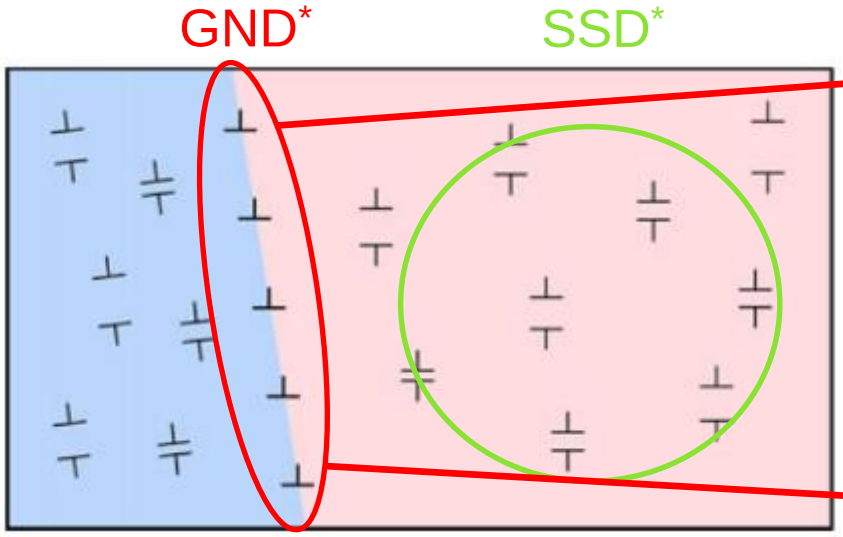
2. Dislocation structures — Link with dislocations

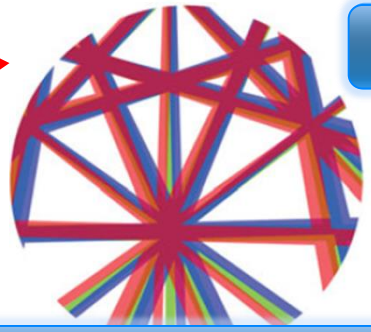
Perfect crystal



Theoretical diffraction pattern

Influence of dislocations on the diffraction pattern





Local misorientation

➔ Rotation of the pattern

LAGB : Low Angle Grain Boundaries

KAM : Kernel Average Misorientation

**GND : Geometrically Necessary Dislocations*

**SSD: Statistically Stored Dislocations*

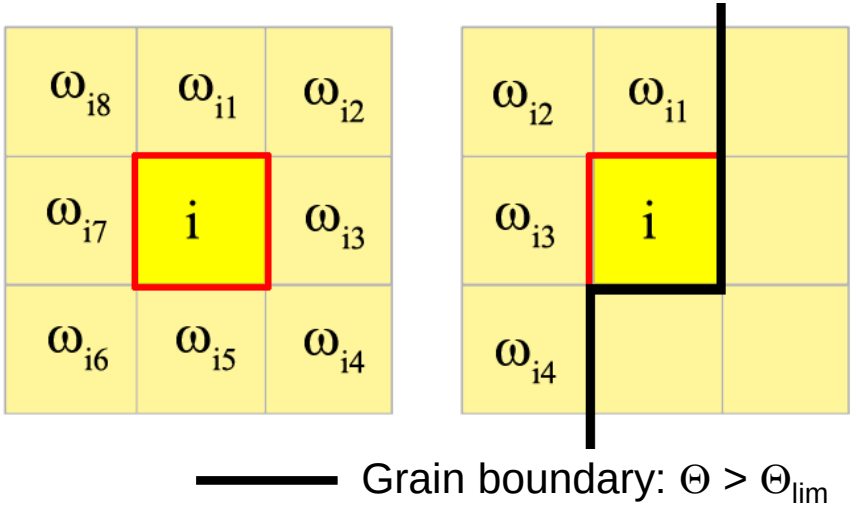
Ref: [WRI11]

Introduction of misorientation criteria

Local misorientation

- Example of Kernel Average Misorientation (KAM)

✓ Misorientation within a subgrain/grain
✓ $0,5° < \Delta\Theta < \Theta_{lim}$
✓ Θ_{lim}= 5° or more, 15° for a grain
✓ Deformation gradient
✓ Link with dislocation densities (GND)
✓ Link with plastic deformation

