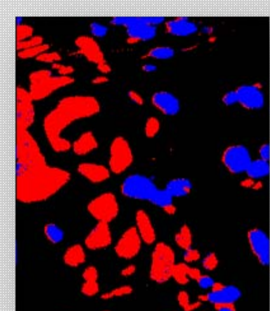
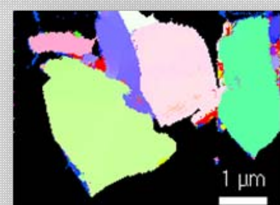
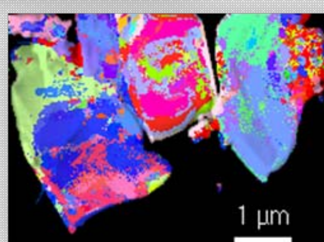
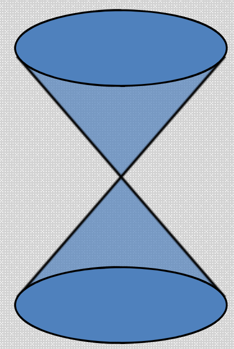
ASTAR (EBSD-TEM like)

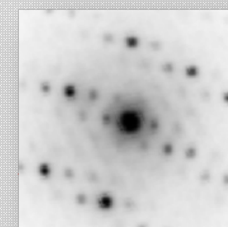
Automatic Crystal Orientation/Phase mapping for TEM

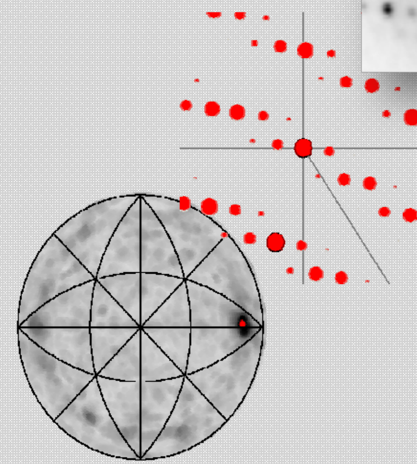








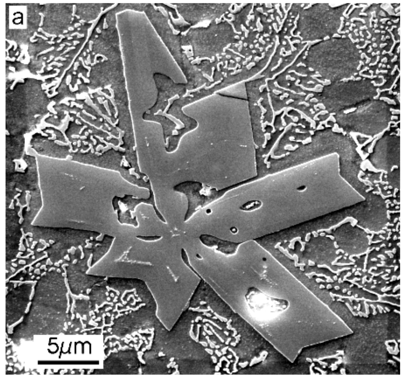


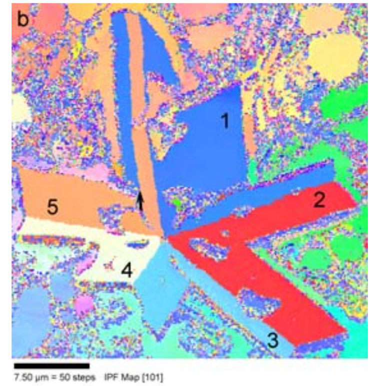


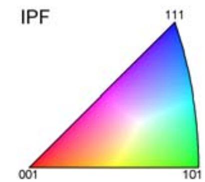
Grenoble INP

NanoMEGAS
Advanced Tools for electron diffraction

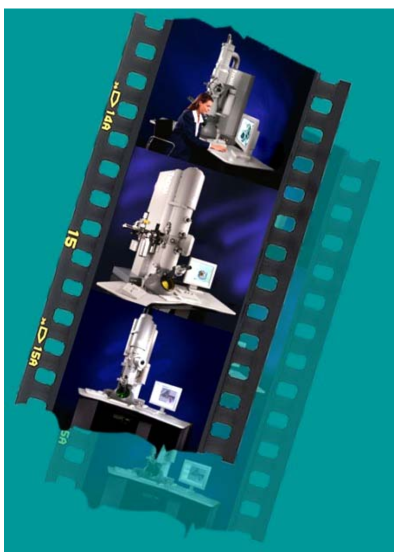
www.nanomegas.com

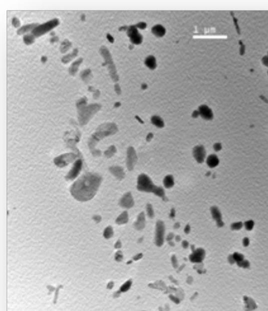


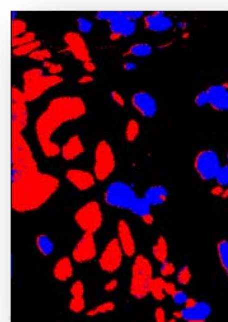


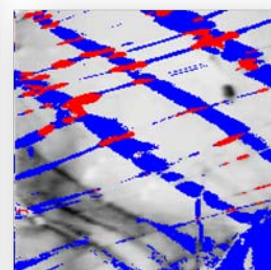


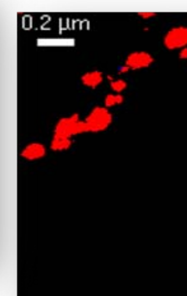
NEW precession application “EBSD” – TEM









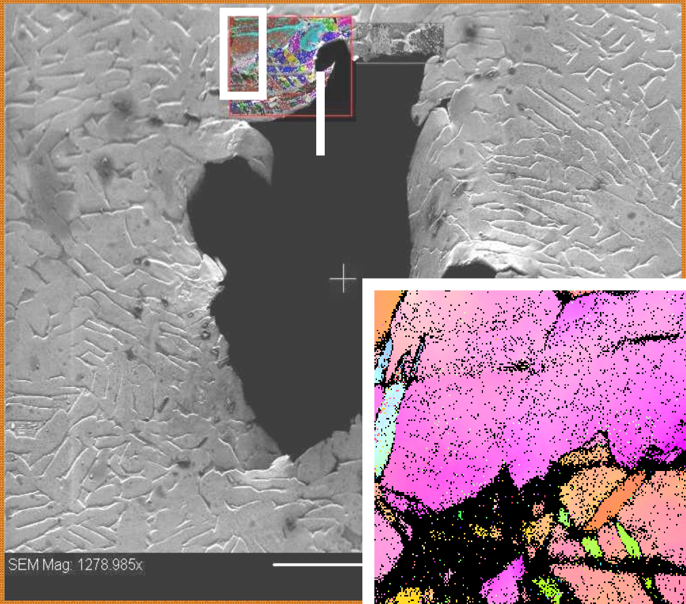


NanoMEGAS
Advanced Tools for electron diffraction

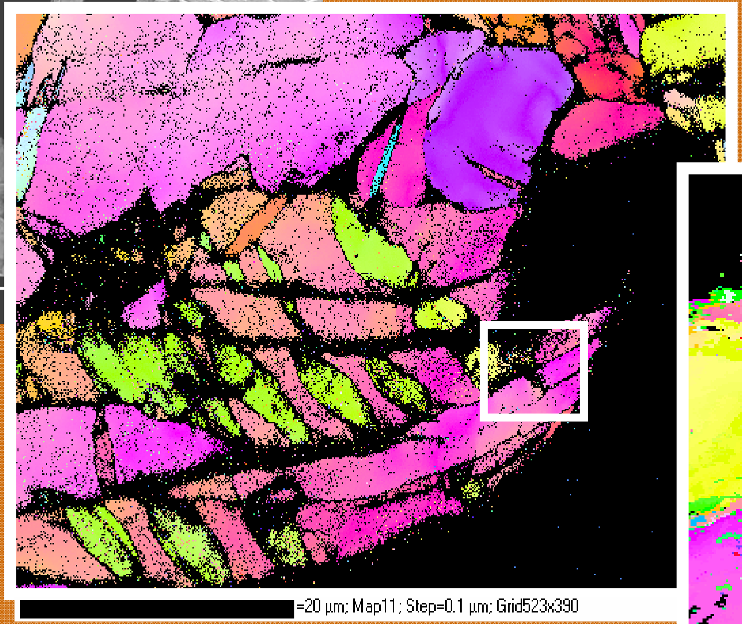
EBSD-TEM : beam is scanned over the sample (eg. 10 μ x 10 μ)

- **spot electron diffraction patterns are collected (<u>NOT</u> sensitive to stress/ strain or surface sample preparation like in EBSD-SEM)**
- **Beam scanning performed by "spinning star" unit / no STEM need**
- **Thousands of experimental spot ED patterns are acquired by a very fast optical CCD camera attached to TEM screen (180 patterns/sec)**
- **Slow scan CCD can also be used (but slow : 20-30 patterns/sec)**
- **Thousands of theoretical ED patterns are generated (templates) from .cif files or commercial databases for all known phases in a sample**
- **Template matching is used (by cross-correlation of all experimental ED patterns with all templates) to generate most probable orientation of every scanned position in the sample.**

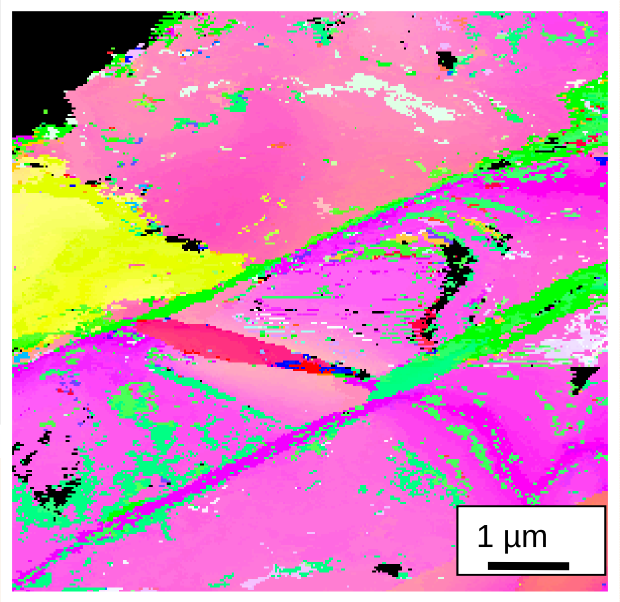
Comparison SEM-(EBSD) vs TEM spatial resolution



SEM orientation map
deformed Ta_6V alloy



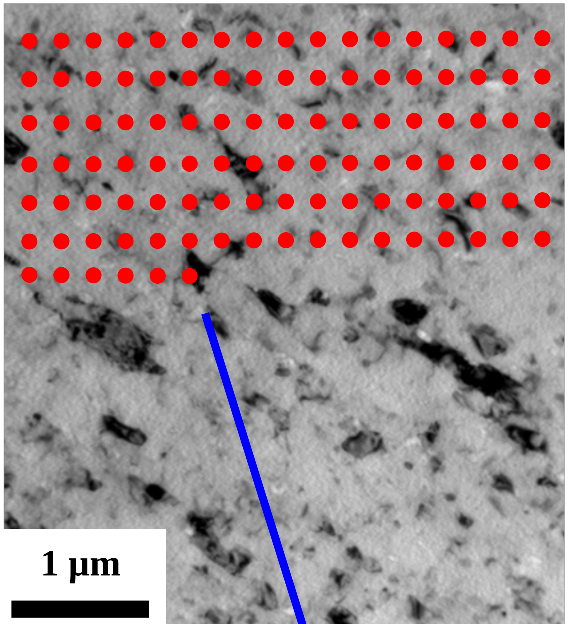
TEM orientation map
(25 nm stepsize)

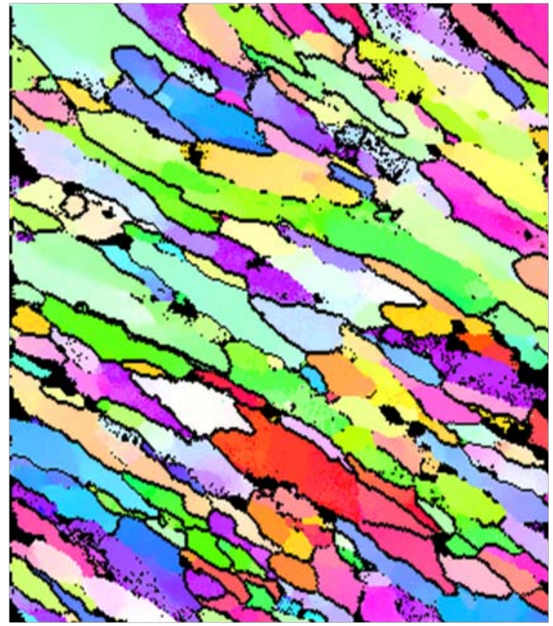


EBSD map (100 nm stepsize)

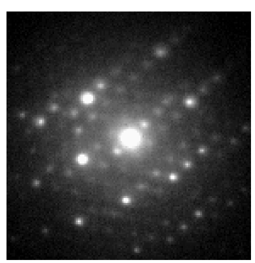
Electron Backscattering Diffraction (EBSD) orientation maps in SEM have usually poor resolution in comparison with TEM maps showing detailed nanostructure

ASTAR : diffraction pattern adquisition



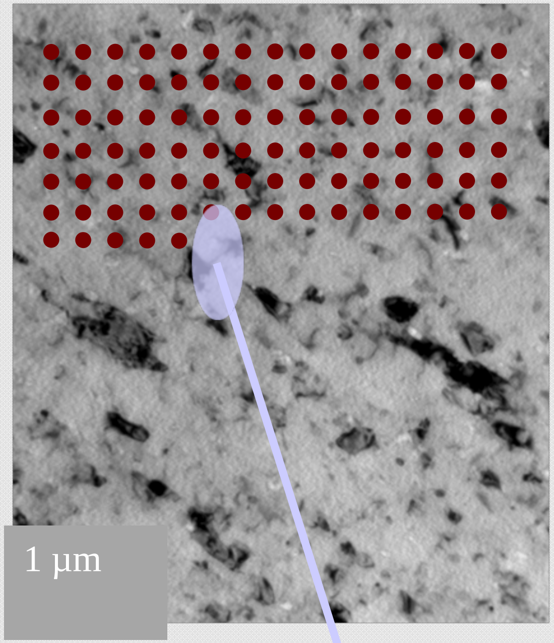


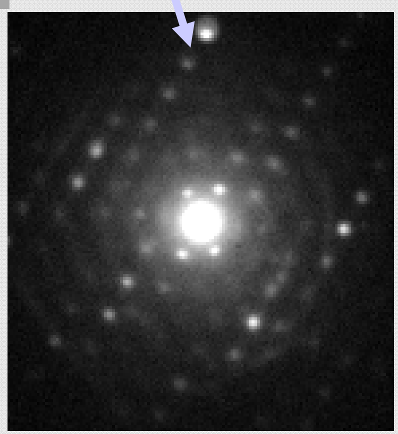
Example :Severely
deformed
7075 Aluminium Alloy



Any TEM –FEG/LaB6 may work with ASTAR

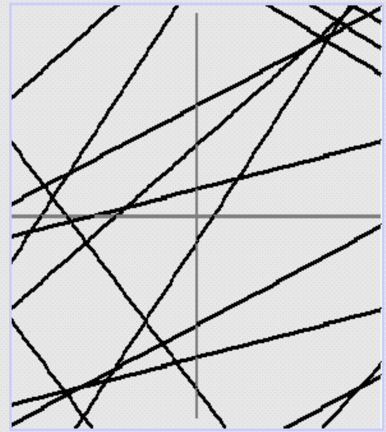
EBSD-TEM : Automated Crystal Orientation Mapping

