EBSD in TEM : Introduction to ASTAR system

Orientation and Phase Mapping with Transmission Electron Microscopes

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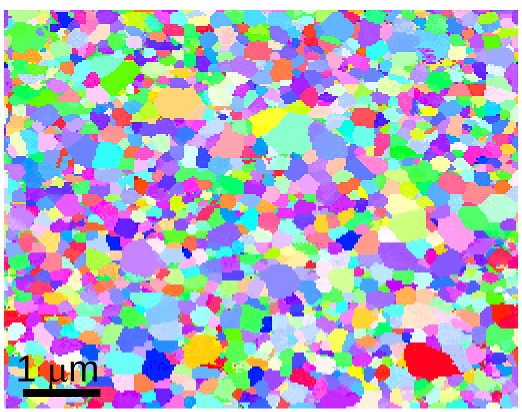
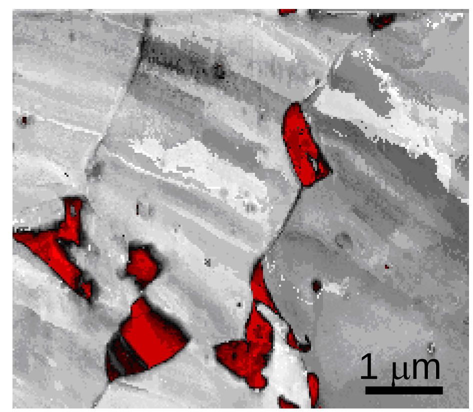
- *ACOM/TEM* : Automated Crystal Orientation Mapping on TEM
- *'DigiSTAR' :* Precession tool for TEM NanoMEGAS Advanced Tools for electron diffraction

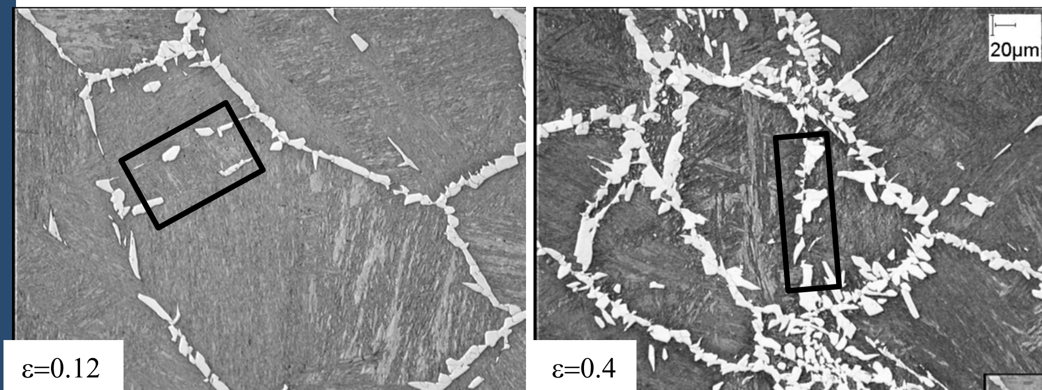
= ASTAR

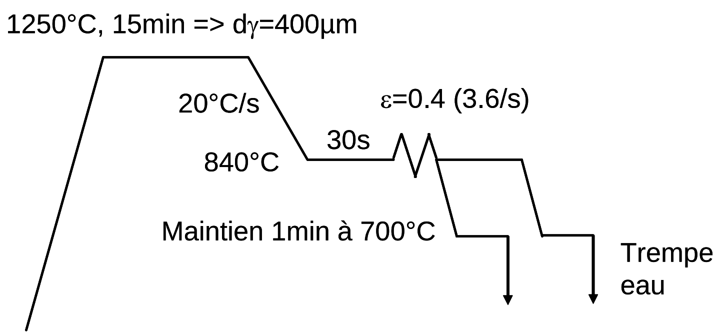


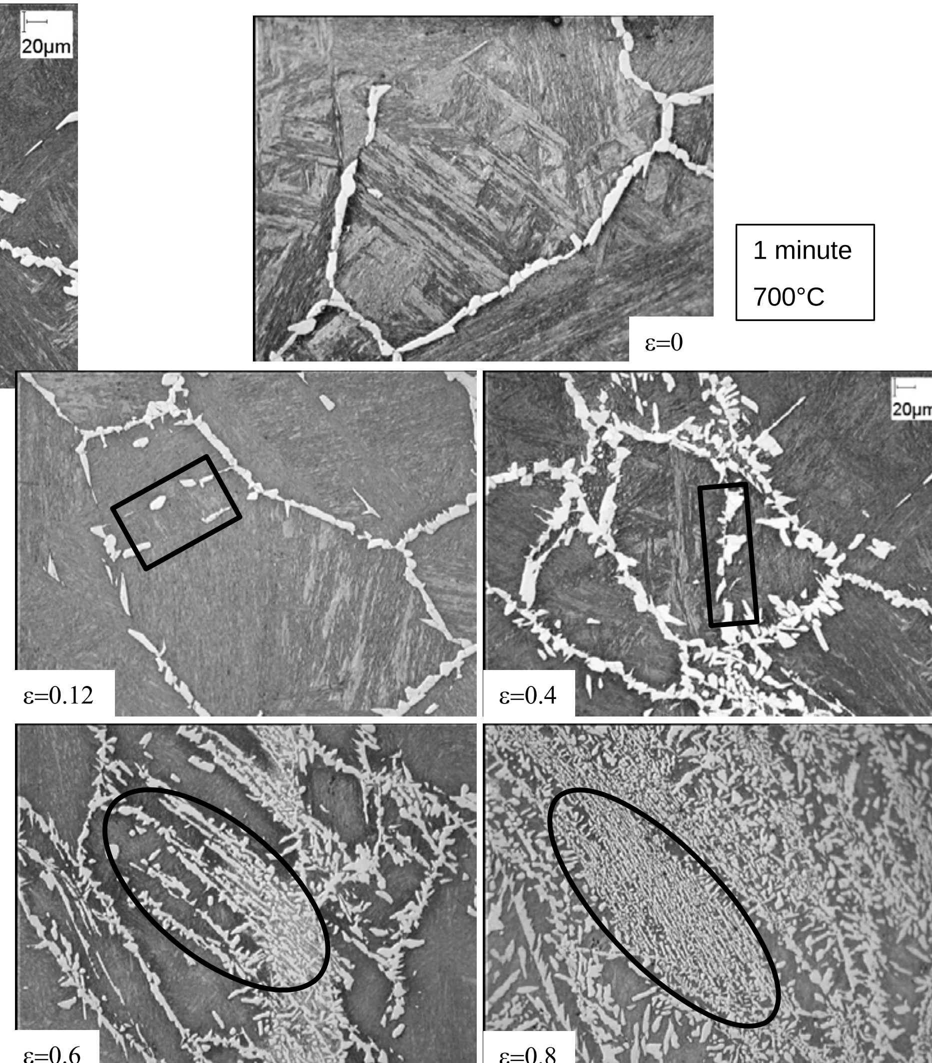
Nanocrystalline Al



TRIP steel with retained austenite

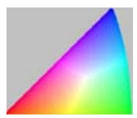

20µm
ε=0.12
ε=0.4


1250°C, 15min => dγ=400µm
20°C/s
840°C
30s
ε=0.4 (3.6/s)
Maintien 1min à 700°C
Trempe
eau

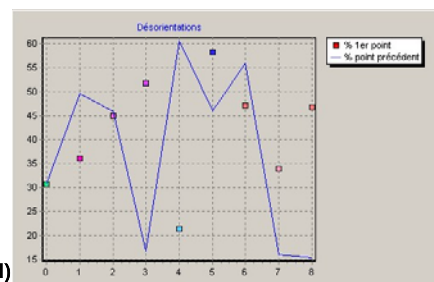

ε=0
1 minute
700°C
20µm
ε=0.12
ε=0.4
ε=0.6
ε=0.8

b) Points

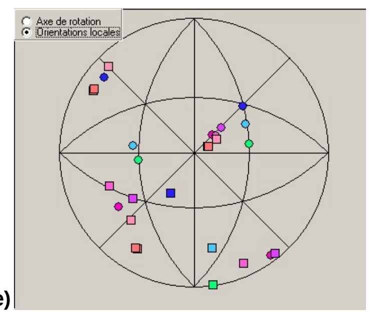
c) Code couleur

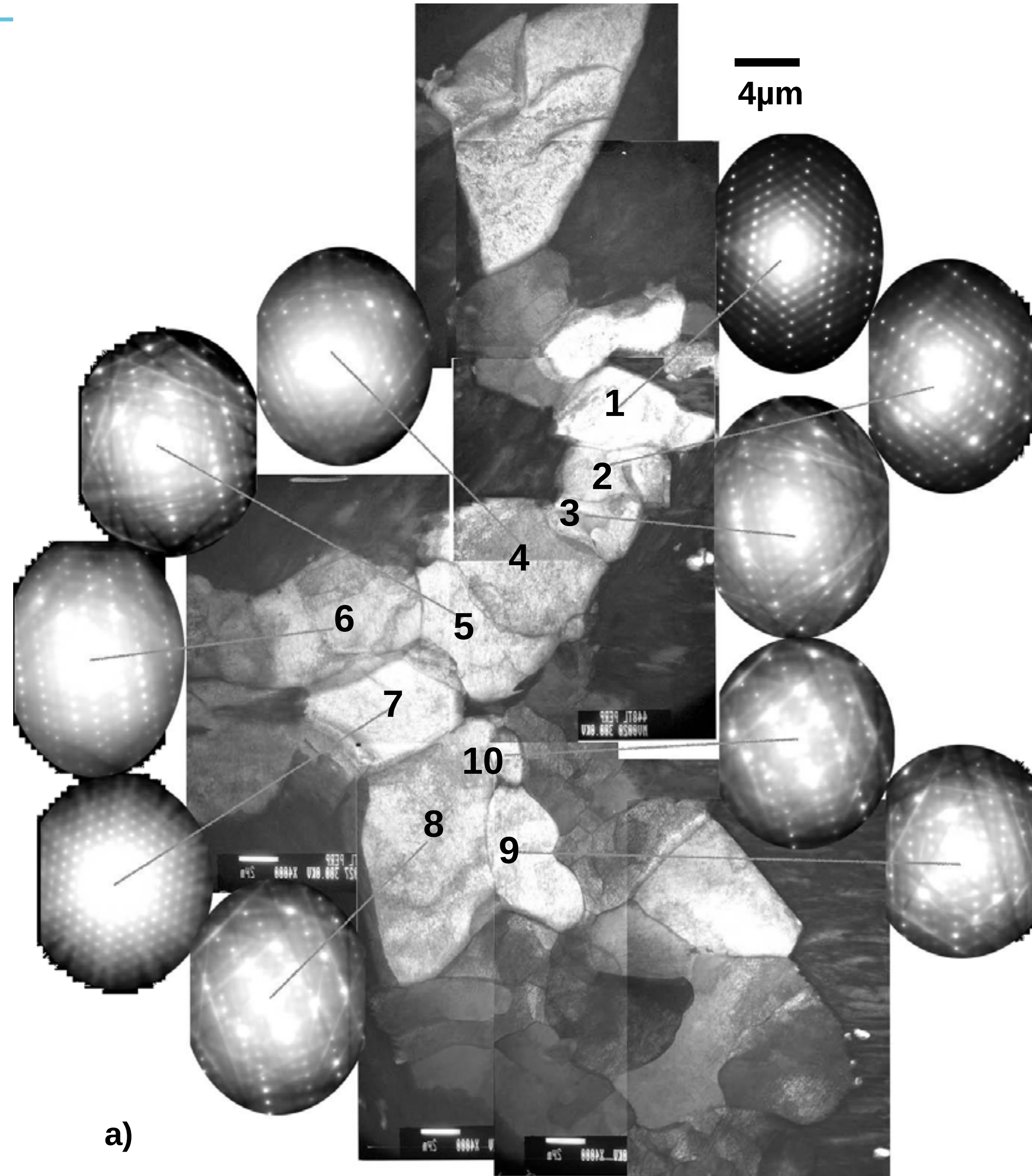


d)



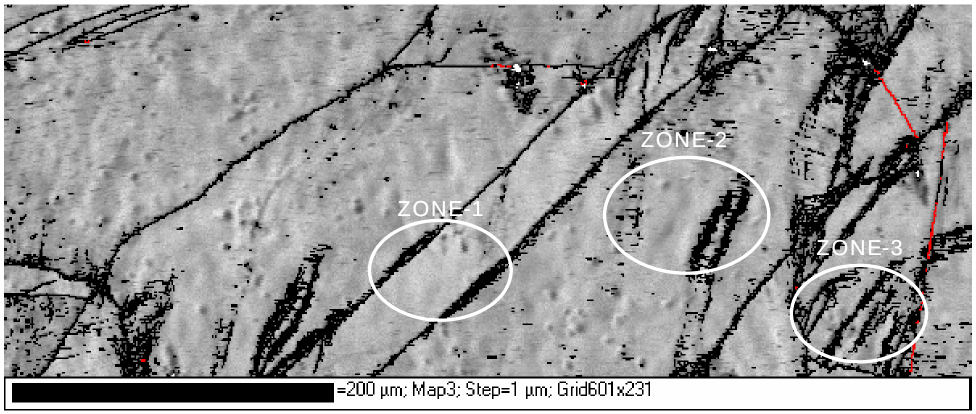
e)



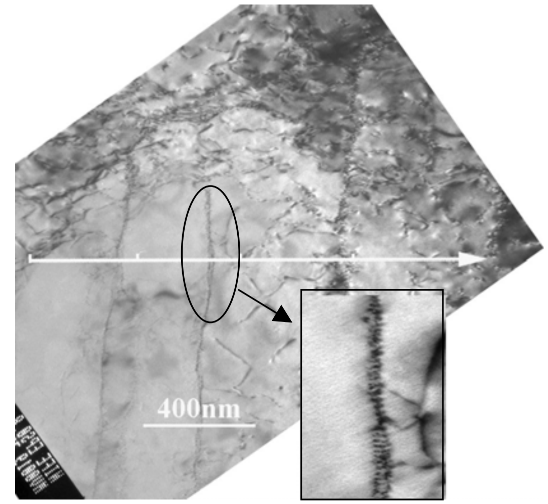


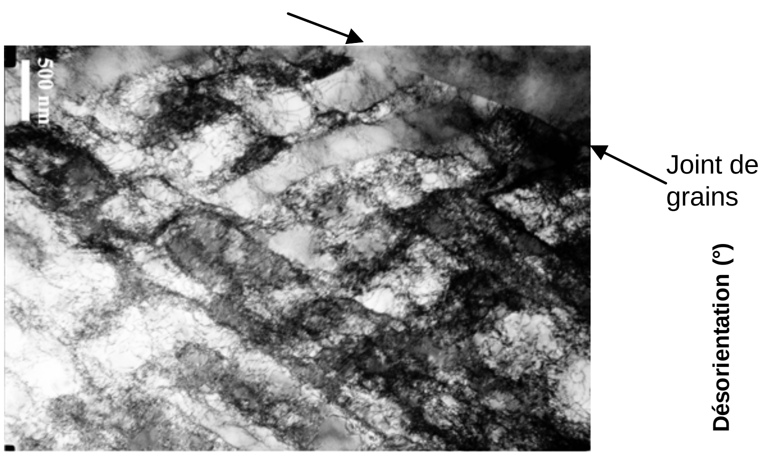
a)

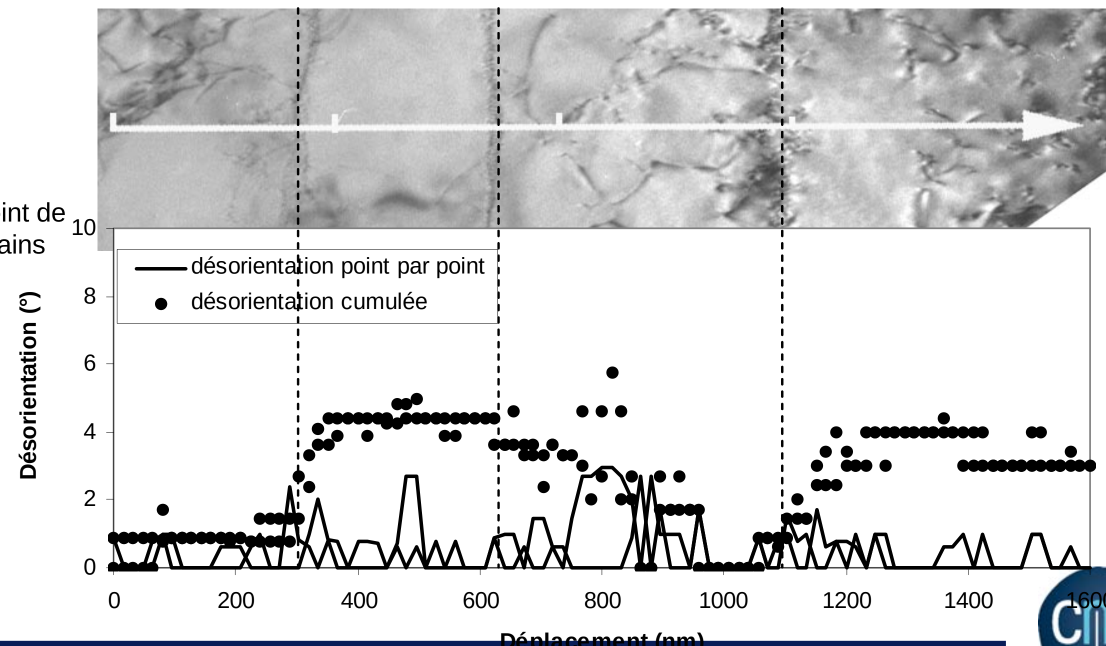
Deformed austenite (Fe-Ni alloy), EBSD quality contrast, and TEM observations



EBSD index quality contrast





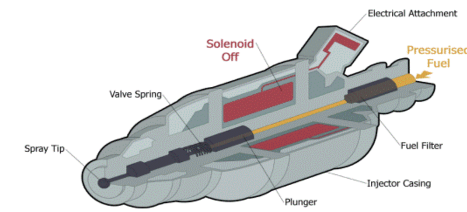


BF TEM observations

1) Example of application in metallurgy

Recrystallization of ferritic stainless steels

Collaboration with N. Meyer, Ugitech



2) ACOM/TEM

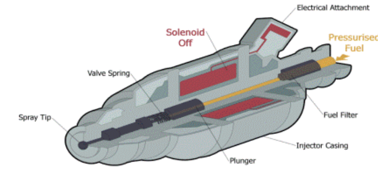
Template matching

Template generation and pattern acquisition

3) ASTAR

Combining orientation/phase identification with Electron Precession

Microstructural optimization for magnetic actuators



- Alloys compositions

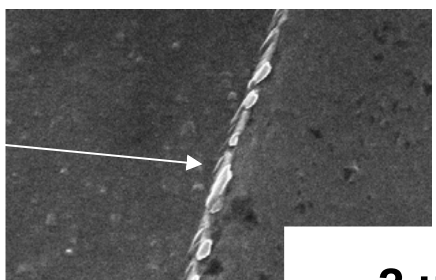
AISI	EN	C	N	Cr	Nb
430	1.4016	0.017	0.028	16.083	0.004
430Nb	1.4511	0.015	0.012	16.284	0.27

- Ferritic Stainless steel 430 (A1 = 880°C)

- Stabilized Ferritic stainless steel: 430Nb (feriitic at all T)