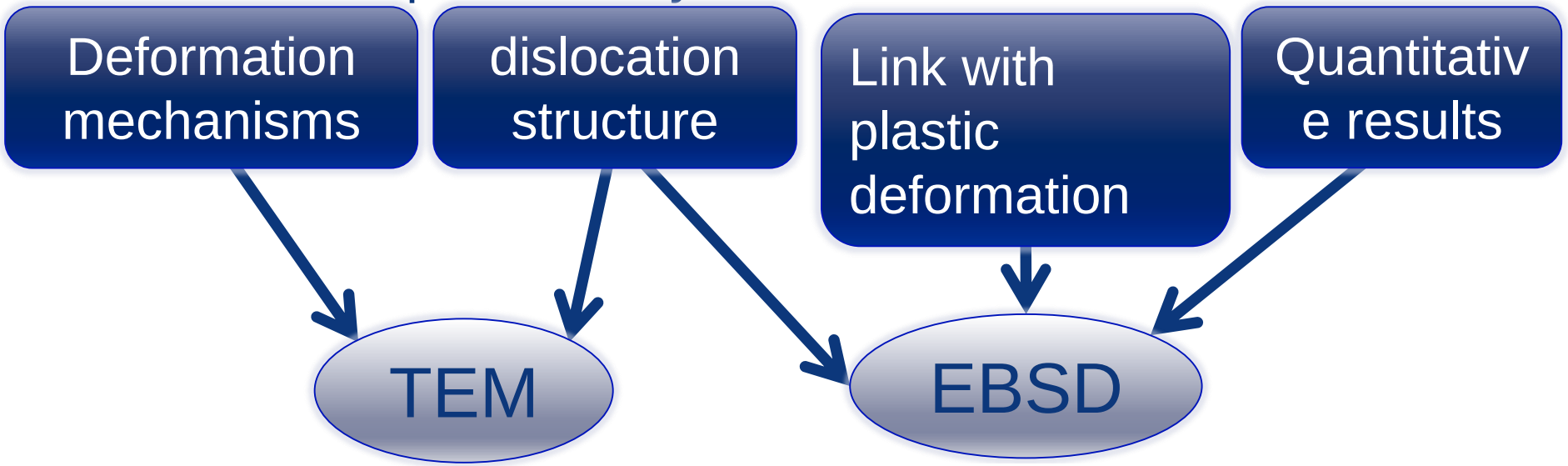


$$KAM_i = \frac{1}{K}\sum_{j=1}^{K} \Delta\Theta_{ij}, \Delta\Theta_{ij} < \Delta\Theta_{lim}$$

⇔ Misorientation average between pixel i and it neighbours j

EBSD and TEM

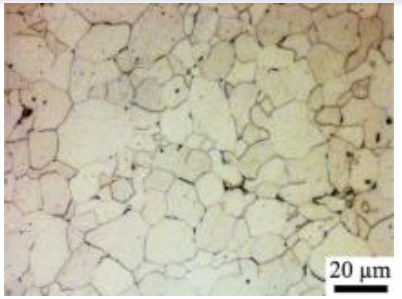
- Between complementarity and substitution



+ Imaging mode ➔ direct image of dislocation structures
+ Understand mechanisms
− Limited to small scale
− Destructive method

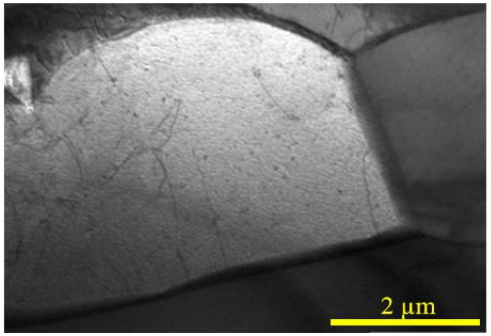
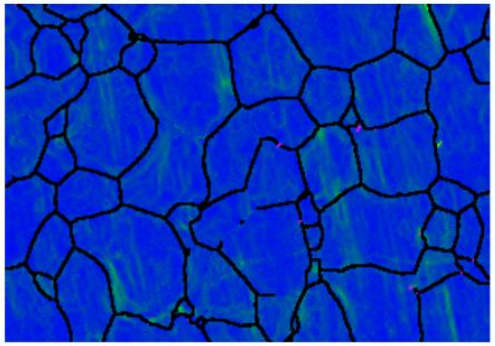
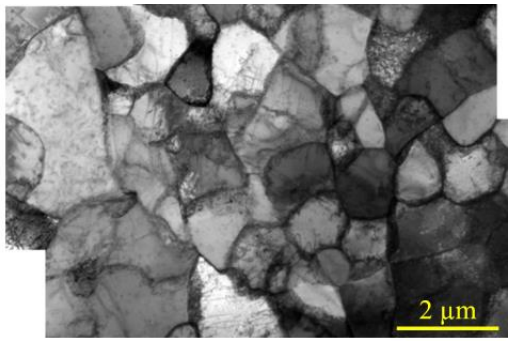
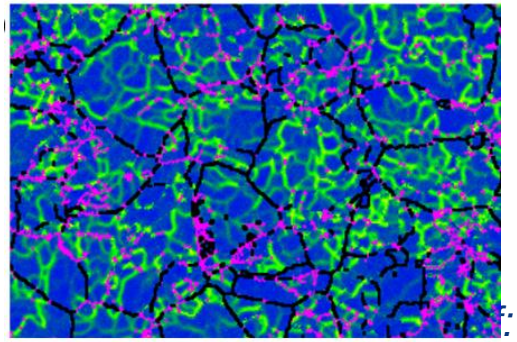
+ Assessment of dislocation densities
+ Quantitative and statistic results
+ Non destructive method (for small samples)
− Influence of experimental parameters ➔ comparative results
− Interpretation of results needed

