Nanobeam diffraction scanning in transmission electron microscopes

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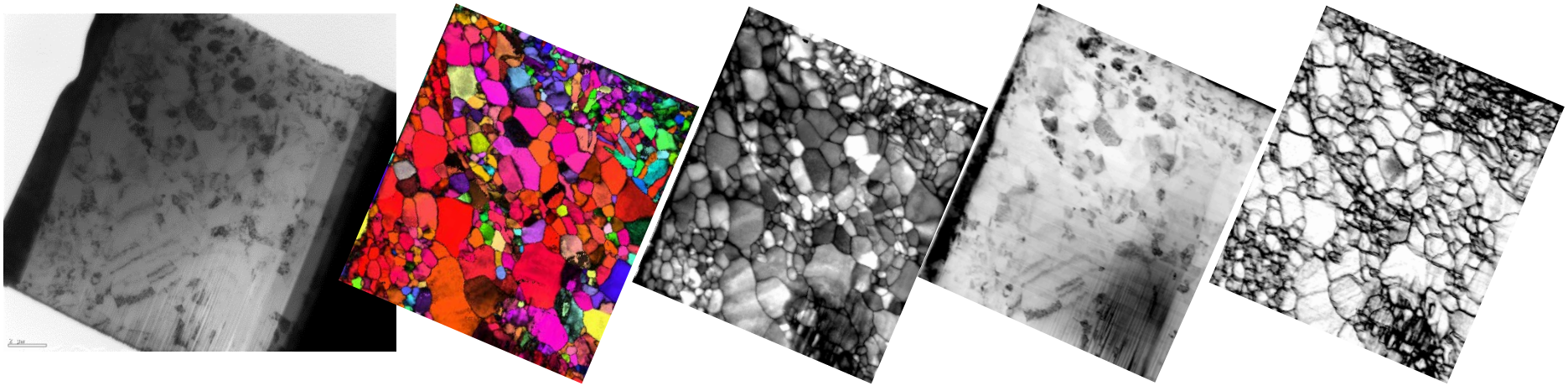
OUTLINES

Dedicated strategies to highlight structural features at nanoscale

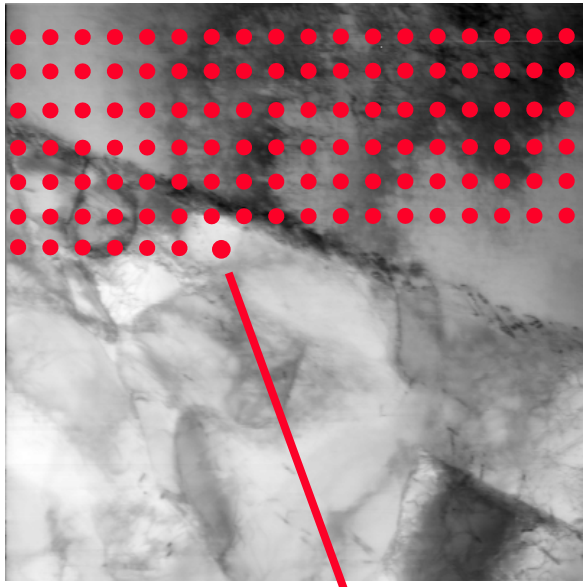
- Orientation and Phase maps
- Combined Virtual Bright- and Dark-Field images
- Correlation coefficient maps.

Mg severely deformed

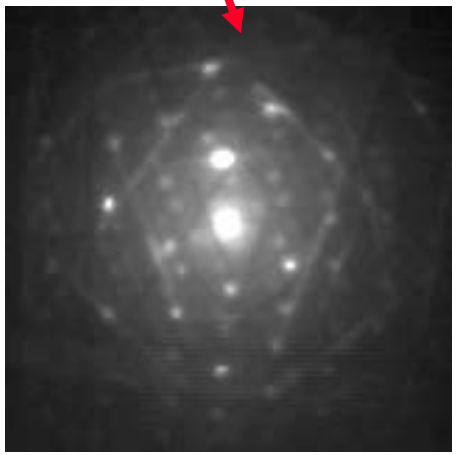


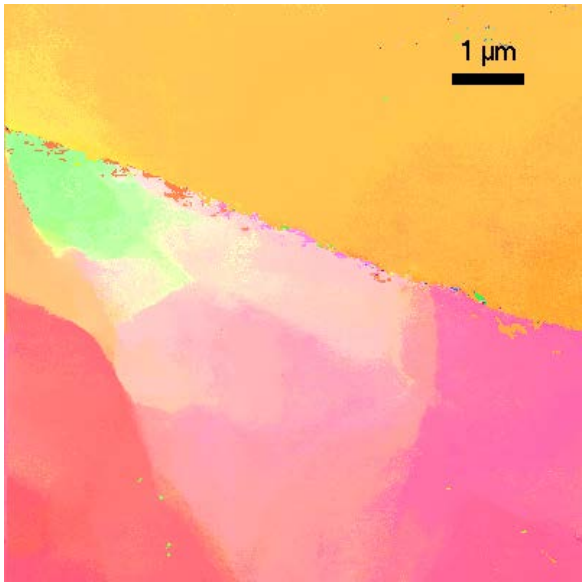
Bright-Field *Orientation map* *Index* *Virtual bright-field* *Correlation coefficient map*

ACOM/TEM: Automated Crystal Orientation Mapping tool

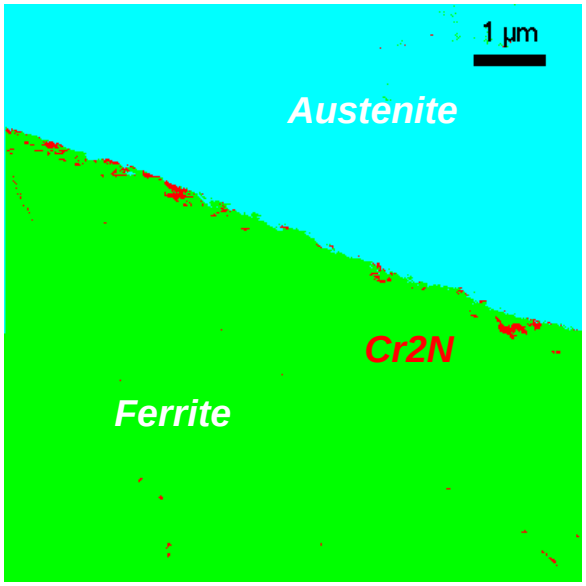


Dual phase steel

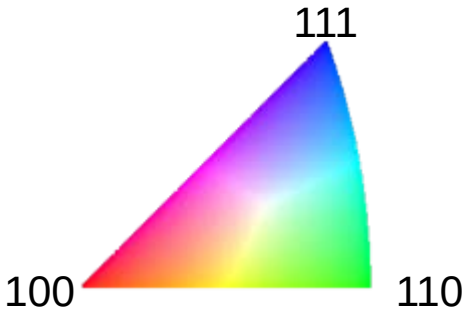




Orientation map



Phase map

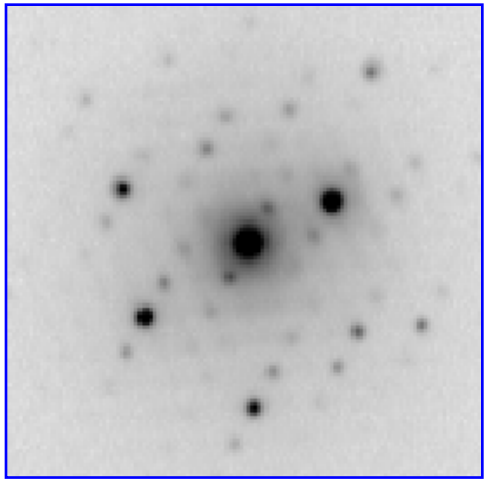


Colour code

- Austenite
- Ferrite
- Cr2N

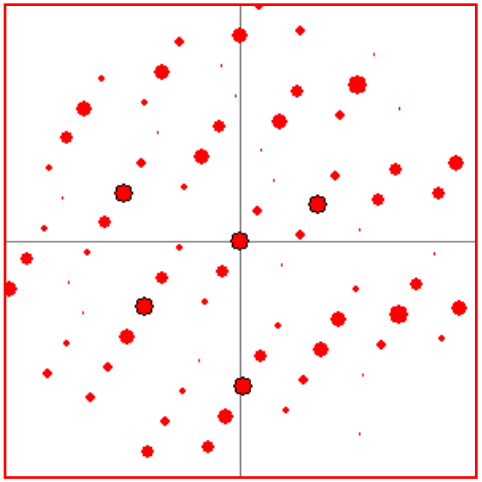
ACOM/TEM : Crystallographic orientation identification

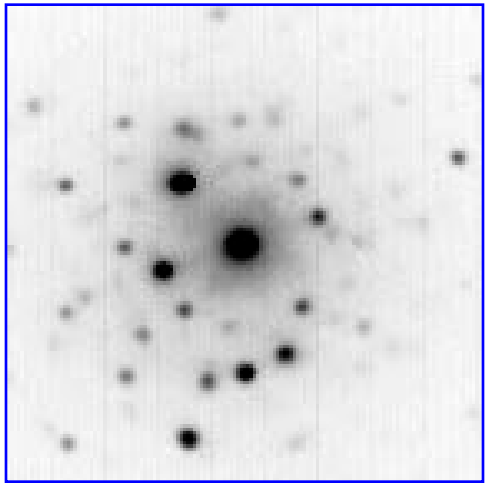
Diffraction pattern for a randomly oriented crystal