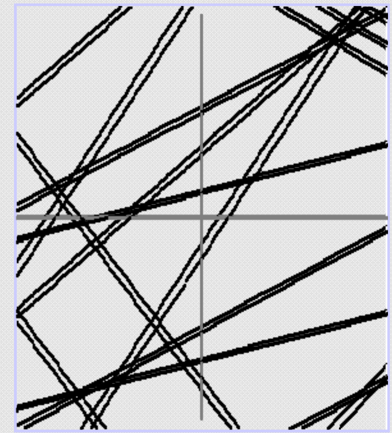




Severely deformed 7075 Al. Alloy

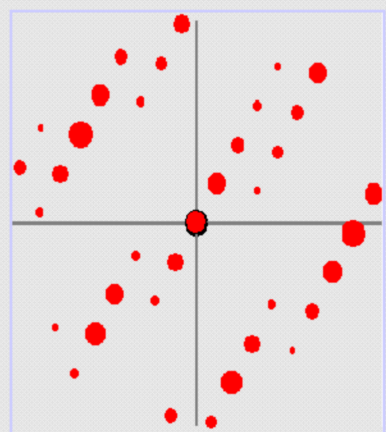
Kikuchi pattern

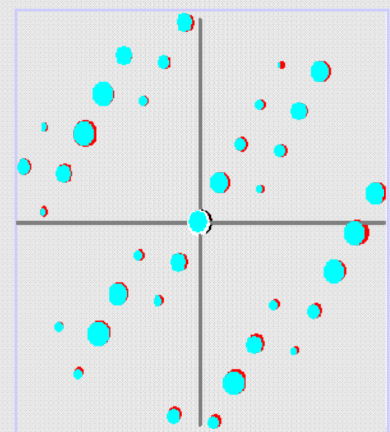




Orientation Ω

Orientation $\Omega + \Omega'(= \Omega + 0.1^{\circ})$



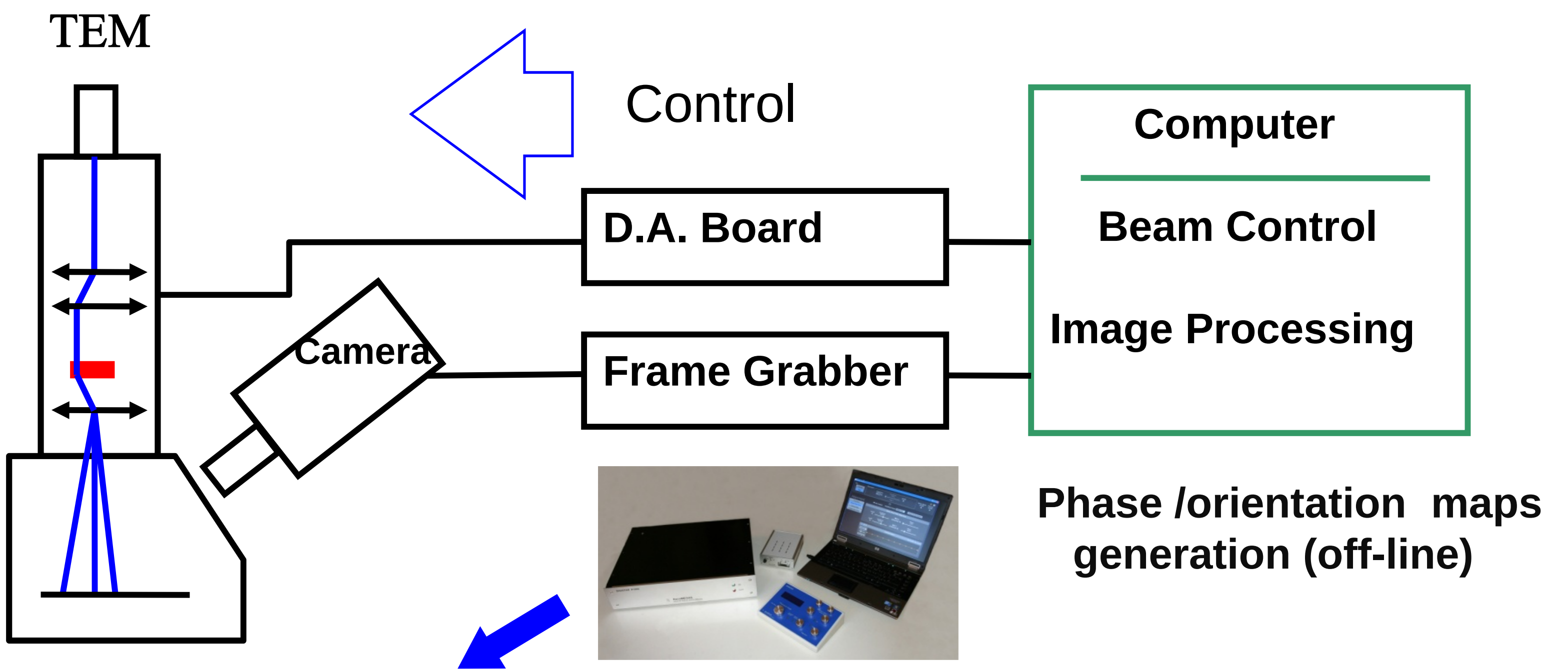


Bragg Spot pattern

NanoMEGAS
Advanced Tools for electron diffraction

Grenoble INP

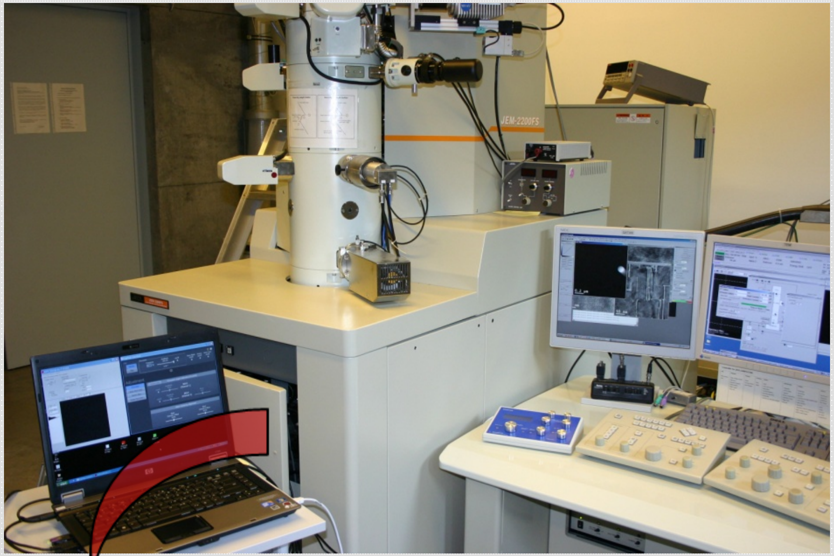
ASTAR (EBSD-TEM Procedure)

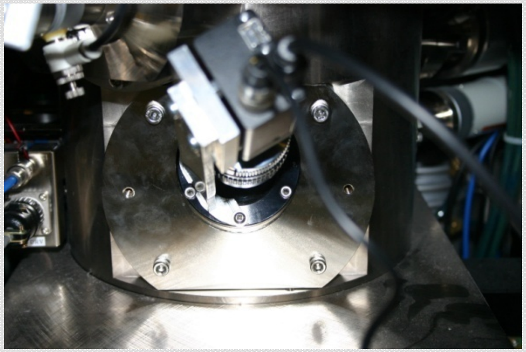


Beam scanning
Dedicated precession unit
« Spinning Star » -DigiSTAR

ASTAR :   Automatic Crystal Orientation
and
phase mapping hardware /software package for TEM

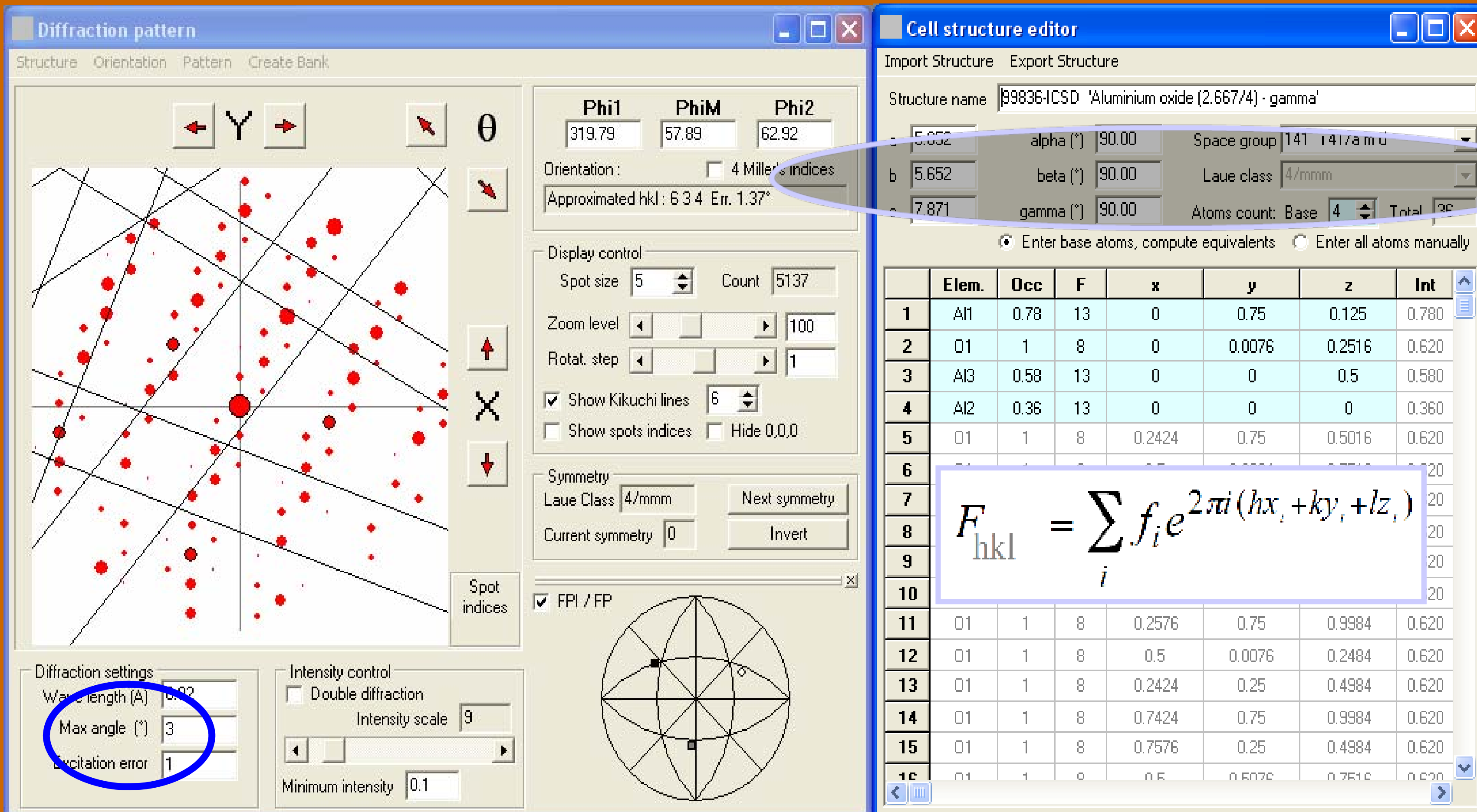
NanoMEGAS
Advanced Tools for electron diffraction





Dedicated fast CCD camera (> 100 patterns/sec) attached to the TEM screen

DiffGen : Template generator



Features: Any crystallographic structure

Laue class adapted to the space group

Structure generator (space group, structure factor equ.)

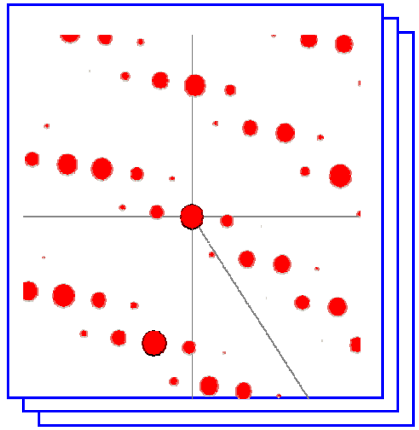
NanoMEGAS

Advanced Tools for electron diffraction

Grenoble INP

ASTAR : crystallographic orientation identification

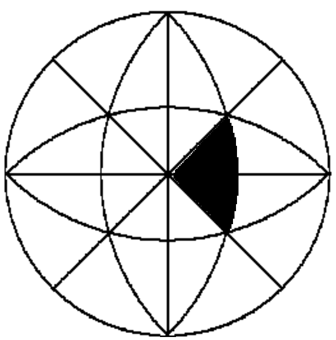
Pre-calculated templates



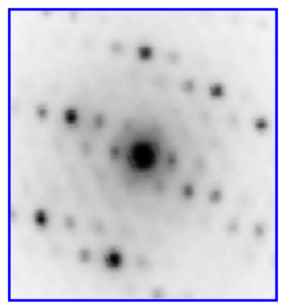
Template generation of all possible simulated orientations (every1º) within stereographic triangle for given crystal lattice(s) and symmetry

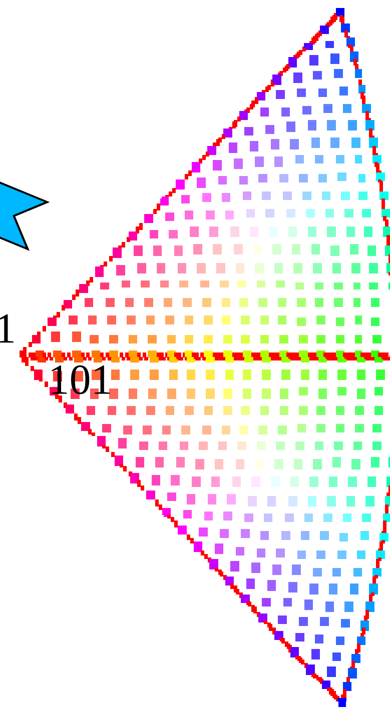
$$Q(i) \sim \sum_{j=1}^{m} P(x_j,y_j)\ T_i(x_j,y_j)$$

Correlation index



Acquired pattern



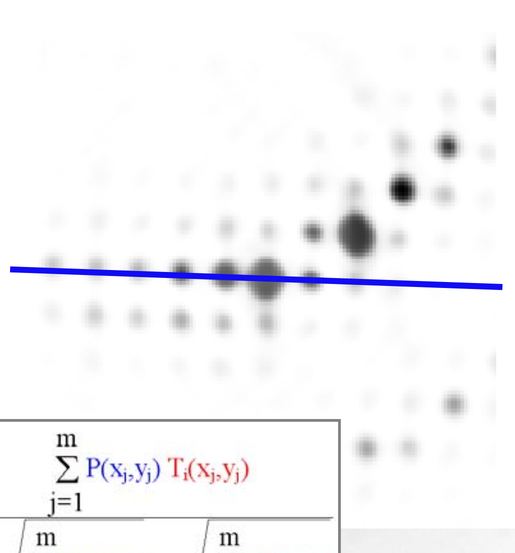


Stereographic projection

Degree of matching between experimental patterns and simulated templates is given by a correlation index ; highest value corresponds to the adequate orientation/phase

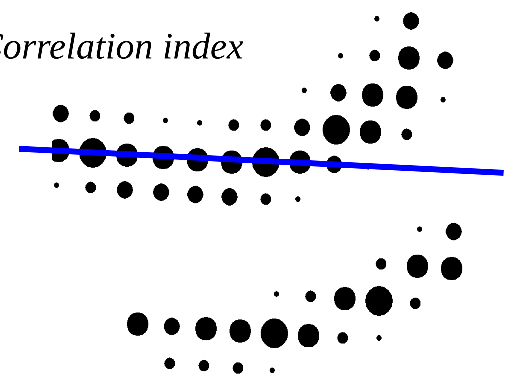
(example ,cubic)**2000~ simulated patterns**

ASTAR : pattern matching by image cross- correlation



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

Correlation index



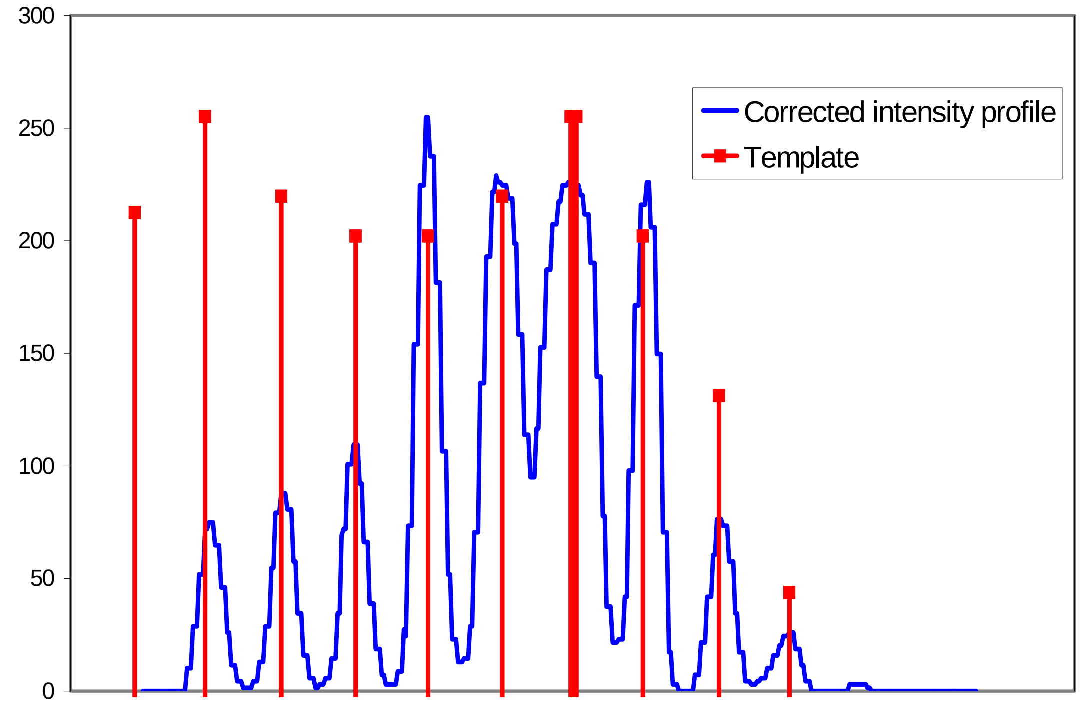
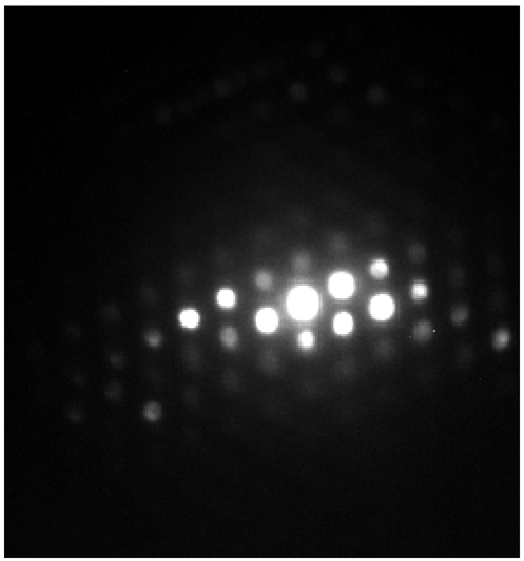


Image treatment

Cross-correlation comparison of all acquired ED patterns with all simulated templates to deduce correct pattern index; degree of matching between experimental patterns and simulated templates is given by a correlation index where highest value corresponds to the adequate orientation/phase.