Cr_2N

% Cr

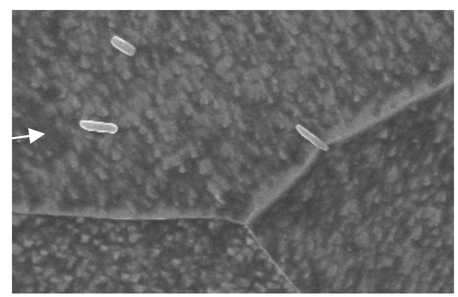


2 µm

Area without Chromium

Nb(C,N)

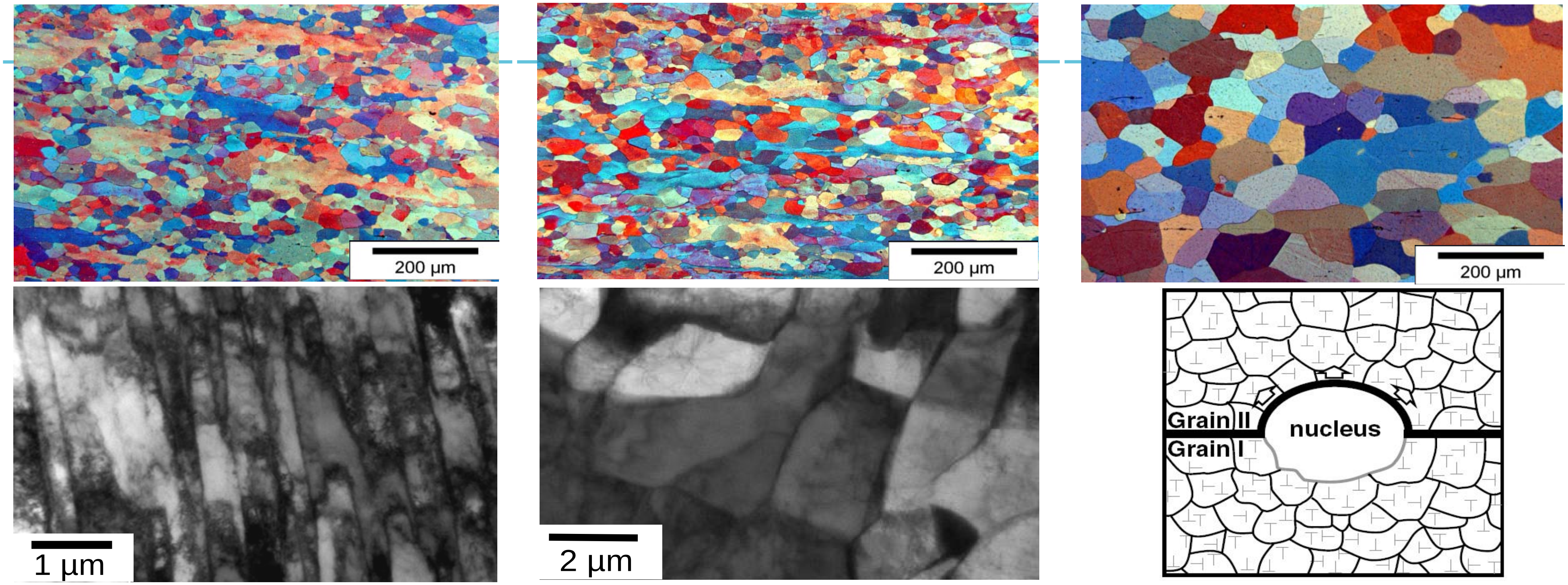
% Cr



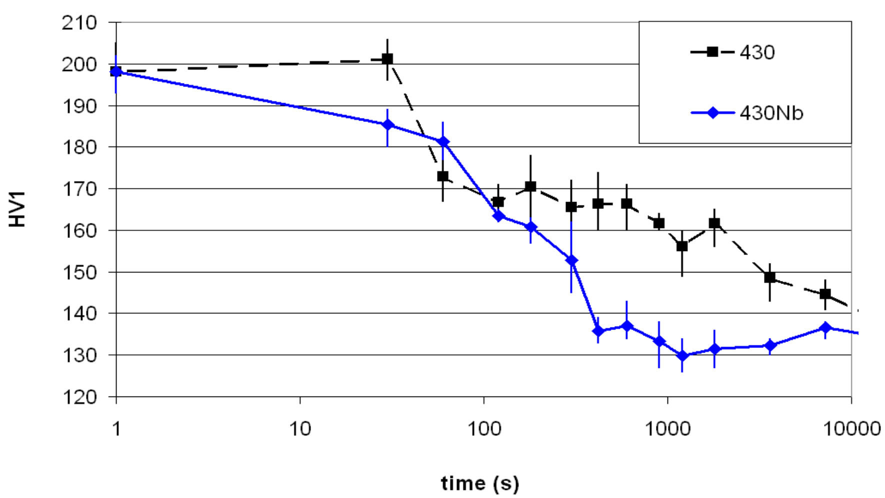
Deformed state | **Static recovery** | **Static recrystallization**

Grain scale

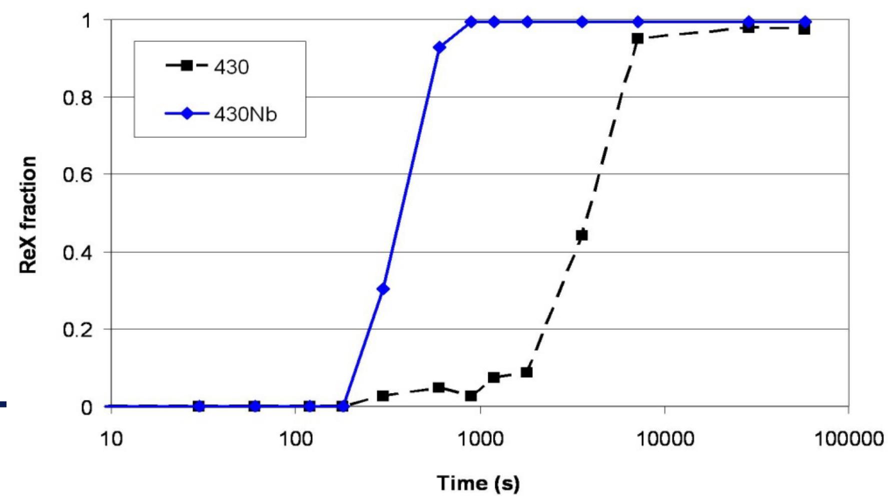
Subgrains



Recovery

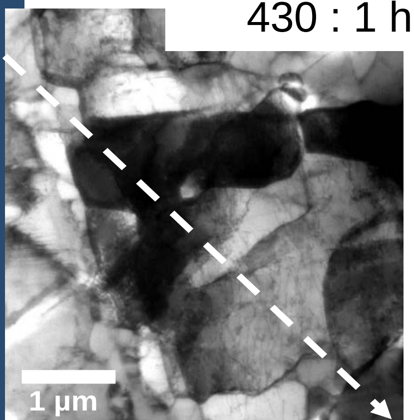


Recrystallized fraction

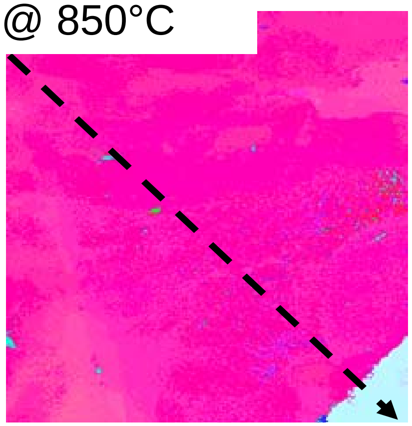


- Recrystallization mechanism
  - Nucleation
    - We did not observed large misorientation, even in recovered area

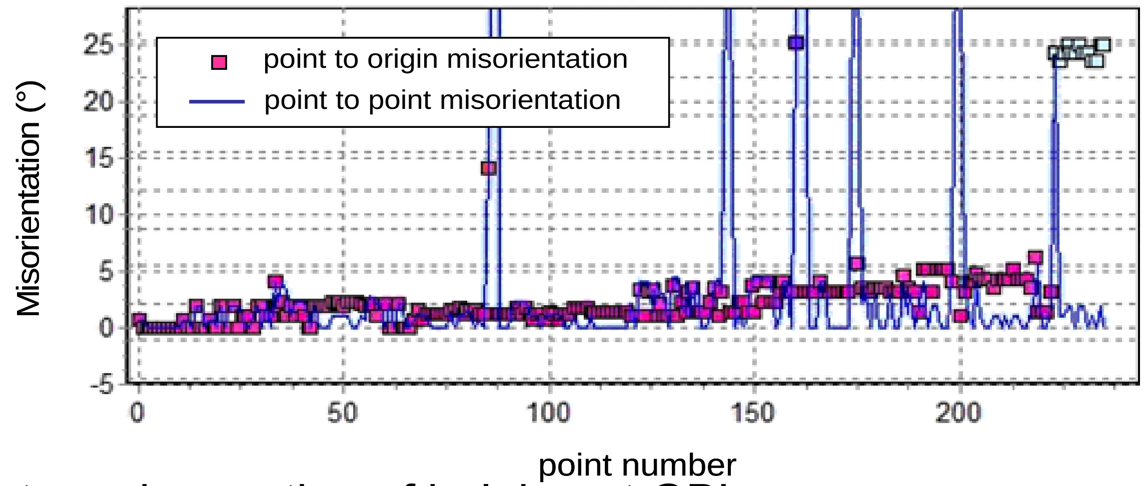
430 : 1 h @ 850°C



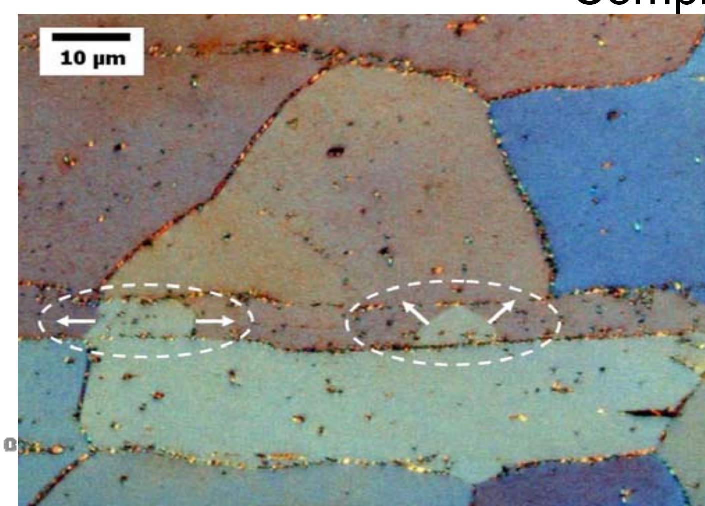
Bright Field



Orientation map



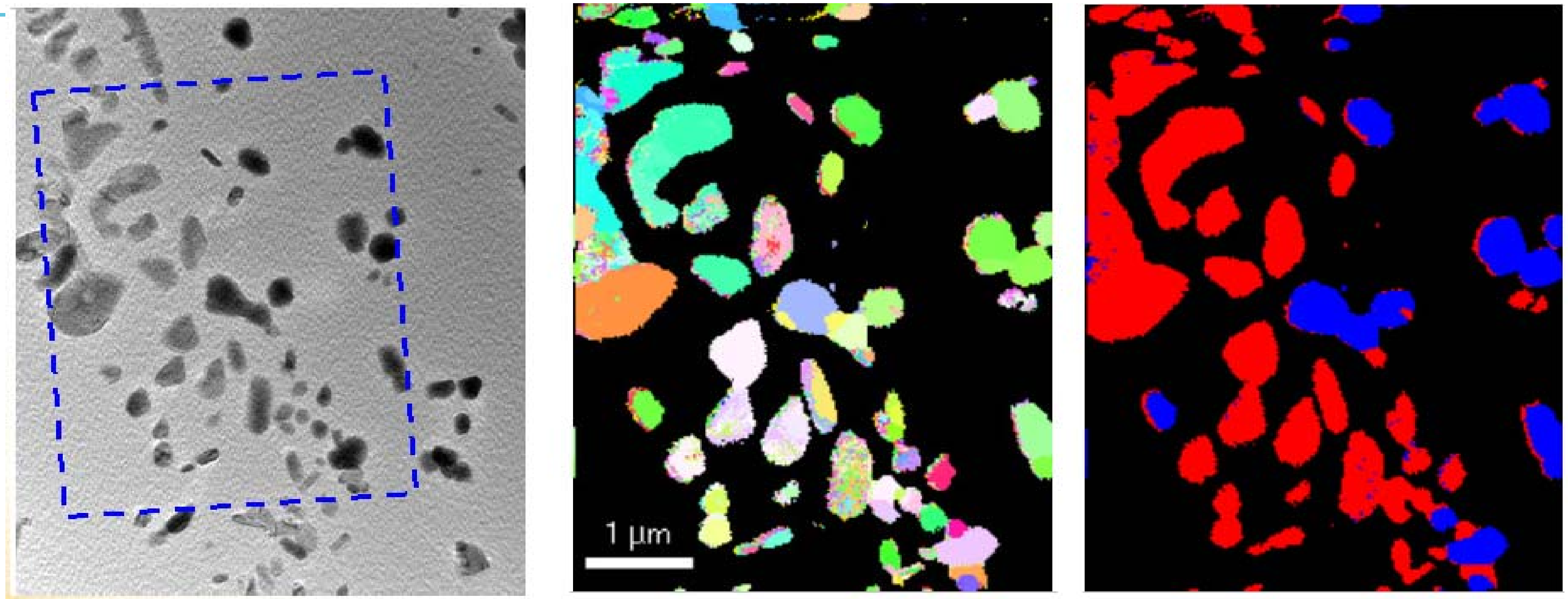
Complementary observation of bulging at GB's



430 : 7 min @ 850°C

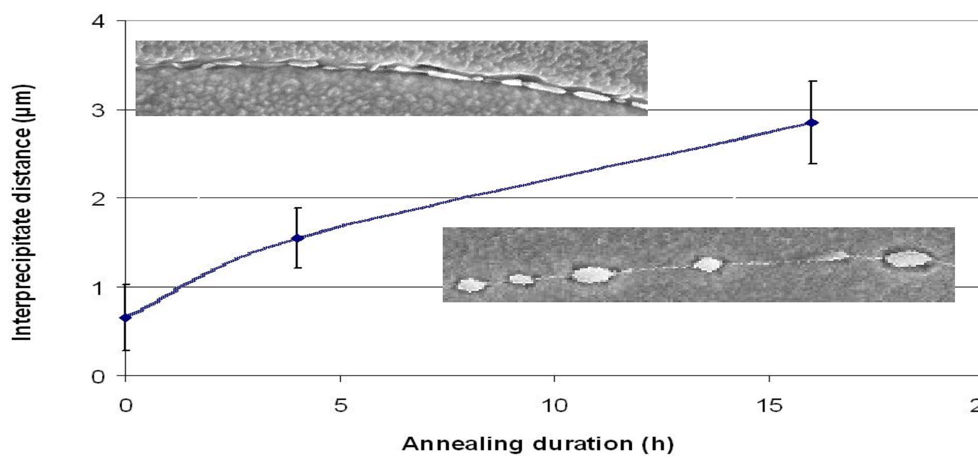
Nucleation mecanisms : SIBM, typical for law strain level

- Identification of the precipitation at the GBs (alloy 430) :



Phase identification: carbides (blue), and nitrides (red)

Recrystallization starts when precipitates have coarsened



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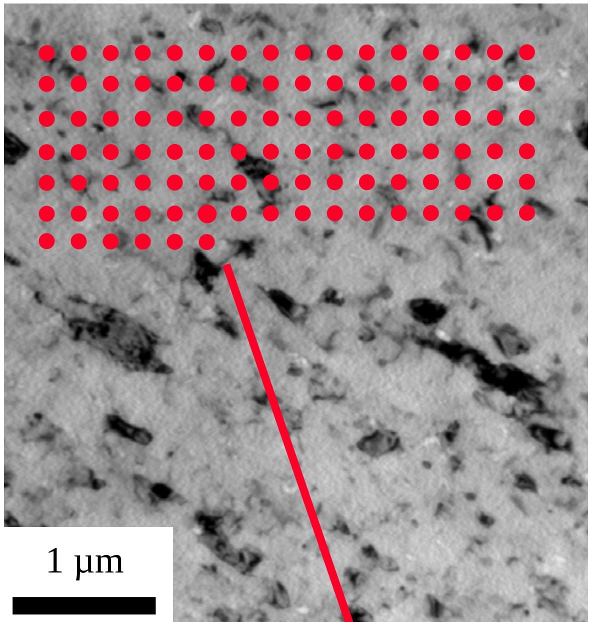
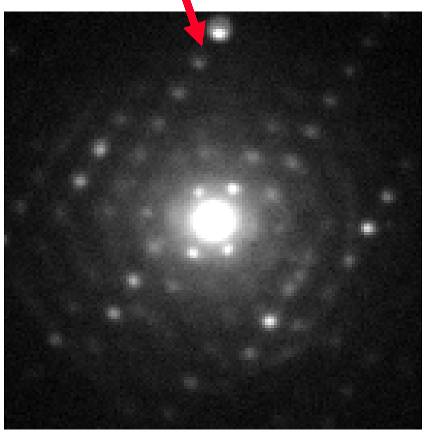
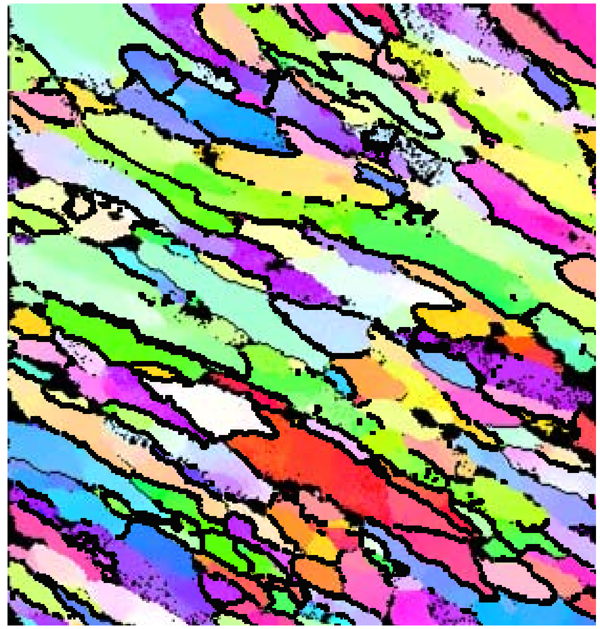
2) ACOM/TEM : Automated Crystal Orientation Mapping

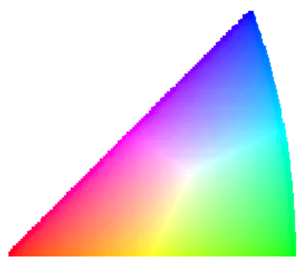




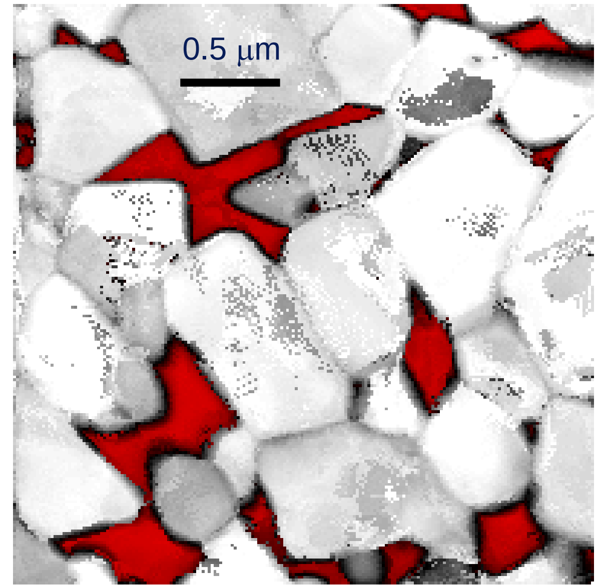
Severely deformed 7075 Al. Alloy



Orientation map (Al alloy)