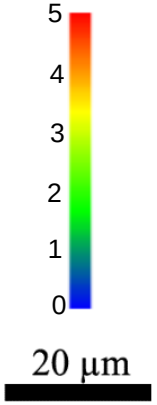
Case of a ferritic steel (1/3)



Low carbon ferritic steel

- ✓ Subjected to cyclic deformation (LCF* fatigue tests)
- ✓ Total strain control : 0.3% < $\Delta\varepsilon_t$ < 1.2%

	TEM	KAM
No deformation		
High deformation, $\Delta\varepsilon_t$ = 1.2%		



*LCF: Low Cycle Fatigue

[LEA12]

Case of a ferritic steel (2/3)

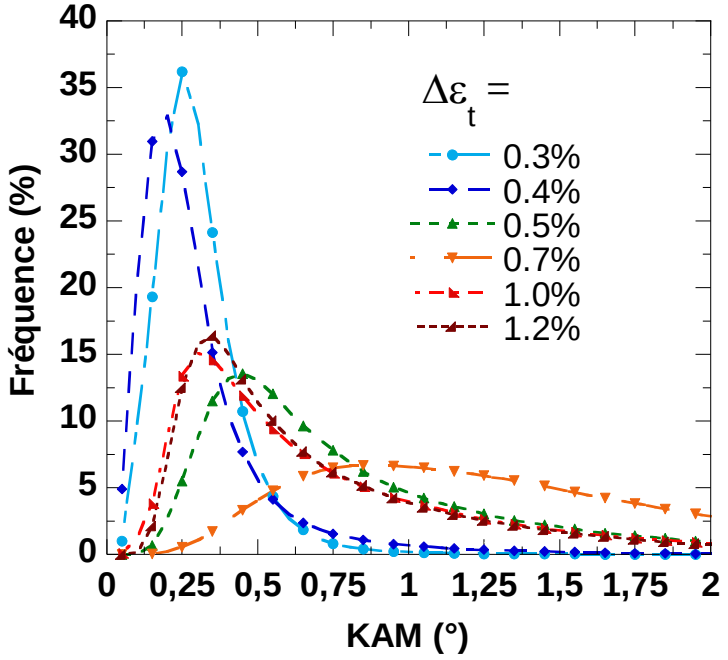
- Take into account the experimental effect