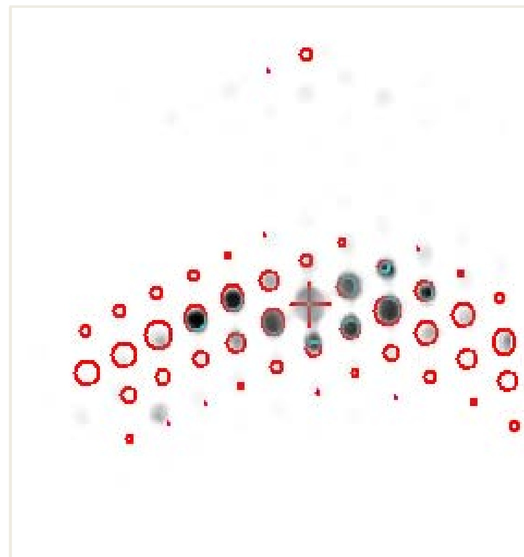
NanoMEGAS
Advanced Tools for electron diffraction

ASTAR identification example : nanocrystalline Cu

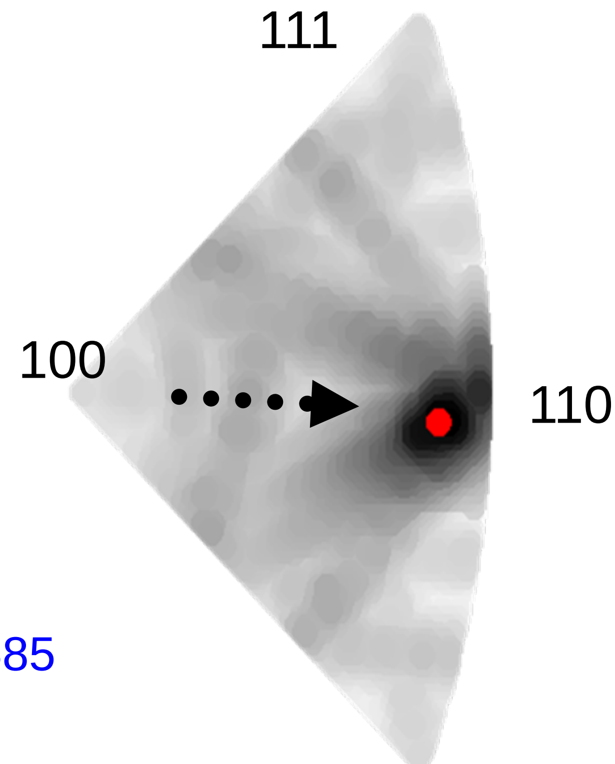


Diffraction pattern

(nanocrystalline cubic copper)



correlation index = 585

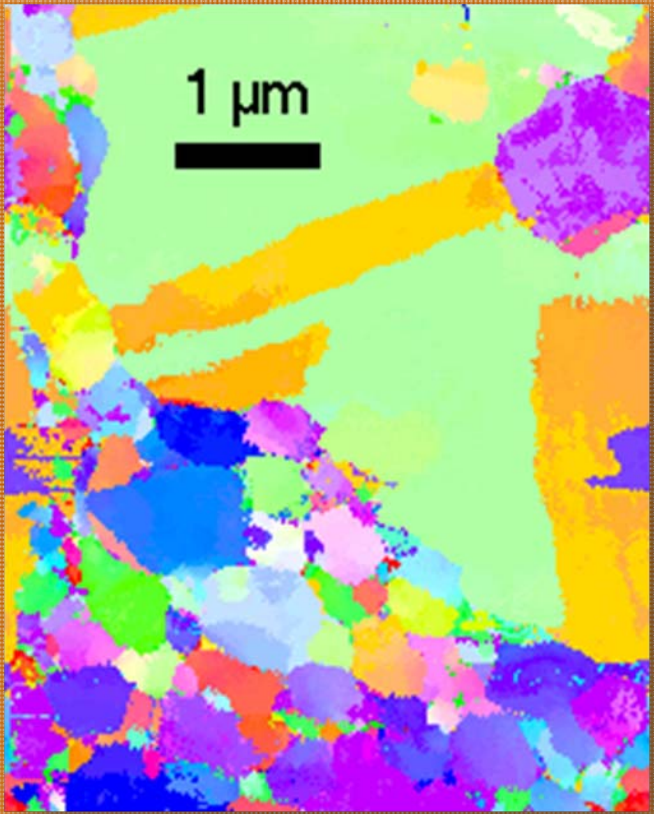


Correlation index map

For a given ED pattern, the correlation index map is calculated for all possible template orientations and plotted on a map that represents a portion of the stereographic projection (reduced to a double standard triangle). That resulting map reveals the most probable orientation for every experimental spot ED pattern (in this case ED pattern is found to be close to 110 ZA orientation)

ASTAR : ultra-fast TEM orientation map

Sample : severely deformed copper



Orientation map

250 x 200 pixel data adquisition

5 min !!

Typical software data analysis time (for cubic)

5-15 min

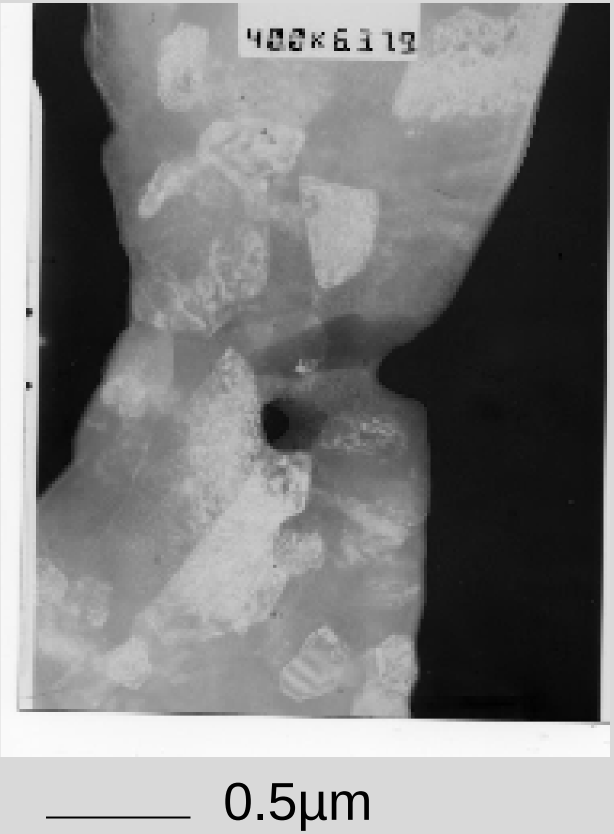
(hexagonal , tetragonal)

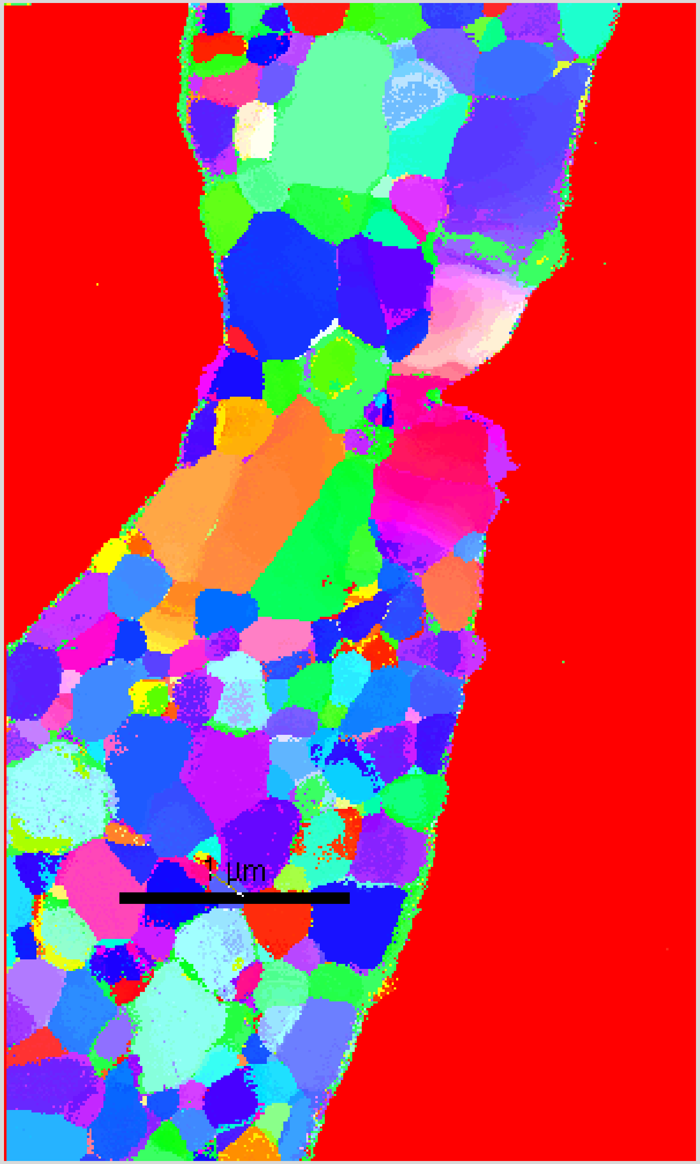
x 3- 4 more time

NBD step 20 nm

NanoMEGAS
Advanced Tools for electron diffraction

Grenoble INP

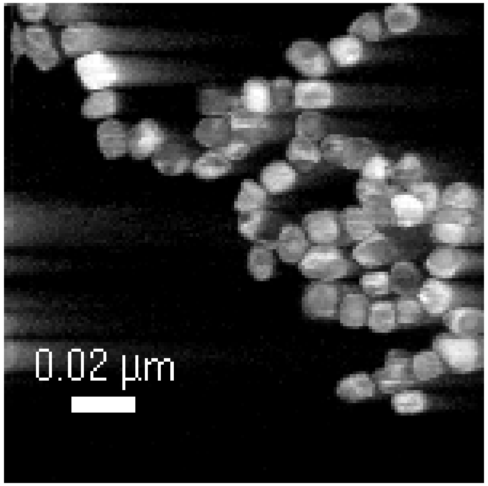


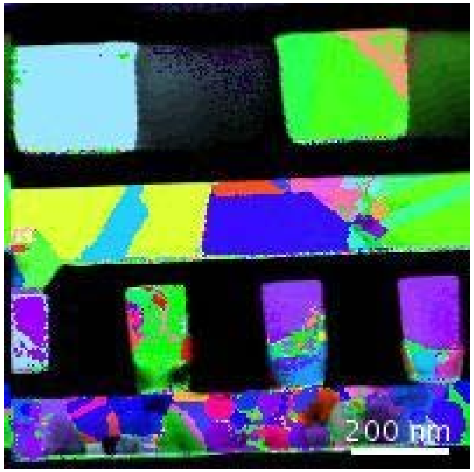


- Comparison of TEM image, and ASTAR results **15 nm resolution**
- CM20 UTwin LaB6 courtresy Prof . S.Godet ULB Brussels

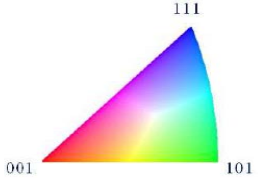
Pt nanoparticles , Cu lines in semiconductors (FEG –TEM)

Orientation (EBSD-like maps) at 1 nm resolution !





Courtesy Jeol Japan- Jeol 2100F



Pt nanoparticles

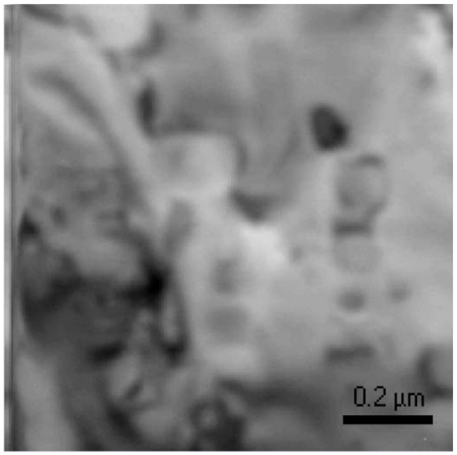
ASTAR phase/orientation TEM device with Jeol 2010 FEG , Jeol 2200 FS ,**1 nm spot size NBD mode**, 150x150 pixel, **step size 1 nm ,** 15 min adquis. time

NanoMEGAS Advanced Tools for electron diffraction

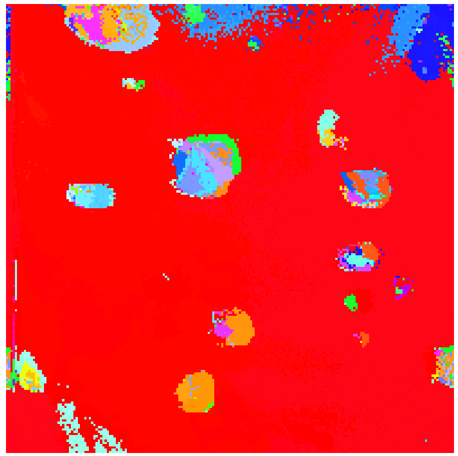
Courtesy Prof. P.Ferreira , Ganesh Univ Texas at Austin
Dr. Holm Kirmse Humboldt Univ Berlin

Grenoble INP

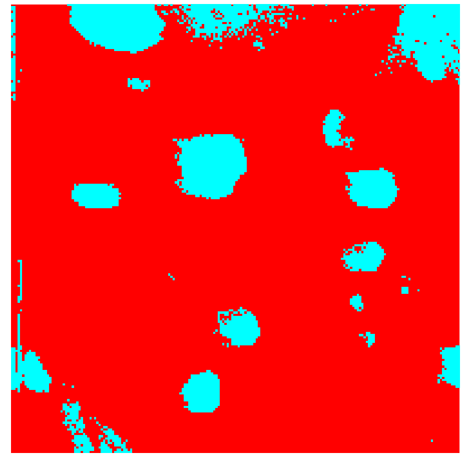
(Mn ,Ga) As clusters in GaAs matrix



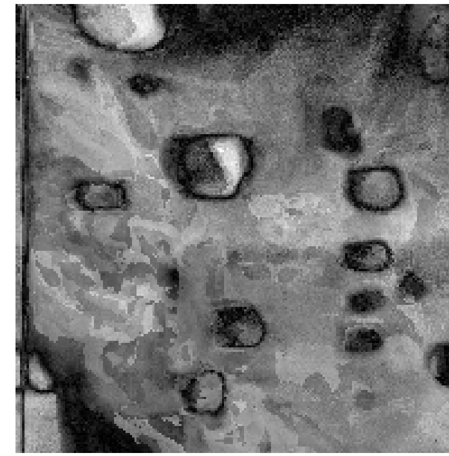
Virtual bright field map



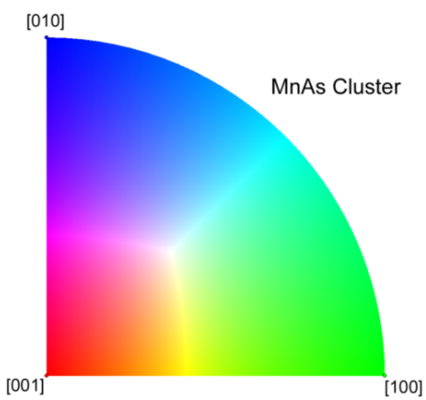
Orientation map



Phase map



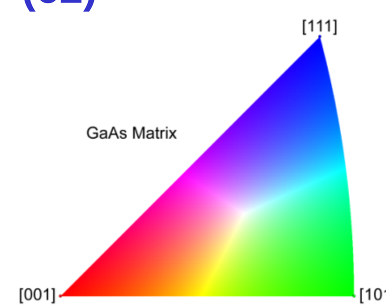
Reliability map



GaAs cubic F-43m (216) a= 0.56533 nm

MnAs β phase orthorhombic $P2_1/n2_1/m2_1/a$ (62)
a= 0.5704 nm, b= 0.3655 nm, c= 0.6365 nm

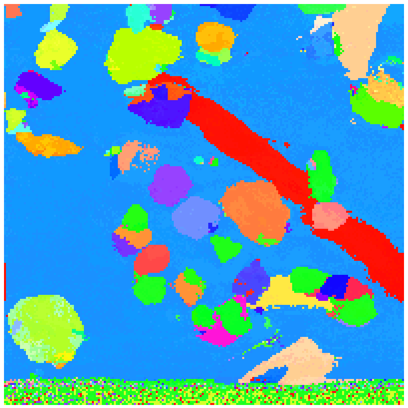
MnAs α phase hexagonal $P6_3/mmc$



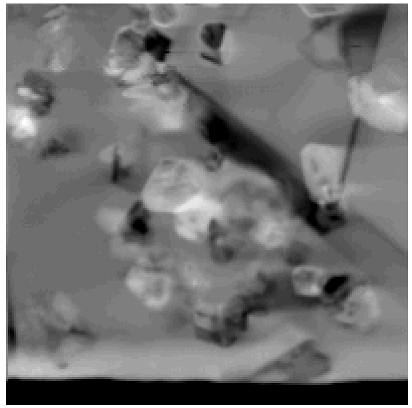
Courtesy Dr. Ines Haeusler Berlin Humboldt University, Germany

Si (matrix) and SiC β 3C (a= 0.436 nm)

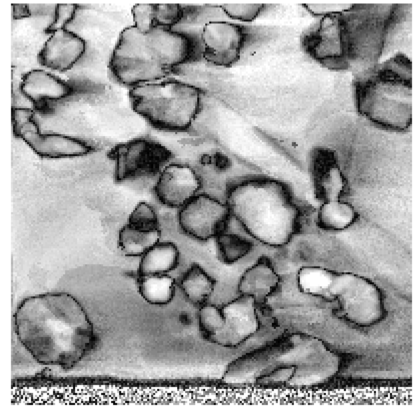
Which orientation relation between matrix – precipitates?