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

Calculated diffraction pattern

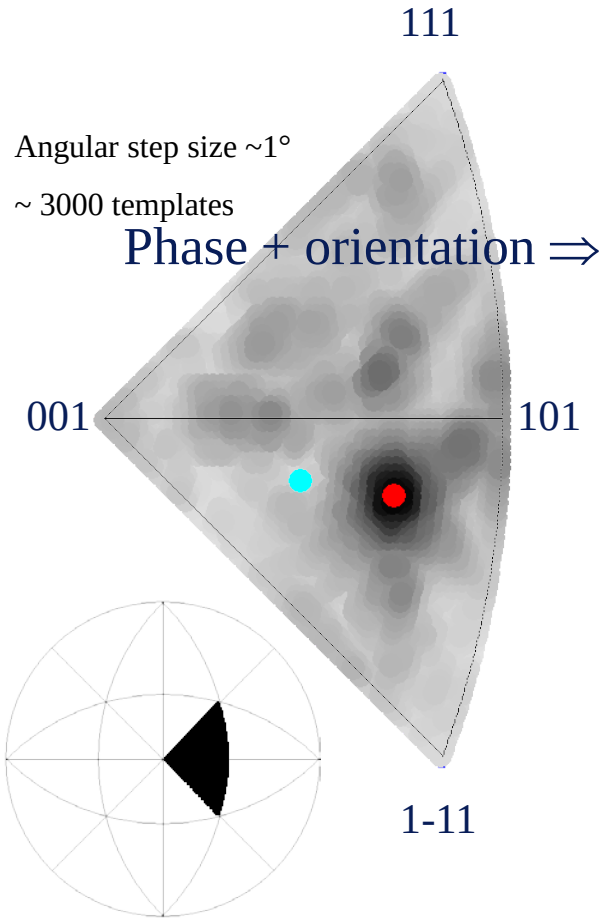




Austenite, ferrite, one or two grains?

ACOM/TEM : the template matching strategy

Stereographic projection (cubic)