➔Define a reference state

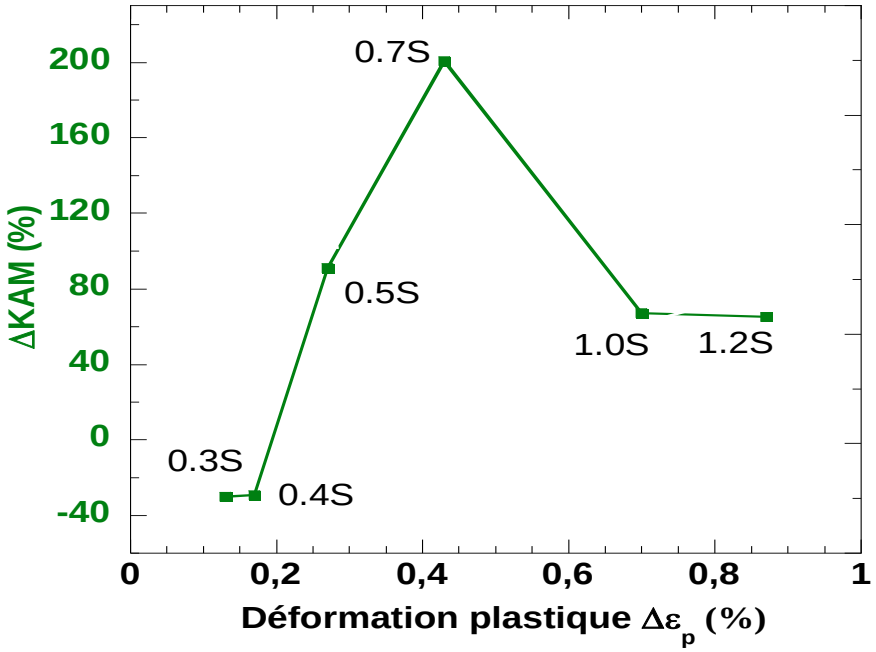
➔Calculate a variation of KAM

$$\Delta KAM = \frac{KAM - KAM_{ref}}{KAM_{ref}}$$

KAM distribution

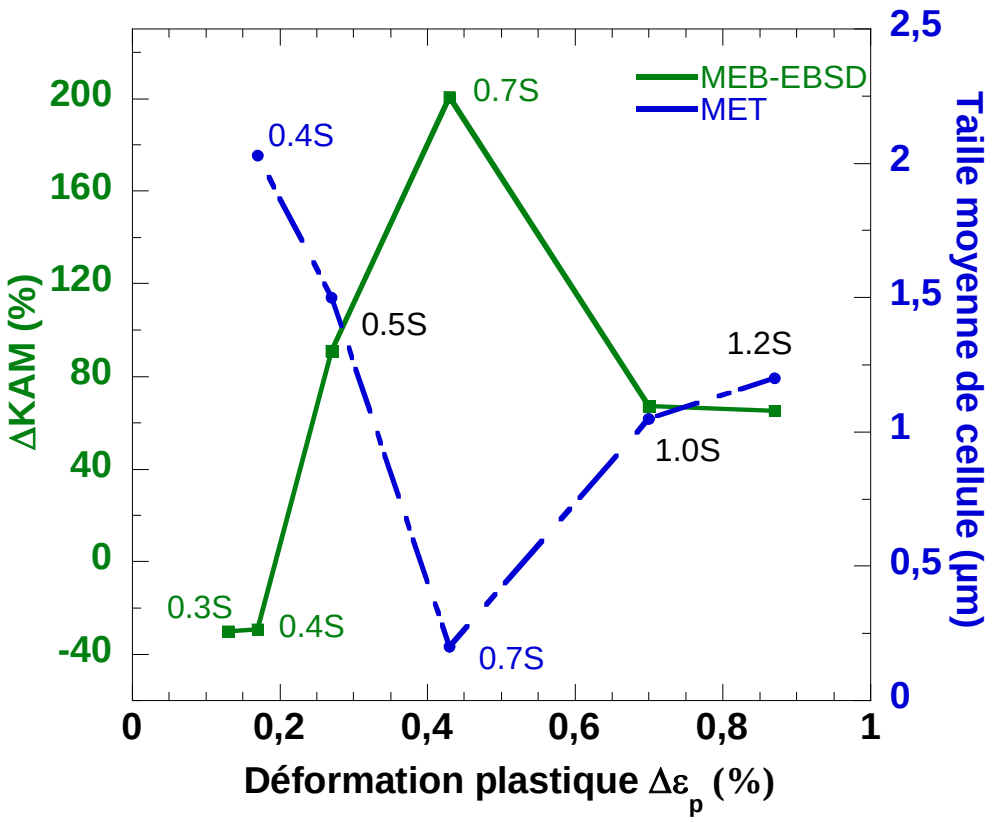


Evolution of ΔKAM with $\Delta\varepsilon_p$



Ref: [LEA12]

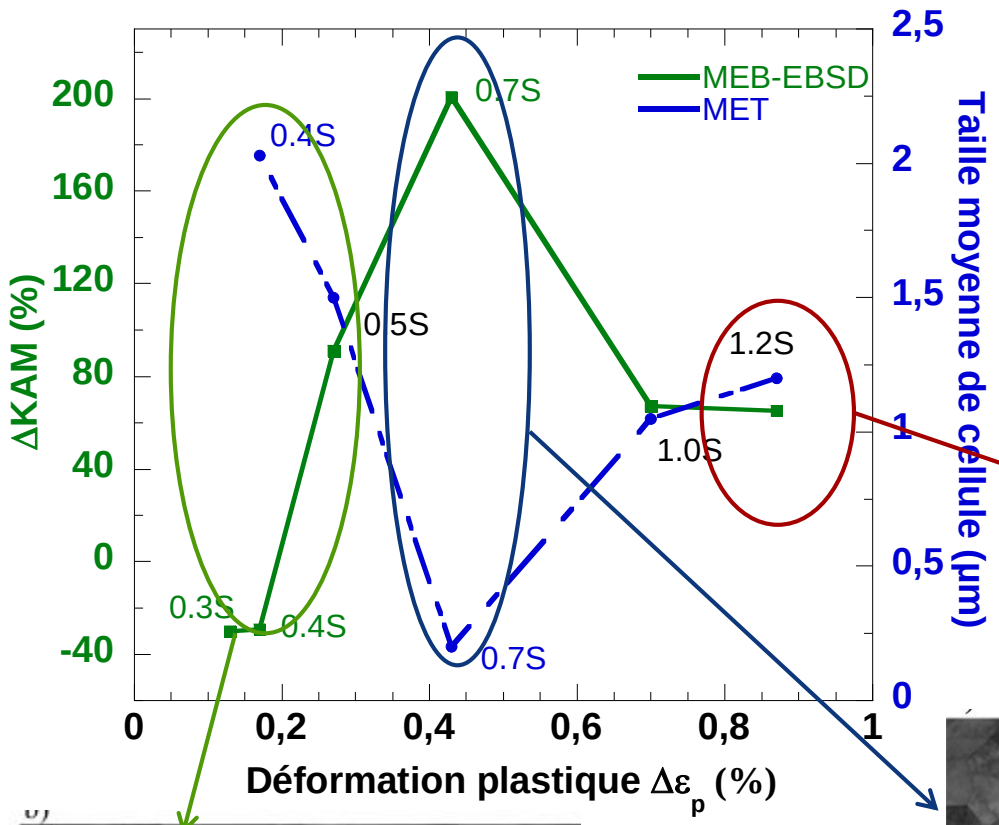
Case of a ferritic steel (2/3)