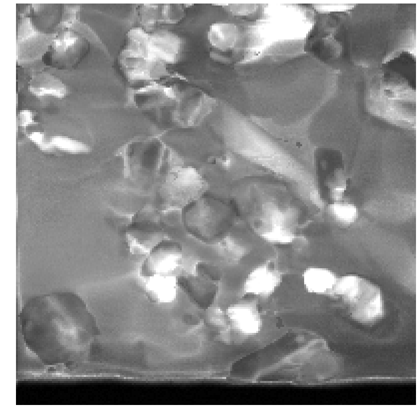
Orientation map



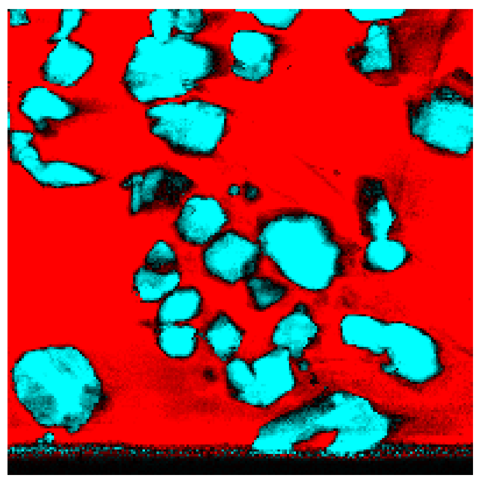
VBF



Reliability



Index



Phase map: Si red, SiC blue

Si cubic a= 0.5428 nm

Grain size 100-500 nm

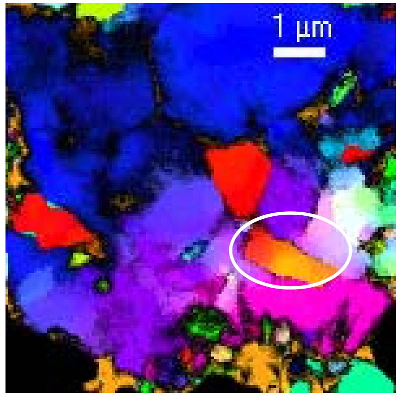
Jeol 3010, 25 nm spot size

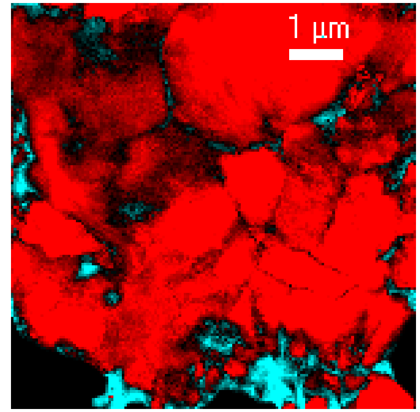
Courtesy T Epicier Univ LYON

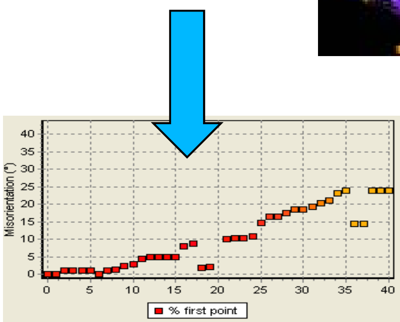
NanoMEGAS
Advanced Tools for electron diffraction

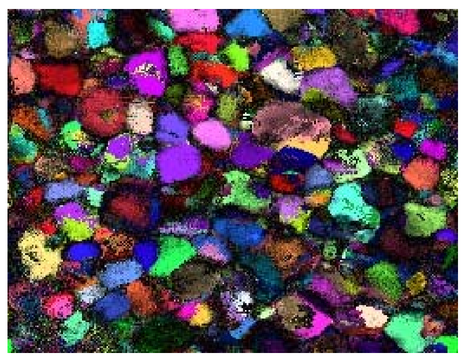
Grenoble INP

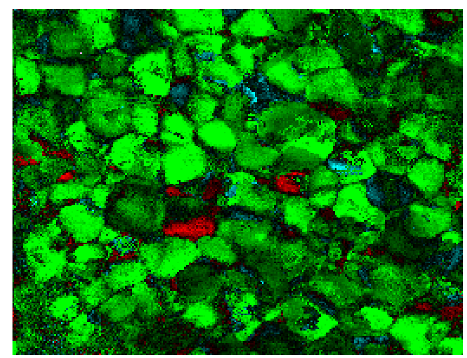
Orientation (a)and phase map of TiNb alloy revealing **cubic phase** (**in red** a= 0.332 nm Im-3m) and **orthorhombic phase** (**in blue** a=0.3215 nm, b=0.485 nm, c=0.462 nm Cmcm)







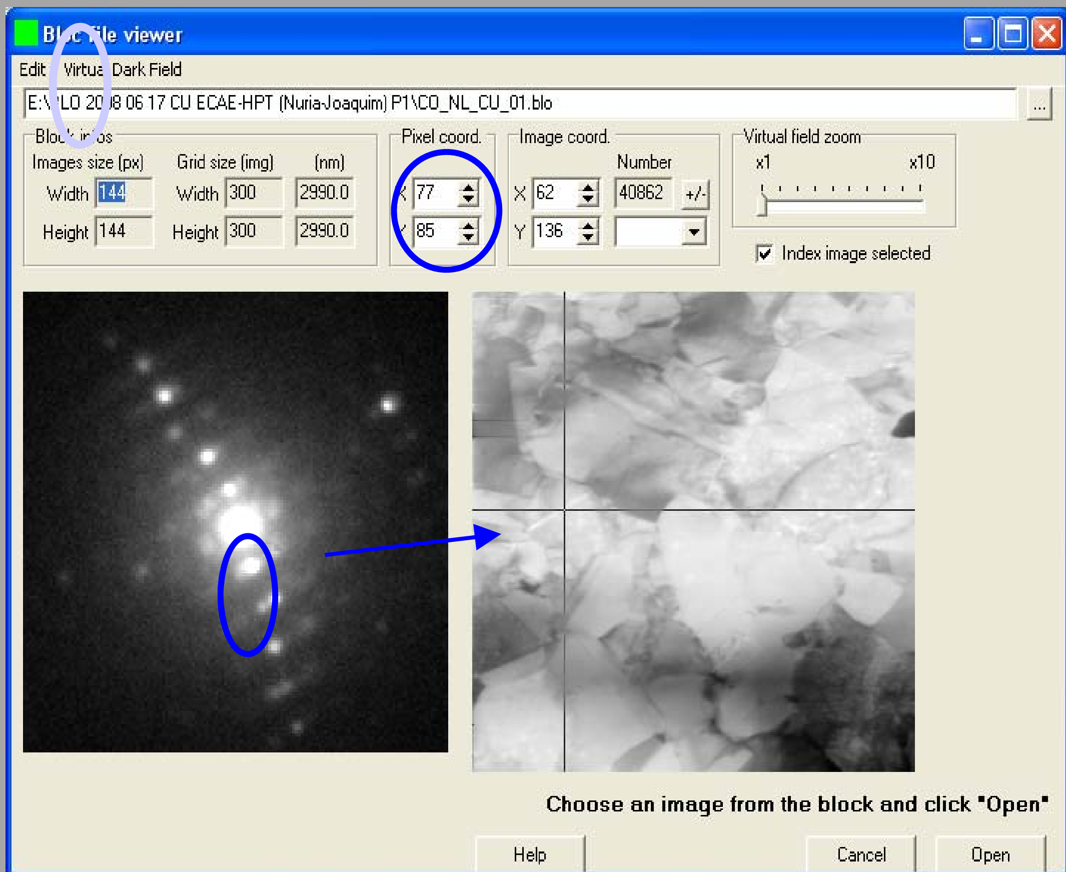




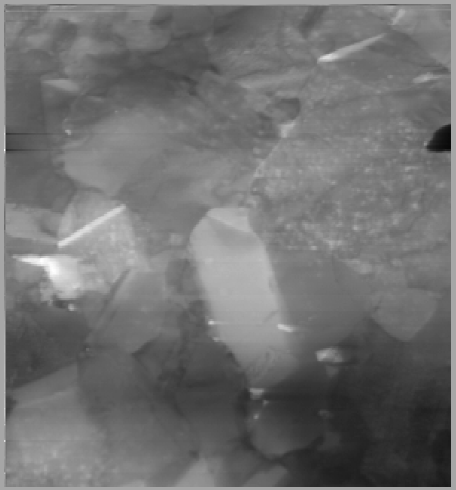
Orientation(c) and phase map d) of AlCu alloy **(in green** AlCu **monoclinic phase** C2/m a=1.206 nm, b=0.410 nm, c=0.691 nm, b = 54,04º **, in red** Al3Cu4 **orthorhombic phase** Fmm2 a= 0.812 nm, b= 1.419, c= 0.999 nm) courtesy Prof.V.Demange V.Dorcet, Univ Rennes France