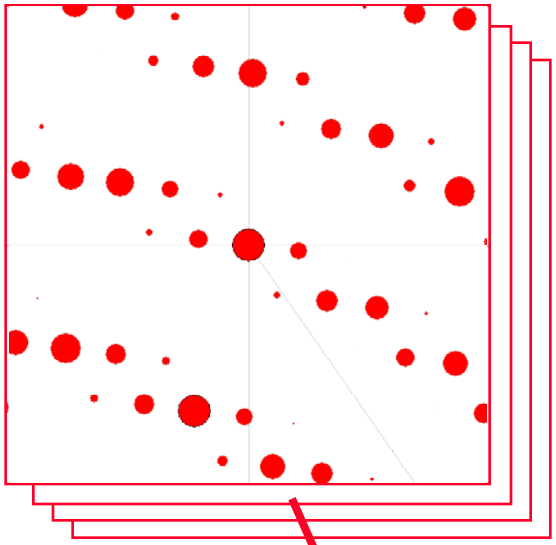


Angular step size ~1°

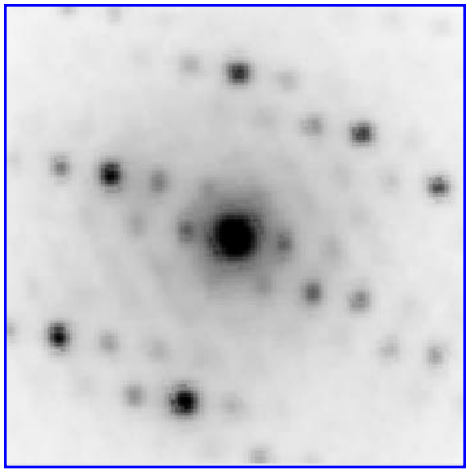
~ 3000 templates

Phase + orientation ⇒

Pre-calculated templates



Acquired pattern



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

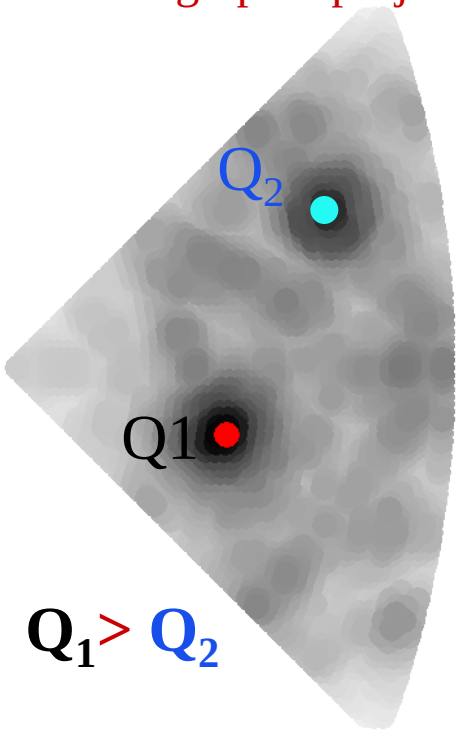
Correlation index

Template Matching

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

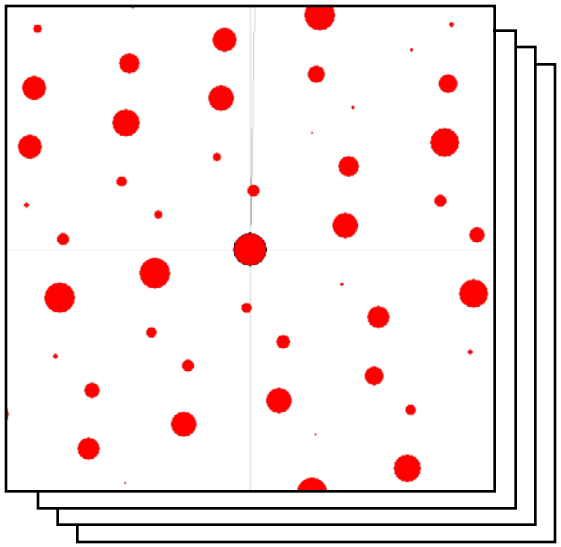
ACOM/TEM : Reliability

Stereographic projection

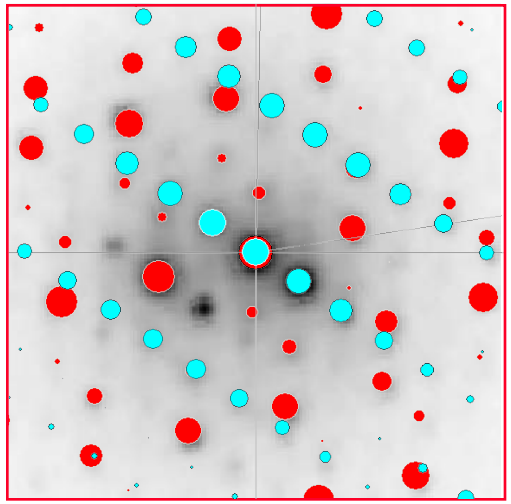


$Q_1 > Q_2$

Templates for copper



Superimposed diffraction patterns at a grain bounday

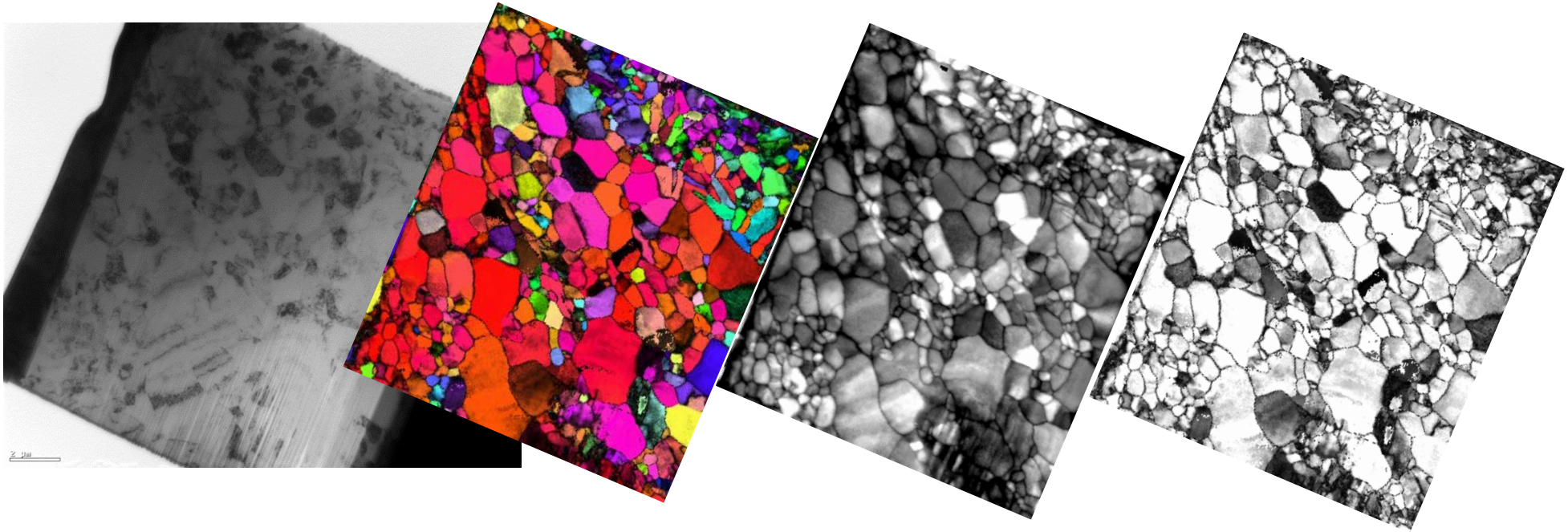


$$R = 100\,(1 - Q_2/Q_1)$$

Reliability

ACOM/TEM: Automated Crystal Orientation Mapping

Mg severely deformed



Orientation + Reliability Correlation Index Reliability

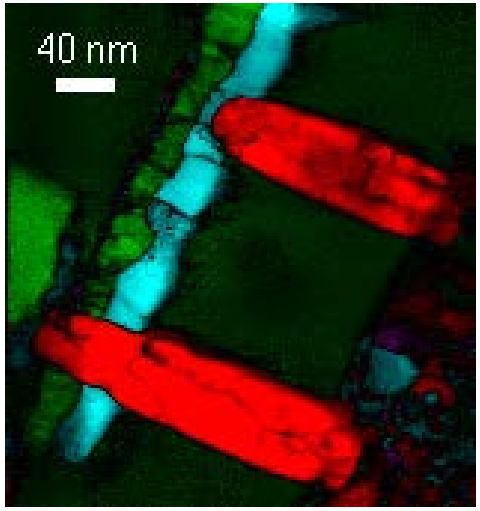
Orientation and phase maps

A nanoscale interconnect device composed of different layers and phases.

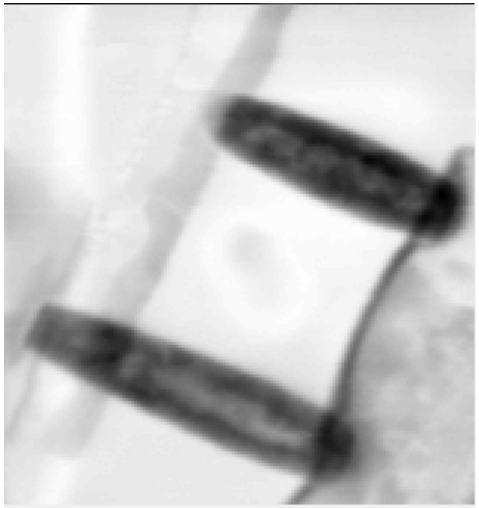
JEOL 2100F

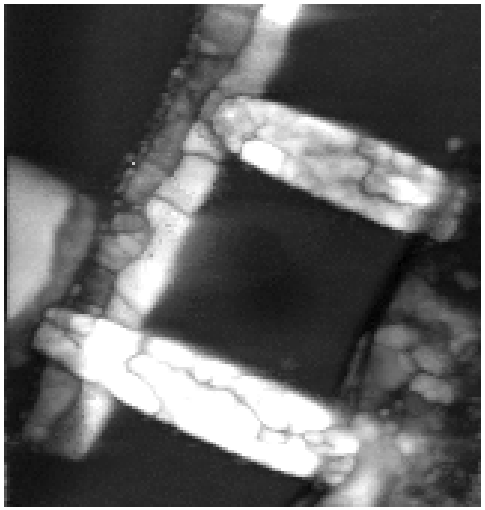
W
Si
NiSi

Phase + Reliability

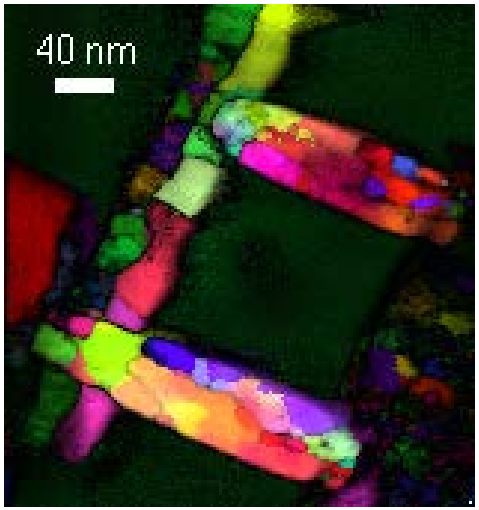


Virtual Bright-Field





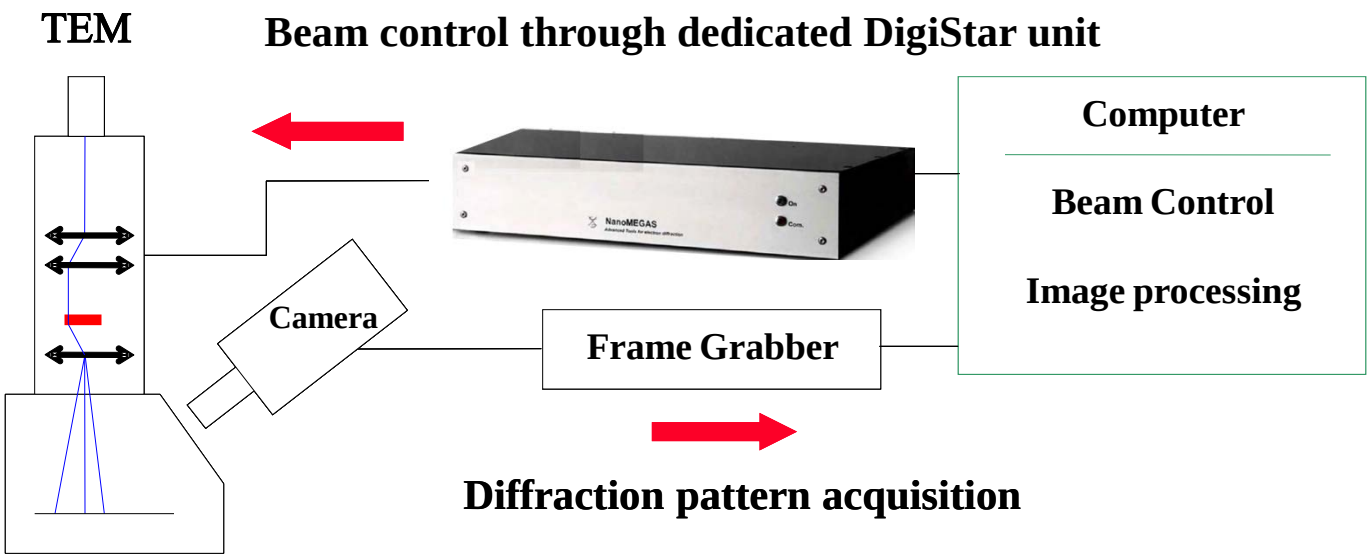
Correlation Index



Orientation + Reliability

Phase reliability

Coupling with the ***NanoMEGAS*** Electron Precession unit (DigiSTAR): **ASTAR**

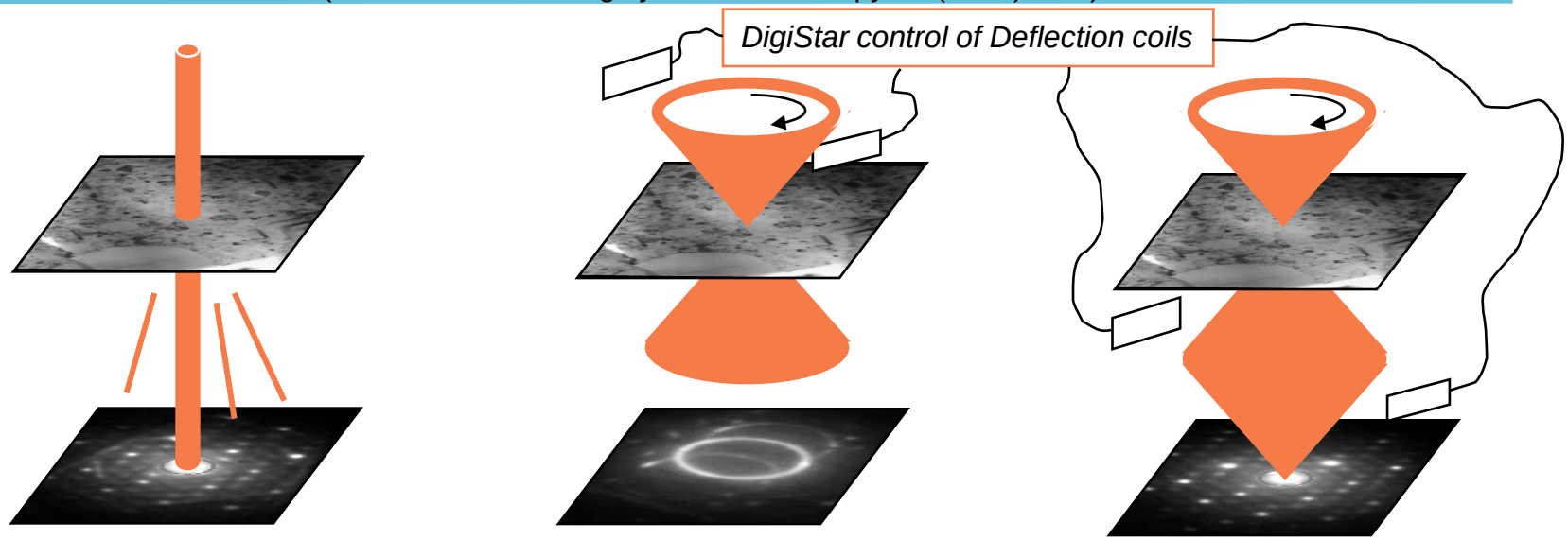


Scanning rates ~100 fps
Precession rate up to 400 hz

Fast optical camera (180 fps)

Precession Electron Diffraction

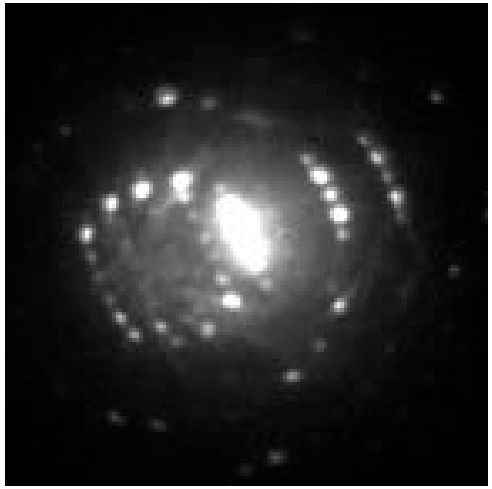
(R. Vincent, P.A. Midgley, Ultramicroscopy 53 (1994) 271.)