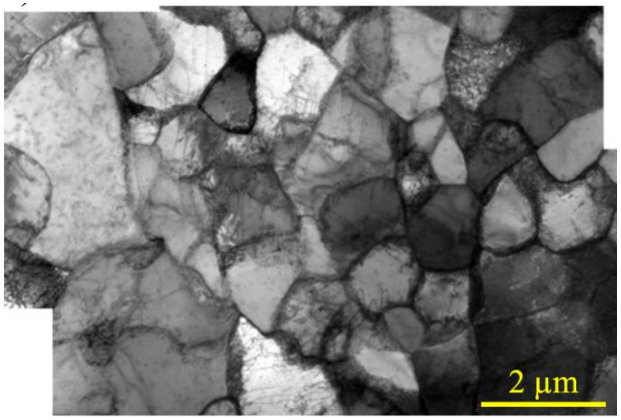
Very good fit between EBSD and TEM results

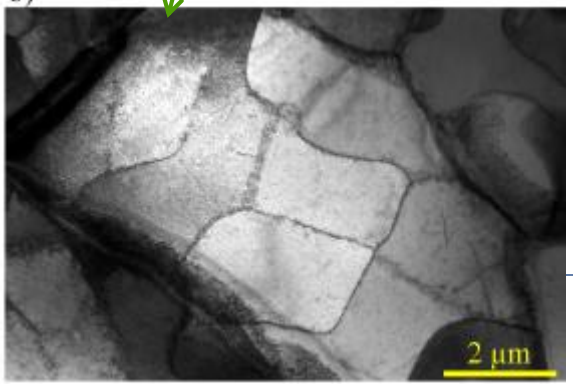
Ref: [LEA12]

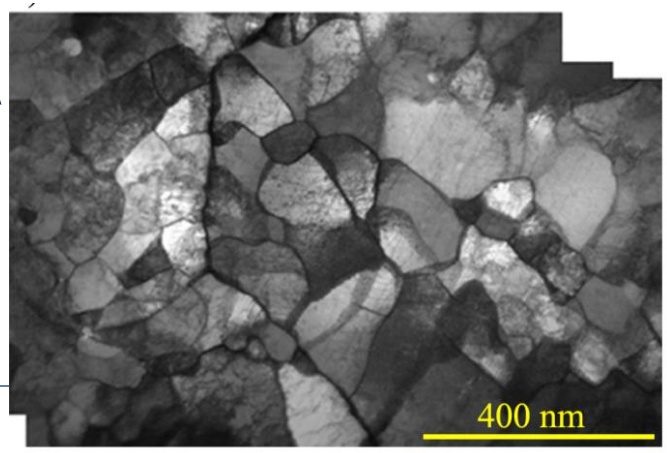
Case of a ferritic steel (2/3)



Very good fit between EBSD and TEM results







f: [LEA12]

Case of a ferritic steel (3/3)

➔Case of CC metal

➔Dislocation structure = cells

Dislocation ←➔ appearance of BD ???

- What about copper?
- What about other dislocation arrangements?
- …

EBSD = “non destructive” ←➔ DC Spark system

Ref: [LEA12]

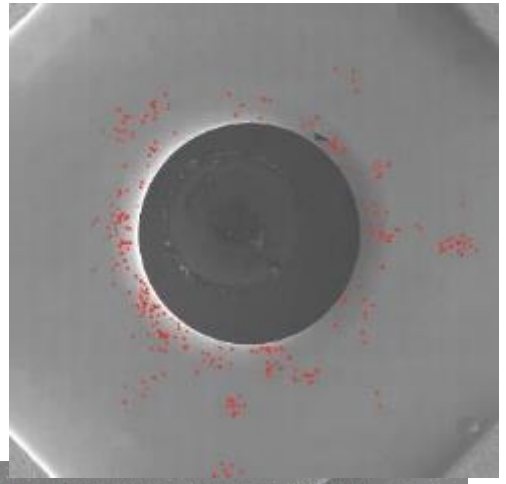
Conclusion

- Microscopy at CERN in the context of CLIC
  - BD* and FE* description
  - Dislocation structure charaterisation of pure copper and link with BD occurency
- Prespective : SEM-FIB and the 3D nanoworld