INDEX : pattern identification software

Diffraction Pattern Block viewer

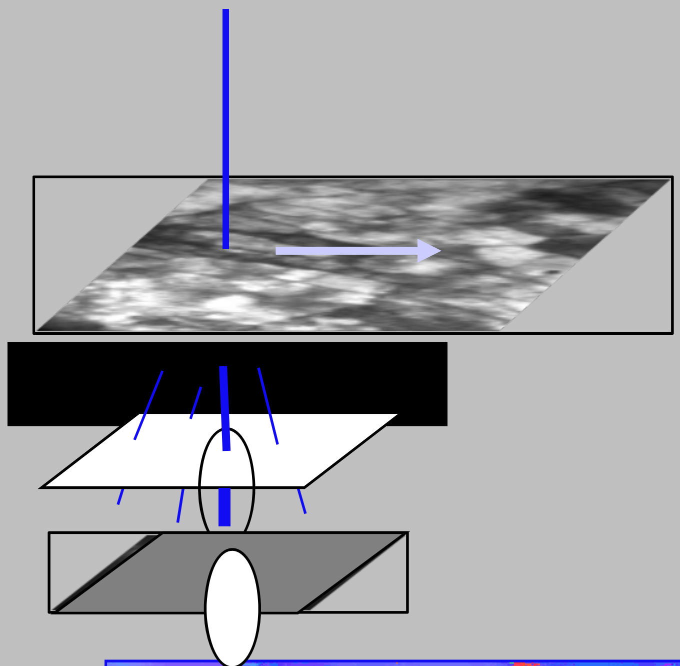


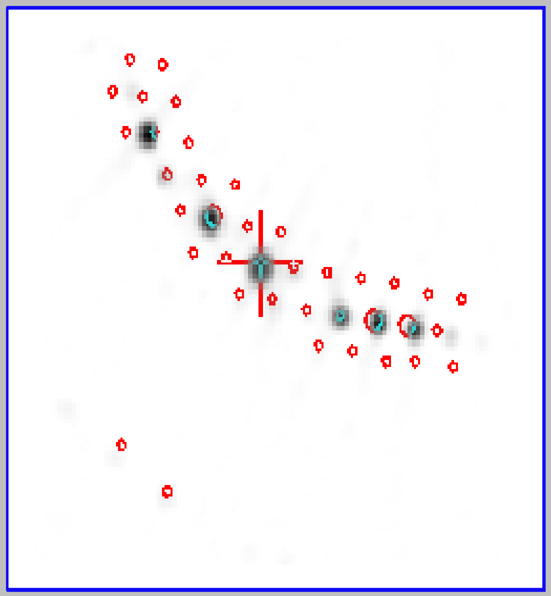
Virtual dark field image

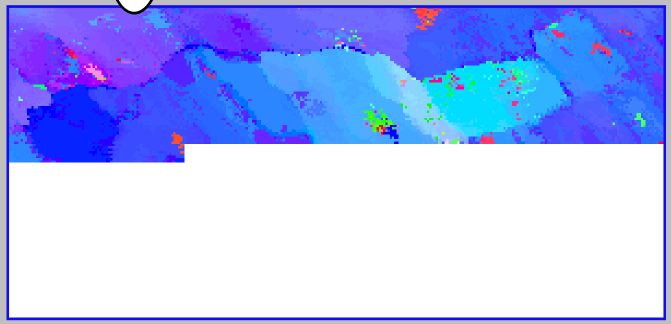


NanoMEGAS
Advanced Tools for electron diffraction

TEMdpa : Virtual Bright Field on-line construction

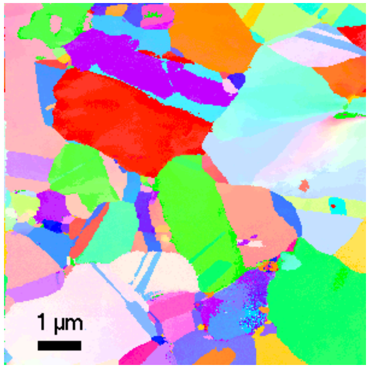


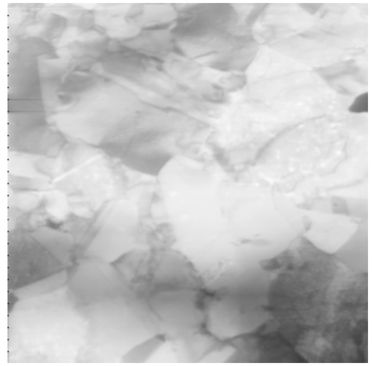




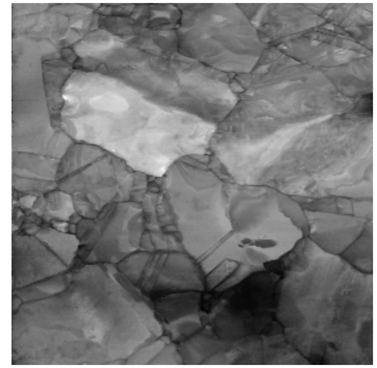
Orientation map

Bright field



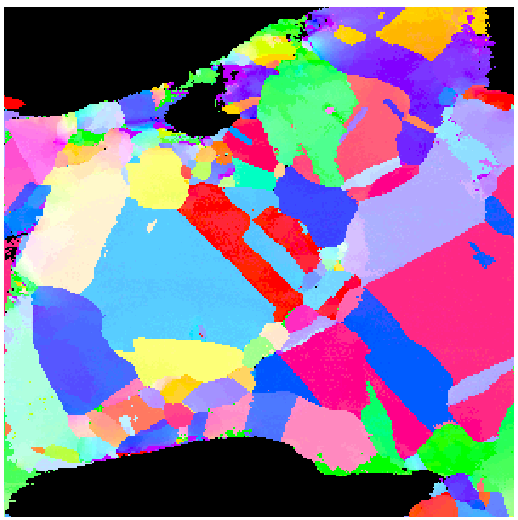


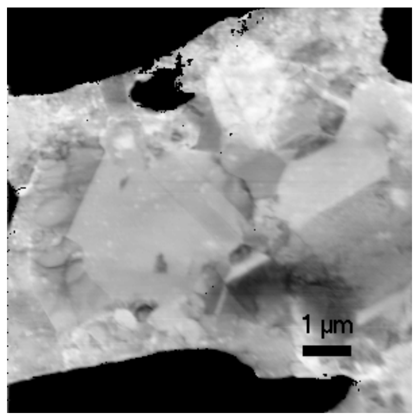
VBF



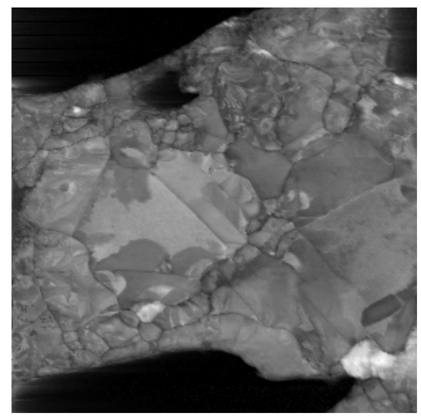
index

reliability

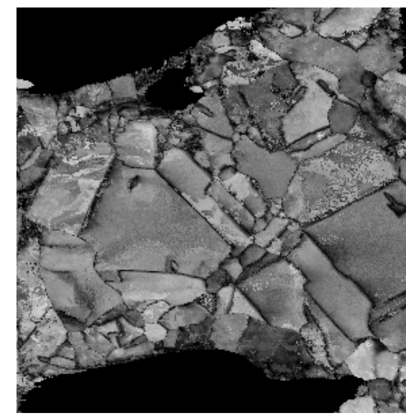




VBF



index



reliability

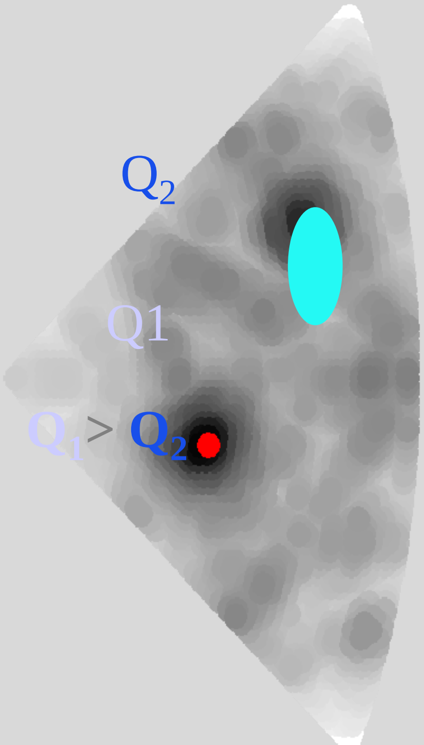
ASTAR (EBSD -TEM) orientation maps : Nanotwins in Cu

CBD mode Jeol 3010 microscope

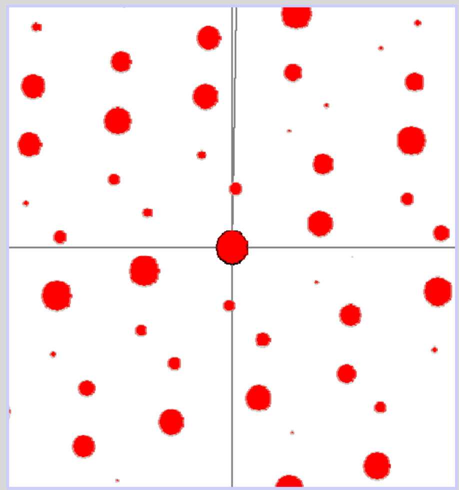
NanoMEGAS
Advanced Tools for electron diffraction

ASTAR : Reliability

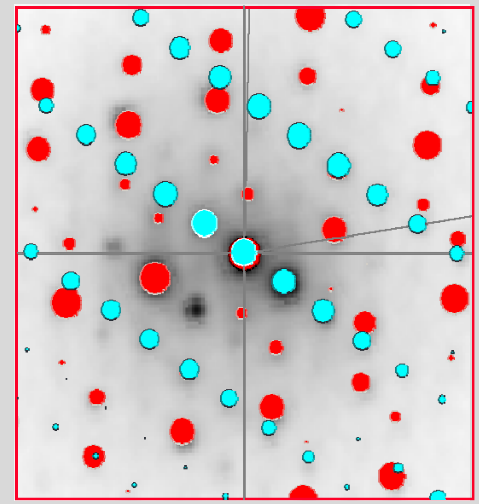
Stereographic projection



Templates for copper



Superimposed diffraction patterns at a grain bounday



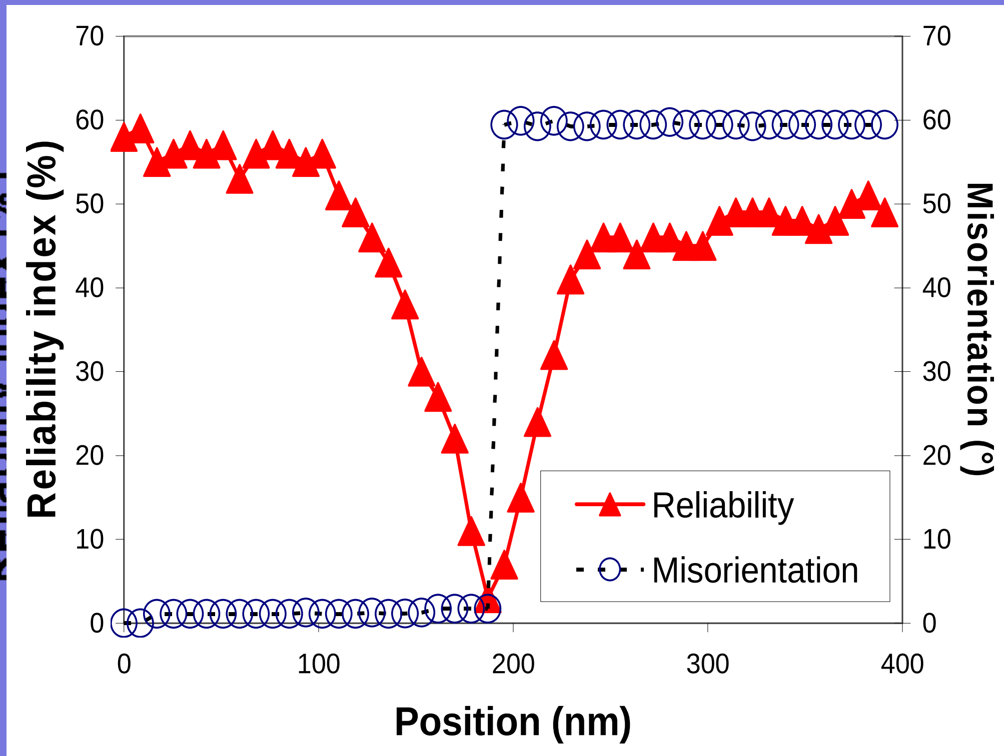
$$R = 100\,(1 - Q_2/Q_1)$$

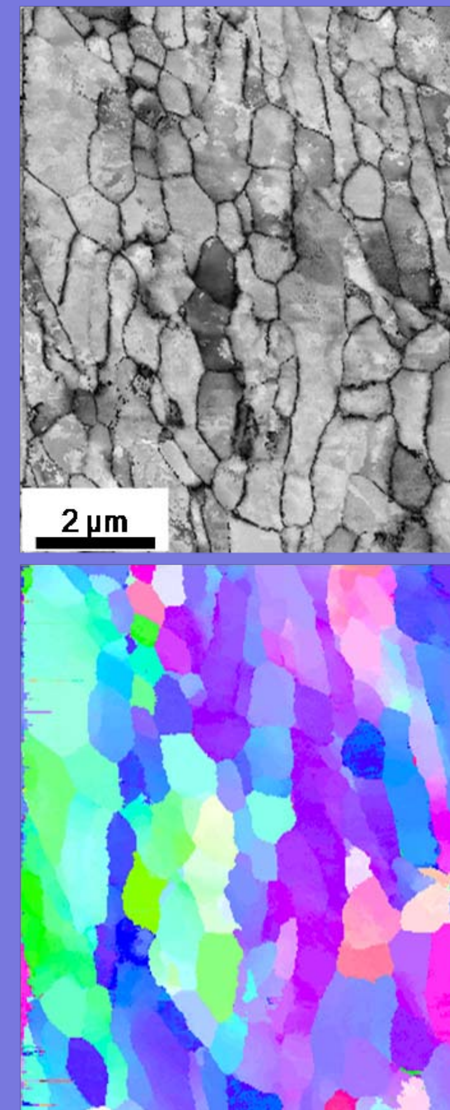
Reliability

Grenoble INP

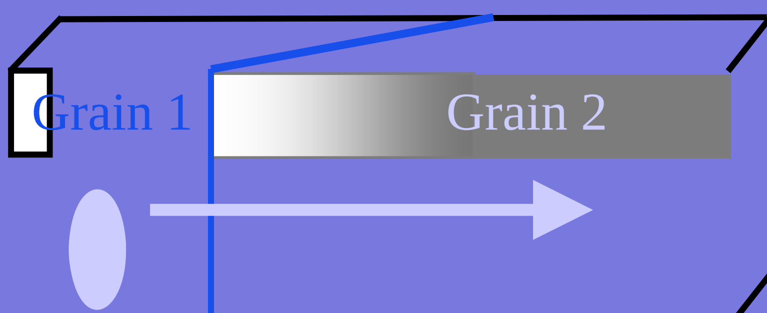
NanoMEGAS
Advanced Tools for electron diffraction

Deconvolution of superimposed Diffraction patterns

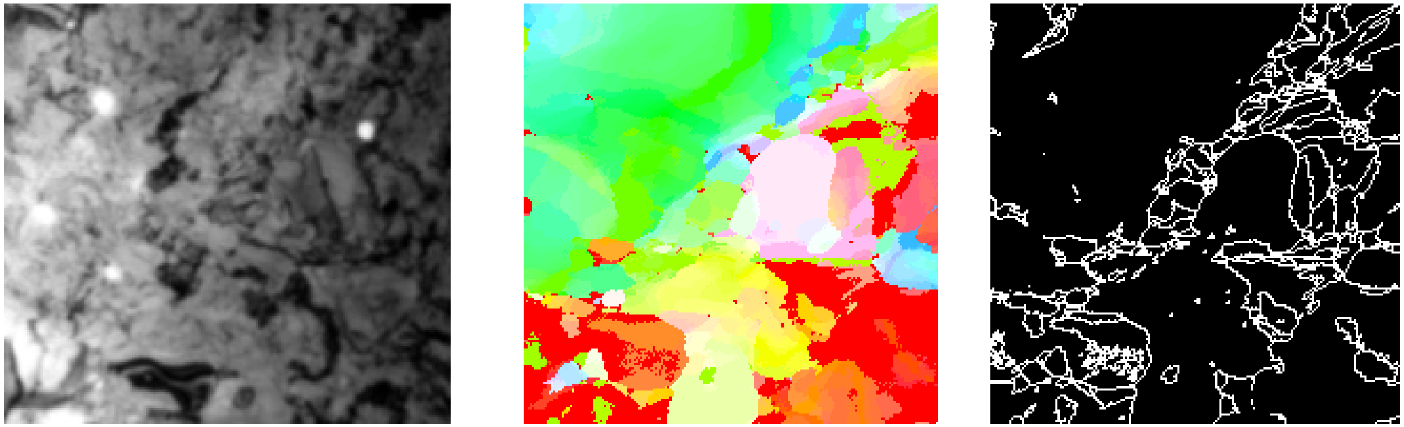




Position (nm)



Imaging Grain Boundaries



Deformed Zr alloy(Zirkaloy 4 rolled down 78%) bright field TEM image (left) showing poor grain boundary contrast, area 5x5 μm (center)same area ASTAR orientation map (right) **highlighted grain boundaries with angle > 15º**

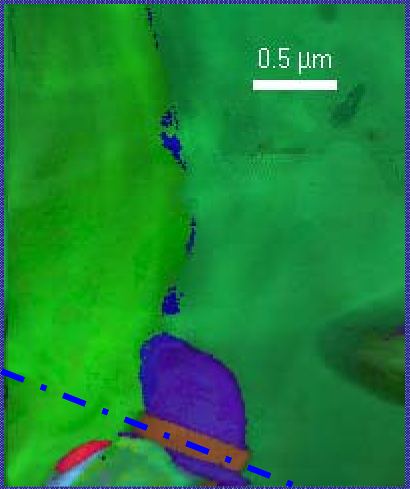
NanoMEGAS
Advanced Tools for electron diffraction

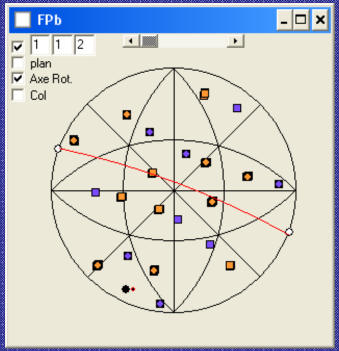
Grenoble INP

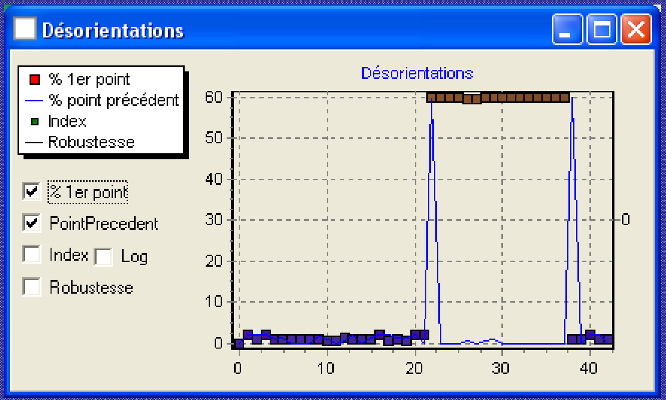
Courtesy Nippon Steel

Additional information obtained during this experiment :

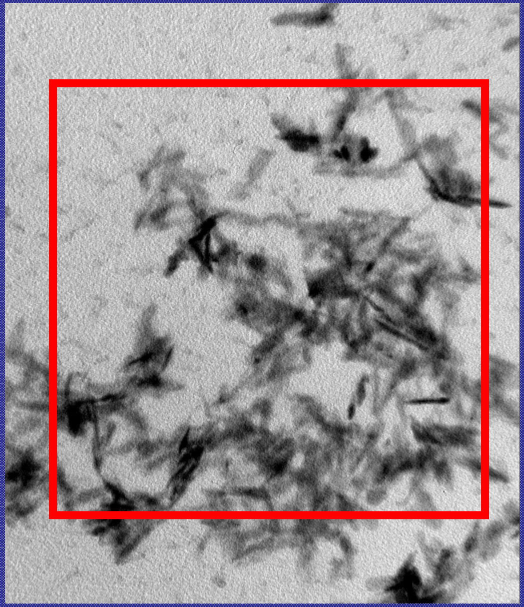
Study of Twins

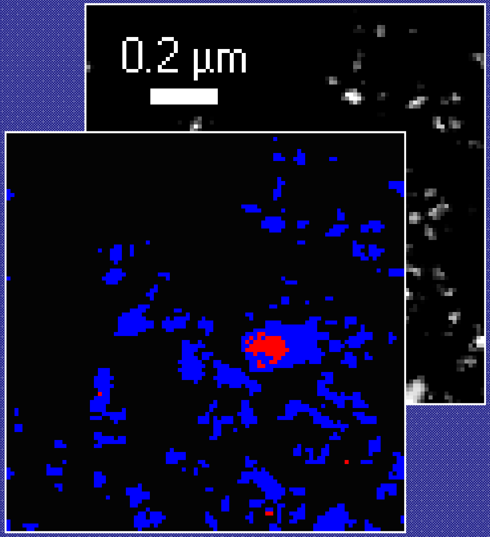






Study of Precipitates



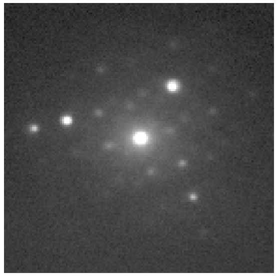


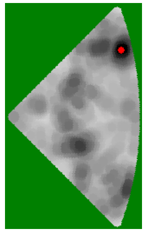
Identification of the various precipitates :

- Cementite (blue)
- TiN (red)

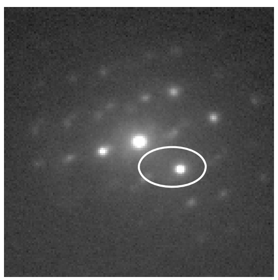
100x100 scanned at a rate of 44 fps (23 min). Step size 12 nm