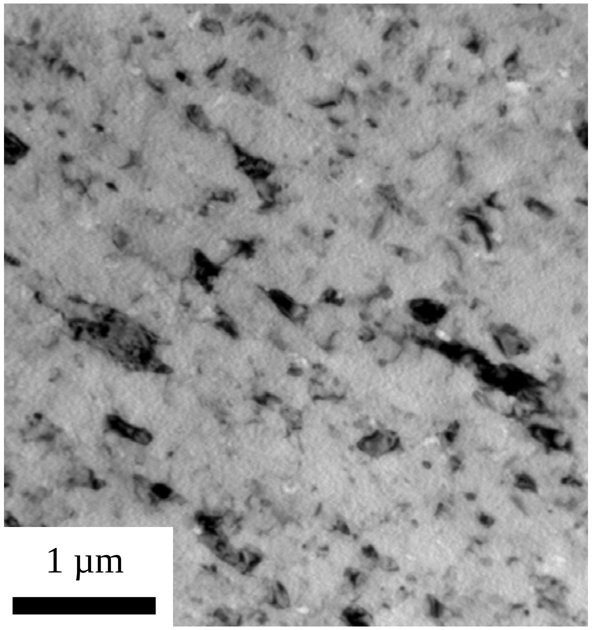


WC-Co

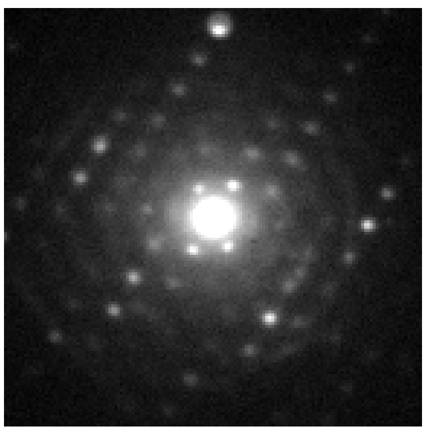


Phase map WC-Co

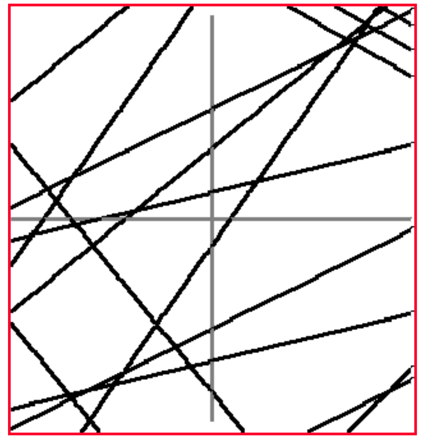
ACOM/TEM : Automated Crystal Orientation Mapping

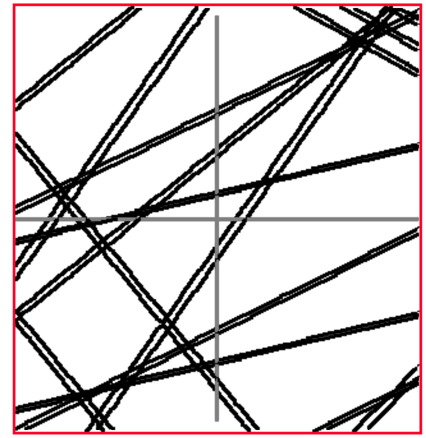


Severely
deformed
7075 Al. Alloy



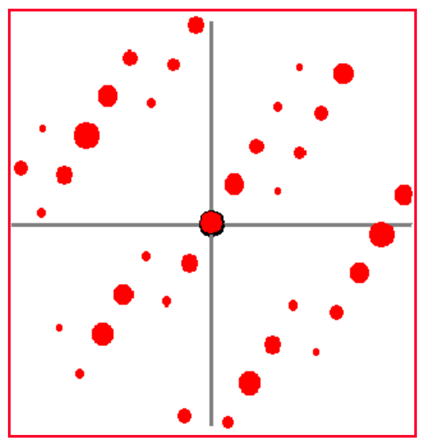
Kikuchi pattern

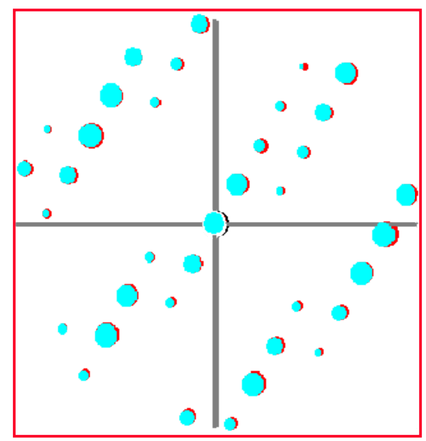




Orientation Ω

Orientation Ω + Ω'(= Ω +0.1°)

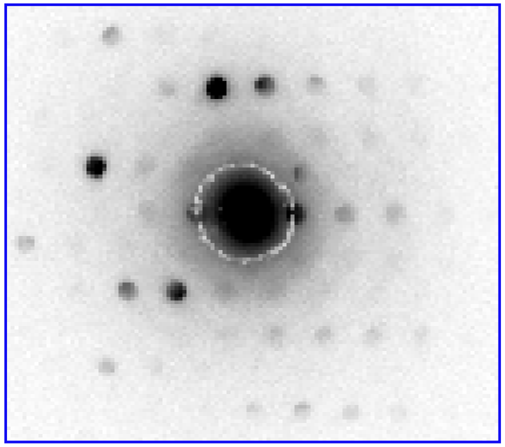




Bragg Spot pattern

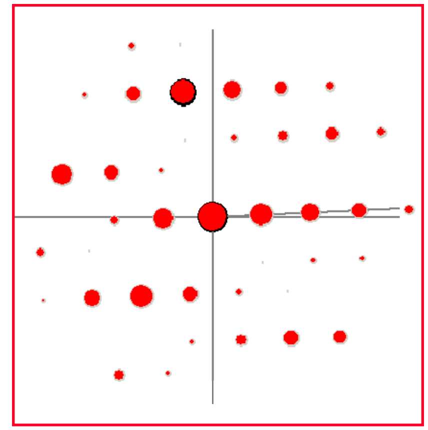
ACOM/TEM : Orientation Indexing

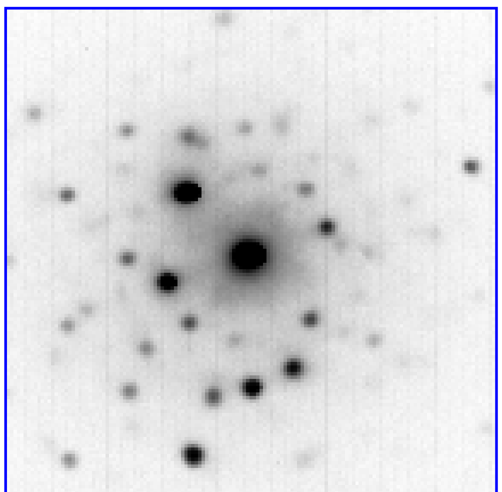
Diffraction pattern for a randomly oriented crystal



orientation
$(\varphi_1,\phi,\varphi_2)$

Calculated diffraction pattern





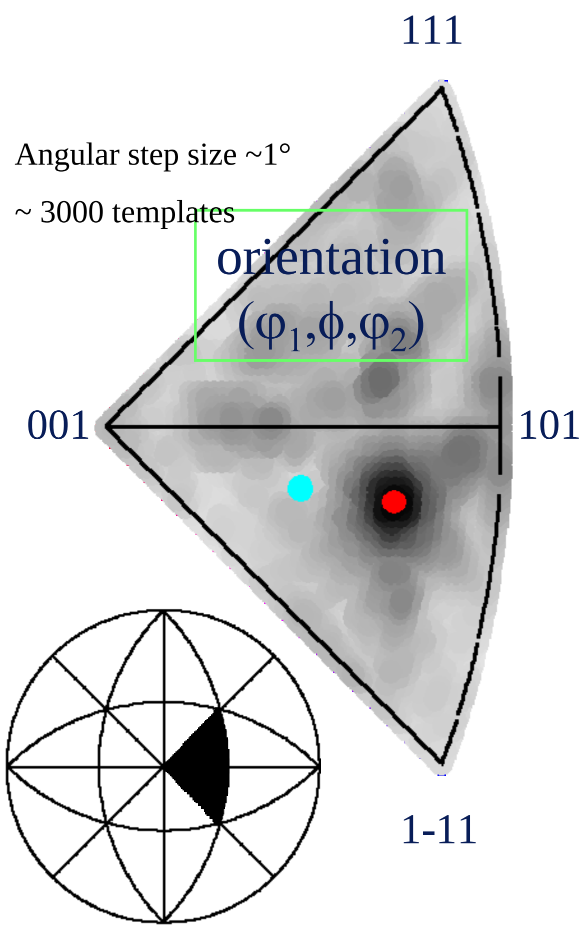
Austenite, ferrite, one or two grains?

ACOM/TEM : Crystallographic orientation identification

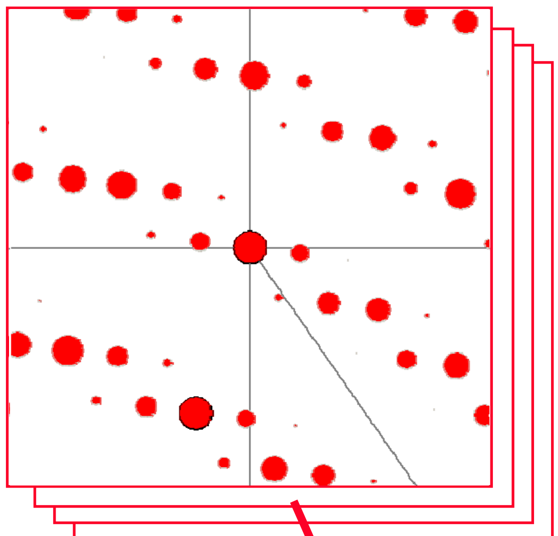
Stereographic projection (cubic)

Angular step size ~1°

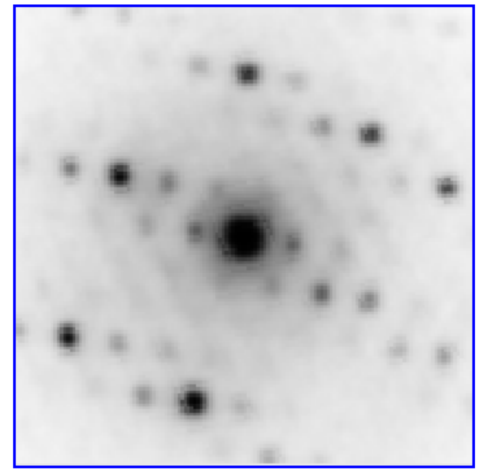
~ 3000 templates



Pre-calculated templates



Acquired pattern



$$Q(i) = \frac{\sum_{j=1}^{m} P(x_j,y_j)\, T_i(x_j,y_j)}{\sqrt{\sum_{j=1}^{m} P^2(x_j,y_j)}\sqrt{\sum_{j=1}^{m} T_i^2(x_j,y_j)}}$$

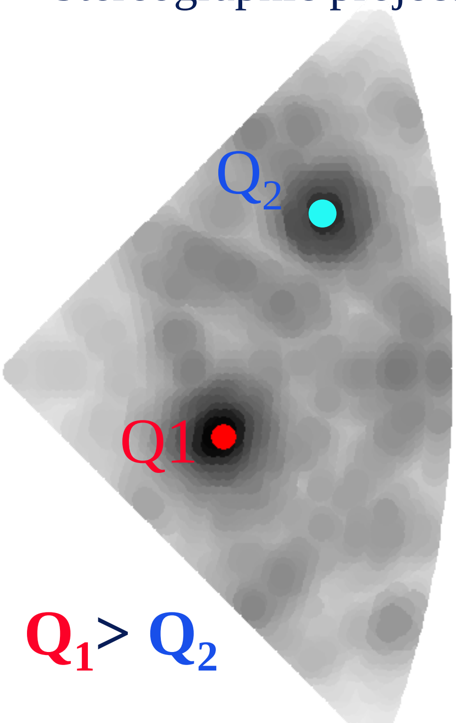
Correlation index

Template Matching

Rauch,E.F. & Dupuy,L. (2005). Arch. Metall. Mater. 50, 87–99.

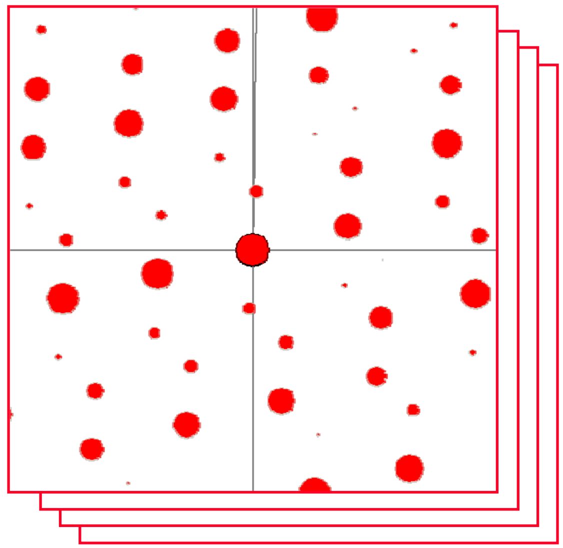
ACOM/TEM : Reliability

Stereographic projection

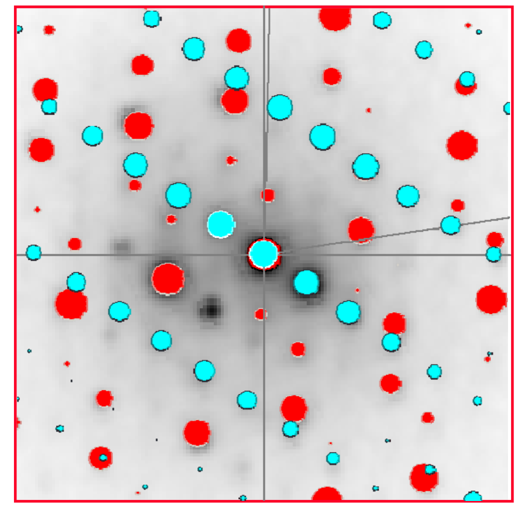


$Q_1 > Q_2$

Templates for copper



Multiple Superimposed diffraction patterns at grain boundaries



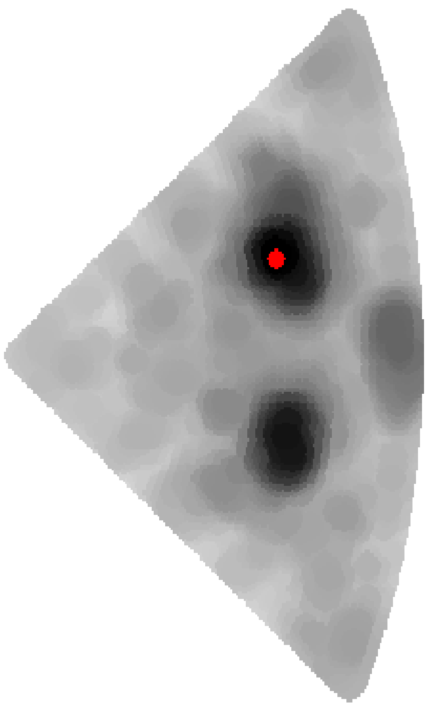
$$R = 100\,(1 - Q_2/Q_1)$$

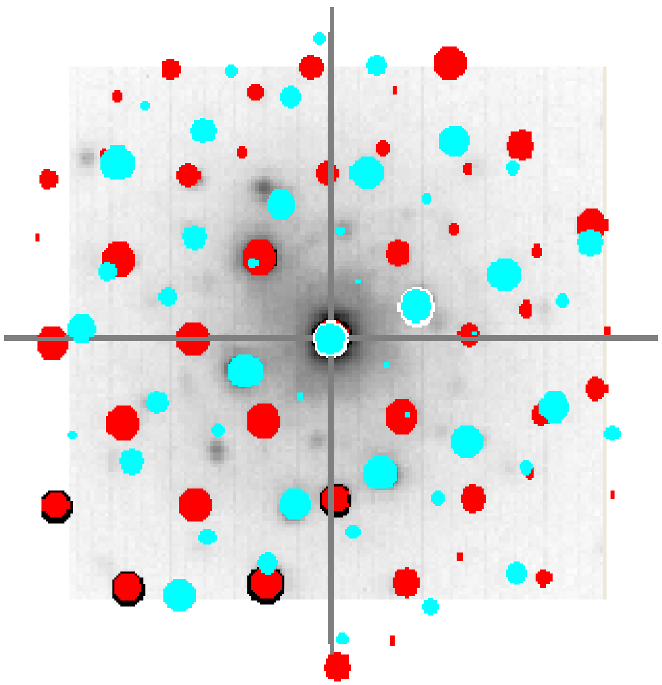
Reliability

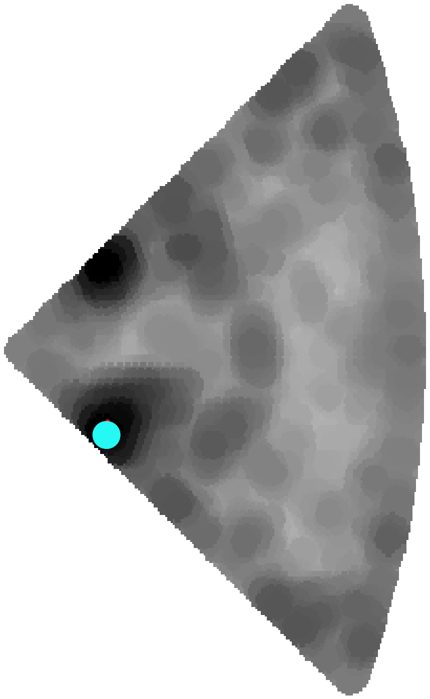
ACOM/TEM : Phase identification and reliability