**BD: Break Down*
**FE: Field Emission*

Microscopy at CERN in the context of CLIC

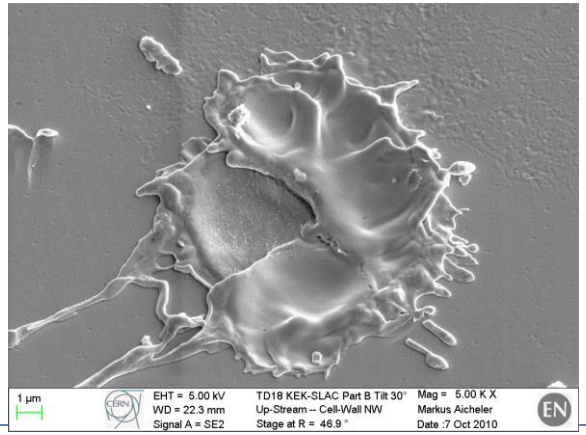
- BD and FE description
  - ✓ Charaterisation and localisation of BD
  - ➔Post-Mortem analysis

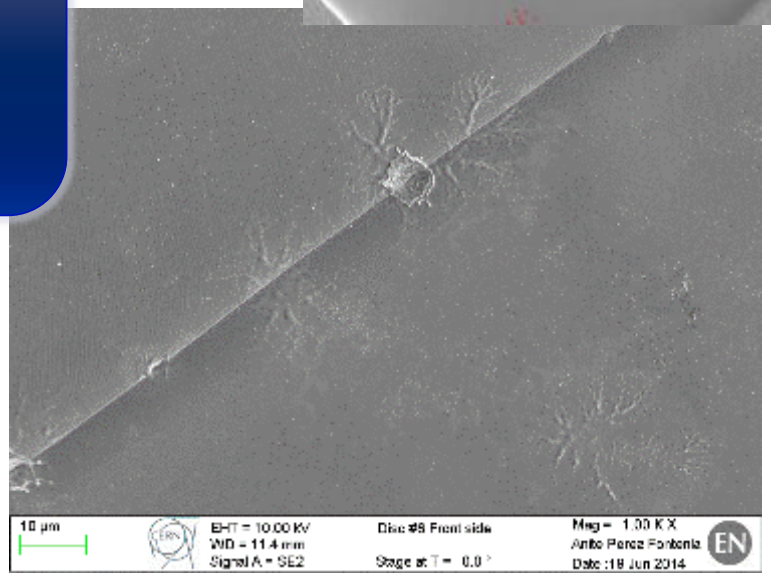


Ana Teresa PEREZ FONTENLA

SEM images review on CLIC structures after testing

Thursday 03, 12:10 - 12:30



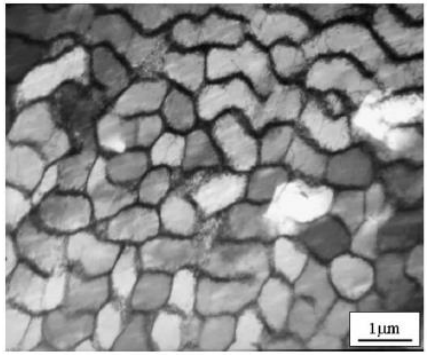


Ref: [PER14]

3. Conclusion — Microscopy at CERN in the context of CLIC

- Dislocation structure charaterisation of copper

  - ✓ Introduction of dislocation structure into Cu-OFE
  - ✓ TEM bibliography ➔ cell structure
  - ✓ Monotonic and fatigue tests + EBSD charaterisation

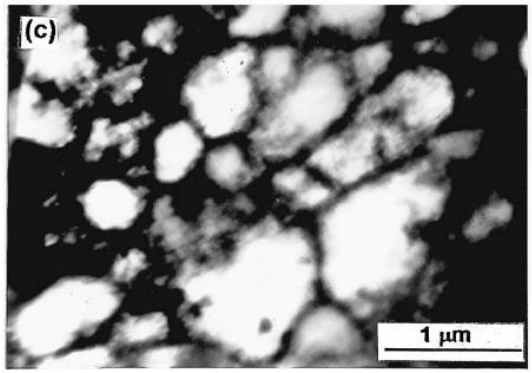


After cyclic deformation

Enrique RODRIGUEZ CASTRO

Identification of dislocations patterns in Cu-OFE for CLIC project by using EBSD

POSTER session



After tension until rupture (monotonic)

- Link with BD occurency
- DC spark system

Ref: [ZHA05] [JIA03]