Twin analysis in deformed steel samples

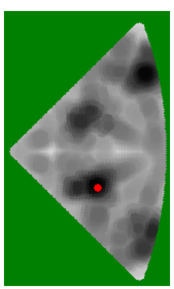


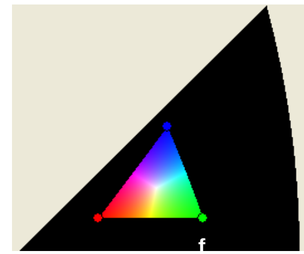


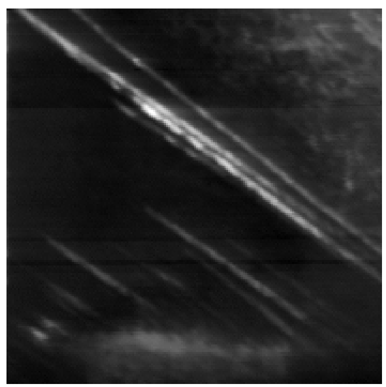
matrix reflections



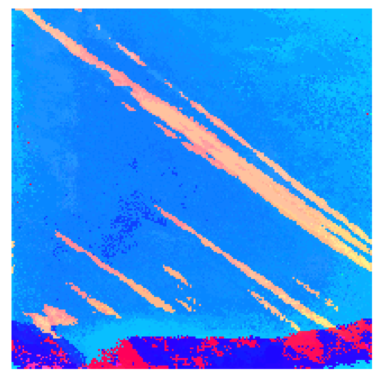
twin reflections

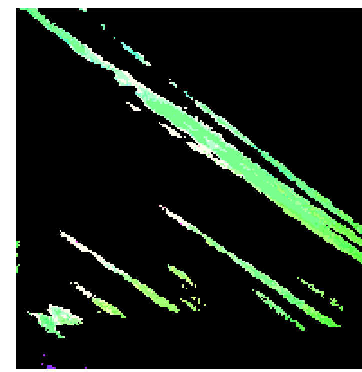






Virtual dark field



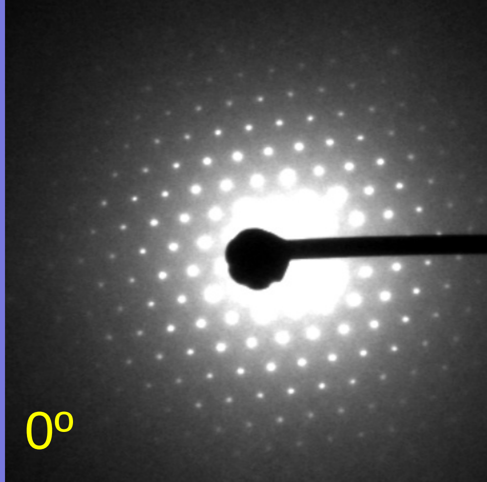


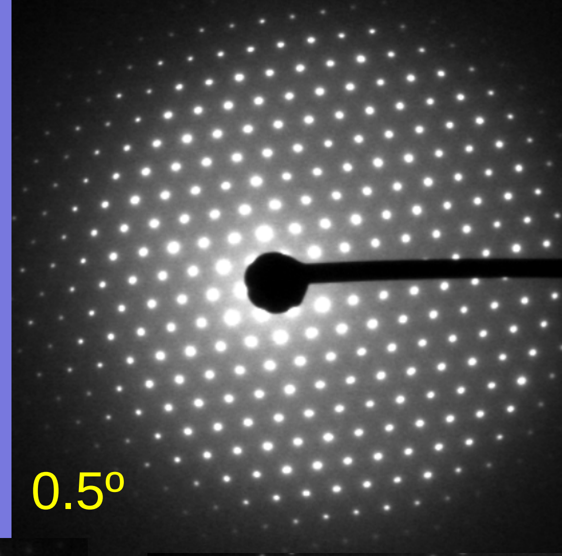
TWIP deformed steel

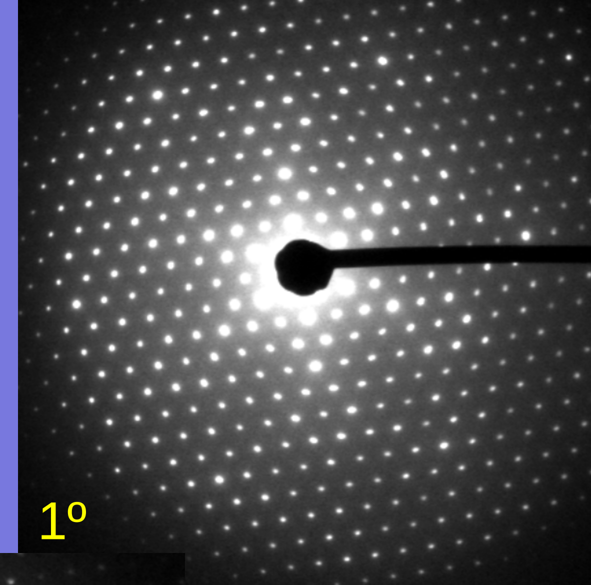
NanoMEGAS
Advanced Tools for electron diffraction

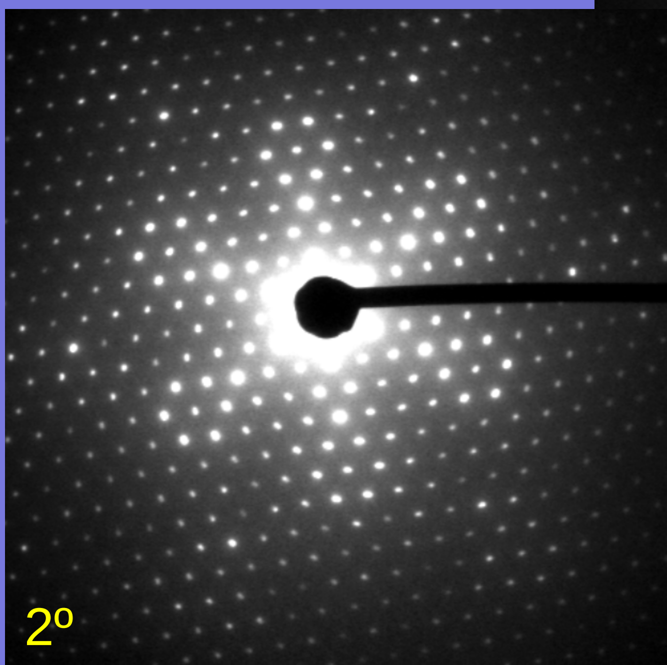
Courtesy Prof. S.Godet Univ Brussels (ULB) Belgium

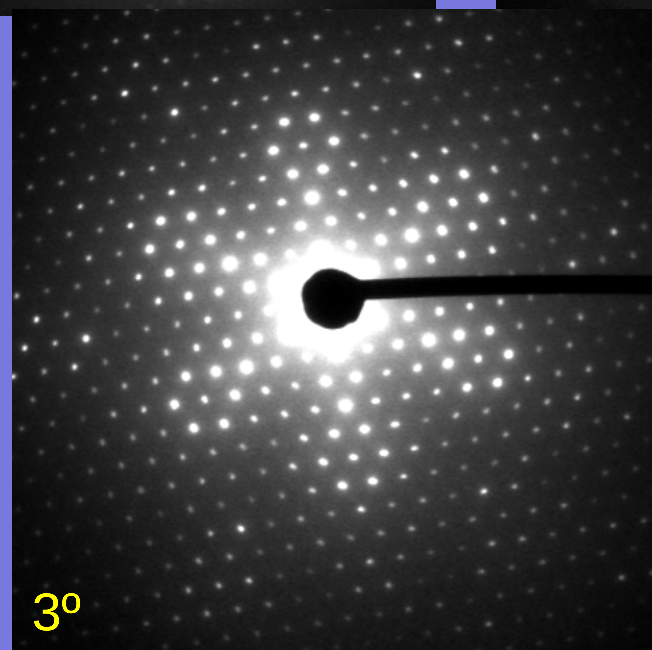
Grenoble INP





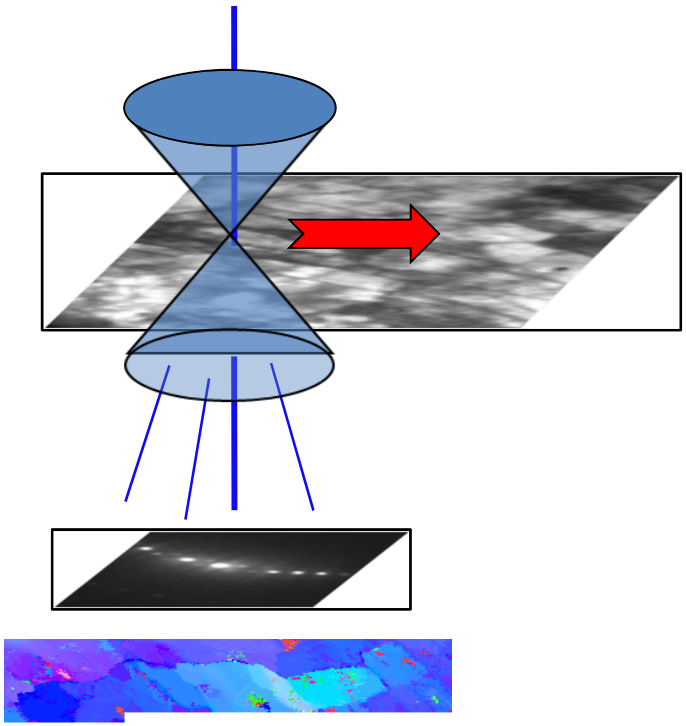




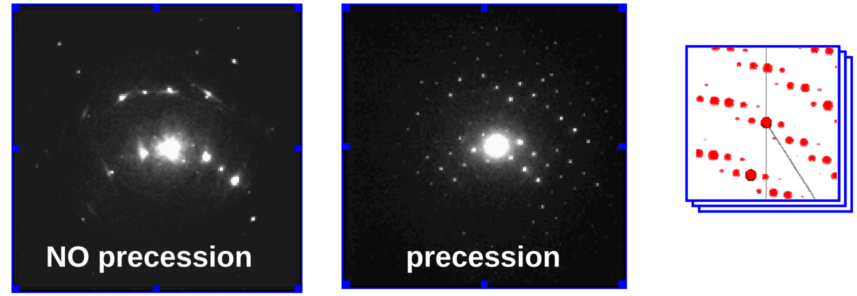


As the precession angle increases from 0° to 3°, the diffraction pattern goes to higher resolution (i.e. more diffraction spots are seen).

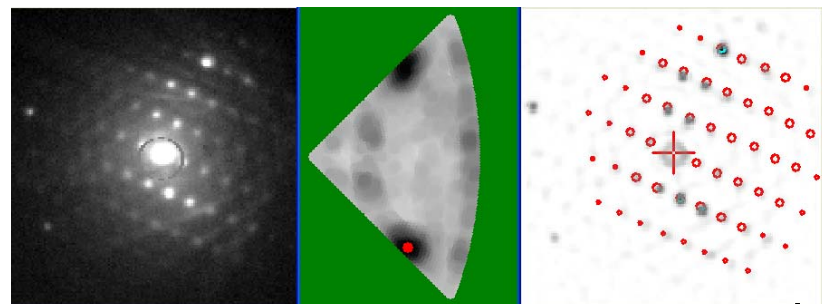
ASTAR : combine scanning with precession



Orientation map

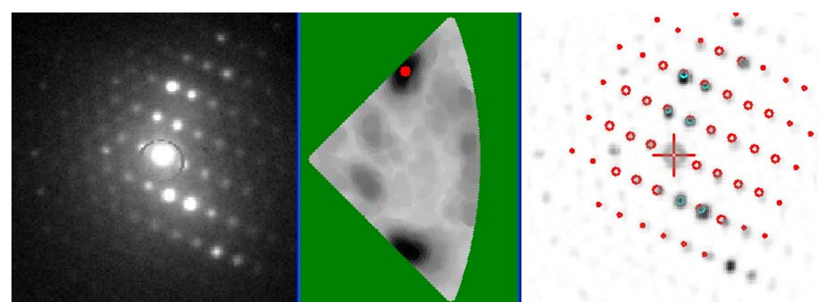


Using precession diffraction the number of ED spots observed increases (almost double) ; correlation index map becomes much more reliable when compared with templates



INCORRECT orientation

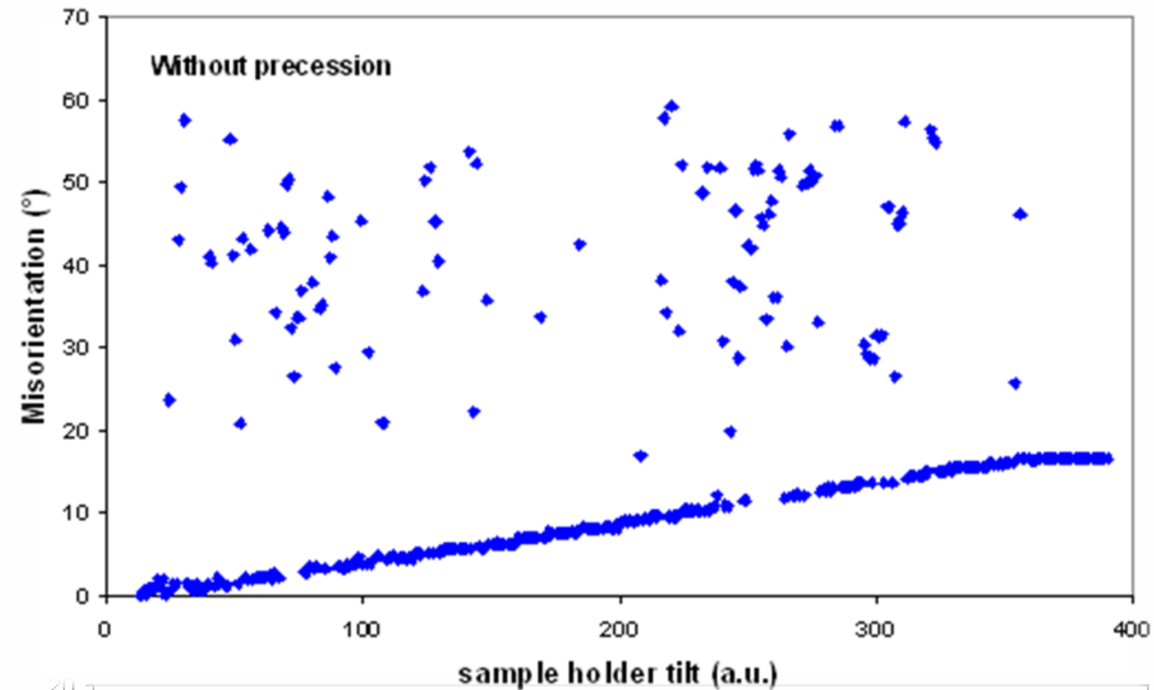
NO precession
(Index 622)

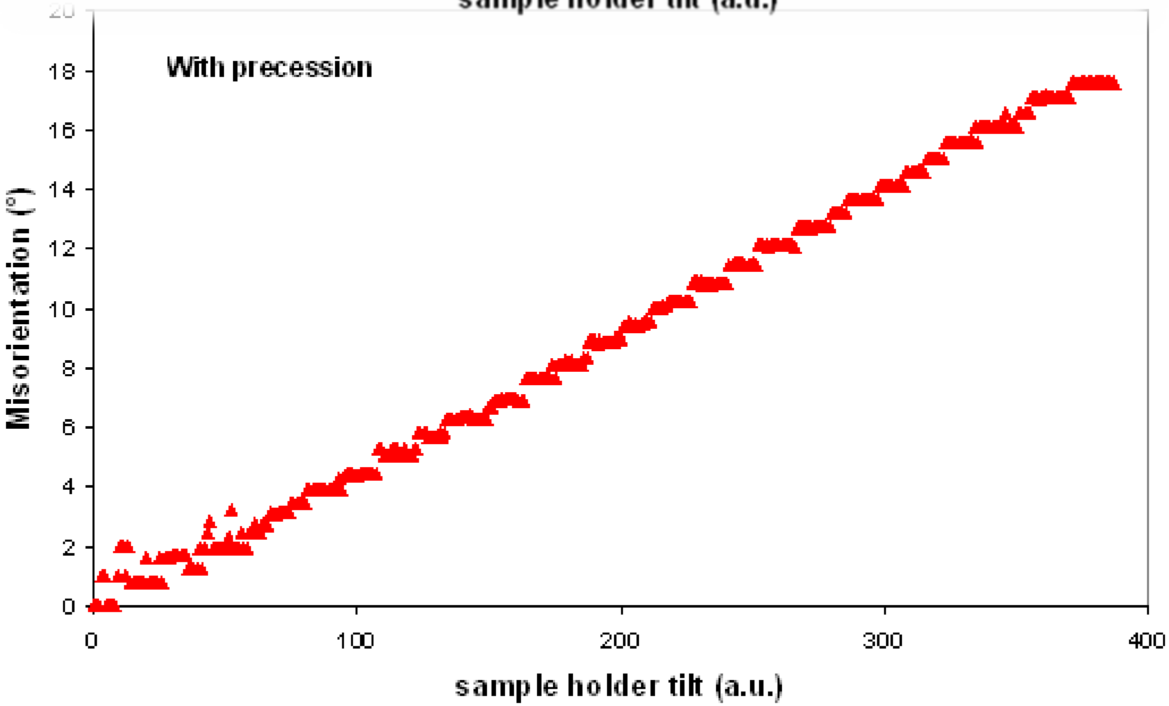


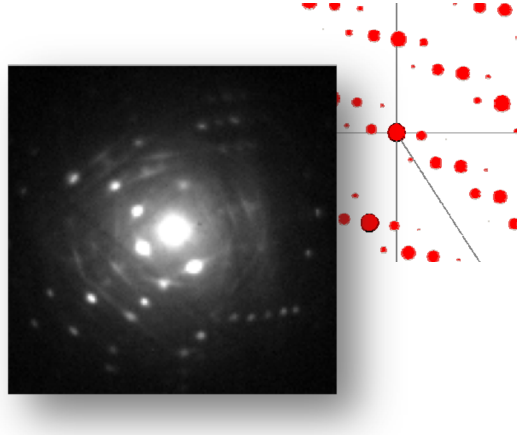
CORRECT orientation

0.5° precession
(Index 745)