Ferrite : Index $I_1 = 553$

Austenite : Index $I_2 = 389$





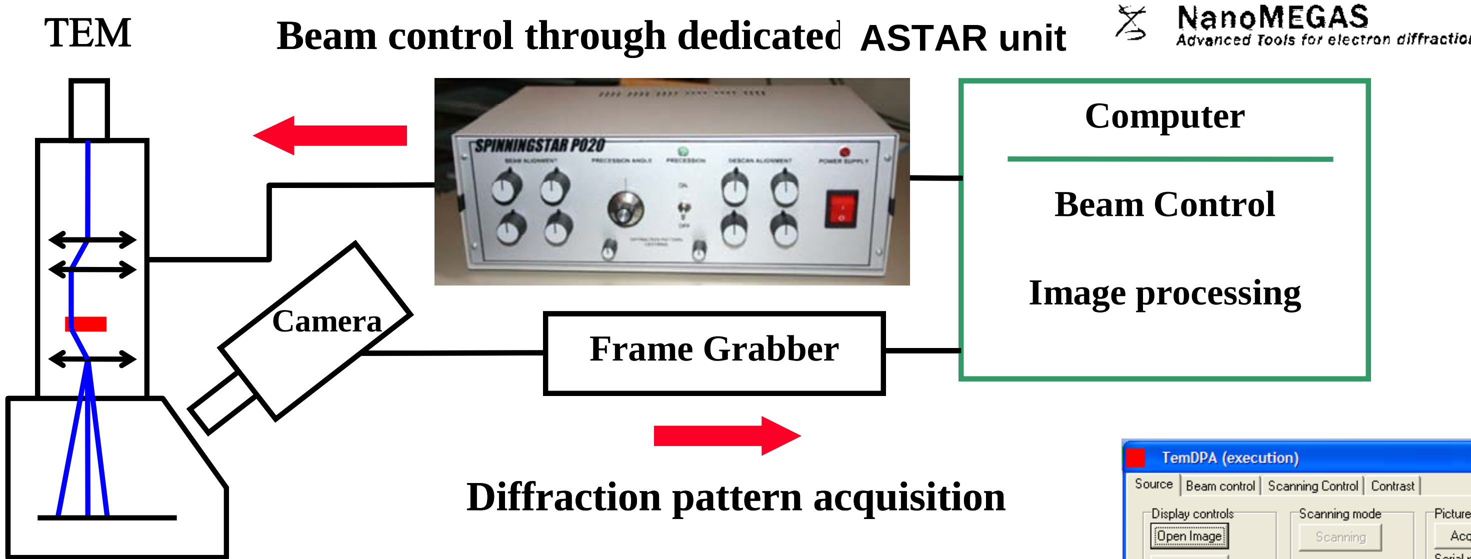


Austenite, ferrite ?

$$P_R = 100\,(1 - I_2/I_1)$$

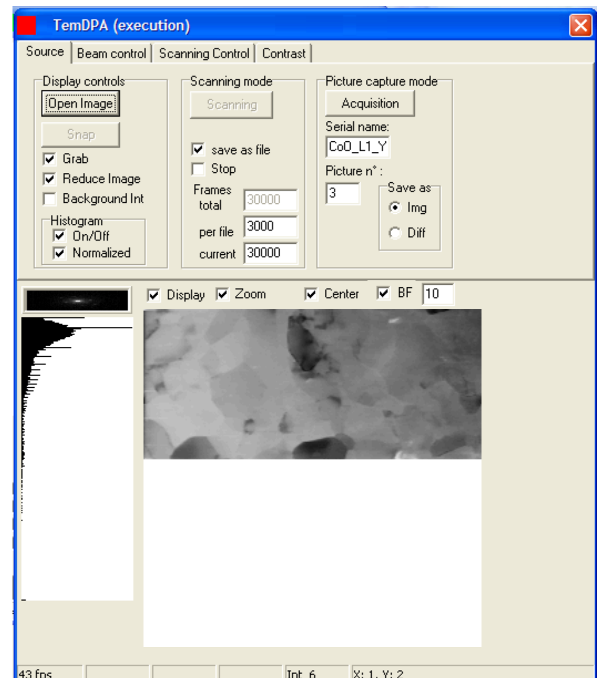
Phase reliability (= 30)

TEMdpa : Diffraction Pattern Acquisition



Features:

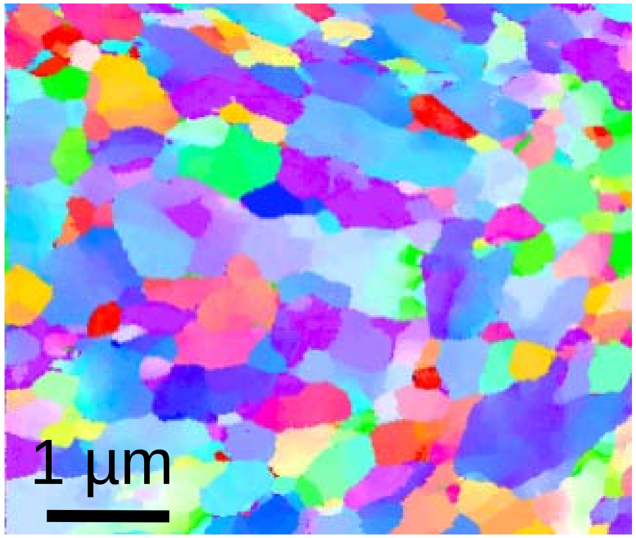
- up to 180 fps
- easy selection of the area of interest
- single or movie type DP acquisition
- brightness, contrast, exposure time control
- On line Virtual Bright Field construction

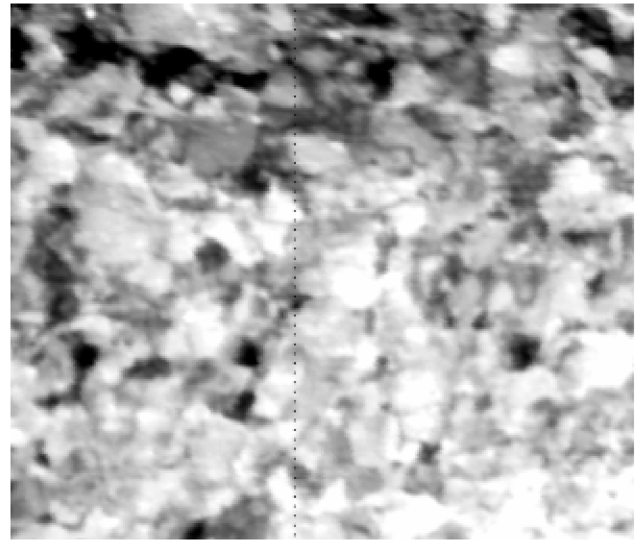


'Bright field' : Heavily deformed Cu(ε=8) (ECAP)

400x300 steps

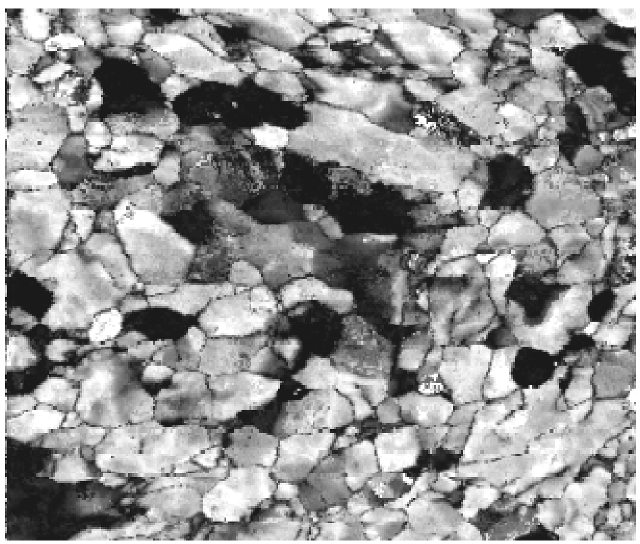
14 nm

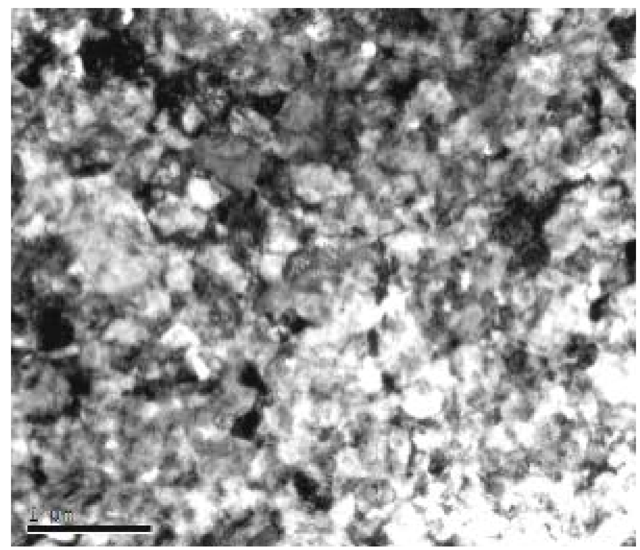




Virtual bright field

Reliability





Bright field

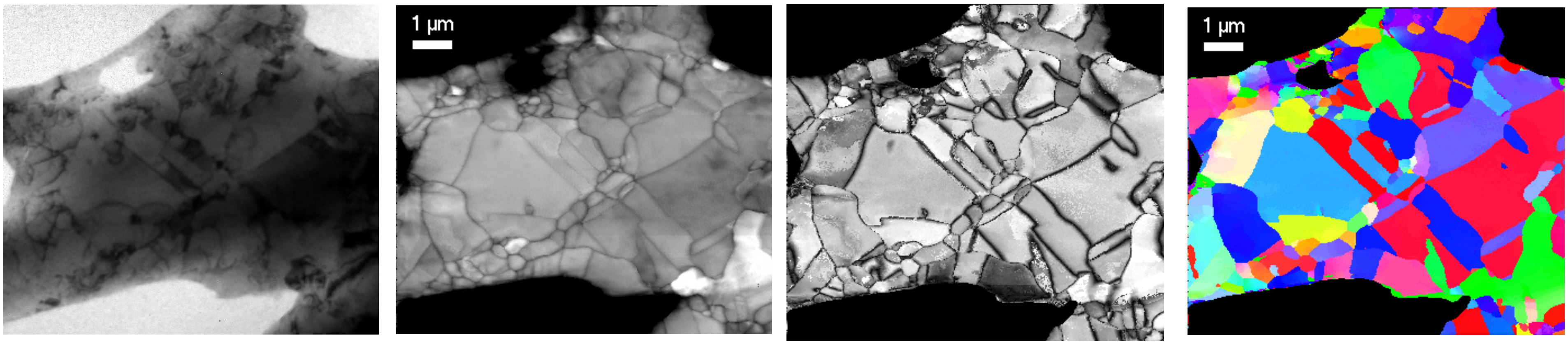
Grenoble INP

cnrs

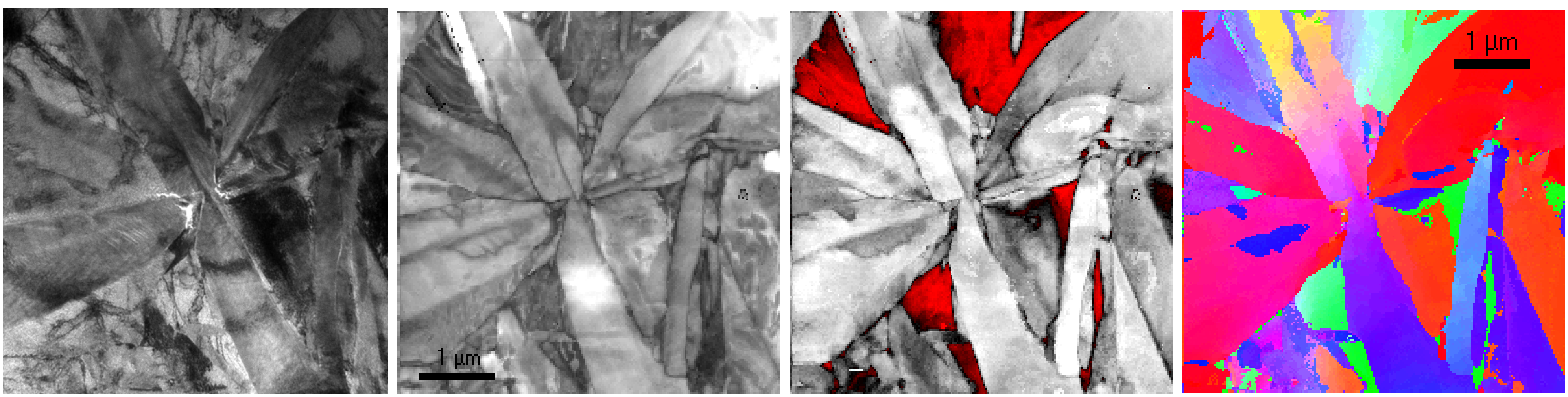
ACOM : some examples of orientation maps

Deformed Cu (coll. N. Llorca – Univ. Barcelona, Spain)

Orientation reliability



Correlation Index | Phase Reliability | Orientations



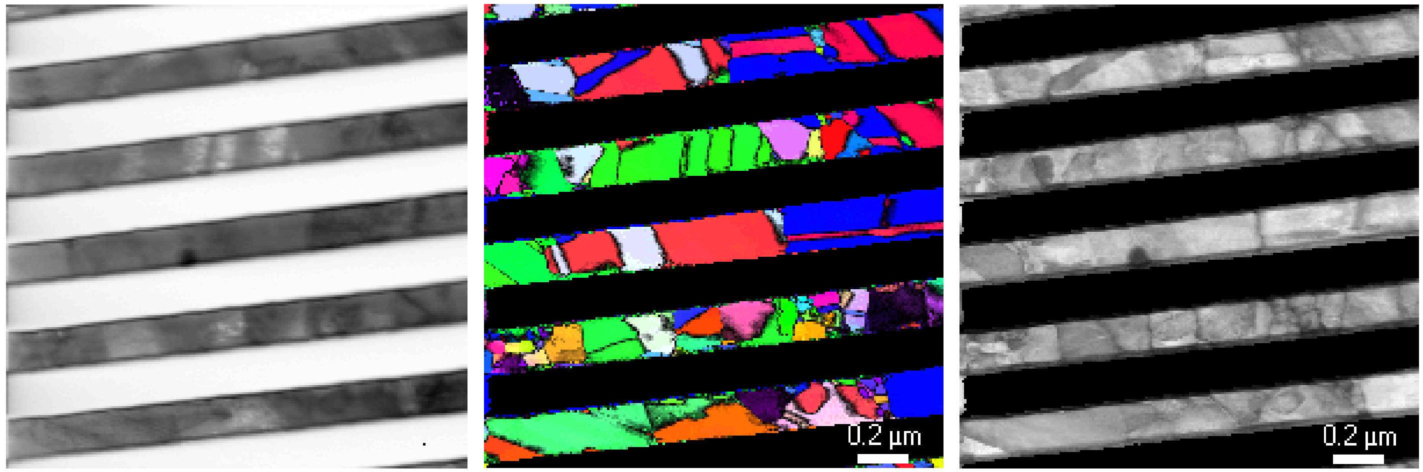
Fe 1.67% C (HT 10 min @ 1100°C, A. Stormvinter - KTH)

Austenite **Martensite**

ACOM/TEM combined to a FEG-TEM

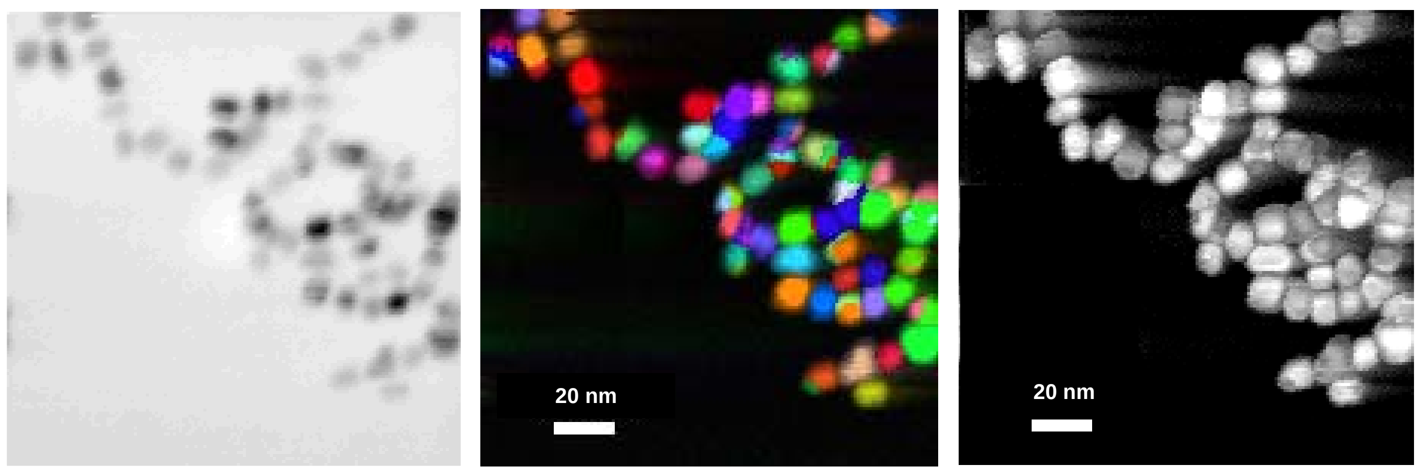
JEOL 2010F
Texas Material Institute
UT, Austin

180 nm Cu lines



Virtual bright field | Orientation map | Correlation Index map

Pt nanocrystals



1) ACOM/TEM :

Template matching

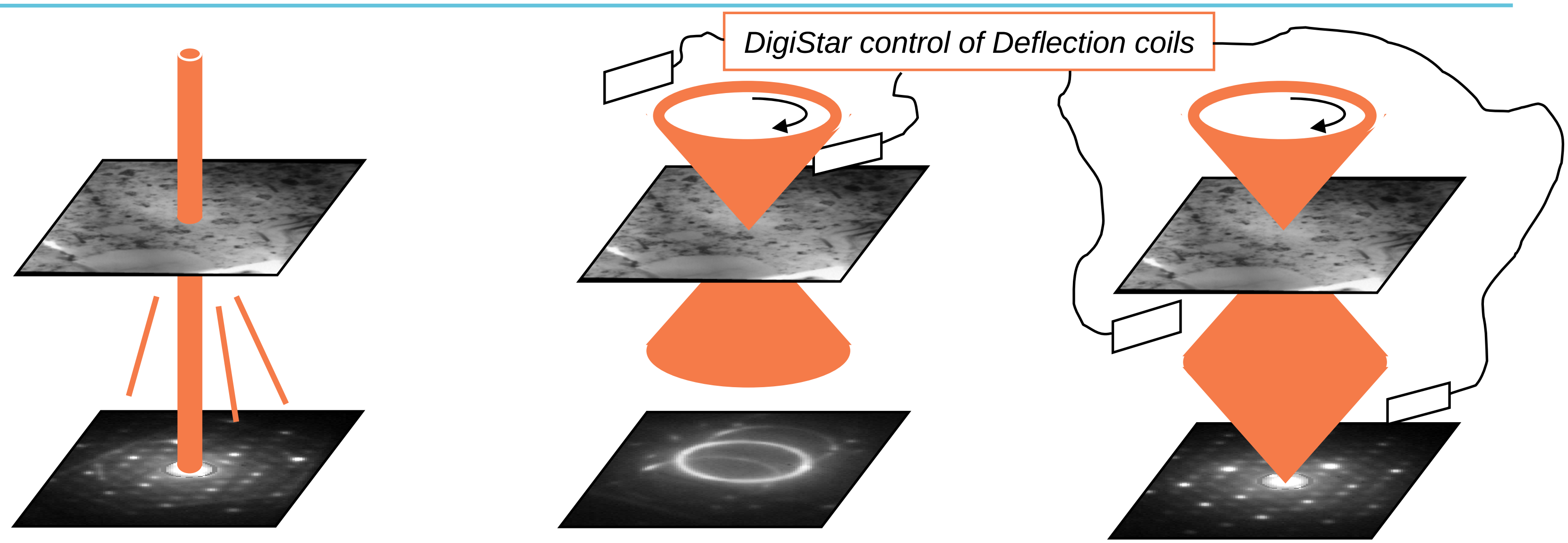
Pattern acquisition and template generation

2) ASTAR :

Combining orientation/phase identification with Electron Precession

Precession Electron Diffraction patterns

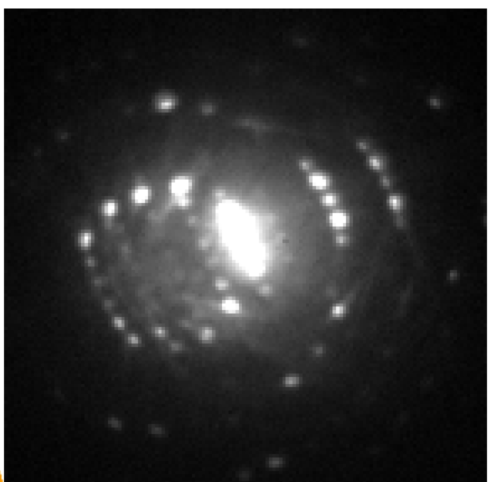
(R. Vincent, P.A. Midgley, Ultramicroscopy 53 (1994) 271.)