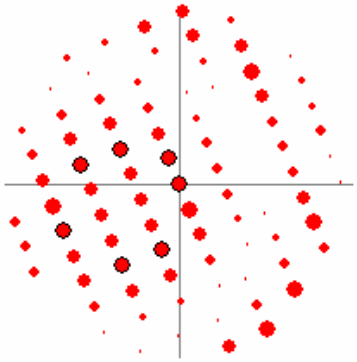


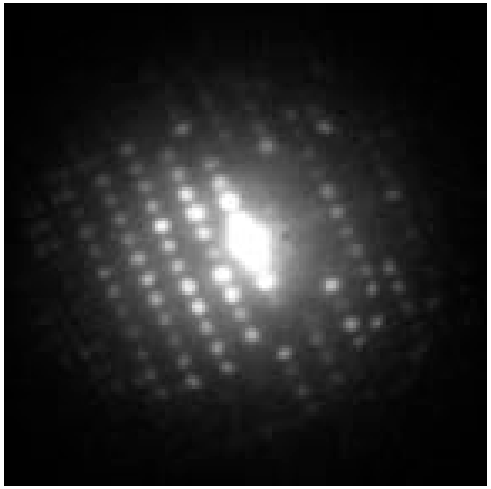
I: Standard setting

X: ½ Precession

Y: Full precession



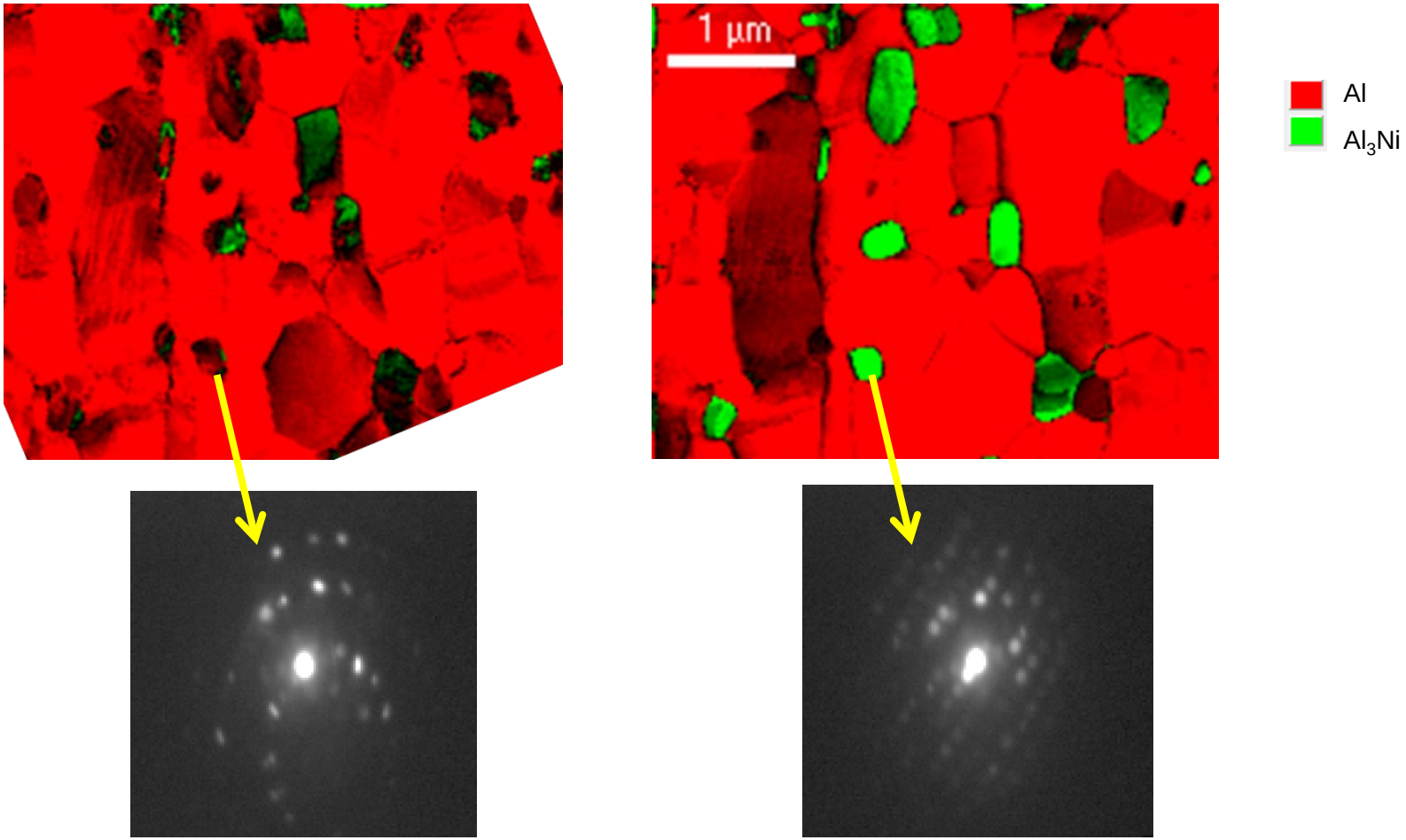




Precession Assisted Phase Mapping

Phase map (colour) combined to phase reliability (grayscale) for Al_3Ni crystals embedded in an Al matrix without (left) and with 0.5° precession (right)

(TECNAI F30 - Courtesy of Prof. A. Gholinea Uni. Manchester UK.)



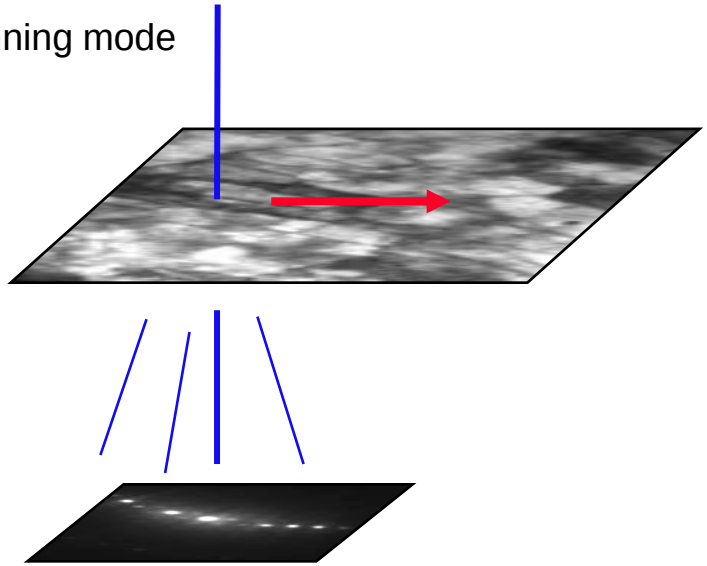
⇒ Combined Virtual Bright- and Dark-Field images