In this example (right) a metal particle gives wrong correlation index without precession due to presence of Kikuchi lines; after applying precession (right lower image), index gets correct value as ED quality improves and Kikuchi lines dissapear



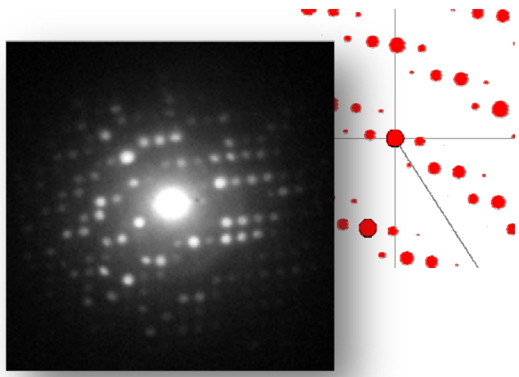




Mayenite mineral

Same crystal tilted 0-20º

400 patterns collected

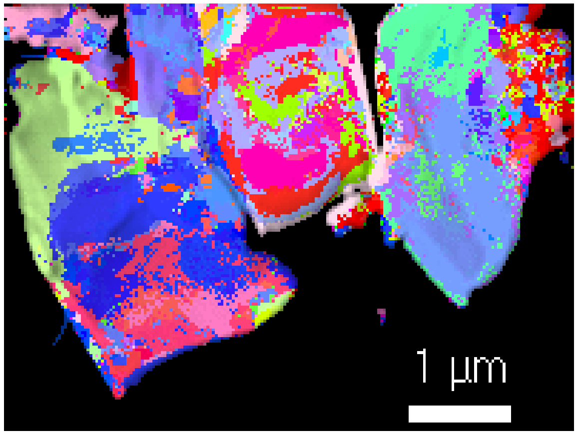


Precession angle 0.25º

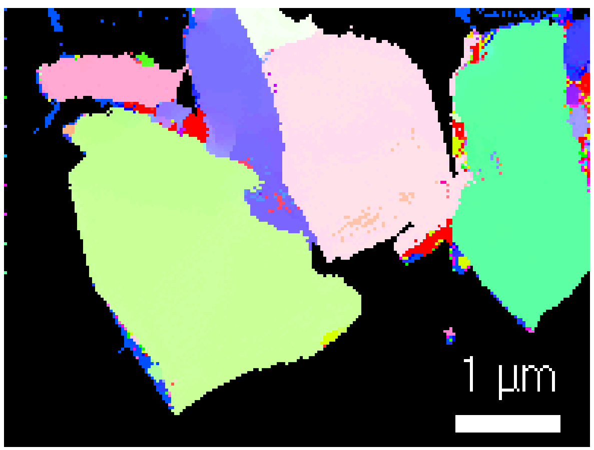
NanoMEGAS
Advanced Tools for electron diffraction

EBSD-TEM : orientation maps with and without beam precession

orientation map, NO precession



beam scanning step 28 nm



orientation map

precession angle 0.25º

In this example three different cubic mayenite crystals *$Ca_{12}Al_{14}O_{33}$* are analyzed with ASTAR ;

orientation map generated without precession results in inconsistent index over areas that must have uniform orientation. On the contrary, orientation maps generated with small precession angle present true uniform orientation over individual grains

CONCLUSION

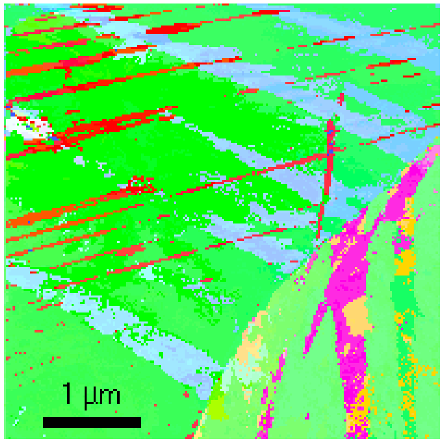
Orientation maps are <u>more precise</u> with precession

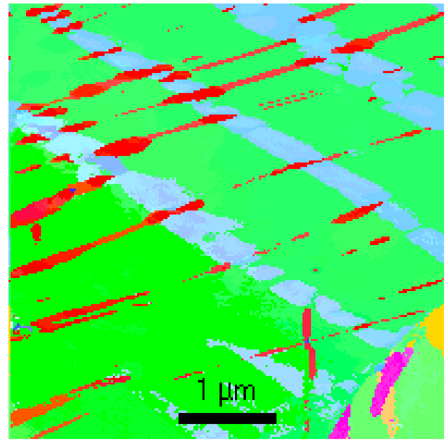
NanoMEGAS
Advanced Tools for electron diffraction

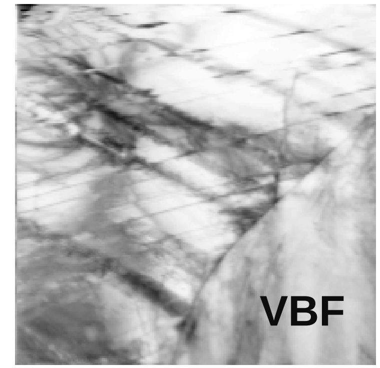
ASTAR : Phase maps with and without precession

3 existing phases: only possible to distinguish by precession

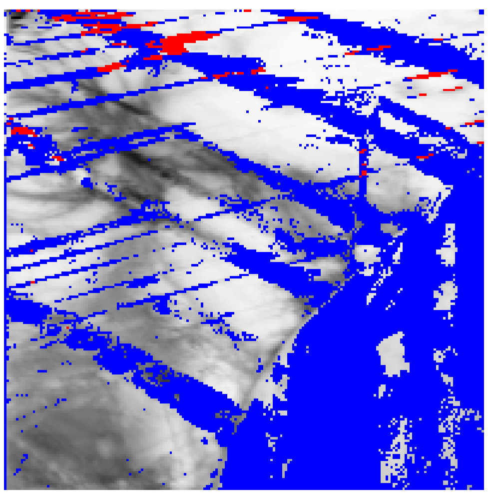
Orientation map

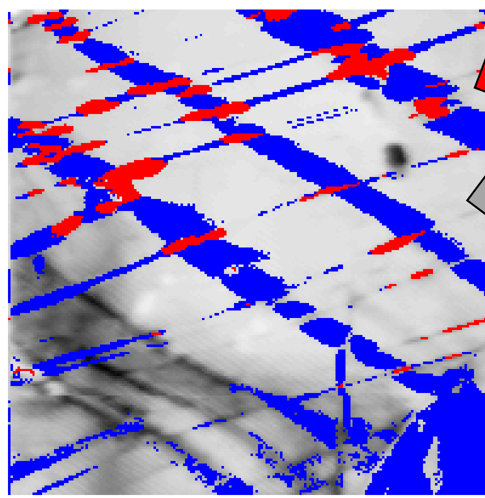






crystal phase map





When stacking faults cross themselves, they produce locally α' martensite structure (a= 2.87 A)

Austenitic matrix with γ fcc structure (a=3.58 A)

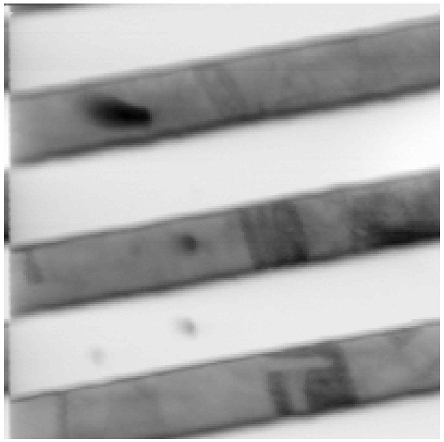
Stacking faults with ε hexagonal structure (a=2.57 c= 4.08 A)

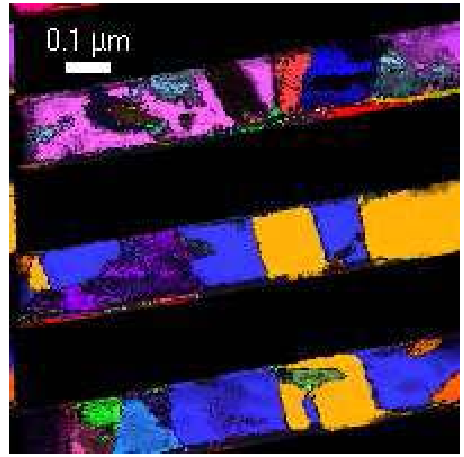
NO precession

precession 0.4º

NanoMEGAS
Advanced Tools for electron diffraction

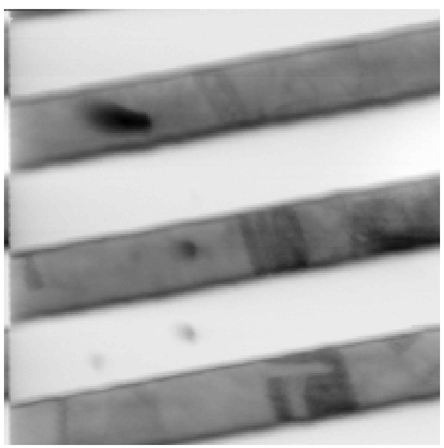
EBSD like-TEM : copper lines   (FEG –TEM)

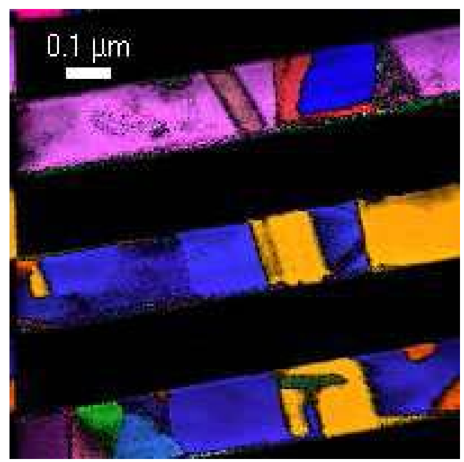


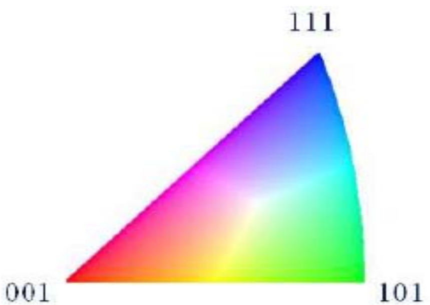


Bright field

No precession







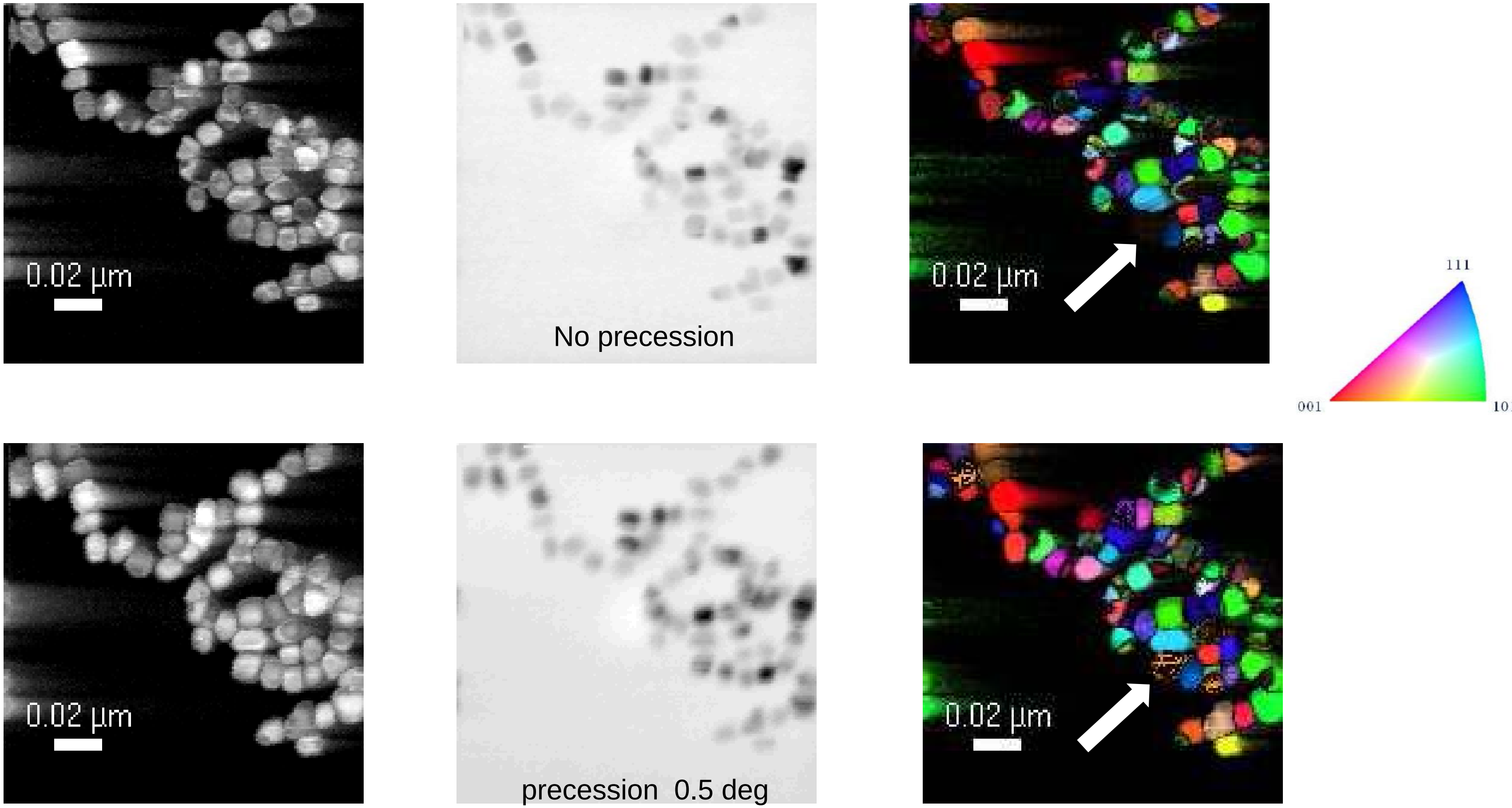
precession 0.6 deg

Jeol 2010 FEG Univ Texas -Austin ,<u>**1 nm spot size** </u> **NBD mode**, 200x200 pixel, **step size 9 nm**

Courtesy Prof. P.Ferreira , Ganesh   Univ Texas at Austin

NanoMEGAS
Advanced Tools for electron diffraction

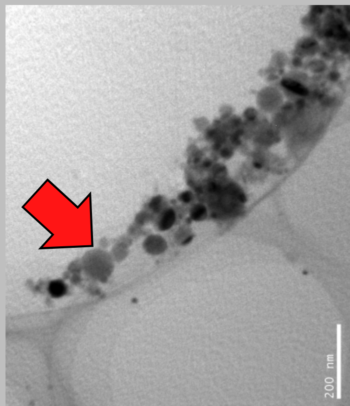
Pt nanoparticles (FEG –TEM)



Jeol 2010 FEG Univ Texas -Austin ,**1 nm spot size NBD mode**, 150x150 pixel, **step size 1 nm**

recent unpublished results

Nanoparticle (50 nm) phase identification



cubic 8.32 A

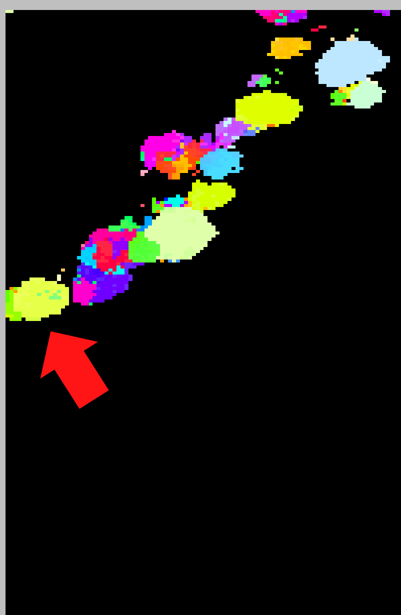
$Fd\bar{3}m$

Magnetite or maghemite ??