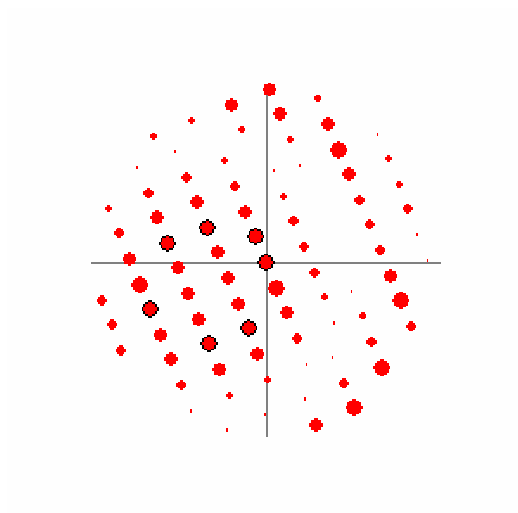


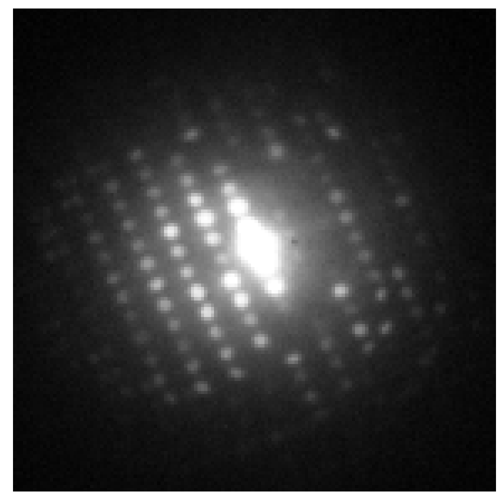
I: Standard setting

X: ½ Precession

Y: Full precession



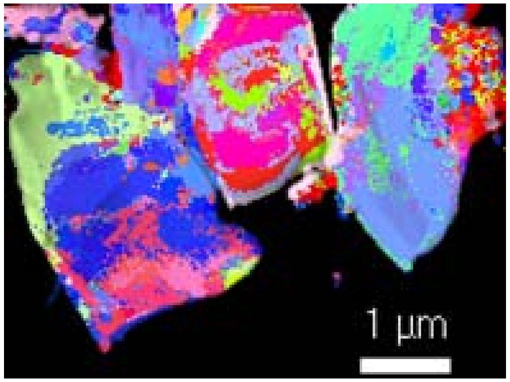


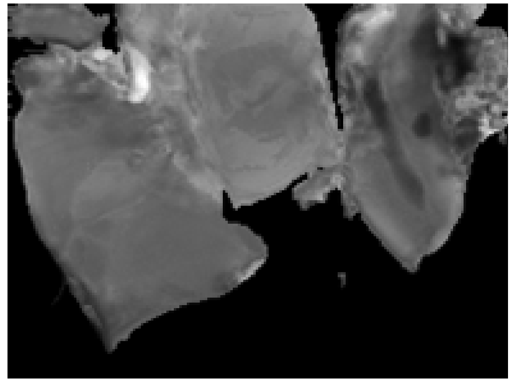
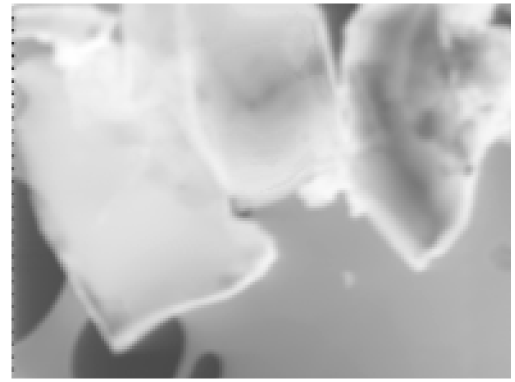


Precession Electron Diffraction patterns

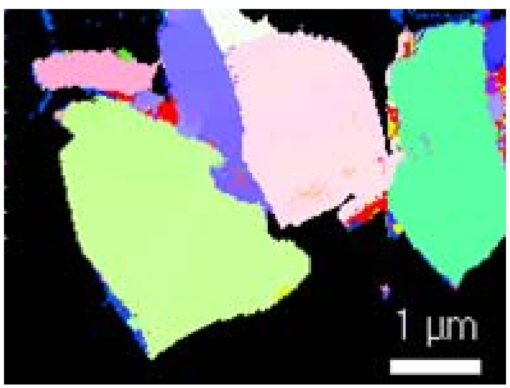
Mayenite crystal ($Ca_{12}Al_{14}O_{33}$) : space group I-43d

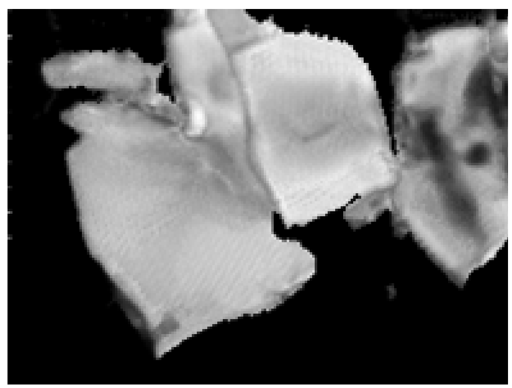
Orientation *Index* *Virtual Bright Field*



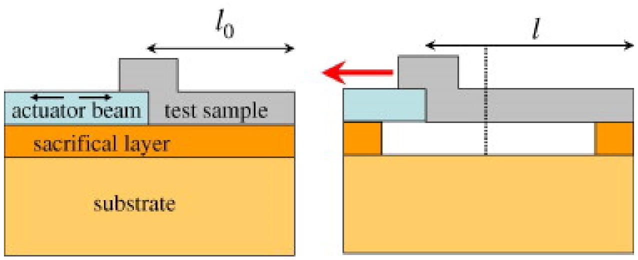
Without precession






With precession precession angle : 0.35º acquisition time 50 min (10 fps)

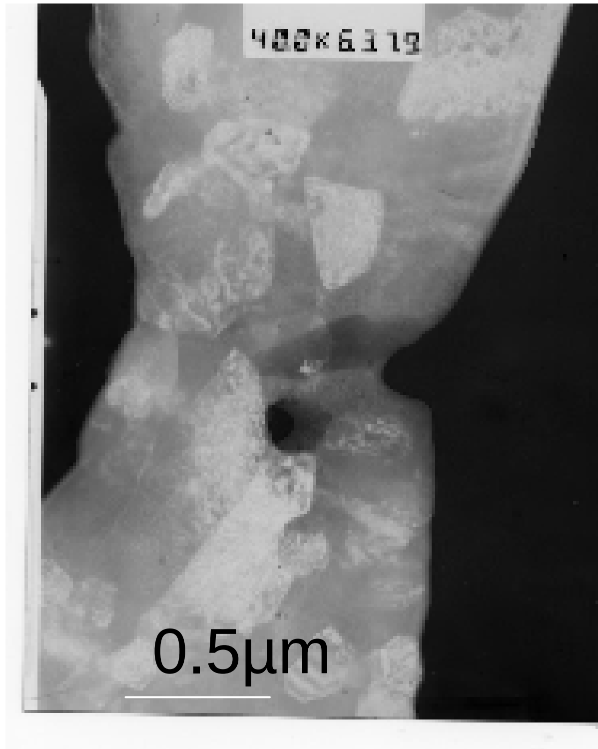
Deformation of Aluminum film

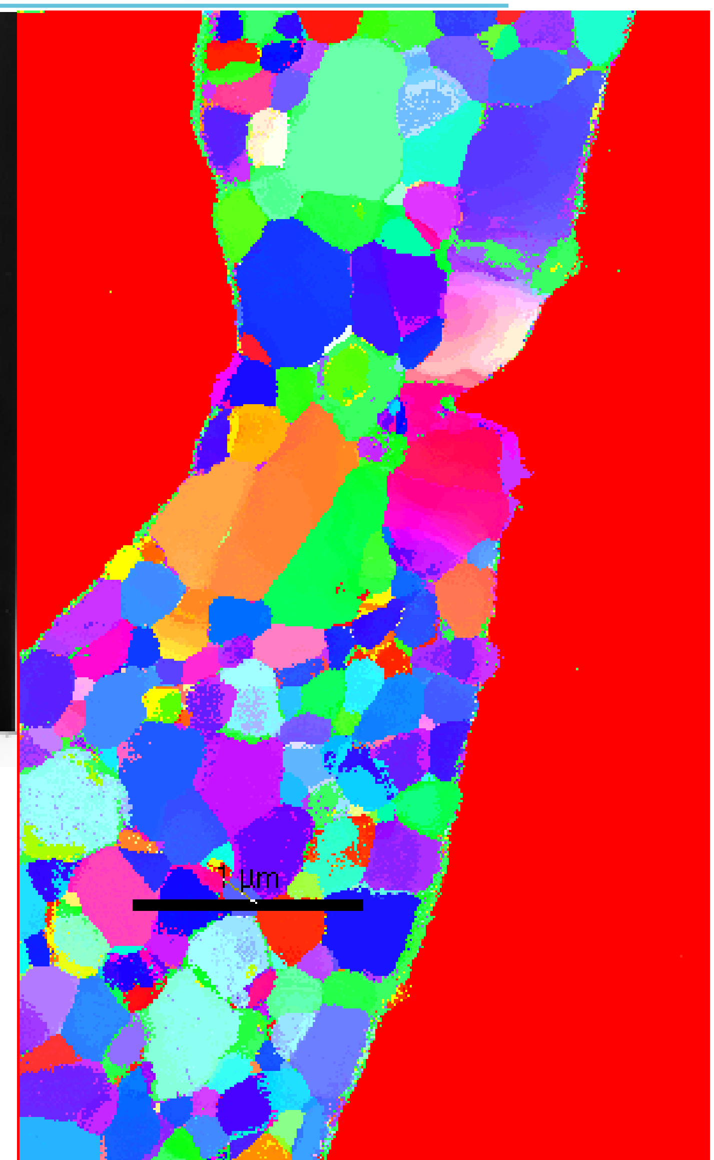


$$\varepsilon^{mech} = \ln\left(1+\frac{\Delta l}{l_0}\right) + (\alpha_{sub} - \alpha)\Delta T$$

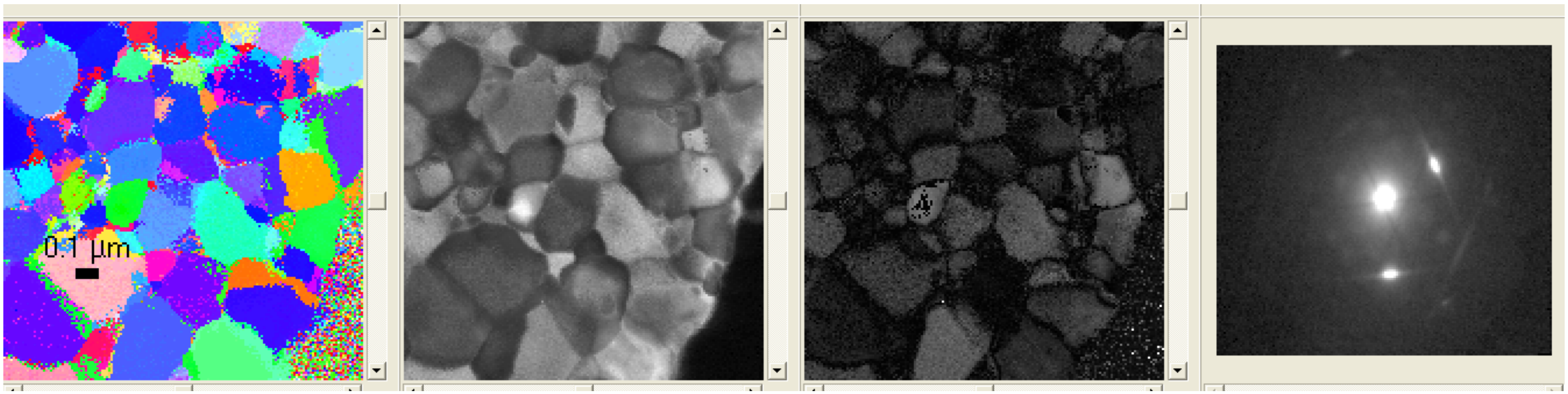
$$\varepsilon_{act}^{mech} = \ln\left(\frac{l_{0act} - \Delta l}{l_{0act} - \Delta l_{free}}\right)$$

$$\sigma_{act} = E_{act}\varepsilon_{act}^{mech} \qquad \sigma = \sigma_{act}\frac{S_{act}}{S}$$

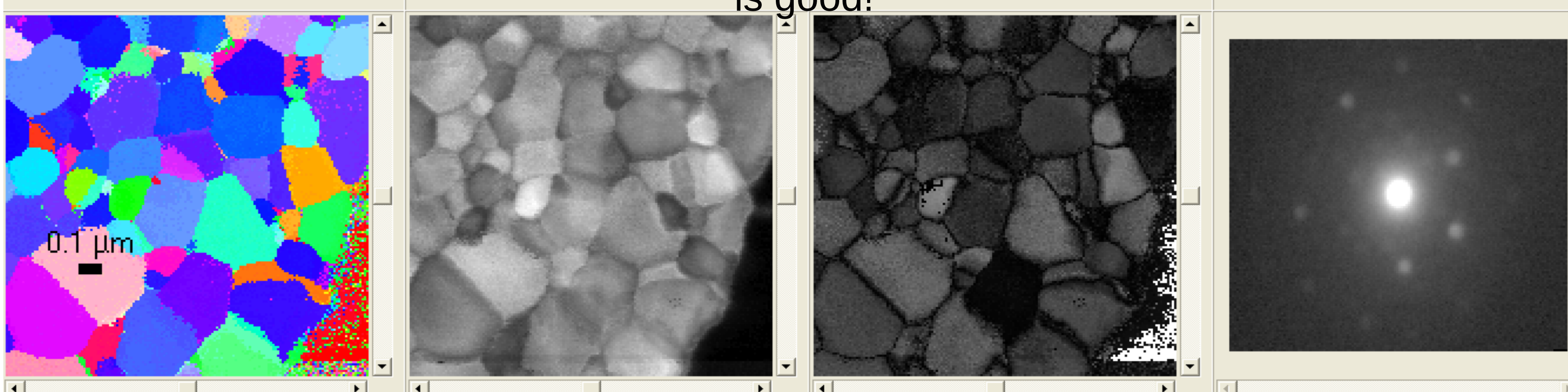




Good results were obtained with a precession angle α= 0.9°



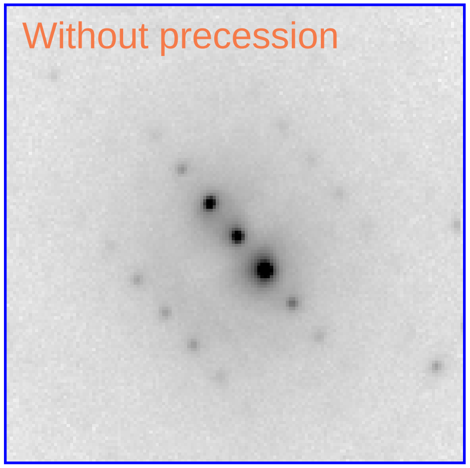
Thick sample, same area, diffraction patterns with kikuchi lines. Without precession, quality is poor, with precession α= 0.9°, diffraction patterns are « cleaned », and indexing is good!

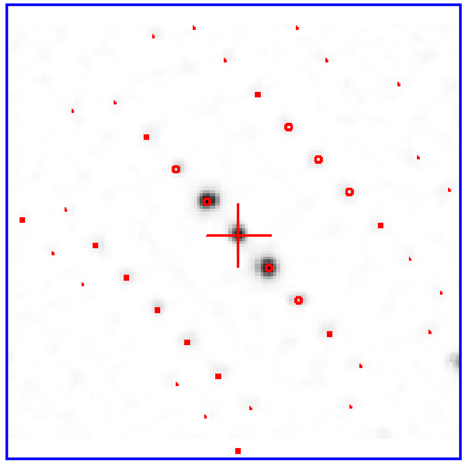


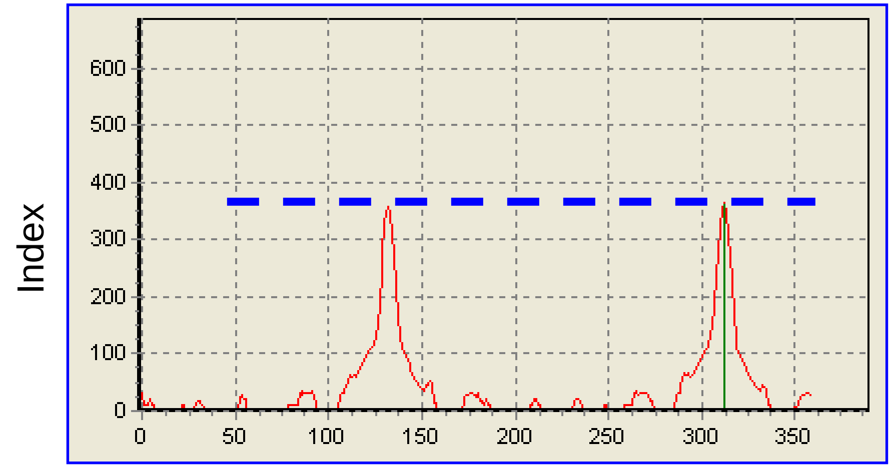
ACOM + Precession: Solving 180° orientation ambiguities

B.c.c. Iron (ferrite), [112]