E.F. RAUCH, M. VERON Eur. Phys. J. Appl. Phys. (2014) 66: 10701

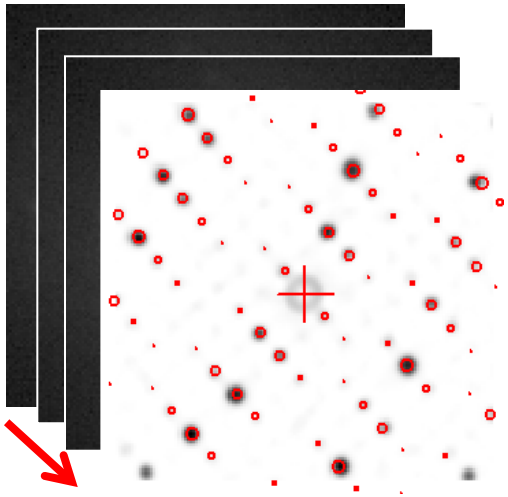
⇒ Correlation coefficient maps

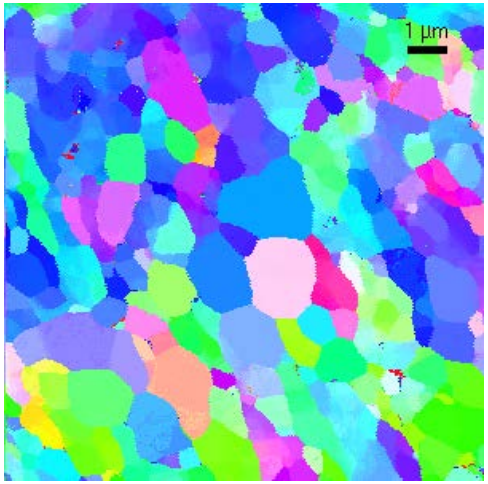
Virtual Bright/Dark Field images

Scanning mode



Acquisition of a stack of diffraction patterns

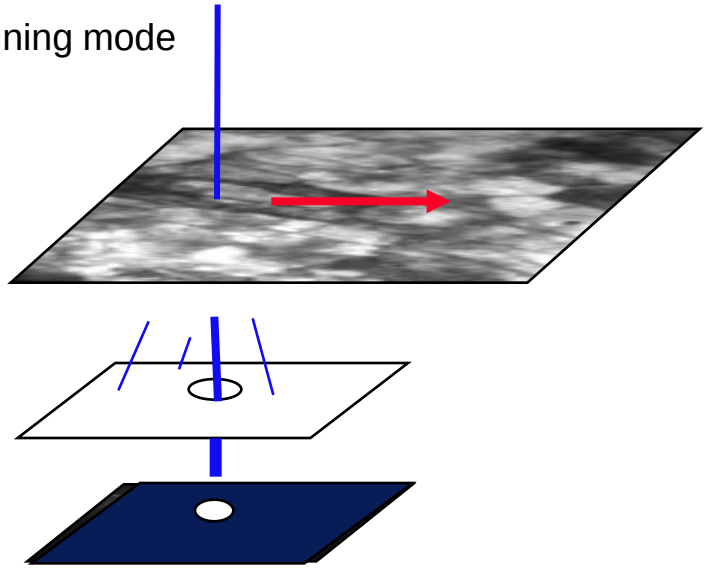




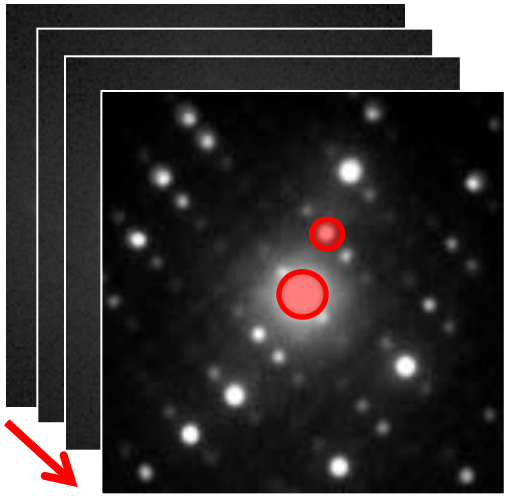
Orientation map

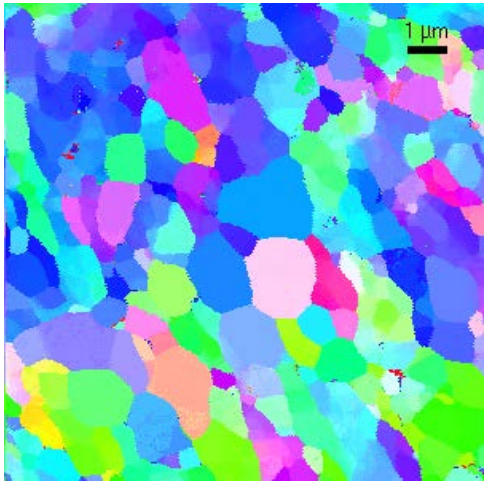
Virtual Bright/Dark Field images

Scanning mode

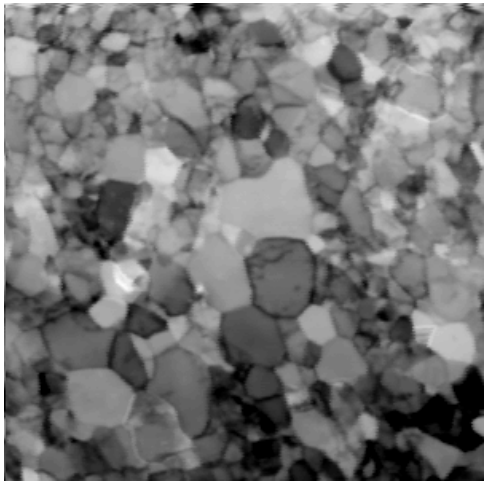


Acquisition of a stack of diffraction patterns

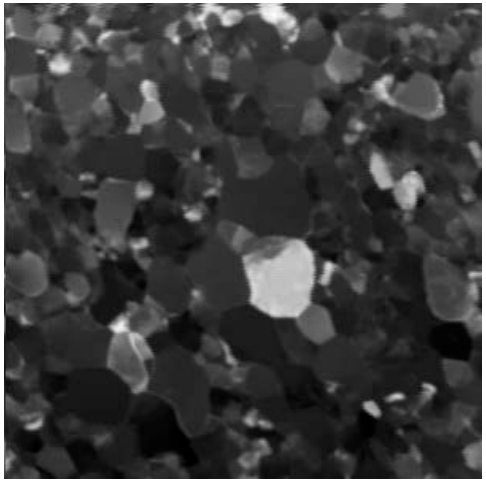




Orientation map

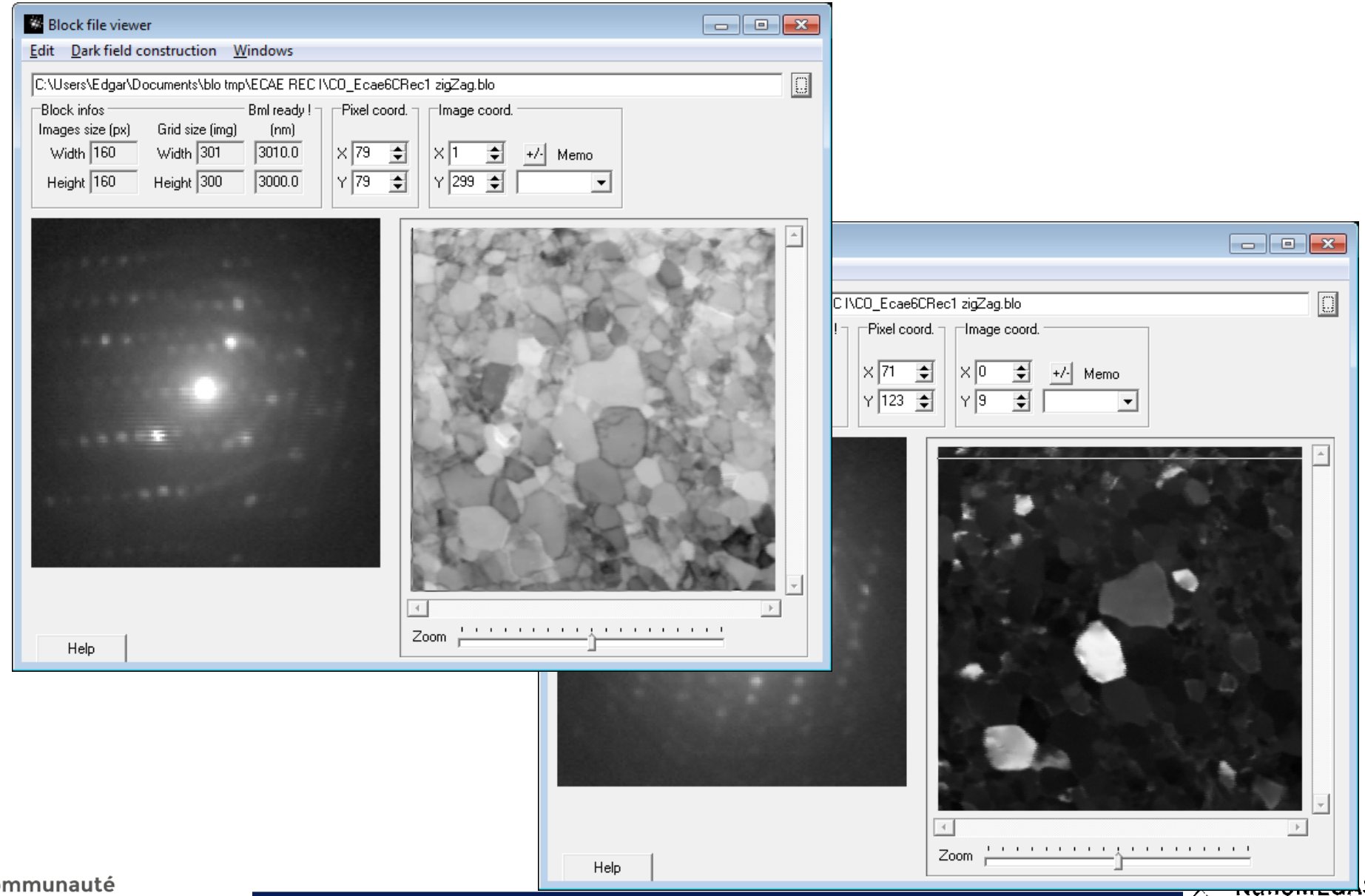