Prespective : SEM-FIB

- SEM-FIB ➔ SEM microscope equiped with a focused ion column

New signals

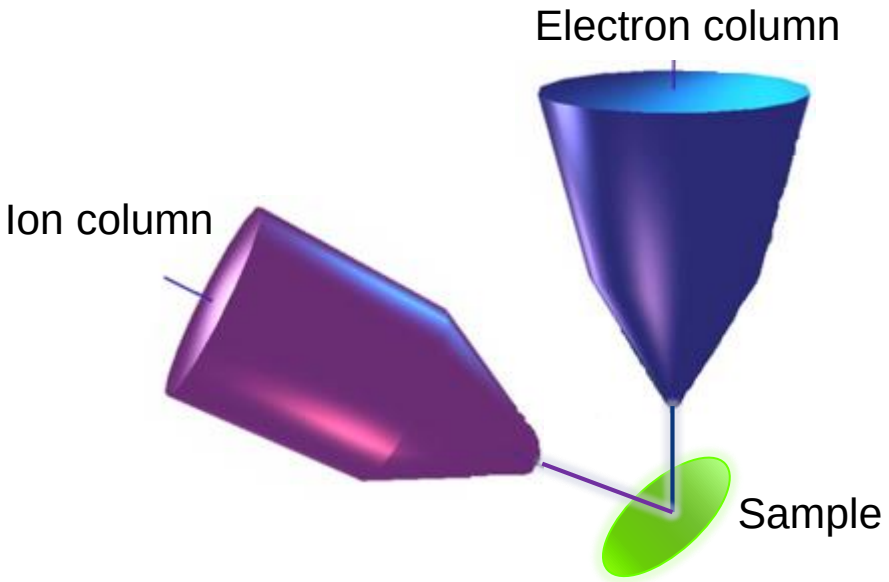
➔ Secondary ions

➔ Secondary electrons

➔ STEM*

Preferential etching

Nanomachining



**STEM: Scanning Transmission Electron Microscopy*

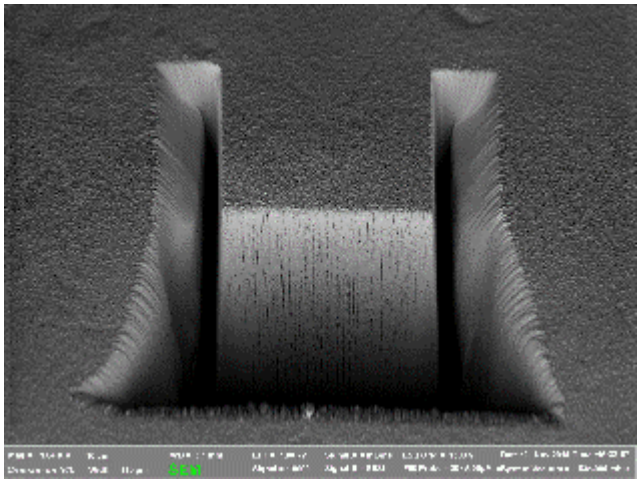
3. Conclusion — Prespective : SEM-FIB

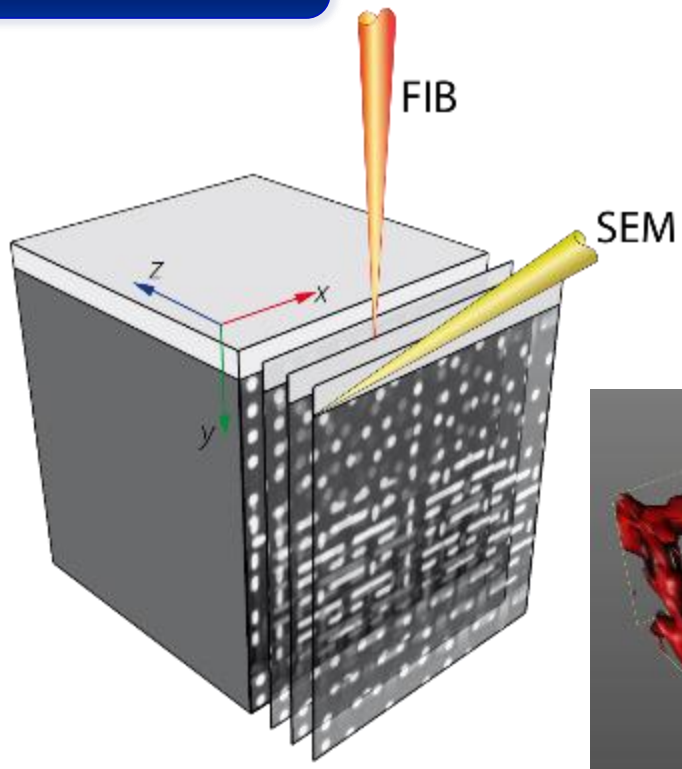
- **Dedicated material preparation**
- **Milling**