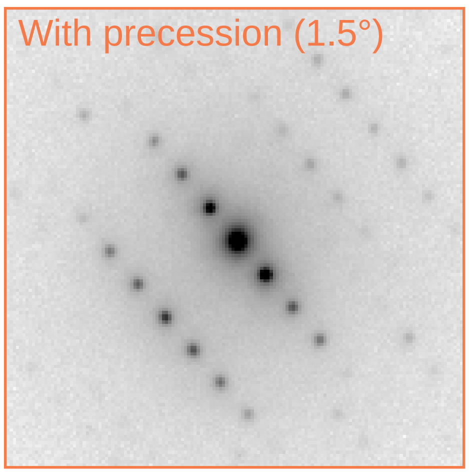
I : Index=317, Reliability=0

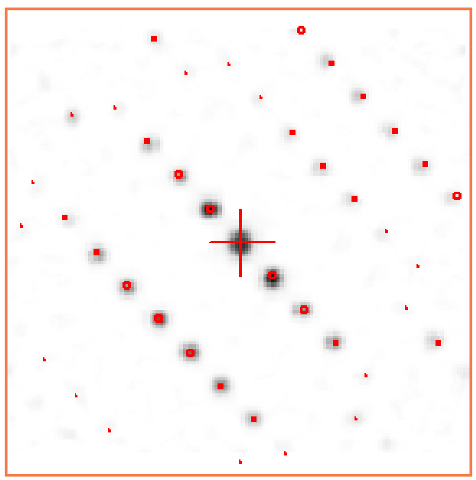


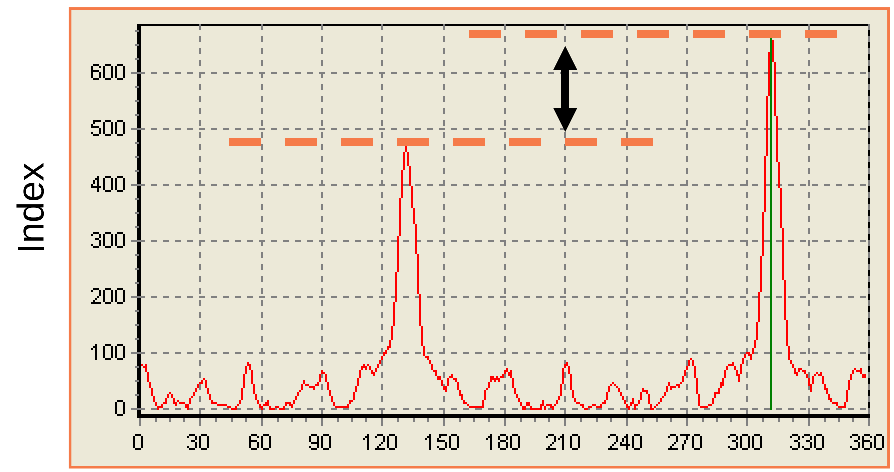




Rotation angle (around the normal axis)

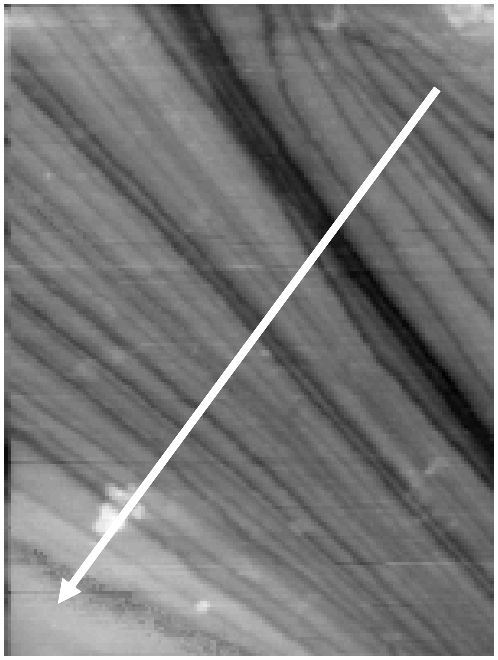


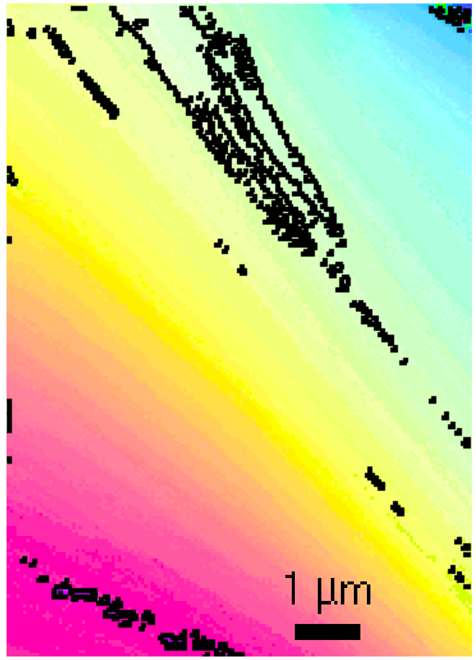


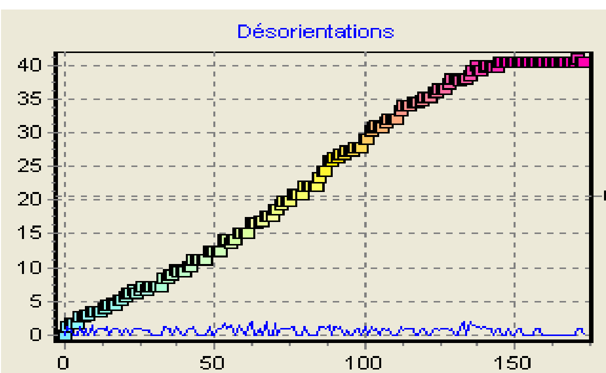


Y : Index=670, Reliability=28

ACOM + Precession: Solving 180° orientation ambiguities



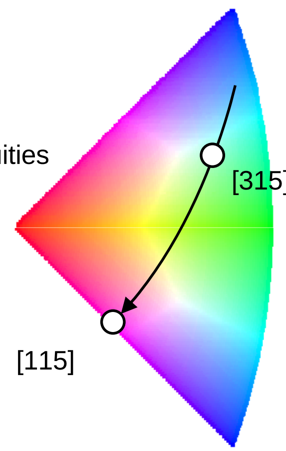




Fe-1.5%Cu (annealed)

Precession angle 0.5°

Orientation ambiguities

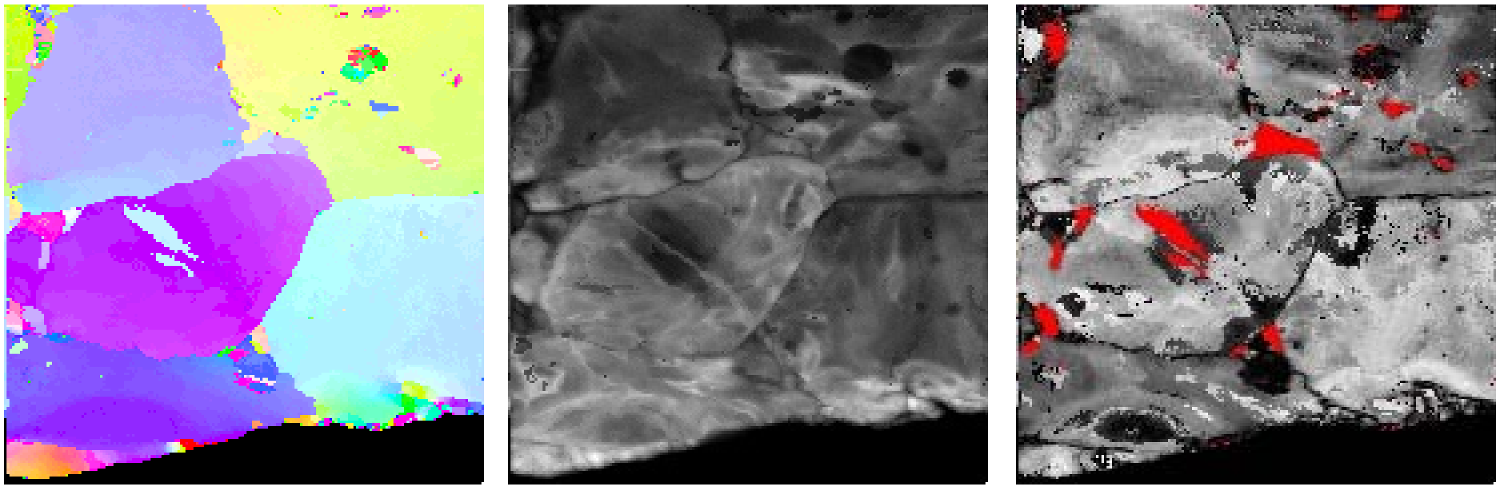


DiffGen : Template generator

Same area with (Y) and without (I) precession

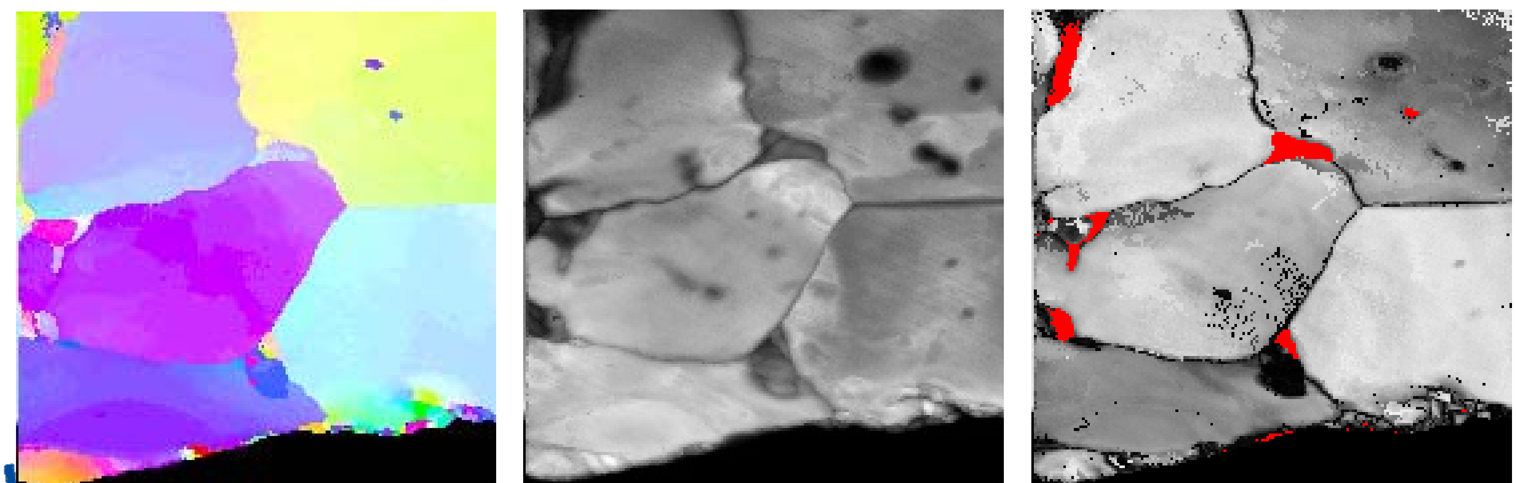
TRIP steel (ferrite + austenite) ; Philips CM120 @ 100hz (6 min),

I



Orientation Index reliability +phase

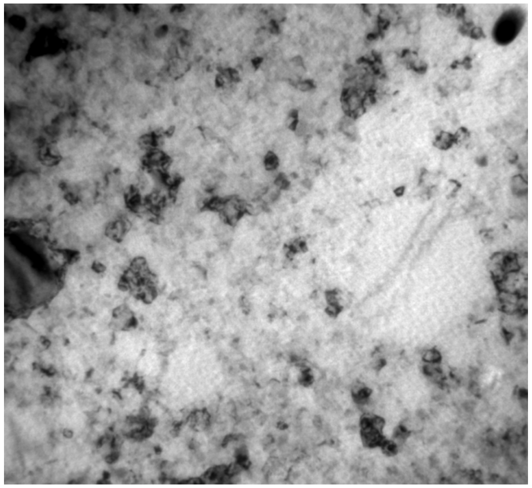
Y



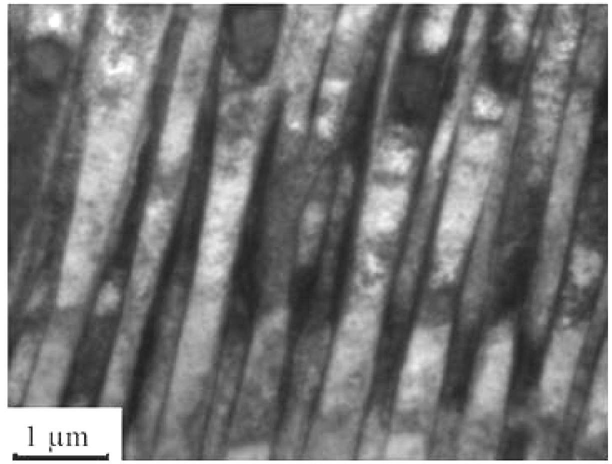
INDEX : pattern identification software

ACOM : some examples of orientation maps

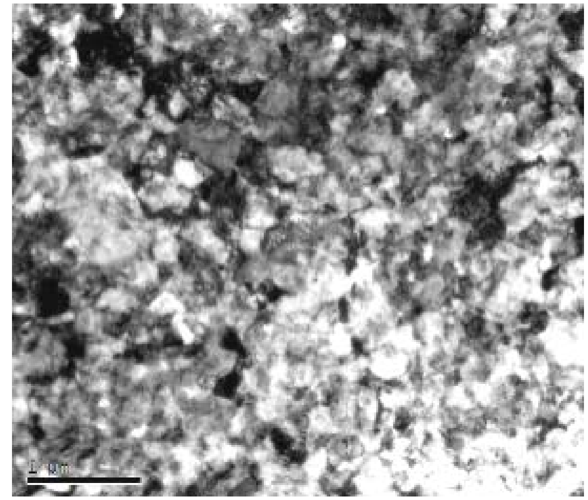
Bright field images



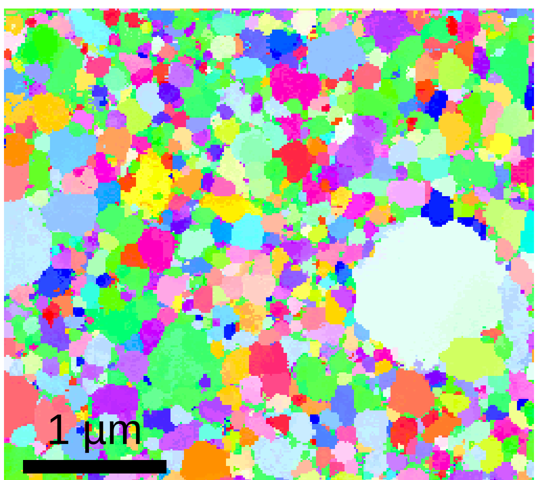
Nanocrystalline Al

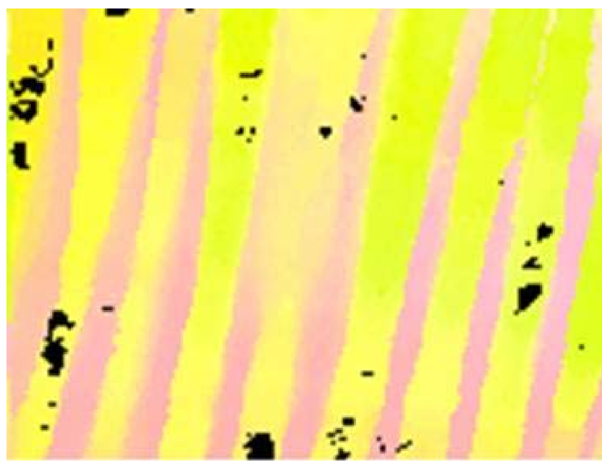


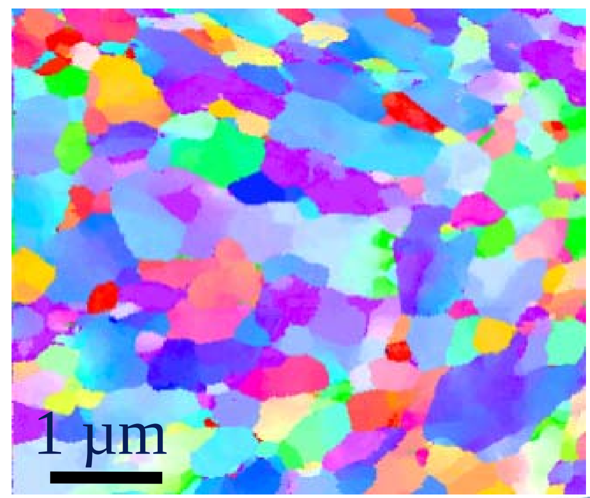
Deformed steel



Severely deformed Cu

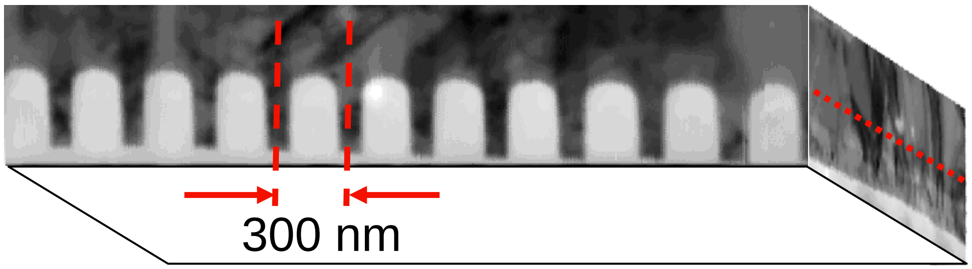






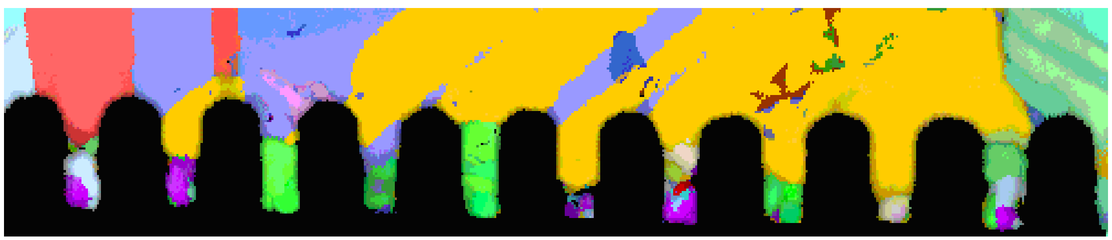
Orientation maps

Grain size and Texture in 80 nm copper lines

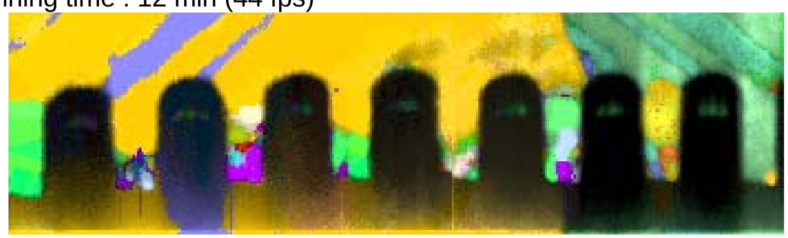


Side view

500x100 steps (6.5 nm each) , Spot size 25 nm
Scanning time : 19 min (44 fps)



300x100 steps (6.5 nm each), spot size 15 nm
Scanning time : 12 min (44 fps)

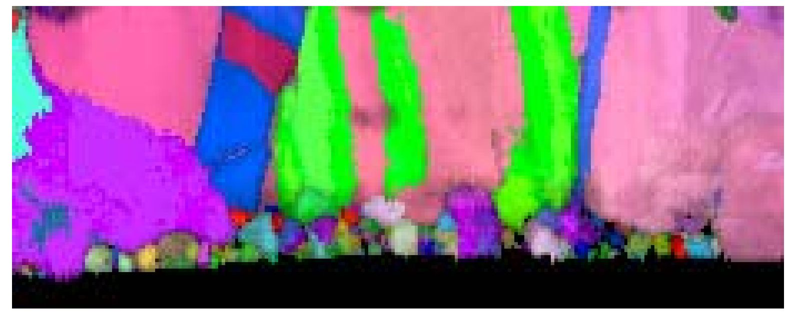


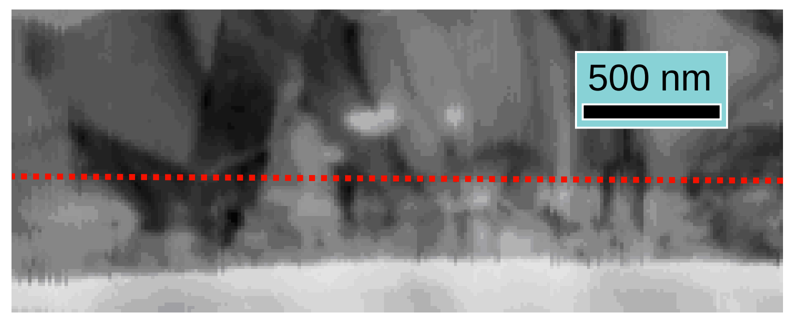
SIDE VIEW (orientation and index superimposed map)
The two scans were performed with different settings
They demonstrated the reproducibility of the identification

CROSS VIEW (orientation map)

Grain size of the order of 30 nm may be identify despite the use of a conventional LaB_6 equipped Jeol 3010 TEM (spot size 25 nm).
A fiber texture was detected within the channel.