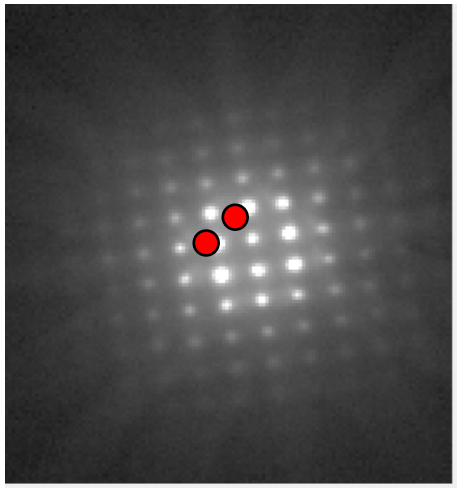
Virtual Bright Field

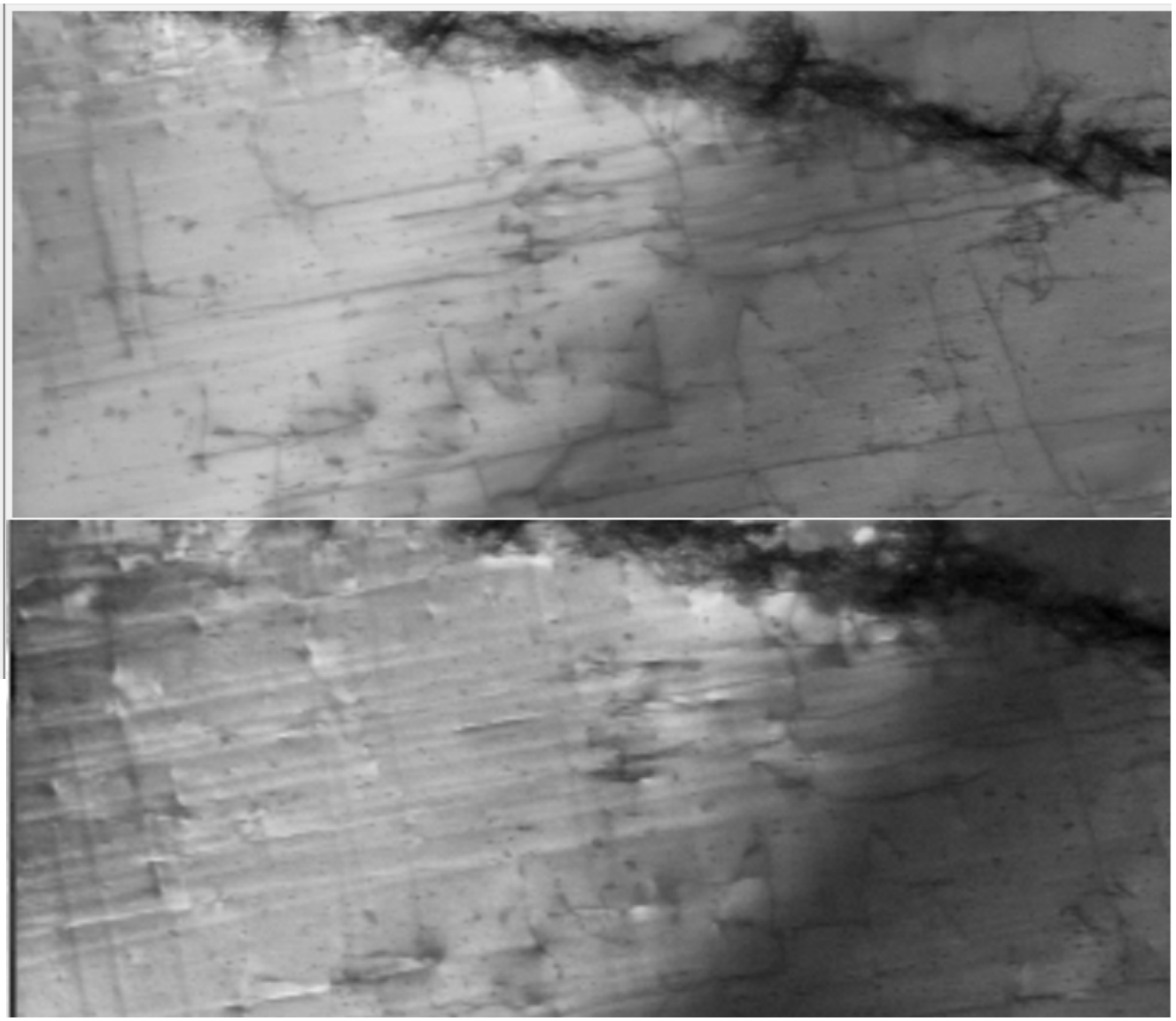


Virtual Dark Field

Virtual Bright- and Dark-field images



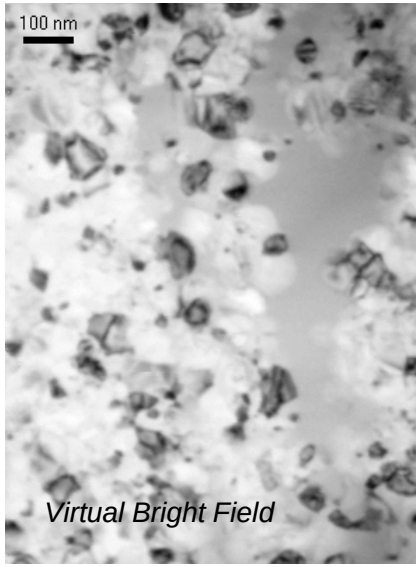


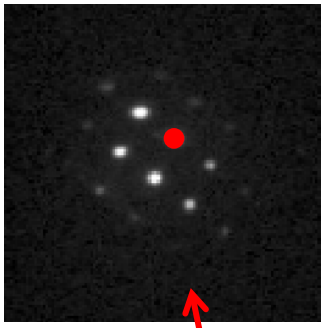


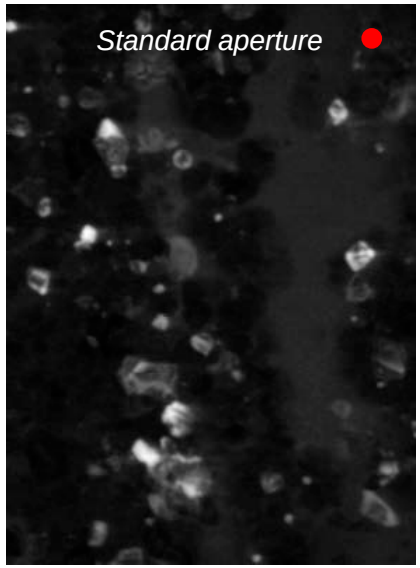
Combined Virtual Dark-Field : free ‘aperture’ design

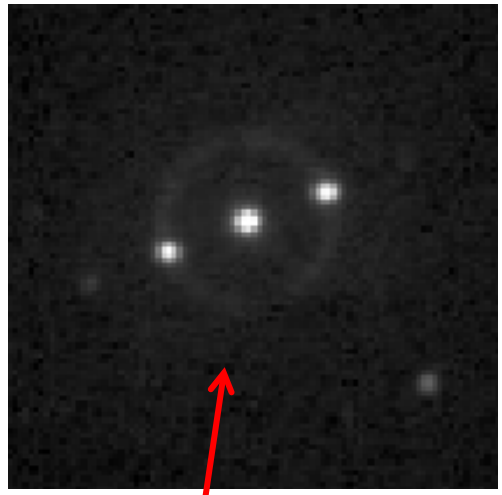
Nanocrystallized partly amorphous NiTi

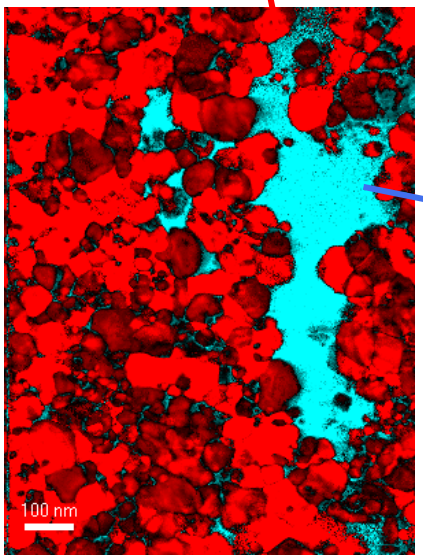
– *credits: E. Manara Courtois, Karlsruhe Inst. Tech.*

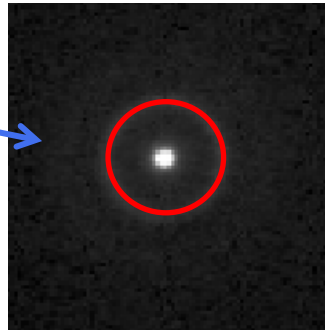


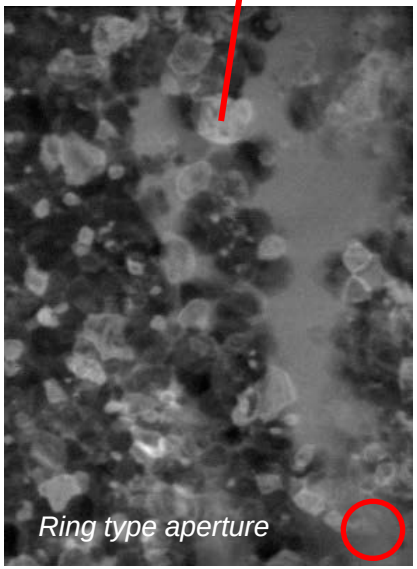












Phase map:

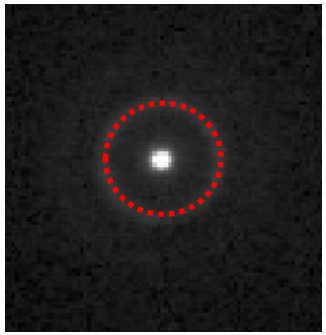
- Crystalized

- Amorphous

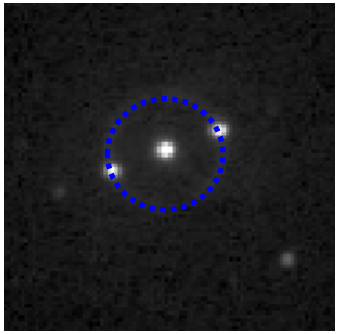
Virtual Dark-Field : free ‘aperture’ design

Nanocrystallized NiTi

– *credits: E. Manara Courtois, Karlsruhe Inst. Tech.*



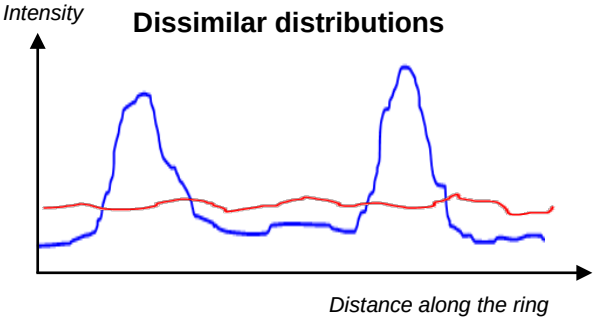
Amorphous

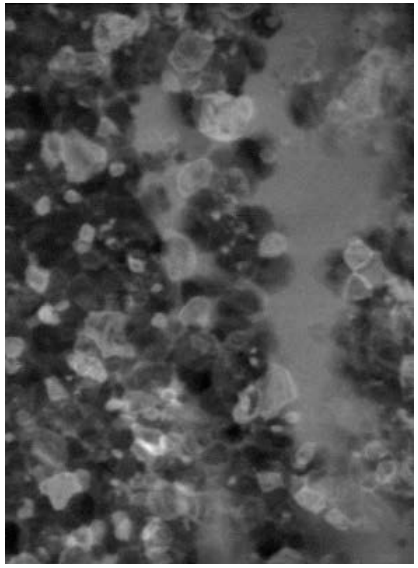


Crystal

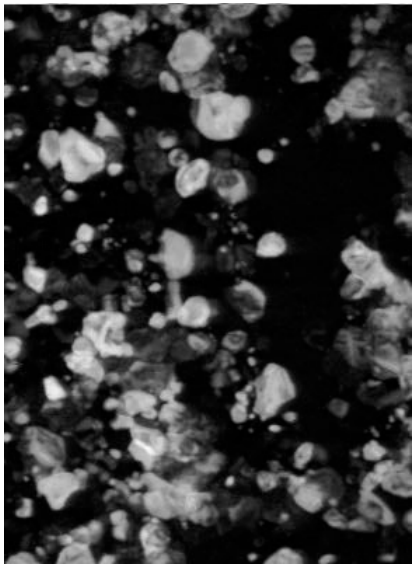
Similar averages

Dissimilar distributions

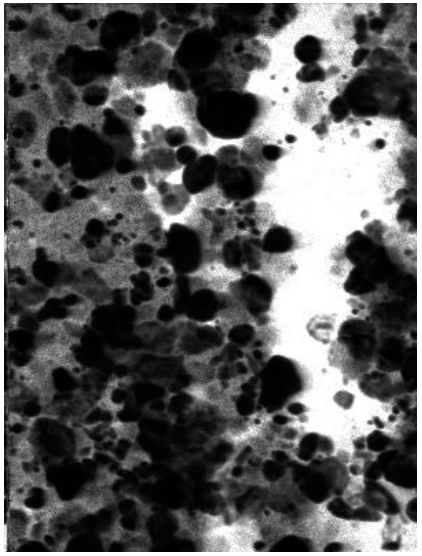




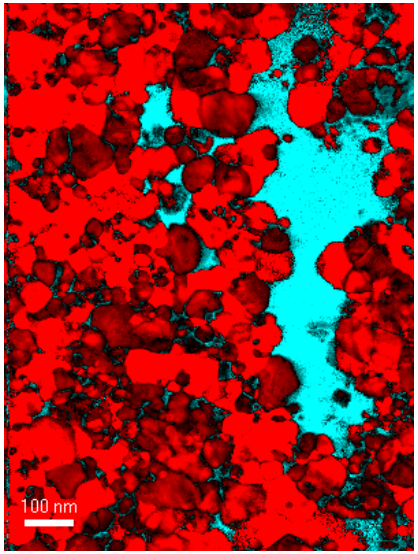
Average intensity



Standard deviation



$$\frac{\text{Average}}{\text{Standard deviation}}$$



Phase map

Correlation coefficient maps

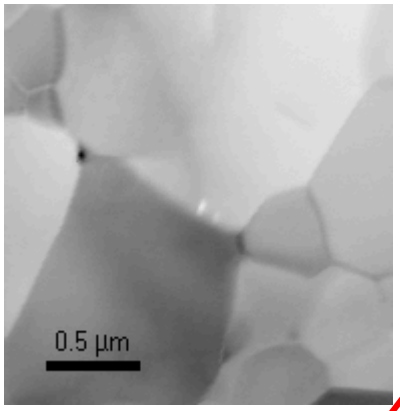
A.K. KISS, E.F. RAUCH, J.L. LABAR Ultramicroscopy 163 (2016) 31–37

S. WANG Microsc. Microanal. 19 (Suppl 2) 2013

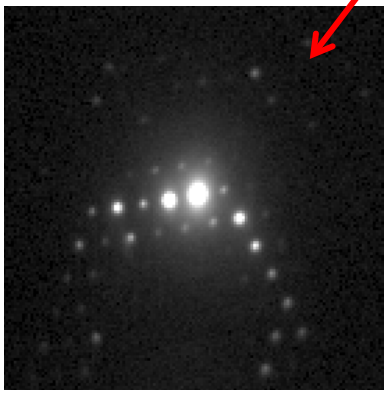
Correlation coefficient maps

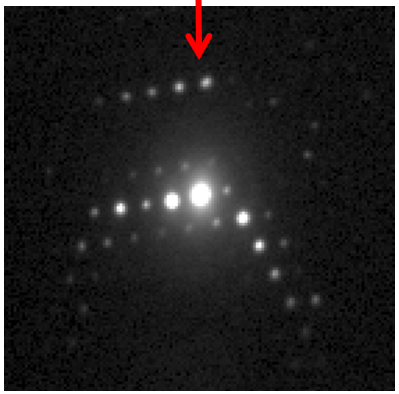
Alumina (Al_2O_3) – credits: S. Lartigue ICMPE, Thiais

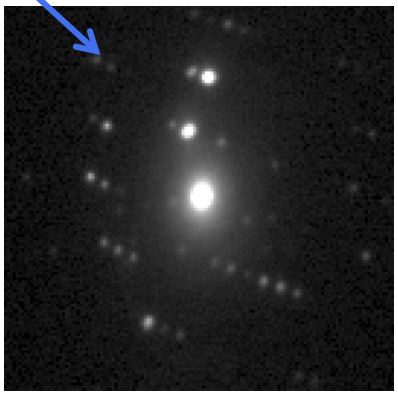
Virtual Bright-Field



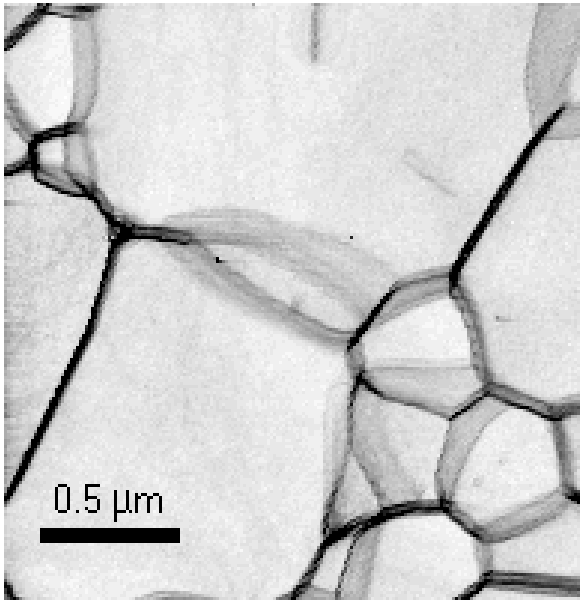
Orientation map







Cross-correlation weights the similarities between successive diffraction patterns

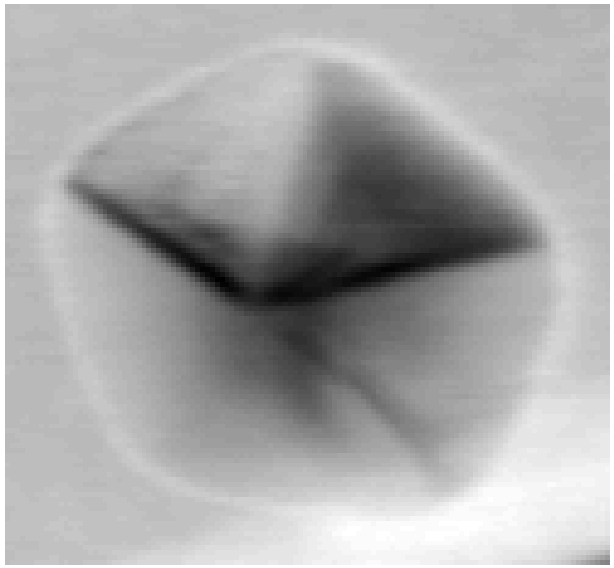


$$C_{iso}(i,j) = \sqrt{\frac{\sum_{x,y}\left[P^{i,j}(x,y) - P^{i+1,j}(x,y)\right]^2 + \sum_{x,y}\left[P^{i,j}(x,y) - P^{i,j+1}(x,y)\right]^2}{2N}}$$