➔ Cross-sections

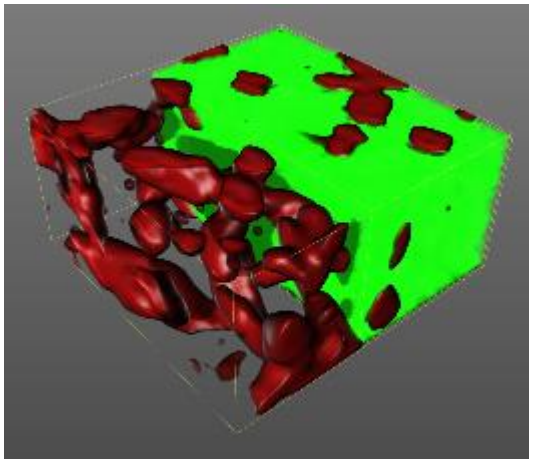
➔ 3D tomography

Preparation of the sample





Acquisistion: Milling and imaging

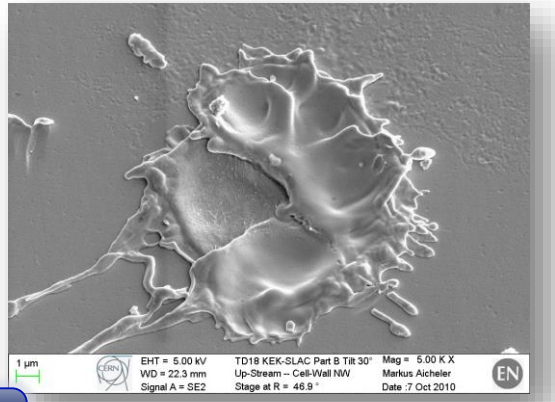


3D reconstruction

Ref: TESCAN

3. Conclusion — Prespective : SEM-FIB

- CERN is actually acquiring a SEM-FIB
- Beginning 2016
- New possibilities for CLIC accelerating RF structure:

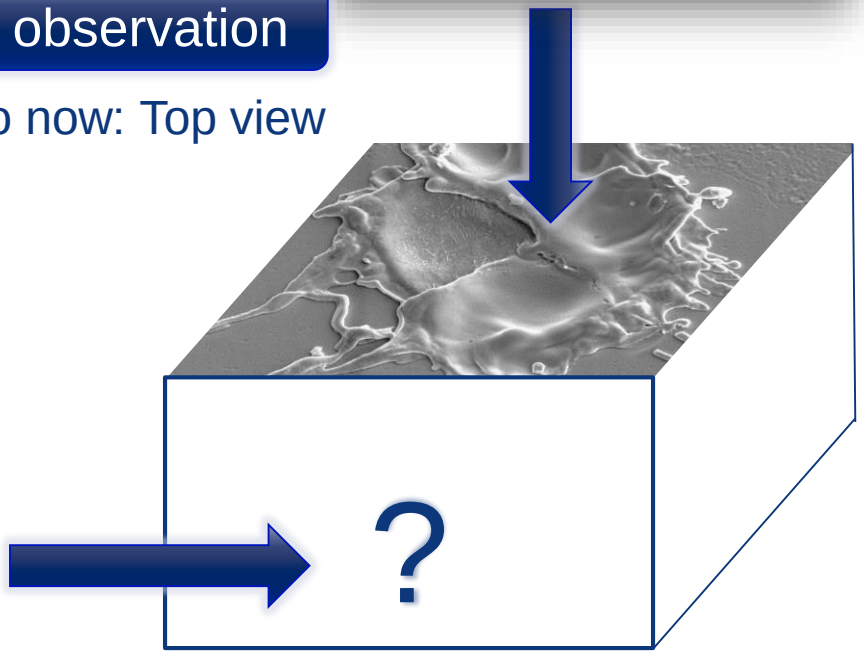


BD observation

Up to now: Top view

- ✓ Delicate preparation on selected area
- ✓ Cross section
- ✓ STEM images
- ✓ …

Prespective:
Cross-section / bulk view
EBSD, KAM



Thank you

***AMMRF**: http://www.ammrf.org.au/myscope/.*
***[BAU10]**: Baudin T., Analyse EBSD Principe et cartographies d'orientations, Techniques de l'Ingénieur, 2010, M 4 138, 1–17.*
***[JIA03]**: Jia W.P. and Fernandes J.V., Mechanical behaviour and the evolution of the dislocation structure of copper polycrystal deformed under fatigue-tension and tension-fatigue sequential strain paths, MSE A 348, 2003, 133-144.*
***[LEA12]**: Léaux F., Relation entre microstructure et fatigue d'un acier ferritique utilise dans l'industrie automobile: élaboration d'indicateurs d'endommagement, Science des matériaux: Université Lille 1 – Sciences et Technologies, 2012*
***[PER14]**: A.T. Perez Fontenla, Post-Mortem analysisTD24 R05 N1 tested in X Box 1, CERN, EDMS:1450676, 2014*
***[WRI11]**: Wright S.I., Nowell M.M. & Field D.P., A review of strain analysis using electron backscatter diffraction. Microscopy and microanalysis, 2011, vol.17, n°3,316–29.*
***[ZHA05]**: Zhang J. and Jiang Y., An experimental investigation on cyclic plastic deformation and substructures of polycrystaline copper, Int. J. Of Plasticity 21, 2005, 2191-2211.*