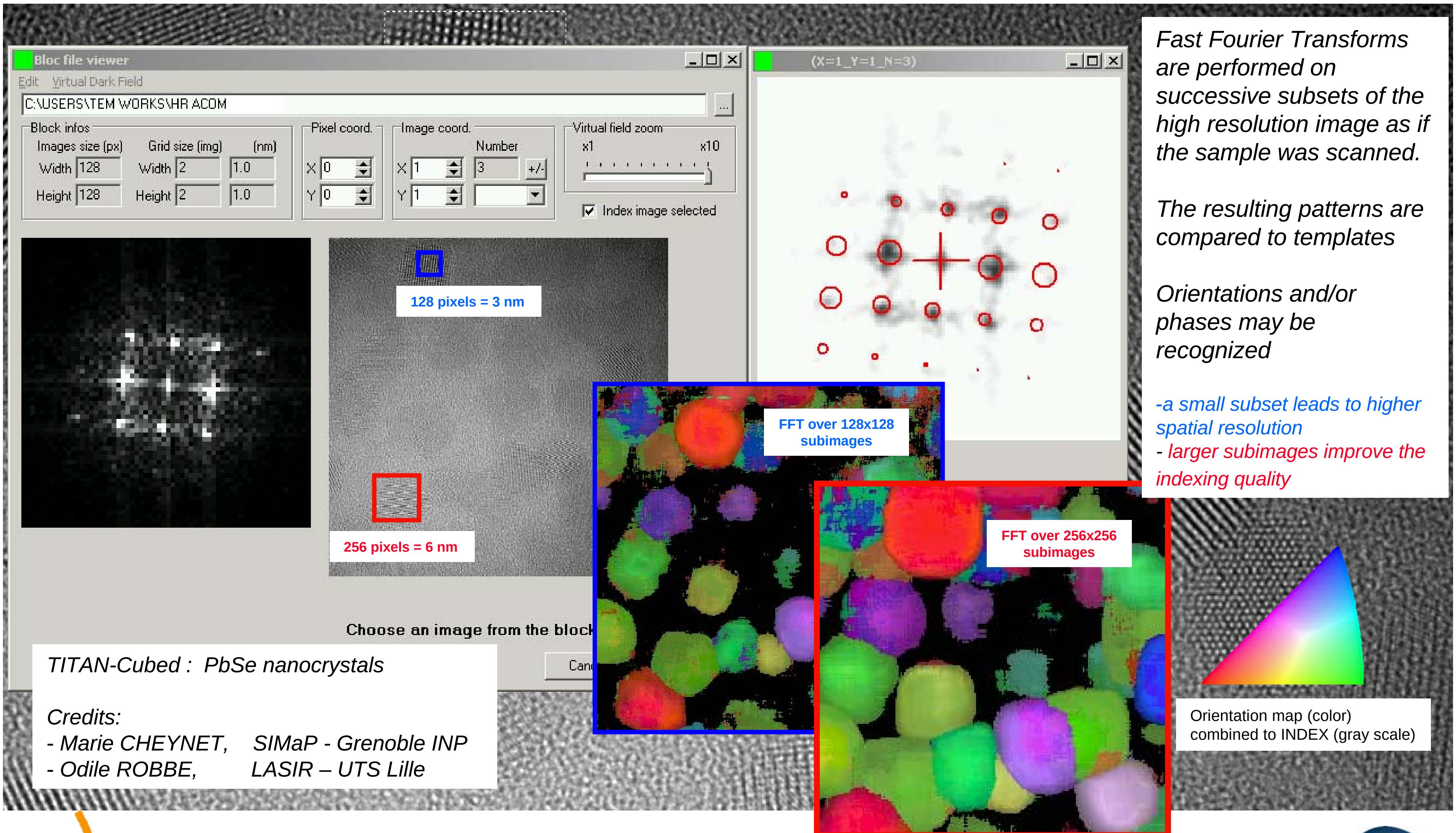
250x100 steps (13 nm each)
LaB_6 equipped Jeol 3010 TEM (spot size 25 nm)
Scanning time : 10 min (44 fps)





Credits:
- Stefan BRANDSTETTER, SIMaP - Grenoble INP

Indexing Fourier transform of High resolution TEM images



Fast Fourier Transforms are performed on successive subsets of the high resolution image as if the sample was scanned.

The resulting patterns are compared to templates

Orientations and/or phases may be recognized

-a small subset leads to higher spatial resolution

- larger subimages improve the indexing quality

TITAN-Cubed : PbSe nanocrystals

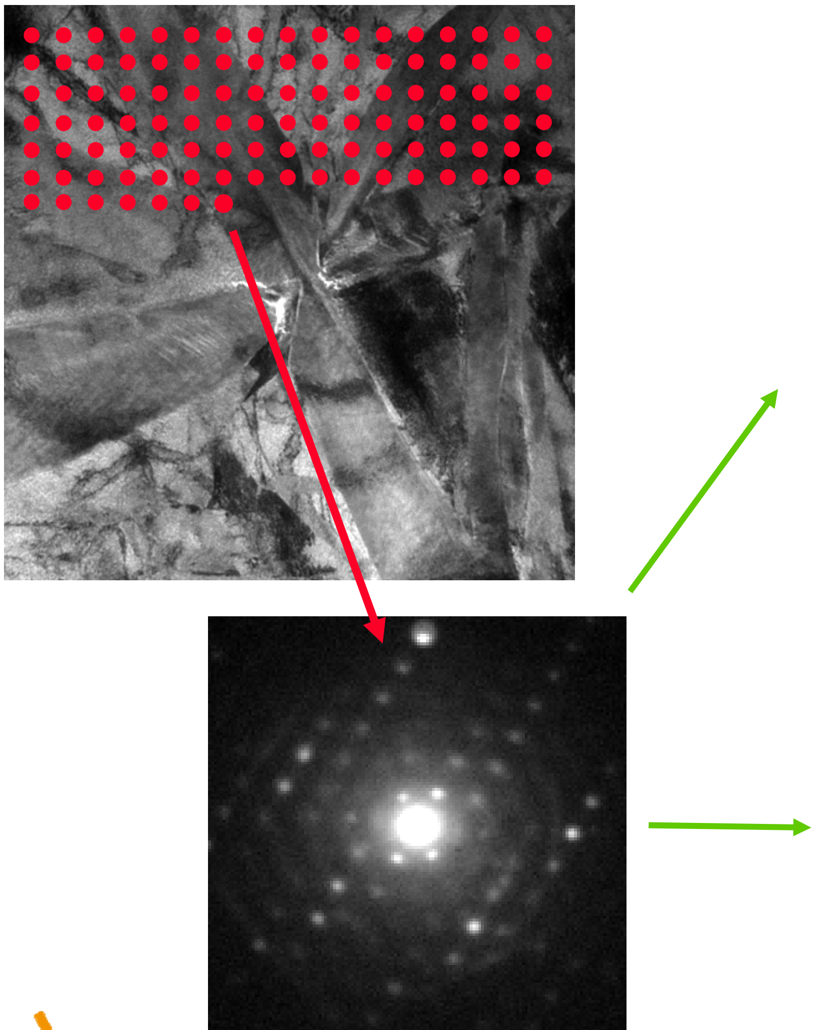
Credits:

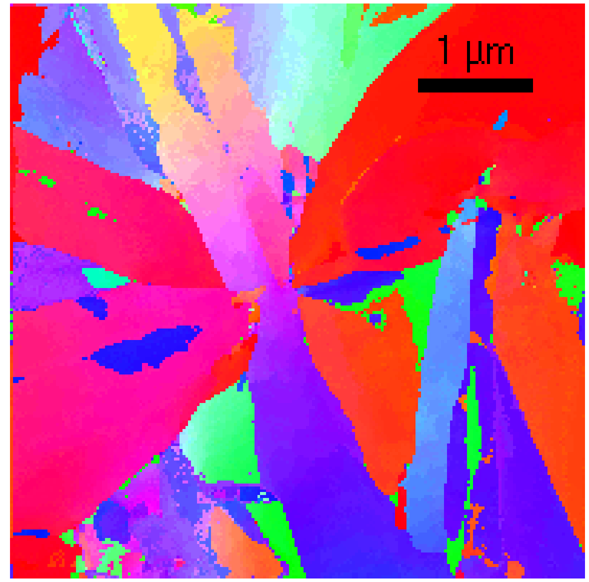
- Marie CHEYNET, SIMaP - Grenoble INP

- Odile ROBBE, LASIR – UTS Lille

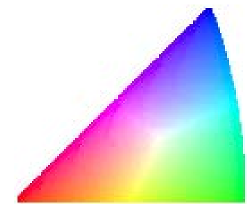
ACOM/TEM : Automated Crystal Orientation Mapping

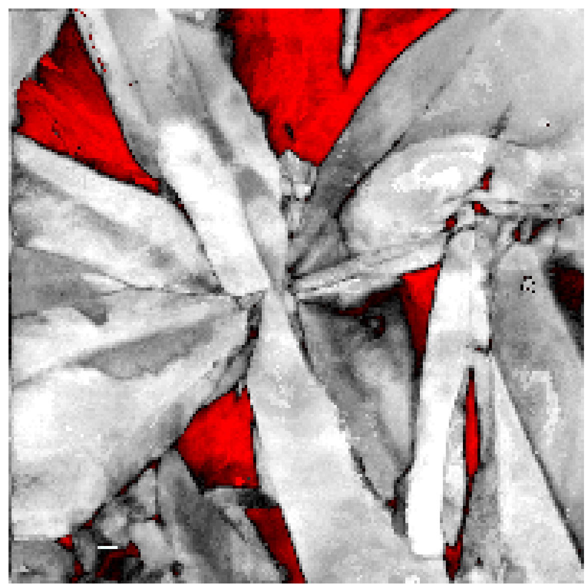
Fe 1.67% C (HT 10 min @ 1100°C, A. Stormvinter - KTH)





Orientation map





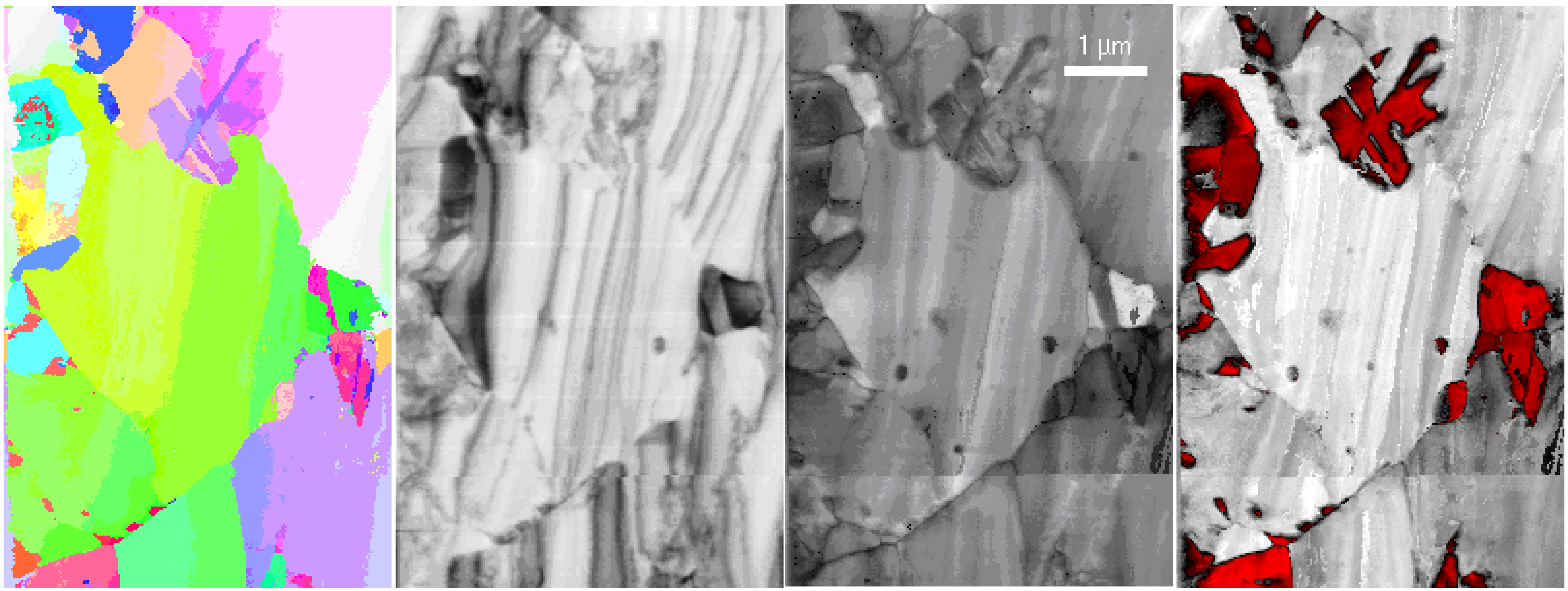
Phase map

Austenite

Martensite

ACOM : orientation and phase maps

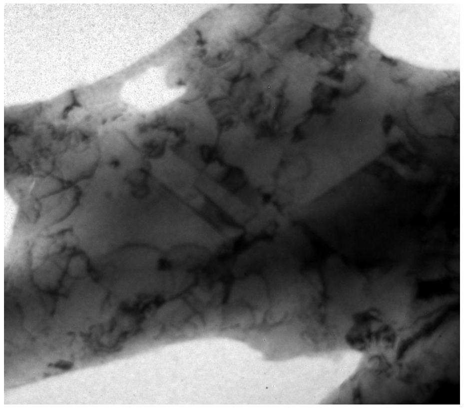
TRIP steel with retained austenite

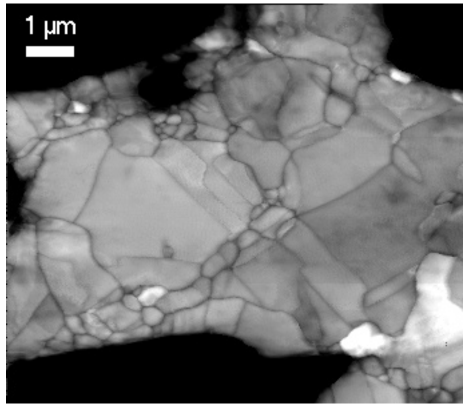


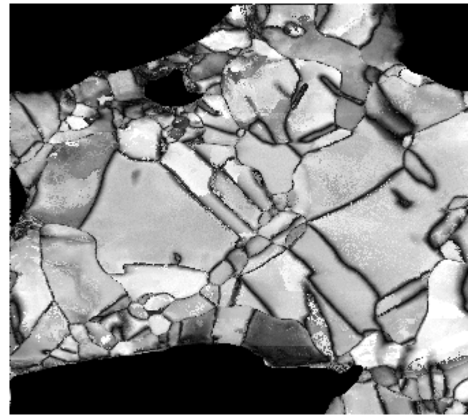
Orientation maps | Virtual bright field | Correlation Index | Phase + reliability

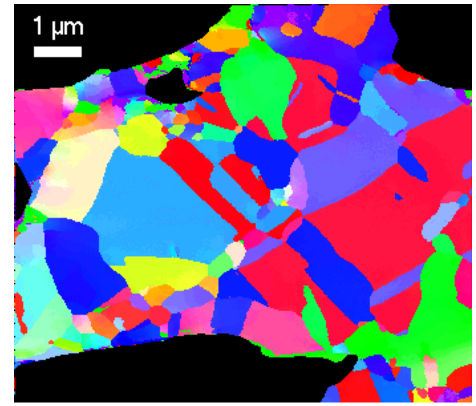
ACOM : some examples of orientation maps

Deformed Cu (coll. N. Llorca – Univ. Barcelona, Spain)

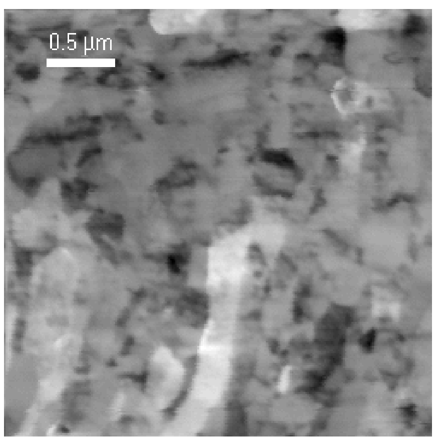


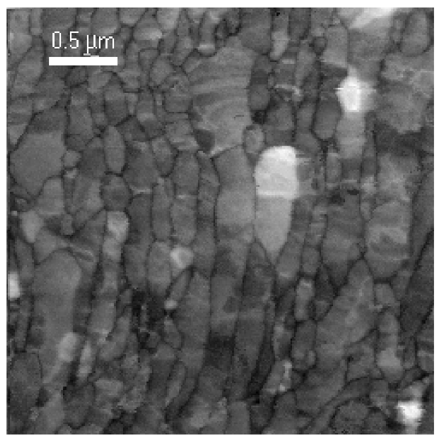


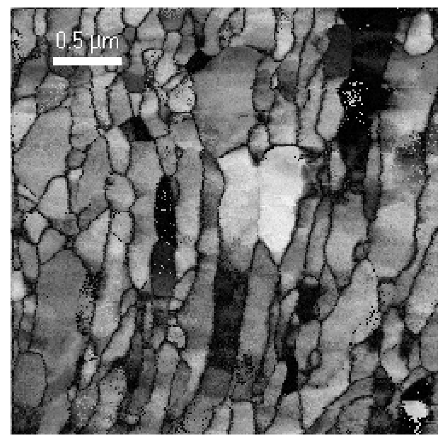


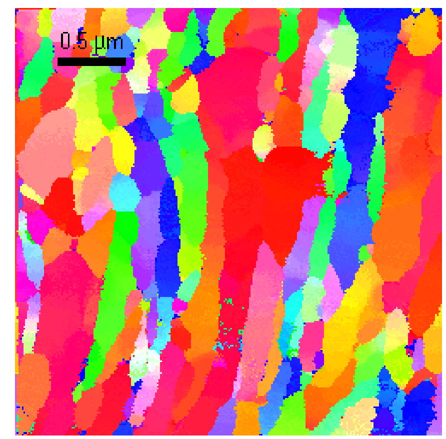


Bright field | Correlation Index | Reliability | Orientations







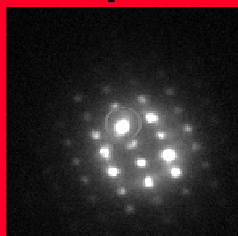


Severely Deformed Fe (coll. S. Descartes – LaMCoS, Lyon - France)

5				9	2	6		
	4	7						
			1				4	5
4			7	1		9		
8	3				4		1	
	8	9						
				6		2		
		2		8		7		