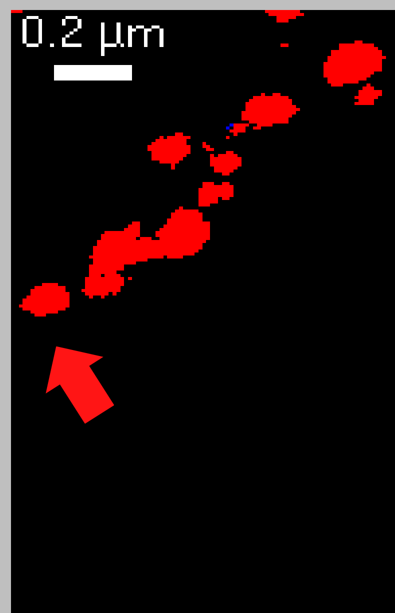
$P4_132$ γ-Fe_2O_3

Fe_3O_4

cubic 8.32 A



Orientation map precession 0.3º



PHASE map precession 0.3º

ALL Nanoparticles

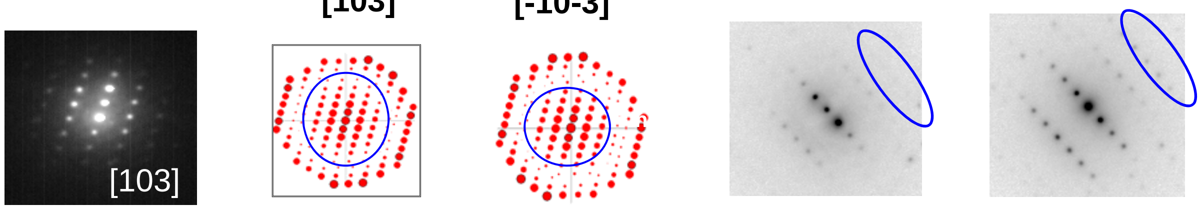
REVEALED AS

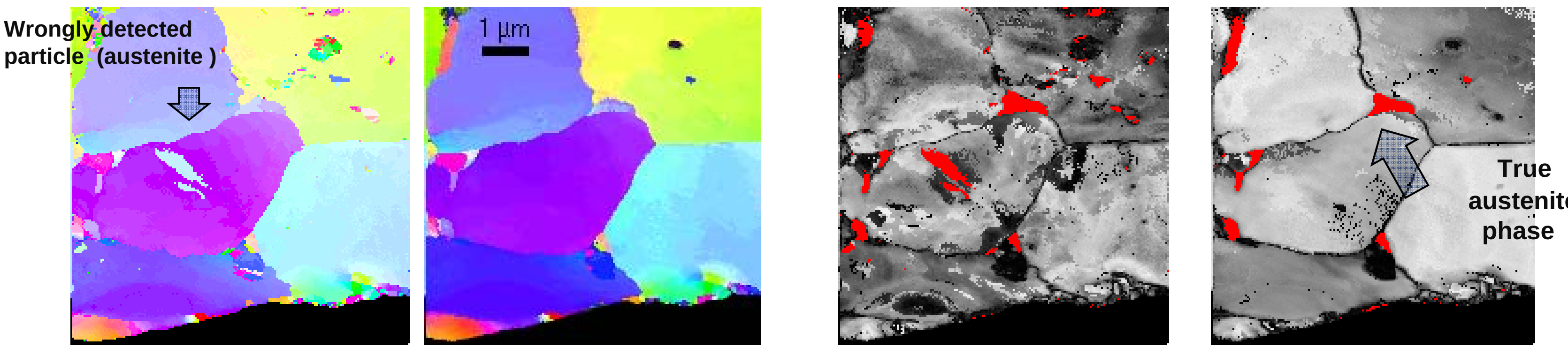
magnetite (RED)

Grain and phase boundaries: solving 180° ambiguity with precession

The ambiguity in the indexing of ED spot patterns arises from the fact that a particular reflection may be indexed either as (hkl) or (-h-k-l). **While this ambiguity is irrelevant for some applications, it becomes important for determination of grain and phase boundary parameters.**

[103] [-10-3]



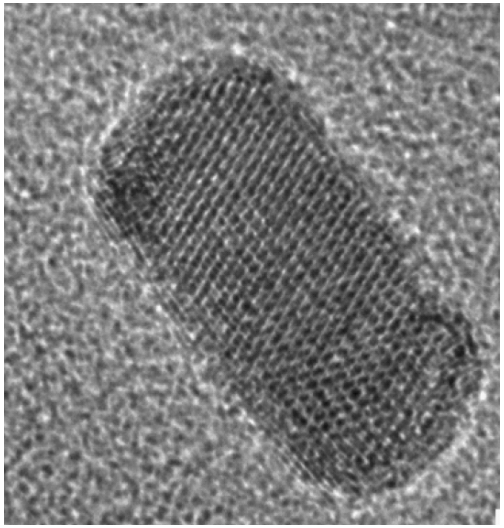


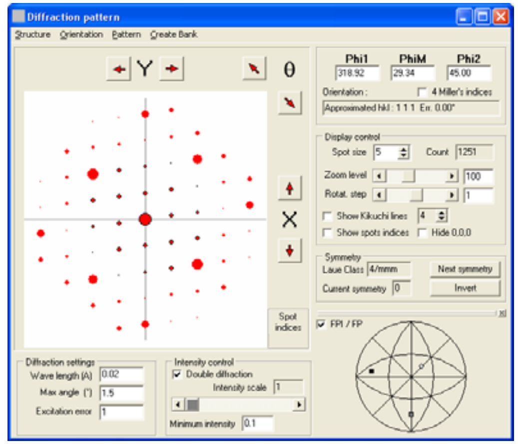
ASTAR + precession

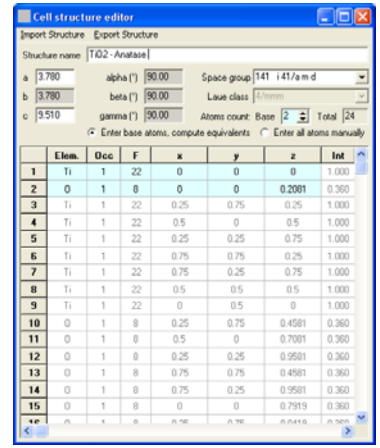
ASTAR + precession

TRIP steel (bcc ferrite + fcc austenite) ; Philips CM120 (6 min scanning),

Indexing high resolution image of anatase - TiO2

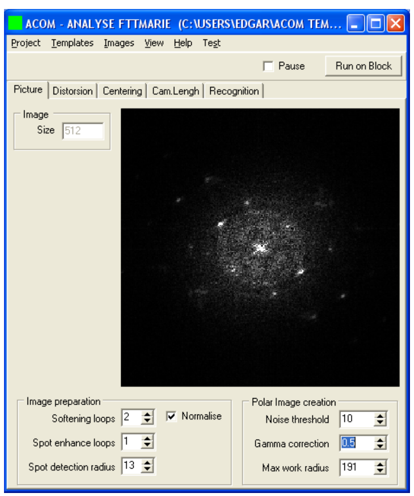


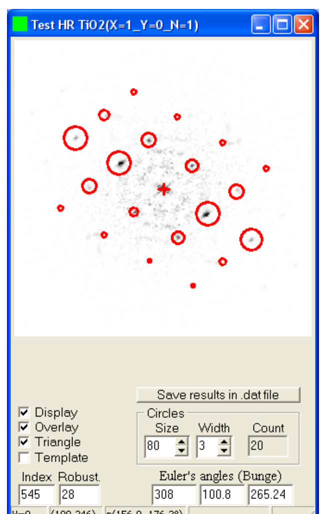


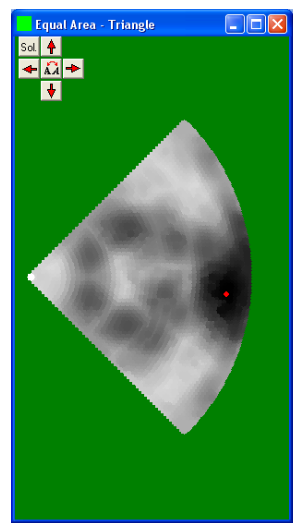


	Elem.	Occ	F	x	y	z	Int
1	Ti	1	22	0	0	0	1.000
2	O	1	8	0	0	0.2081	0.360
3	Ti	1	22	0.25	0.75	0.25	1.000
4	Ti	1	22	0.5	0	0.5	1.000
5	Ti	1	22	0.25	0.25	0.75	1.000
6	Ti	1	22	0.75	0.75	0.25	1.000
7	Ti	1	22	0.75	0.25	0.75	1.000
8	Ti	1	22	0.5	0.5	0.5	1.000
9	Ti	1	22	0	0.5	0	1.000
10	O	1	8	0.25	0.75	0.4581	0.360
11	O	1	8	0.5	0	0.7081	0.360
12	O	1	8	0.25	0.25	0.9581	0.360
13	O	1	8	0.75	0.75	0.4581	0.360
14	O	1	8	0.75	0.25	0.9581	0.360
15	O	1	8	0	0	0.7919	0.360

it is possible to detect automatically the nanoparticle orientation: close to [100]



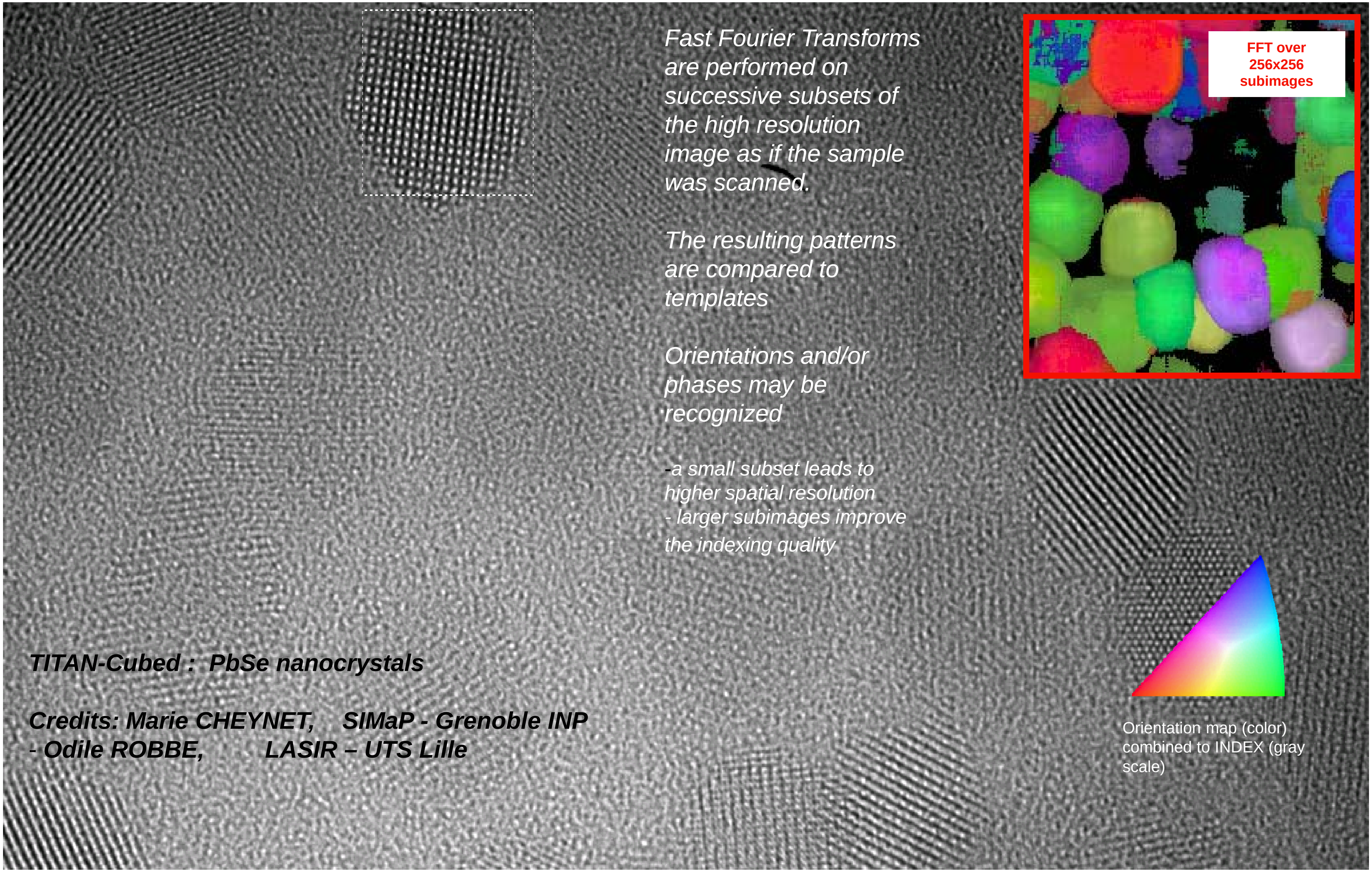




NanoMEGAS
Advanced Tools for electron diffraction

Grenoble INP

ASTAR : Phase /orientation mapping HREM images



Titan Cs corrected HREM : PbSe nanoparticles

Precession adds a value to EBSD-TEM technique :

- **ED patterns acquired with precession contain less dynamical effects , more spots and when compared with templates give much better correlation-reability than ED patterns without precession**

- **Correlation index for many reflections highly increases even at small precession angles (eg 0.2 º -0.5º)**

- **Orientation maps for several materials are of much better quality with precession**

- **Phase maps for several materials are of much better quality with precession (much less artifacts or ambiguities)**

- **In orientation-phase maps "180º ambiguity " for cubic crystals can be solved using precession**

1st precession electron diffraction user meeting

NanoMEGAS
Advanced Tools for electron diffraction

IC CNR Istituto di Cristallografia

Martina Franca 8-9 May, 2008

WORKSHOP ELECTRON PRECESSION EBSD-TEM

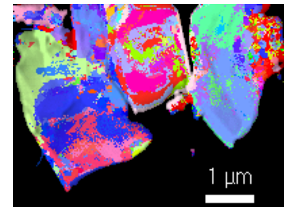
M&M Portland USA 1-5 August 2010

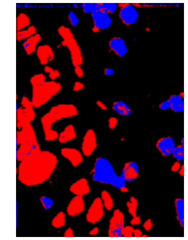
ICM17 Rio de Janeiro Brazil 22-23 September 2010

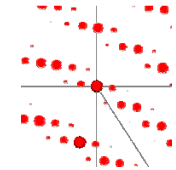
Electron Crystallography in Physical and Biological Science
(AsCA2010 Satellite Meeting, KBSI, Daejeon) 29-30 October 2010

www.nanomegas.com

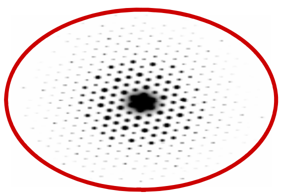
TEM ELECTRON DIFFRACTION SOLUTIONS

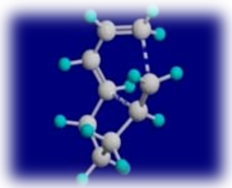


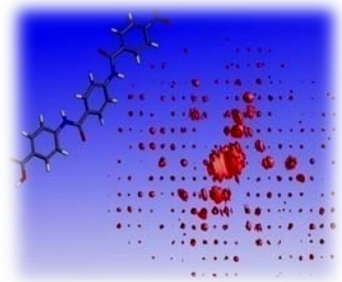




AUTOMATIC ORIENTATION / PHASE MAPPING

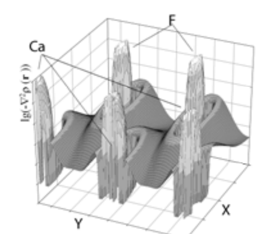






3D PRECESSION DIFFRACTION TOMOGRAPHY

PRECESSION UNIT
« DigiStar »
Basic platform



ELECTRON DIFFRACTOMETER-

NanoMEGAS
Advanced Tools for electron diffraction