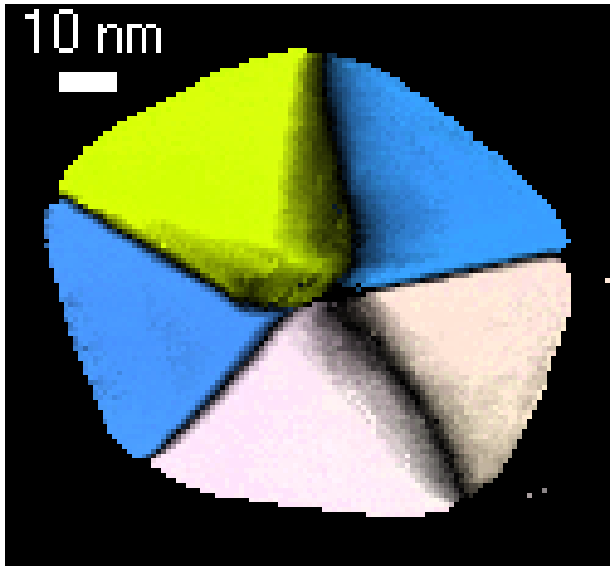
Correlation coefficient maps

Multi-twinned gold particles (JEOL ARM)

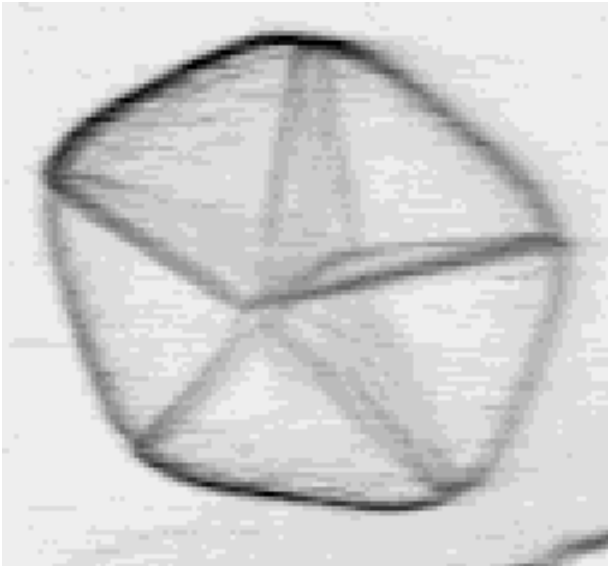
Credits: Arturo Ponce, UT San Antonio,Texas



Virtual Bright-Field

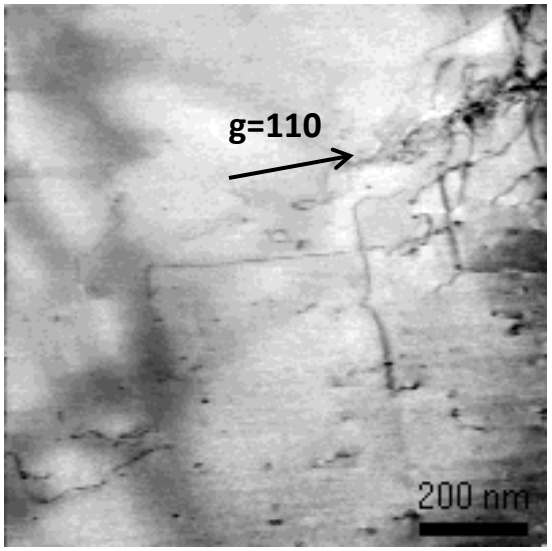


Orientation map

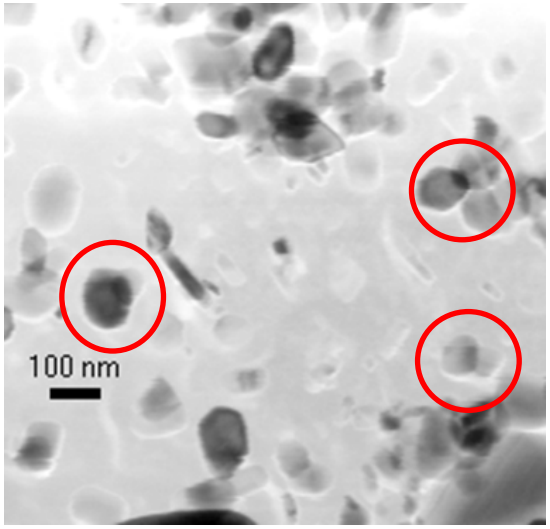


DP correlation coefficient map

Correlation coefficient maps

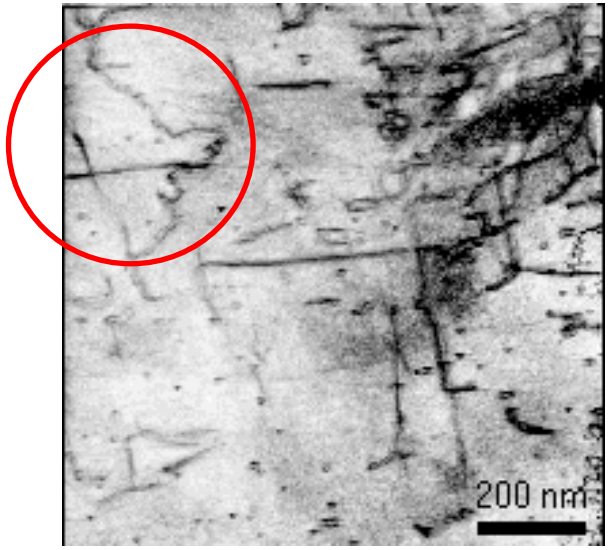


Superimposed precipitates→

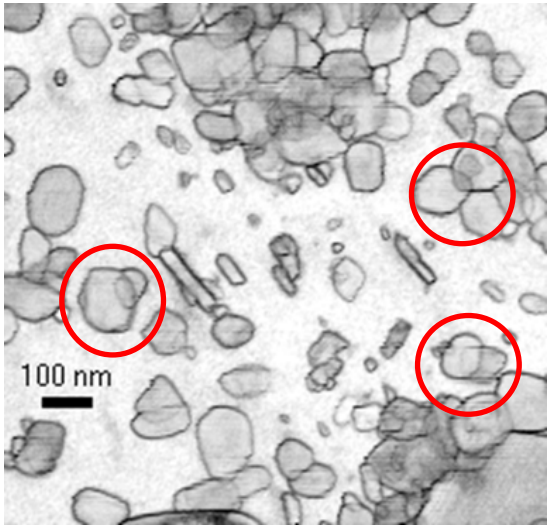


← Hidden dislocations

Virtual Bright-Field



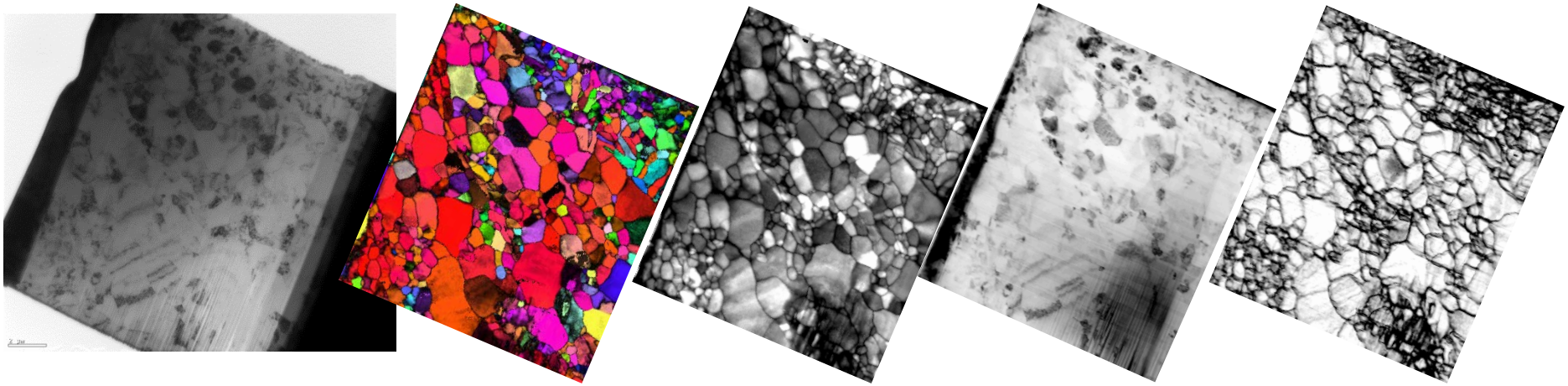
DP correlation coefficient map



Credits: Fanny MAS, SiMAP

To sum up

Mg severely deforrmed



Bright-Field *Orientation map* *Index* *Virtual bright-field* *Correlation coefficient map*