ACOM/TEM : four steps

TEMdpa.exe



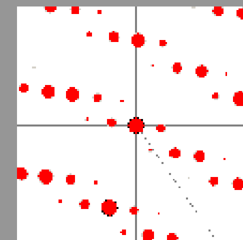
On line

(hardware)

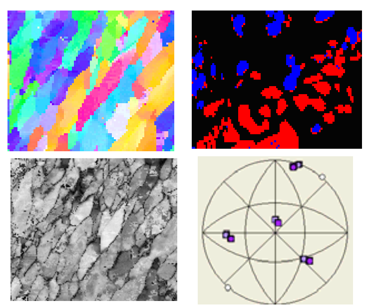
INDEX.exe

$$Q(i) = \frac{\sum_{j=1}^{m} P(x_j, y_j)\, T_i(x_j, y_j)}{\sqrt{\sum_{j=1}^{m} P^2(x_j, y_j)}\,\sqrt{\sum_{j=1}^{m} T_i^2(x_j, y_j)}}$$

DiffGen.exe

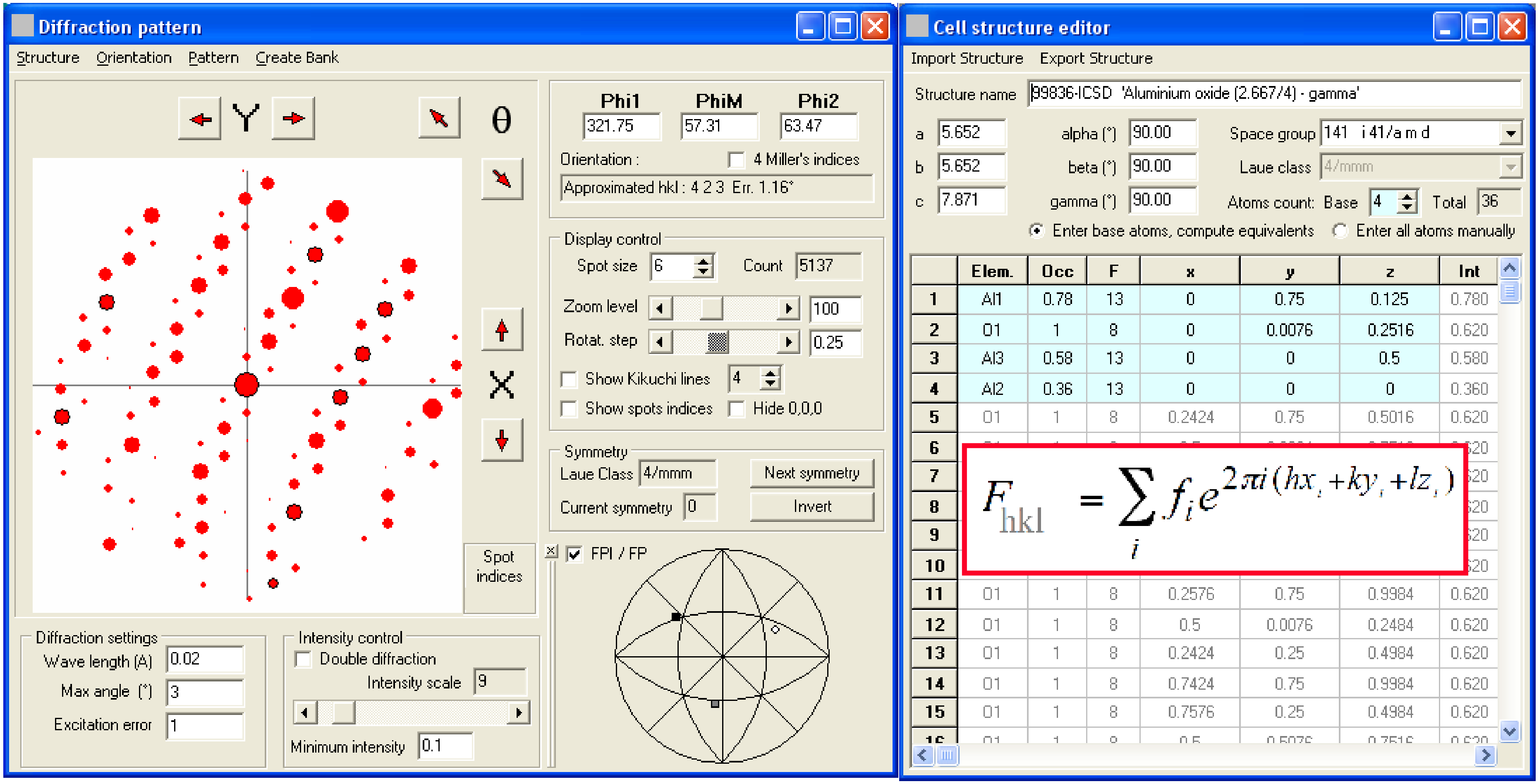


MapViewer.exe



Off line

DiffGen : Template generator

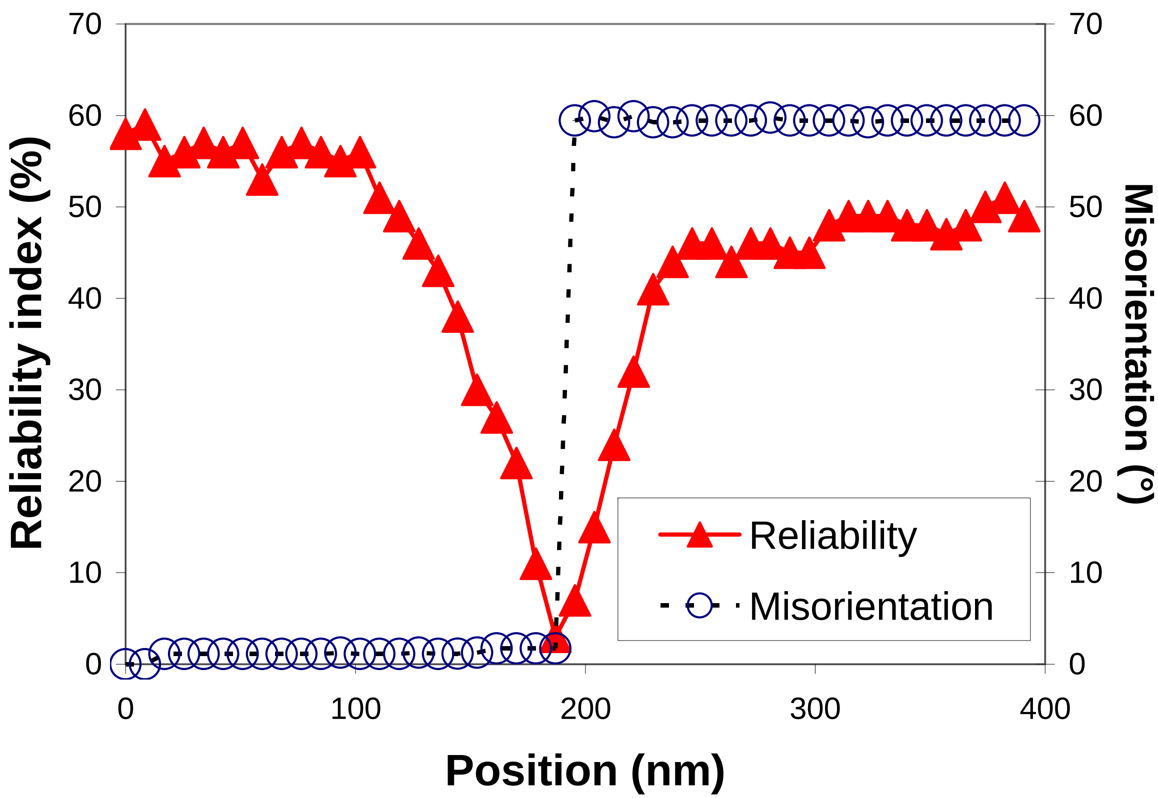


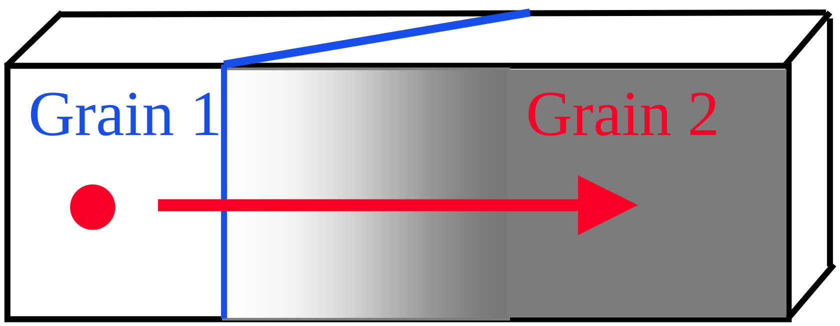
Features: Any crystallographic structure, any orientation

Laue class related to the space group

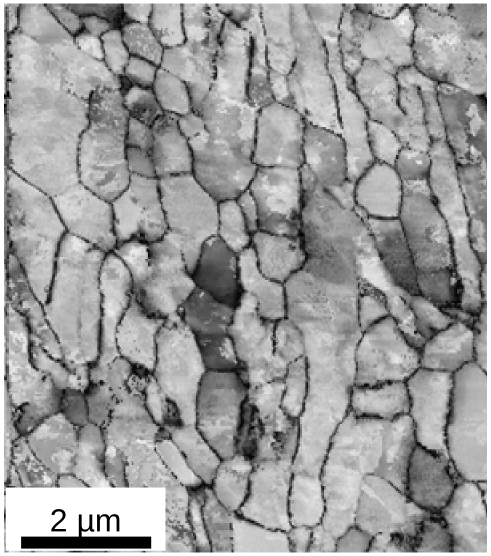
Structure generator (lattice, cell, structure factor)

Deconvolution of superimposed Diffraction patterns





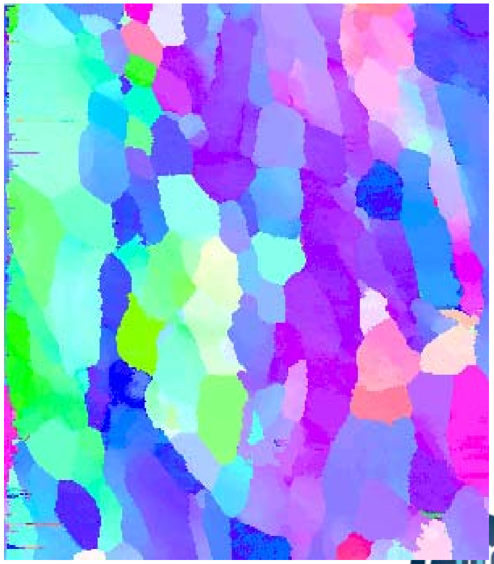
reliability



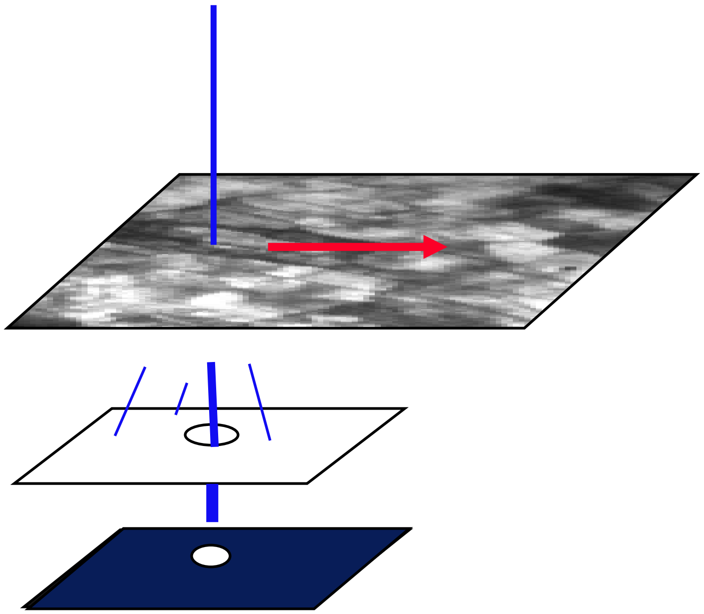
Fe - 60 ppmC severely deformed

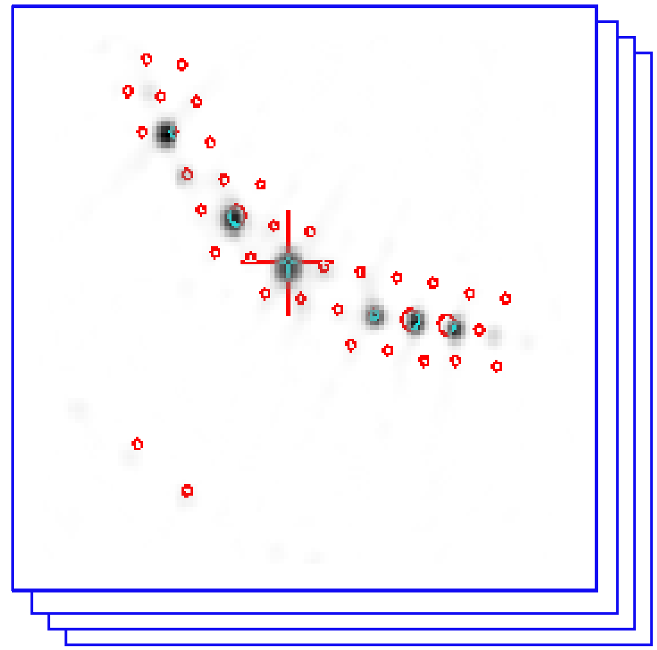
Bright field

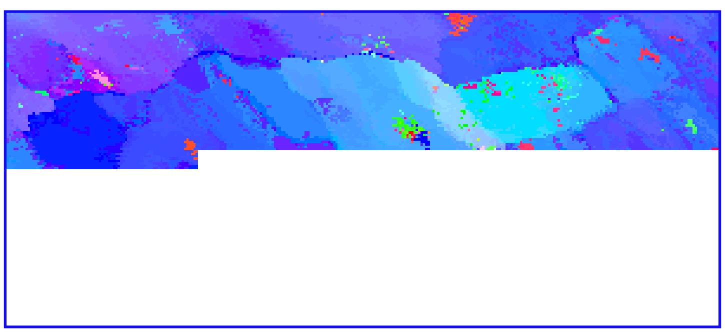
Orientation map



TEMdpa : Virtual Bright Field on-line construction



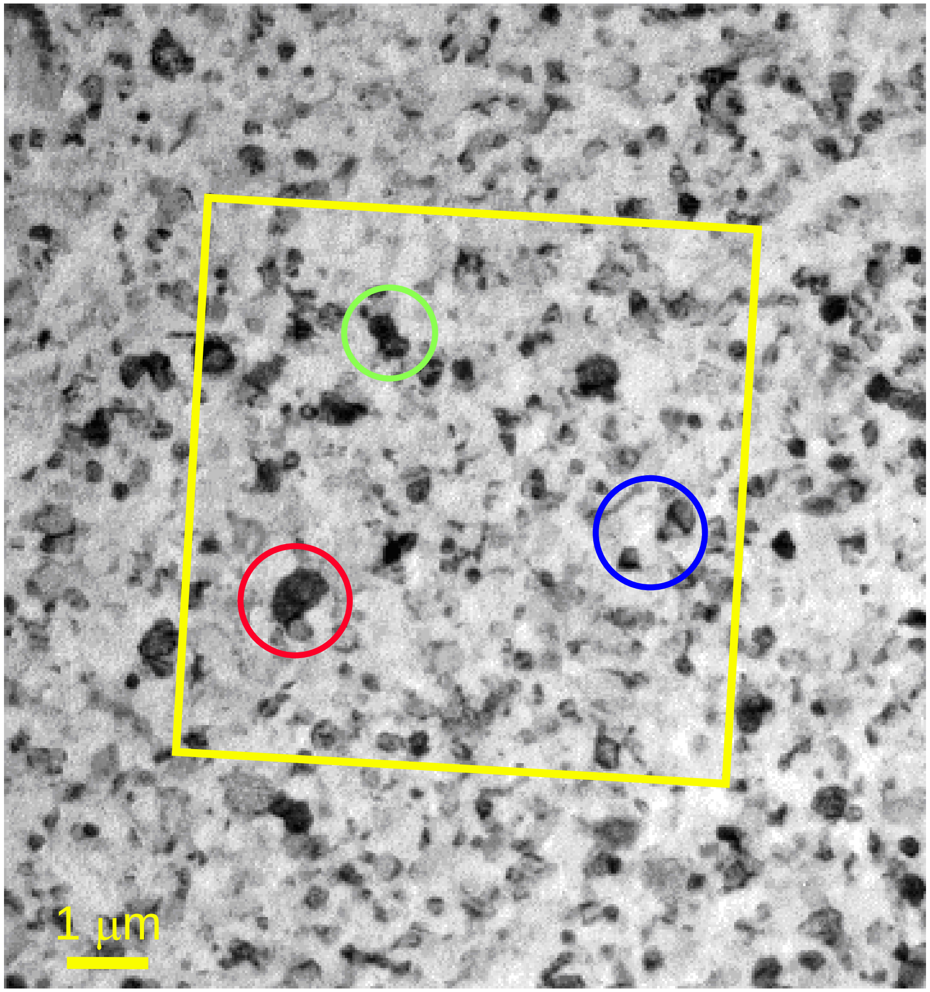




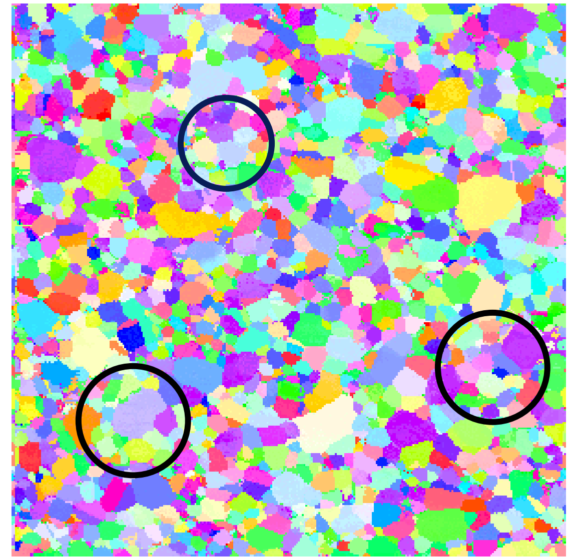
Orientation map

‘ Bright field ‘

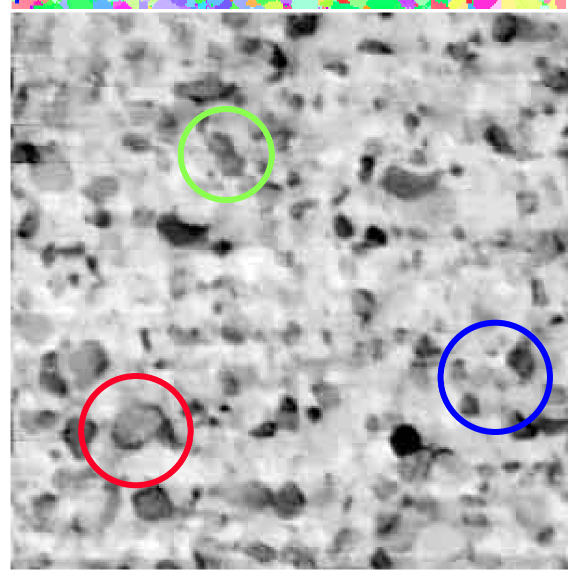
TEMdpa : Virtual Bright Field on-line construction



Bright field image



Orientation map



Virtual bright field image

Aluminium (mean grain size 200nm)